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A FACTORIAL APPROACH
TO THE STUDY OF
ALKALI-SILICA REACTION IN CONCRETE

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Thesis submitted to The City University
for the Degree of Doctor of Philosophy
in the Department of Civil Engineering

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DECLARATION

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ABSTRACT

This thesis describes a research project which investigates certain aspects of the alkali-silica reaction in concrete. A factorial approach was adopted for the experimental stages of the investigation. Such an approach is based on proven statistical techniques and has the advantage of allowing any interaction between the experimental factors to be studied in addition to studying the main effect of each separate factor.

The experimental work was carried out in three stages. The first and second stages comprised physical tests which involved the monitoring of expansion in the test specimens. The third stage involved chemical analysis to determine the alkali content of the test specimens.

The main parameters which were included in the investigation were:-
(a) the alkali content of the Portland cement
(b) the use of pulverised fuel ash as a cement replacement material
(c) the amount of reactive aggregate
(d) the free water content of the mix
In addition, the use of pulverised fuel ash as an admixture and the different effects produced by the two most commonly used reactive aggregates in laboratory tests (namely Pyrex glass and Beltane opal) were studied.

In general the experimental work was carried out using standard test methods as specified by the British Standards Institution and the American Society for Testing and Materials. In particular the "mortar bar method", as described in A.S.T.M. C227, was used extensively. Where necessary the standard test methods were adapted to suit the experimental nature of the investigation.

It is shown that the main effects of the experimental factors are all highly significant and thus have a considerable contribution towards the expansion of the mortar bar specimens. Moreover, the level of interaction between certain of the factors is also shown to be significant. This indicates that, in some cases, the effects of the individual factors are not independent. The experimental results also show the different responses produced by the two reactive aggregate materials.

With respect to the use of pulverised fuel ash, certain time dependent trends are discussed which may suggest some long term instability in the beneficial effect which pulverised fuel ash can achieve by reducing expansions. The factorial approach assisted in highlighting this time dependent effect in the experimental data.

The chemical analysis results have shown that pulverised fuel ash tends to increase the alkali content of the mortar bars. This is discussed with respect to the mode of operation of pulverised fuel ash in inhibiting expansion due to alkali-silica reaction, which is considered to be one of chemical reaction and not a simple dilution effect. An explanation of the contribution of pulverised fuel ash and the differences noted between its use with Pyrex glass and Beltane opal is hypothesised in terms of the relative affinity of each of these three materials for reaction with the hydroxyl ion present in the pore fluids.

TO HELEN AND MY PARENTS

CHAPTER 1 INTRODUCTION

The phenomenon of Alkali-Silica Reaction (A.S.R.) in concrete has been known for almost half a century since it was first reported by Stanton in 1940 (1). It is part of the larger group of deleterious reactions known as Alkali Aggregate Reaction (A.A.R.) which may occur in certain concretes. In addition to A.S.R. the A.A.R. group also includes Alkali Carbonate Reaction and some very slow siliceous reactions, however only A.S.R. is considered here.

As the name A.S.R. suggests, the reaction is considered to be associated with cements which contain a relatively high proportion of alkali and with aggregates which contain a significant proportion of reactive silica. The alkaline materials found in cement are the oxides of the metals Sodium and Potassium, which form their respective hydroxides when dissolved in water. Reactive forms of silica include Opal, Chalcedony and Tridymite, which can occur in rocks such as Cherts, Siliceous Limestones and Rhyolites. More recently strained Quartz and some geological glasses have been shown to be reactive.

In the intervening years since Stanton's early work, the problem of A.S.R. appears to have increased if measured by the number of cases being documented. Whether this is due to an increase in its occurrence or simply due to an increased awareness of the problem is open to speculation. Most certainly, concrete structures suffering from the effects of A.S.R. are now known to exist in countries previously thought to be free of the problem. In particular a number of such structures are now documented in the U.K. (2-7) whereas earlier work (8-10) expressed a high degree of confidence in the opinion that there was little chance of such occurrences.

There are two factors which are worthy of note as possibly having contributed to this apparent increase in A.S.R. Firstly, as transportation costs have risen there has been a trend towards the utilisation of locally-won aggregates, whenever possible, rather than using material already known to be satisfactory from practical experience. In many instances such aggregates were not tested rigorously enough before being considered to be fit for use, particularly where phenomena such as A.S.R. had previously been absent.

Secondly, with the increased use of the dry process kiln in the cement production industry and the associated employment of flue gas recirculation to enhance economy, the alkali content of cements has tended to increase. Further, the relative proportions of the various main components in cements are different for modern U.K. cements in comparison to cements manufactured in the past (11). In particular the ratio of tricalcium silicate (C_3S) to dicalcium silicate (C_2S) is much greater than it was say 25 years ago (12).

It is somewhat ironic that the former possibility above should be put forward as a probable cause of an increase in the rate of occurrence of A.S.R. when in the earliest research on the subject (1) the use of untried and untested locally-won material was already found to be the source of some of the first documented cases. Obviously the early warning went unheeded.

Once the reaction had been recognised and found to be fairly widespread in countries such as the U.S.A., it became an important topic for research. In particular three aspects were seen to have great importance:-

i) to establish the exact nature of the reaction

ii) to devise suitable laboratory techniques capable of assessing the potential reactivity of any given cement, aggregate or cement/aggregate combination.

iii) to find ways of combatting the adverse effects of the reaction.

Whilst many advances have been made in all of these fields, to date there is neither an absolutely foolproof laboratory test nor an undisputed description of the mode of reaction.

Classically the reaction has been described in terms of the alkaline material in the cement combining with the reactive silica in the aggregate to form a gelatinous compound. This gel has an affinity for water, any free water being readily absorbed. Since the intake of water is accompanied by an increase in the volume of the gel, internal pressures are set up in the concrete matrix. Consequently, the concrete expands and extensive cracking and surface spalling usually occur. In addition, the gel may be extruded from the cracks. Whilst the foregoing description is generally accepted, the causes of the reaction and mechanism itself are not universally defined. However, most researchers have agreed that for a deleterious effect to be manifest the three parameters alkali, reactive silica and free water must all be present. If one of these three parameters is missing then no deterioration due to A.S.R. will occur. For example, should the free water not be present, then the actual reaction itself may still take place but there will be no expansion process. Under these conditions the silica gel could lie dormant until a source of water becomes available, when rapid expansion may occur.

Many laboratory tests have been devised which attempt to assess the reactivity of aggregates, cements or particular aggregate/cement combinations. The most important and widely used of the tests are petrographic examination of aggregates (13), the gel pat test (14), the rapid chemical testing of aggregate (15), the rock prism test (16), the concrete prism test (17) and the mortar bar test (18).

The latter test has been used most widely to assess the performance of materials in this context and also as a research tool to investigate the reaction itself. It is commonly regarded as the most reliable test available but has the disadvantage of requiring considerable time before meaningful results are obtained. Due to the complexity of the reaction, however, even the mortar bar test can give misleading results. In particular some U.K. aggregates found to be reactive in practice have shown no sign of expansion when tested according to this method (19). With other tests, particularly the rapid chemical test, the converse has often been true since aggregates known to be sound in practice have been designated potentially reactive (20).

The serious and destructive nature of the reaction renders remedial work virtually impossible unless the affected concrete is either non-structural or of limited extent so that a complete section can be replaced. Consequently there has been greater interest in formulation of methods of prevention rather than cure. The most straightforward means of ensuring that disruption does not occur being the removal of one or all of the three conditions necessary for the phenomenon.

It is widely accepted that the choice of a non-reactive aggregate permits the safe use of a high alkali cement and conversely that the reduction of the alkali content of the cement below a certain limit can allow reactive aggregates to be used safely. However, some difficulty is experienced when trying to define this safe limit of alkali content. Based on his own early work Stanton (1) suggested a limit of 0.6% by weight for the acid-soluble alkali content expressed as equivalent Sodium Oxide (Na_2O_{eq}). More recently Hobbs (21) expressed a limit for acid- soluble Na_2O_{eq} content in terms of kilogrammes per cubic metre of wet concrete, defining the dividing line between safe and deleterious mixes to be at 3.0kg Na_2O_{eq}/m^3. The third factor in the deterioration process, water, can be removed in certain instances by waterproof membranes and well designed drainage. Due to the possible formation of the dormant hydrous alkali-metal silicate gel, however, this solution on its own cannot be considered completely stable since disruption may occur should water eventually become available.

In situations where economic restraints or circumstances dictate that it is not practicable to remove any of the three main parameters, other solutions are required. Mineral admixtures such as pulverised fuel ash (p.f.a.), ground granulated blast furnace slag (g.g.b.f.s.), limestone flour and silica fume have been shown to have beneficial effects. The results of some research (10, 22) has even shown that certain reactive aggregates can be effective as preventative treatments when ground to a suitably fine particle size.

CHAPTER 2 THE SCOPE OF THE PRESENT WORK

The research described in this thesis can be divided into two sections, each dealing with a separate aspect within the topic of A.S.R., namely its physical and chemical aspects. It is appropiate to describe first the scope of the physical section of the work, since this was largely completed before the major chemical work was started. Consequently the former imposed certain restraints on the latter with respect to the specimens which were available for chemical analysis.

After an extensive initial literature survey, certain parameters associated with A.S.R. were selected for examination. In addition it was proposed to use a statistically based method of experimentation in an attempt to explain the sometimes contradictory conclusions drawn from the results of some previous investigations. The basis of the chosen method of statistical experimentation is known as Factorial Analysis which allows the experimenter some insight not only into the effect of each parameter of the experiments but also the interaction between pairs of parameters and larger groups where applicable. Factorial Analysis has rarely been used in research with concrete (23) and, as far as the author is aware, never when investigating A.S.R.. Hence a further consideration of the work was to examine the advantages to be gained by use of this method of experimentation.

The final choice lay in the selection of a laboratory test able to monitor the effects of A.S.R.. Here it was felt important to follow a recognised standard test method in order to exclude as much extraneous variation as possible and also to facilitate direct comparison of the results with other appropriate published data. Because of its widespread use, both in the construction industry and in research projects, the 'mortar bar method' was selected as the main technique for the physical investigation.

The selection of this method, based on the regimes of the American Society for Testing and Materials (A.S.T.M.) C227-71 (18) and C441-69 (24), was made in the absence of any British Standard test specification, and it ensured that practical significance for the work was retained.

The section of the work under the heading 'Physical Investigation' is sub-divided into two stages, these being entitled Series 1 and Series 2. Series 1 represents a small group of preliminary experiments designed to create a level of understanding and appreciation for the subject and the procedures before embarking on the main group of experiments in Series 2.

For both groups of experiments the three parameters listed below were chosen for investigation:-

i) Alkali content of cement.

ii) Partial replacement of cement content using pulverised fuel ash.

iii) Reactive aggregate content (Pyrex glass).

The use of pulverised fuel ash as a replacement for part of the cement content of the mix was included to determine its usefulness in combatting the deleterious nature of A.S.R.. This was a point of some considerable controversy at the time of the experimental work.

Additionally, Series 2 included extra parameters to extend the scope of the work. In the main group of experiments in Series 2 the free water content of the mix was introduced as a parameter. In a smaller set of parallel experiments the Pyrex glass reactive aggregate was replaced by the other most commonly used research material, namely Beltane opal, thus affording some comparison of the effects produced by these two reactive aggregates. Further experiments were also conducted, outside the statistical framework of the main group of experiments in Series 2, which focussed attention on important issues arising from Series 1 and external sources.

The output from the physical tests of Series 1 and 2 is in the form of linear expansions of the mortar bars at the discrete ages selected for the measurements.

The chemical aspect of this research is a logical extension of the analytical techniques used to classify the individual components of the mortar mixes. Its conception emanated directly from the need to gain further understanding into the mechanism of A.S.R. and in particular the way in which p.f.a. affects the reaction. Whilst the results from the expansion tests, utilizing Pyrex glass as the reactive aggregate, were always positive in identifying p.f.a. as beneficial in reducing expansion, some of the Beltane opal mixes produced an opposite effect. It was also noted that the p.f.a. itself contained a higher level of the alkaline material than was found in the Portland cements which were used, whereas some research has concluded that the p.f.a. reduces expansion simply because it dilutes the alkali component of the reaction. It seems most likely that the high level of alkali in the p.f.a. produces the possibility that the level of alkalis in the pore fluids is actually enhanced with p.f.a. mixes.

The chemical tests comprised the analyses of the mortar bars themselves and also the water from the bottom of the storage containers. This enabled the alkali content of the whole system to be studied and in particular to determine if there was any tendency for the alkalis to be leached out of the bar during the A.S.T.M. mortar bar test.

CHAPTER 3 ALKALI-SILICA REACTION, A REVIEW OF PUBLISHED RESEARCH

3.1 DISCOVERY OF THE REACTION AND SOME EARLY RESEARCH

The potential for the deleterious alkali-silica reaction must have existed since the time when aggregate, Portland cement and water were first used to manufacture concrete. However, it was not until the pioneering work of Stanton, published in 1940 (1), that the reaction was first discovered. Stanton's early research, carried out as a materials and research engineer for the State Division of Highways in Sacramento, California, was initiated due to the failure of a concrete road pavement within two years of its construction circa 1936-37. Strong similarities between this very rapid failure and other distressed structures in the same area, such as bridges and sea walls, lead to an extensive investigation, both in the field and in the laboratory. Before this time, any distress caused by A.S.R. would have been attributed to other mechanisms which produce outward features similar to some, but not all, of the features caused by A.S.R..

Stanton concluded that the failure of the structures which he investigated was linked to a chemical reaction between certain mineral constituents present in the aggregate and by-products found in the cement. His laboratory studies revealed that Portland cements containing a relatively high proportion of alkaline material in the form of sodium and potassium oxides were the most reactive, producing excessive expansion in the laboratory specimens when used with the reactive aggregates. Under his particular experimental regime Stanton found expansion to be more or less proportional to the acid soluble alkali content (expressed as total equivalent sodium oxide, as explained in the footnote to Tables 5.4 and 5.8) of the cement.

With respect to the particular rock types found to be reactive, Stanton concluded that siliceous magnesian limestone, cherts and siliceous shale were the most vulnerable and that chalcedony, chalcedonic silica and opaline silica were the minerals most likely to be involved. Many of the structures investigated contained siliceous magnesian limestone aggregate.

Stanton acknowledged that with the siliceous magnesian limestone the reaction, at least in part, may be due to the magnesium carbonate content. Thus two types of alkali-aggregate reaction may be present, namely A.S.R. and A.C.R. (alkali-carbonate reaction).

Stanton's work also drew attention to a product of the reaction which was described as a 'soft viscous substance'. This substance was analysed to be a silica gel containing some form of complex sodium silicate and was held responsible for the expansion and cracking because of its ability to take in moisture and thereby increase in volume.

The earliest published British research on the subject of A.S.R. was produced in 1952 by Jones of the Building Research Station (22). This was the first in a series of five National Building Studies research papers, on the overall topic of "Reactions between Aggregates and Cements", which were published by Jones and Tarleton in the period between 1952 and 1958 (8, 9, 10, 14, 22). The subjects covered by this research programme included the following:-

i) a review of the field investigations carried out by Stanton and updated to include additional work by other authors.

ii) laboratory testing of U.K. Portland cements and U.K. aggregates.

iii) trials with the different forms of laboratory test methods being developed by various researchers.

iv) a study of the effect of the reaction on the physical nature of concrete.

v) investigation of the mechanism of the reaction and the chemical and physical consequences of the reaction.

Much of the work described in Jones' earliest paper produced conclusions along similar lines to those of Stanton's, as might be expected since this paper was primarily a collection of American experiences on the topic. In addition to opal and chalcedony, Jones listed tridymite and cristobalite as reactive forms of silica and included certain volcanic rocks in his list of potentially reactive species.

In the following stage of his work (8), Jones confined his field of interest to A.S.R. and paid particular attention to the concrete materials being used at that time in the British construction industry. The connection of the deleterious reaction with siliceous aggregates was of particular interest, since this posed questions regarding the suitability of aggregates such as flint, which is widely used in the U.K., when used in conjunction with a high alkali cement.

Data on the composition of British Portland cements, covering an extended period of time, showed that alkali contents generally fell within the range 0.5% to 1.0%, when expressed as a percentage, by weight, of total equivalent sodium oxide. Rare exceptions were noted at both ends of the scale. Since Stanton's work, among others, had indicated that cements needed to have alkali contents below 0.6% to safeguard against A.S.R., the potential for the reaction appeared to be present in respect of most British cements.

The work on British aggregates revolved around those types considered to be the most likely candidates for A.S.R. and resulted in the statement that 'In general, it may be said that, with the exceptions stated, there is reason to believe that little trouble would arise with British aggregates'. The exceptions referred to had been found to be 'reactive' in the laboratory testing reported by others, though only to an extent which may have been considered marginal in comparison to the troublesome American aggregates and often only when used in a mixture with inert material.

Following this survey of published data, Jones and Tarleton (9, 10) extended the work to gain first-hand experience with the materials commonly used in British concretes. Using the mortar bar test method two series of experiments were undertaken, the first using a medium alkali cement and the second using a high alkali cement. Based on the results obtained, in the concluding remarks it was stated that 'the normal British aggregates so far tested, when used as whole aggregates, are not expansively reactive with high alkali cements at normal temperatures'. In particular, Thames Valley flint was shown to be innocuous whether used as whole aggregate or diluted using an inert material.

3.2 THE CHANGING U.K. SITUATION

Following the discovery of the A.S.R. problem in the U.S.A. and the early British research discussed above, there was a period of little activity as regards British interest in the subject. Confidence was high in the apparent immunity of British aggregates from the deleterious effects of A.S.R.. This confidence was further substantiated by the lack of any known example of A.S.R. in the U.K., with the construction industry believing that there was no reason why this situation should change.

However, in 1971 A.S.R. was diagnosed as the cause of concrete disruption in a dam in Jersey, Channel Islands. The dam, named Val de la Mare, is part of the principal storage reservoir for the island and was found to be exhibiting signs of distress which, after exhaustive investigation, were attributed to A.S.R. This discovery of disruption to the concrete was made some nine years after completion of construction in 1962 which would probably indicate that the materials were not 'violently' reactive, unlike some of the early failures examined by Stanton in the U.S.A.

The case study of Val de la Mare dam was first reported by Coombes in 1975 (2) and later by Coombes et al in 1976 (25, 26, 27). In these reports extensive information is presented on the remedial action which was taken. This action included the infilling of cracks by grouting and the anchoring of the most seriously affected section of the dam to the foundation rock. The remedial work was practicable only because of the lack of severity of the damage and the type of structure involved, and was not a cure but represented the beginning of a continuous process of monitoring and corrective action.

By 1977 thoughts of A.S.R. on the U.K. mainland were beginning to awaken, stimulated by articles such as that by Figg (28) warning of the possible dangers in using sea-dredged flint aggregates. In 1978 the first of a series of review papers by different authors was published by Palmer (4) which highlighted the first recorded cases of A.S.R. on the U.K. mainland, these having been diagnosed some two years earlier in 1976.

Around this time the intensity of British interest and research into A.S.R. saw a dramatic increase, headed by the two main research institutions, namely the Building Research

Establishment (B.R.E.) and the Cement and Concrete Association (C.α C.A.). Initially this period of activity could be looked upon as one of catching up with the information already available from the extensive research carried out in the U.S.A. and certain other countries. Publications concerning the situation in the U.K. followed from Gutt and Nixon (5) of B.R.E., a second contribution by Palmer (7) and a catalogued review by Allen (6), the latter two authors being from the C. α C.A.

The earliest problems with A.S.R. on the U.K. mainland were not considered to be very serious or of much consequence. The structures involved were relatively low-key, incorporated locally won (often sea-dredged) aggregates and were confined to the South West part of the country. However, with the advent of each of the reviews mentioned above, both the number of structures found to be affected and the locations of the problem became more widespread. Isolated occurrences have now been confirmed in many diverse areas of the country but particularly in the Central Midlands and the South West. Both these areas of higher concentration of A.S.R. cases are known to be associated with Portland cement works which produce a relatively high alkali product.

The severity of the problem has also increased with respect to the type of structures involved. For example, road bridges in the South West (29, 30), Derbyshire (31, 32), West Midlands (29), Warwickshire (33) and Surrey (34); dams in Scotland (4, 6, 34); a car park in the South West (35) and an hospital also in the South West (36) have all been found to be seriously affected.

Despite early and continued assurances that the number of structures affected was small in comparison to the total number of concrete structures existing, and that there was no reason to expect a vast increase in the problem (37, 38) the number of cases of A.S.R. being diagnosed has continued to grow. Comparatively recently reports have suggested that hundreds of structures might be involved (39, 40). To a certain extent this increase in the number of cases being diagnosed was to be expected, indeed Palmer in his initial report (4) forecast that this would happen simply due to the new awareness that the possibility of A.S.R. actually existed. Previously, the deterioration would have been ascribed to other possible causes, without the fullest examination being carried out, since A.S.R. would never have been considered.

A more contentious issue is the question of why the problem has been allowed to occur. Certainly the use of locally won, economic but untried aggregates (particularly unwashed sea or river-bed dredged ones) have played a significant part. That this should be put forward as a contributory cause of the problem in the U.K., over thirty years after the same diagnosis was put forward by Stanton in the U.S.A., is surely demonstrative of a serious lack of awareness by the construction industry.

The other possible aspect of the source of the problem lies with the cements which are now in use. Here, there is considerable disagreement regarding the changes which have occurred over recent years. On the one hand, opinion linked to the cement manufacturing industry, would consider that no significant changes have occurred over the last few decades either in cement composition or the cement properties which are relevant to A.S.R.. However, other research (11, 12, 41, 42) may show that this is not the case, particularly with respect to the relative amounts of dicalcium silicate and tricalcium silicate and even the alkali content itself.

For some time A.S.R. has been known to occur in countries such as Denmark, Germany, Iceland and South Africa, in addition to the U.S.A. It appears, however, that the U.K. does not stand alone as a country where the infestation has only recently been discovered. Lately, for example, it has been confirmed that the Tarbela Dam project in Northern Pakistan exhibits evidence of the reaction (43). It is reported that the approaches to one of the two main spillways are affected and that this forms part of the earliest construction stage of the project which was carried out using materials which had not been tested for alkali aggregate reactivity.

3.3 PROCESSES INVOLVED IN A.S.R.

3.3.1 Mechanism of Chemical Reaction and Expansion

Commencing with Stanton's early work (1) the mechanism of A.S.R. has been linked with alkalis originating from the cement, in the form of sodium and potassium oxides, and certain forms of reactive silica when present as mineral constituents of concrete aggregates. Stanton considered that these two materials reacted together to form a viscous gelatinous substance analysed to be a type of complex sodium silicate. Subsequently, many investigations have been carried out into the actual chemical process involved and, whilst all those which have been consulted tend to agree that the presence of these two components signifies the potential for reaction, there have been some differences in the actual mechanisms postulated.

Jones and Tarleton (14) produced their 'tentative reaction mechanism' in the form of an opal particle reacting with alkalis to give an 'alkali-silica gel'. In more recent times, however, there has been general agreement that the principle reaction is not directly between the positive alkali metal ions (Na^+ and K^+) and the reactive silica but rather that it is the negatively charged hydroxyl counter ions (OH^-) which first react with the silica particles (12, 44, 45, 46, 47). These negative ions are produced when the alkaline substances go into solution in the pore fluid within the concrete. In addition, hydroxyl ions are also produced when some of the calcium hydroxide, released during cement hydration, enters into solution in the pore fluids. The Na^+, K^+ and Ca^{2+} cations are then drawn into the reacting particle to maintain electrical neutrality.

The importance of the alkali metal oxide content of the cement is, therefore, that it enhances the hydroxyl ion content of the pore fluids. This creates a pH value above that normally expected for concrete, which is determined by the solubility of the ever present calcium hydroxide. The chemical composition of the gel is now thought to be a complex and variable combination of calcium, sodium and potassium silicates (48). Analyses have shown there to be greater proportions of calcium in the gel than either of the other two alkali metals. This may be due to the greater electrical attraction of the Ca^{2+} cation when maintaining the electrical balance during the reaction or, alternatively, due to ion exchange between the Na^+ and K^+ ions and the Ca^{2+} ion after formation of the gel.

For expansion, and consequently damage, to occur it has always been understood that a third ingredient is necessary, namely water. The silicate gel takes in the water and expands causing internal pressures to be manifest. If these pressures become large enough to exceed the strength of the concrete then rupturing occurs.

Stanton's work showed that specimens made from a potentially reactive cement/aggregate combination did not react or expand when stored continuously under water. However, the same combination of materials was found to produce a deleterious effect when stored in a high humidity environment, or when subjected to alternate wetting and drying. With present knowledge, the explanation of this apparent anomaly would appear to be that when stored under water, the concentration of the hydroxyl ion in the pore fluids would never reach the level required for reaction. That is, effectively the large volume of water negates the effect of the high alkali cement.

There has always been a lack of agreement with respect to the way in which the overall process of the reaction progresses and in particular the mechanism by which the water is taken in by the alkali silicate gel. Jones (22) referred to work by other authors which ascribed the pressure build up to the formation of a semi-permeable membrane on the surface of the reacting particle, which then resulted in osmotic action. In the same paper other authors saw the effect as one of simple absorption of water into the gel as the chemical reaction proceeded.

There still does not appear to be a universally accepted and proven description of the mechanism of expansive pressure nor the severity of expansion to be expected (48). Diamond (49) considers that A.S.R. comprises two distinct stages. The first stage is described as purely chemical and involves the reaction of the alkali with the siliceous material, normally taking place within the boundary of the reacting particle. The second stage of the reaction is then the physical or physiochemical absorption of fluid into the gel formed during the first stage. This may produce local swelling at the site of the reacted particle and, if swelling continues, overall expansion and cracking may follow. This two stage approach, however, is not confirmed by Dent-Glasser (50) who describes the complex physiochemical process in concise and understandable terms. It is postulated that the reaction is a continuous process incorporating the imbibition of water as it progresses.

To summarise, from information available in current literature, expansive A.S.R. can take place in concrete when the three parameters of high alkali cement, reactive silica in the aggregate and sufficient water are all present. The fundamental reaction which takes place is between the reactive silica and hydroxyl ions (OH^-) in the pore fluids. As the hydroxyl ions react with the aggregate particles, so positive ions are attracted to the reaction site, thus maintaining electrical neutrality. These positive ions are the Na^+ and K^+ ions derived from the alkalis in the cement, in addition to the calcium cations (Ca^{2+}) derived from the calcium hydroxide released during the hydration of any Portland cement. The resulting gel product has an affinity for water, causing imbibition to occur as the reaction proceeds. Consequently, the gel product swells and, due to the attack of the hydroxyl ions on the structure of the siliceous particles (enhanced by the probable occurrence of the reaction sites within existing flaws in the poorly crystalline material), the aggregate particles are weakened and are thus able to swell and crack. This enables more hydroxyl ions to reach reactive sites and hence the process continues as the aggregate particles decompose.

3.3.2 Symptoms of Reaction and Mechanism of Damage

The main outward evidence of alkali-silica reactivity is cracking [5, 47], which enables the concrete to dissipate the internal forces developed by the expanding gel. In unrestrained concrete the cracks characteristically form a random pattern, often referred to as 'map cracking' because of its similar appearance to details such as roads, rivers and county boundaries shown on maps. In concrete which is restrained by, for example, reinforcement or prestressing tendons, the crack pattern is modified accordingly, with cracks tending to run parallel to the reinforcement. A more unusual sympton caused by the build up of expansive forces can be the physical movement of a particular section of a structure. This feature was noted on Val de la Mare Dam where misalignment occurred in certain sections of a concrete handrail along the crest walkway bridge (2).

Another common feature of the reaction is the exudation of gel from the surface of the concrete, in particular through any cracks. This gel often produces a white stain on the surface of the concrete as it dries out from its fluid state and reacts with carbon dioxide in the atmosphere. In some instances, where there is limited surface abrasion, the cracks can fill with gel and eventually form a ridge of hard material standing proud of the concrete surface (47).

Where the reaction takes place with discrete and fairly large reactive particles contained in the aggregate a feature known as surface 'pop-outs' can develop. This occurs when a reactive particle lies close to the concrete surface and the expansive forces cause a conical piece of concrete to be forced away from the main body. The reactive particle is normally found at the base of the 'pop-out'. In more severe cases extensive surface spalling can develop.

The physical damage to concrete caused directly by A.S.R. can therefore be any combination drawn from the list of cracking, surface pop-outs and surface spalling, the exudation of gel being more of an aesthetic problem. However, in laboratory studies it has been shown that despite even severe cracking the compressive strength of the concrete is often maintained (51). This possibility that the primary damage to the concrete might not render the structural member unserviceable was also recognised by Stanton (1). However, Stanton further commented that the initial cracking caused by A.S.R. may have very serious consequences with respect to the resulting lack of durability against subsequent secondary action from other factors. Frost action, reinforcement corrosion, salt ingress causing cement deterioration and greater susceptibility to carbonation may all result to compound the deterioration of the concrete.

3.3.3 Additional Factors contributing to the Extent of Reaction

Any factor which affects the quality of a concrete must have an effect on the extent of damage occurring due to a reactive aggregate/cement combination. Whether the effect is simply concerned with the physical behaviour of the concrete, and therefore its ability to withstand the disruptive influence of A.S.R., or whether it is of more fundamental relevance to the reaction itself is the important difference.

Stanton (1), Diamond (52) and Hobbs (53, 21) have all carried out research into the effect of the grain size of the reactive aggregate and in so doing have compared their experimental results with other published information. For his part, Stanton concluded that the expansion of mortar bars, containing siliceous magnesian limestone as the reactive component, tends to increase as the grain size of the reactive material is decreased, until a limit of maximum expansion is reached. After this limiting value Stanton found that further decreases in particle size quickly lead to virtually zero expansion occurring. Maximum expansion was measured for specimens containing reactive material with a particle size ranging from 170μm to 600μm, whereas a particle size of less than 170μm was found to yield negligible expansion. The work which yielded these conclusions was carried out using a fixed percentage (5%) of reactive material mixed with a known inert aggregate.

In a bid to establish why there should be such an abrupt end to the trend for increasing expansion with decreasing particle size, Diamond carried out similar tests using reactive material in seven size fractions between 150μm and 20μm. According to Stanton's experience all size fractions within this range would produce negligible expansion. However, Diamond found that even for the smallest particle size of 20-30μm significant expansion occurred. One complication to the direct comparison of these two investigations is that the reactive aggregate used by Diamond was an opaline material obtained from the Faroe Islands and therefore did not match the reactive material used by Stanton.

The later work of Hobbs was formulated with the foreknowledge of these apparently irreconcilable observations. Moreover, reference can also be made to observations which show that expansion increases with increasing particle size up to a maximum size of 0.5mm (54). Using Beltane opal as the reactive constituent, Hobbs studied the effect of particle sizes within the extremes of the aggregate grading specified for the A.S.T.M. mortar bar test (18). This test and the aggregate grading is fully discussed in the experimental part of the present research. Thus, for reactive material in five size fractions between 4.8mm and 150µm, Hobbs observed that expansion increases with decreasing particle size.

The lack of consistency of the observations gained from these independent research projects is further indication of the complexity of the reaction. One conclusion which must be drawn from these research programmes, when considered collectively, is that different reactive aggregates do not necessarily behave in a similar manner.

Relatively little information has been published concerning the influence of water/cement ratio on A.S.R. Some early comment on this aspect was made by Jones and Tarleton (14) based on observations which showed that expansion increases with increasing water/cement ratio, up to a maximum expansion for a ratio of 0.40. After this maximum expansion there was a small decline noted up to a ratio of 0.44 followed by a steady level of expansion up to the maximum ratio used of 0.56.

This pessimum effect for expansion with respect to water/cement ratio has also been observed by Lenzner (55). However, in this case, the ratio found to give maximum expansion was 0.60. It is perhaps worthy of note from Lenzner's results, that the reduction in expansion, caused by increases in the water/cement ratio above the pessimum value, was large when compared with the reduction in expansion caused by similar decreases in the water/cement ratio below the pessimum value.

Some results have failed to demonstrate this pessimum effect related to water/cement ratio, for example Dahms (56) refers only to an increase in damage with increasing ratios.

Highlighting the dependence of the effect of water/cement ratio on the amount of reactive aggregate present, Hobbs (21) produced a 'family' of curves relating the water/cement ratio to expansion. The emphasis placed on the corresponding comment that the curves are unique to the particular mixes which were tested is noteworthy, a point also mentioned by Lenzner.

By designing experimental work in a particular manner and manipulating the results accordingly, expansion caused by A.S.R. can be compared with other mix parameters, such as aggregate/cement ratio, or derived parameters, for example alkali/silica ratio (21). Properties such as porosity (8, 9, 10, 14, 22) and permeability may also be relevant. In particular, the permeability of the concrete must play a part in determining how freely the pore fluids traverse the concrete mass and allow the hydroxyl ions to reach the reactive sites. Moreover, it has been shown that in concrete where evaporation is possible from one surface only (e.g. concrete foundation bases) an effect can be produced whereby the alkali concentration is increased near to the open surface (19). This effect is obviously controlled to some extent by the permeability of the concrete and may be responsible for the occurrence of A.S.R. in an otherwise innocuous mix.

Whatever method is chosen for expressing the mix proportions to enable the effect of different parameters on expansion due to A.S.R. to be evaluated, the most important factors still remain the alkali content of the cement and the reactive silica contained in the aggregate. If each of these are present in suitable quantities and a source of water is available then A.S.R. will occur and all other parameters of the mix will only have a secondary effect.

3.4 LABORATORY RESEARCH INTO A.S.R.

3.4.1 Research Materials

The requirement for reliable but rapid laboratory tests into certain aspects of A.S.R. necessitates the use of reactive materials other than those which might be used in normal structural concrete. These materials can involve the artificial enhancement of cement alkali content or the use of an aggregate constituent which is highly reactive but unlikely to be used under practical circumstances.

Much of the research on A.S.R. has been carried out using either crushed Pyrex glass or crushed Beltane opal as the reactive aggregate. Beltane opal is a naturally occurring siliceous rock obtained from the Beltane Quarry in Napa Valley, Sonoma County, California. It has been shown to contain approximately 80% potentially reactive siliceous material (57), principally cristobalite and tridymite. A number of research projects have shown Beltane opal to exhibit a strong pessimum content of less than 100% when used in a mixture with a known inert aggregate. This pessimum content yields the maximum expansion under the particular conditions employed and has been found to be generally below 10% by weight of total aggregate. Further, it has been implied (21, 45, 53) that above the pessimum content expansions are progressively reduced until, beyond approximately 20% Beltane opal content, a level of negligible expansion is maintained.

Further research, using Beltane opal, by Hobbs (21) and by Gaze and Nixon (58) has determined that the ratio between the total equivalent alkali content and the reactive silica content (i.e. the Na_2Oeq/SiO_2 ratio) also has a critical value corresponding to maximum expansion. Figures 3.1 and 3.2, which are drawn from the data published by Gaze and Nixon, show this pessimum effect for expansion results taken on mature specimens at ages 4 months and 10 months respectively. At earlier ages of measurement the effect was absent within the range of alkali contents employed, as shown by Figure 3.3 drawn from the 3 month results.

Pyrex glass has also been found by certain authors (59) to exhibit a pessimum type of behaviour. However, in the majority of cases, maximum expansion has been gained with the use of 100% Pyrex glass as the aggregate.

The problem arising from these two highly reactive materials is the selection of which one more accurately mirrors the behaviour of reactive aggregates in the practical sense. Nixon and Gaze (60) claim that Pyrex glass appears to behave in a similar fashion to the reactive cherts found in the U.K. Conversely, Hobbs (61) considers that any information gained from research using Pyrex glass relates only to mixes containing this material and that Beltane opal is more suitable since opaline materials are commonly found to be the reactive constituents of natural concrete aggregates. Providing a third opinion, Figg (59) states that neither Beltane opal nor Pyrex glass act like a 'real' aggregate and are therefore both of limited use for relevant research. He suggests that the use of fused silica as the reactive medium may be more appropriate.

Whatever the outcome of this controversy, from the practical sense there is no current substitute for testing the actual cement/aggregate combination that is proposed for use. However, even this safety measure can produce anomalies in determining whether or not the combination will produce a deleterious effect. For example, certain U.K. aggregates, which are known to be reactive in the field and responsible for structural deterioration, have not reacted expansively in laboratory tests (19). Conversely, it has also been reported that some U.K. aggregates which have a long history of successful use in concrete have been categorised as reactive in laboratory tests (59). However, this latter case related to chemical tests rather than physical expansion tests.

3.4.2 Research Methods

It is unfortunate that direct comparisons between the results from independently carried out research projects are often not valid because standardised laboratory procedures have not been followed. Specimen type, specimen size, reactive aggregate and storage temperature are often different for two projects designed to study the influence of the same variable. This dilemma is typified by the different observations regarding the effect of particle size which were highlighted in an earlier section. In this way the need for close control on all factors inherent in any research project, but which are not the subject of the particular investigation, becomes apparent. If results are to be both repeatable and reproducible, whilst also accurately defining the effects of the factors being studied, then all other factors must be held constant. In addition, it is implied that the particular effects obtained may be limited to the set of experimental conditions chosen for the work being carried out. This emphasises the need for careful consideration of the most relevant conditions.

Recently, two critical reviews have been published on the variety of standard test methods which are currently available for studying A.S.R. (20, 62). Primarily these reviews considered the tests from the point of view of their practical viability in determining the likelihood of A.S.R. occurring in structural concrete. Such practical considerations must, however, also be relevant to the choice of appropriate test methods for research work.

Earlier work on the available forms of rapid and accelerated laboratory tests was carried out in the U.K. in the 1950s (14, 22). At this time some of the recognised standard test methods of today were only in their development stage.

The types of test which may be conducted fall into three broad categories, namely petrographic examination, chemical testing and physical expansion testing. In the practical situation Sims (20) emphasises the need for some kind of petrographic examination of a suspect aggregate as a prerequisite stage in any testing programme, if only to help choose the most suitable further tests to apply. For the type of research involved in the experimental section of this particular investigation, however, this is not of particular usefulness.

Of the remaining two categories of tests, the most widely known standard methods which are currently available are as follows:-

i) the mortar bar test (A.S.T.M. C.227-81) (18)
ii) the concrete prism test (C.S.A. A23.2-14A) (17)
iii) the rock cylinder test (A.S.T.M. C.586-69) (16)
iv) the rapid chemical test (A.S.T.M. C.289-71) (15)
v) the gel pat test (14)

Of these five tests the mortar bar method is widely regarded as the most reliable in testing for A.S.R. Anomalies can sometimes occur, as referred to earlier, when reactivity in the field cannot be reproduced in the laboratory. In addition, the rapid chemical test is applicable for use with rapidly expanding siliceous aggregates and has the advantage of speed in producing results. However, from the literature it would appear that this test is somewhat less reliable in determining a reactive aggregate, the errors tending to be on the conservative side and therefore causing some innocuous aggregates to be condemned as potentially reactive.

The other test in the above list which produces very rapid results is the gel pat test, which was regarded by Jones and Tarleton (14) as useful for gaining an early indication of materials likely to cause A.S.R. This early British research laid down a recommended test procedure for using this method but this has never been converted into a formally published standard.

Associated with the mortar bar method, described in A.S.T.M. C.227-81, is another form of the test which is designed to test the effectiveness of mineral admixtures in reducing expansion due to A.S.R.. This usage is described in A.S.T.M. C.441-69 (24) and involves the use of Pyrex glass as the reactive siliceous aggregate.

The two remaining test methods mentioned above are not generally considered to be particularly suited for use with rapidly expanding siliceous materials. Their particular field of suitability is for either slowly expanding siliceous materials or alkali carbonate reactivity (62).

The comparatively recent discovery of A.S.R. in the U.K. is perhaps responsible for the lack of any British Standard test methods. In B.S. 882: Part 2: 1973 'Coarse and fine aggregates from natural sources', there is no mention of any form of alkali-aggregate reaction. Under these circumstances most research work in the U.K. has been carried out using either the A.S.T.M. methods or individually developed procedures.

From the practical viewpoint, it must be emphasised that accelerated test procedures cannot reproduce field conditions or truly simulate field behaviour. However, the methods discussed have been found to provide reasonable estimates of field durability provided that they are not taken as strict pass/fail criteria. Both the published reviews on test methods which have been referred to recognise this situation. The level of reactivity to be expected in the field and the expansion which can be tolerated in service is a matter for enginering judgement based on the interpretation of the test results.

3.5 WAYS OF AVOIDING A.S.R.

It has been discussed that for a deleterious reaction to occur, that is A.S.R. with resultant damage to the stucture concerned, three parameters must all be present, namely a reactive aggregate, a cement containing sufficient alkalis and a supply of moisture. Removal of any of these parameters should therefore ensure avoidance of damage and represent the most simple and effective course of action.

In practice all Portland cements contain alkalis, though the amount is very variable, and in many structures it is difficult to remove all sources of moisture. The easiest and most definite solution is, therefore, the use of an aggregate proven to be inert. Whilst it is difficult to argue any flaw in the logic behind this statement, it is not always possible to implement this course of action due to non-availability of a suitable material within the economic constraints of the particular contract. Moreover, aggregates themselves, being a natural resource, are available only in finite quantities and eventually supplies of the ideal materials will expire. Already local shortages are being noted (63).

In certain applications the removal of the source of moisture may be practicable. An important consideration is that the moisture need not necessarily be in the form of 'running' water, the worst case being an intermittent supply, but that high humidity (above 75%) or heavy condensation can be equally dangerous (48). Also, in mass concrete structures, residual construction moisture within the concrete itself can be sufficient for expansion to occur. In other types of structure the avoidance of A.S.R. by the removal of the source of moisture may involve a continued risk. The highly reactive cement/aggregate combination which remains will chemically react, at least in part, and form the alkali-silicate gel. This product may then represent the potential for damage if, at any later stage, a source of moisture becomes available due to an unexpected change in the environment surrounding the structure or the failure of the external moisture protection system.

Where a potentially reactive or untried aggregate is, for particular reasons, specified for use in an unavoidably moist environment, then of the three main parameters involved in A.S.R., only the alkali content of the cement remains available as a means of avoidance. From the earliest published research (1) the use of a low alkali cement has been recommended as an adequate safeguard, a figure of 0.6% Na_2O_{eq} having been considered to be the maximum allowable cement alkali content. Since the richness of the mix also has an effect on the amount of alkali which is present in the concrete, more recently it has been suggested that a limit on alkali content should be expressed in terms of the amount of alkali present in the overall mix (21). Based on the acid soluble alkalis, the proposed limit in the U.K. is 3.0kg Na_2O_{eq}/m^3 of wet concrete (7, 21). In other countries different limits have been proposed, however, generally these have been in the range of 2.5kg Na_2O_{eq}/m^3 to 4.0kg Na_2O_{eq}/m^3. To some extent the chosen limit has depended on whether concrete or mortar specimens were used to carry out the research programme used to investigate the problem.

It has already been mentioned that conditions extraneous to the concrete itself may produce localised concentration of alkalis. This may result in reaction occurring at particular locations in the concrete when, 'on average', the overall cement/aggregate combination is satisfactory. Such a phenomenon can therefore negate an attempt to avoid A.S.R. by reducing the overall alkali level. Nixon et al (19) showed that the movement of moisture through concrete, towards a drying surface, can be responsible for the concentration of alkali metal ions. Some of the early failures in the U.K. which were discussed by Palmer (4) were concrete bases with most of their mass below ground and only their top faces exposed, thus offering a classic example of conditions likely to cause alkali concentration. Other types of structure, such as water retaining structures and road pavements, also offer the potential for water migration and hence alkali concentration.

In addition to the direct methods of avoiding A.S.R. there are a number of mineral admixtures which have been suggested as having a beneficial effect. Such materials have been included into the concrete mix as an additional component, as a partial replacement of the cement content or as a partial replacement of the fine aggregate content. The most commonly used admixtures are ground granulated blast furnace slag (g.g.b.f.s.) and pulverised fuel ash (p.f.a.). The latter is widely available as a waste material from power stations which are fuelled by pulverised coal and its use is reviewed in depth in Section 3.6 which follows.

For some considerable time, the introduction of p.f.a. and g.g.b.f.s. into reactive mixes have been studied as ways of reducing the destructive expansion arising from A.S.R..

However, they do not represent the complete list of suitable mineral admixtures, one of the most recent materials to be tried being silica fume (64) (also known as silica flour). In certain applications this by-product of the silicon and ferro silicon manufacturing industry has been shown to be very successful in reducing expansions (51, 65). The silica fume is incorporated into the concrete mix in similar fashion to p.f.a. and g.g.b.f.s., that is as a partial replacement of the cement content, but requires much smaller percentages to be used (5-10%).

Other research has shown that under some circumstances even the reactive aggregates themselves can be used to combat A.S.R. if employed in a finely divided state. Jones (22), referring back to Stanton's work of 1942, mentions that shales and opaline cherts can reduce expansion to a negligible amount when finely ground and used to replace 15 to 20% of the cement content. Later, Jones with Tarleton (10) confirmed this beneficial effect by using highly reactive malmstone. However, the reductions in expansion were fairly limited and did not result in a negligible level being achieved even with a 20% replacement.

A wide variety of other naturally occurring materials have been shown to have expansion reducing potential, in particular materials with a pozzolanic ability. For example, Tang Ming-shu et al (66) carried out research using a tuff which was found to be a more effective admixture than either p.f.a. or g.g.b.f.s. when used at the same cement replacement level.

Working on a different concept, Sprung and Adabian (67) also found mineral admixtures to be successful in reducing expansion. This research utilized the admixtures as a partial replacement for the aggregate rather than the cement, the authors considering this to be the only way in which the true value of these materials may be judged. P.f.a., trass, ground cristobalite and a silicon-glass dust were all found to be successful in reducing expansion to varying degrees. The most efficient material was the silicon-glass dust, which is similar to the silica fume mentioned earlier, requiring a replacement level of less than 5% to prevent severe expansion.

3.6 THE USE OF PULVERISED FUEL ASH TO PREVENT A.S.R.

3.6.1 The Effectiveness of P.F.A. in Reducing Expansion

In Britain, pulverised fuel ash, in the literal sense, is all the solid waste material produced by burning pulverised coal. However, the term p.f.a. is also used to refer to a particular portion of this material, known otherwise as 'fly ash', which makes up approximately 80% of the total. Fly ash comprises the particles which are fine enough to be carried by the flue gases and therefore require collection using mechanical separators and electrostatic precipitators. The remaining 20% proportion of the total material is known as furnace bottom ash which is a clinker formed when the larger ash particles fuse together. With respect to A.S.R. it is the fly ash which has beneficial effects and consequently in this thesis any reference to p.f.a. implies the fly ash portion only.

During the last ten years or so, and specifically since the first occurrence of A.S.R. in the U.K., there have been numerous published research projects which have included a study into the effectiveness of p.f.a. in reducing expansion due to the reaction (38, 51, 58, 60, 61, 66 to 71). Many of these investigations have been conducted in the U.K. In general the results obtained have been favourable to the use of p.f.a. which has been recommended as a suitable admixture for use with reactive mixes by two recent reports (48, 72). These reports present advice and guidance notes on the subject of A.S.R. to the construction industry.

It is also worthy of note that a wealth of research is still in progress on the use of p.f.a., as demonstrated by the amount of relevant information to be found in the proceedings from a recent international conference on A.S.R. (73) held in Denmark in 1983. This is possibly an indication that the use of p.f.a. is still not universally accepted nor the full extent of its effect understood.

Despite the above generalisation, not all the research has reached positive conclusions regarding the effect of p.f.a. in this context. Evidence from field studies, referred to by Hobbs (61) has sometimes shown pozzolans to be ineffective in controlling concrete deterioration. In one particular case cited, the use of a pozzolan was claimed to be responsible for an increase in both the number and width of cracks in a section of concrete road pavement. The type of pozzolan, however, was not defined. Resulting from field observations such as these, it has been recommended that the effect of p.f.a. with the particular aggregate/cement combination under consideration should be laboratory tested before being used in an actual construction.

This point has also been raised after a series of laboratory tests made in the U.K. (60).

From his laboratory investigation, Hobbs (61) concluded that p.f.a., when used as a partial replacement for the cement content in a reactive mix, can sometimes cause an increase in expansion due to A.S.R. The experimental results behind this conclusion were gained from specimens incorporating Beltane opal, as the reactive aggregate, at contents other than the pessimum value. Other features of this research are that the reactive portion of the aggregate was only included in the 150-300μm particle size range (previously found to be the most highly reactive size (53)) and when p.f.a. was used the amount of reactive aggregate was adjusted to maintain a sensibly constant ratio between the water soluble alkalis and the reactive silica content. In addition, the specimens were stored at a temperature of 20°C. More commonly the success, or otherwise, of p.f.a. has been judged without making any adjustments to the reactive aggregate content.

In a subsquent study Hobbs (38) acknowledged the beneficial contribution which p.f.a. can make. It was found that with a 30% to 40% cement replacement level long term expansion due to A.S.R. could be reduced. However, the results still showed that very early expansions could be greater than those produced by a corresponding control mix containing no p.f.a. and also that the amount of reduction in the long term was variable. An important conclusion reached by Hobbs from this work was that for concrete exposed to external moisture, damage due to A.S.R. is unlikely to occur if the acid soluble alkali content of the mix is kept below 3.0kg Na_2O_{eq}/m^3 by the substitution of p.f.a. for part of the cement content. In this limiting value for alkali content the p.f.a. is assumed to make a contribution of 0.2% by weight. The inclusion of this contribution to the mix alkali content from the p.f.a. represents another controversial aspect of the use of p.f.a.

Research work which has resulted in conclusions supporting the ability of p.f.a. to reduce expansions has been carried out in a number of overseas countries, such as Germany (67) and South Africa (51). This work has included the use of local materials in conjunction with Pyrex glass, Beltane opal and local natural reactive aggregates.

In Germany, Sprung and Adabian investigated the use of p.f.a. as a replacement for up to 60% of the fine aggregate content in reactive mixes containing pure opal in the grain size <0.09mm substituted into a known inert aggregate. The authors found this procedure resulted in expansion reductions and consider that it is the only true way to judge the performance of an admixture.

The South African work, reported by Oberholster and Westra, included experiments using both Pyrex glass and locally occurring natural aggregates which were known to be reactive. Initially the effect of p.f.a. was tested using Pyrex glass and the method described in A.S.T.M. C441. The results were positive in showing that p.f.a. was very effective in reducing expansive A.S.R. The follow-up tests, using a natural aggregate from the Malmsbury Group, were designed to examine whether similar effects could be obtained in relation to a natural material. It was found that a 20% replacement of the cement by the p.f.a. was equivalent in expansion reduction to using a cement which just satisfied the limit of 0.6% total acid soluble alkali content for low alkali cements.

As mentioned earlier, at the B.R.E. there is a large, on-going U.K. research programme concerning the use of p.f.a., from which various progress reports have been published (58,60). In addition, a wide variety of p.f.as. were tested by Buttler, Newman and Owens in 1980 (69). This latter work was carried out using Pyrex glass as the reactive aggregate and produced results which supported the view that all good quality p.f.a. will reduce expansion when used as a cement replacement admixture. One unusual outcome of this particular investigation was the suggestion that p.f.a. may show an 'optimum' characteristic. That is, there may be a maximum replacement level above which the beneficial effect of p.f.a. is curtailed or even superseded by a slight rise in expansion with further increases in the amount of p.f.a. used. This optimum replacement level was found to be in the range of 30%-35% replacement.

The B.R.E. work has explored not only the question of whether p.f.a. in general reduces expansions but also the relative effects of different p.f.as. in comparison to their alkali contents. In addition, the work includes the use of Pyrex glass, Beltane opal and U.K. reactive chert.

From the initial work with Pyrex glass all the p.f.as. which were tested were found to dramatically reduce the expansions measured on mortar bars when used to replace 10%, 20% or 30% of the high alkali cement content. For the use of p.f.a. to be an improvement on employing a low alkali cement, a replacement level of 20% or 30% was found to be necessary. The relative effectiveness of the p.f.as. was found to have a slight correlation with their alkali contents but was more conclusively governed by their pozzolanicities. For effective expansion reduction in mortar bars containing the reactive natural chert aggregate a replacement level of 30% was found to be required to improve on the use of a low alkali cement.

3.6.2 The Mechanism of P.F.A. when used as a Partial Cement Replacement

In the above discussion the consensus of opinion is shown to be that, in all but the most unusual situations, p.f.a. is a viable method of controlling damage due to A.S.R. However, the mechanism by which this effect is achieved remains a matter for considerable debate and disagreement. Two possibilities are considered to exist, namely that the p.f.a. acts either as a diluent for the alkaline material available for the reaction or alternatively that the p.f.a. has a more fundamental effect by inhibiting or modifying the reaction itself.

The notion that p.f.a. simply dilutes the alkali concentration because it is used to replace part of the cement content is complicated by the fact the p.f.a. also includes alkalis. Indeed, many p.f.as. could be seen to provide a much larger alkaline potential than the cement which it replaced when the total alkali content of the p.f.a. is considered. However, most of the alkalis within the p.f.a. are in solid solution and, as such, are probably not relevant to the reaction. Consequently there are two other expressions of alkali content which are used for p.f.a., namely the water soluble and available alkali contents. The latter value expresses the amount of alkali likely to be released from the p.f.a. for dissolution into the pore fluids of a concrete during the pozzolanic reaction and is therefore probably the most relevant to A.S.R. However, if the p.f.a. does actually undergo a reaction this may amount to the same as the total alkalis.

The dilution effect is supported by Hobbs following investigations at the C. α C.A. (38, 61) which were discussed earlier in connection with the 3.0kg Na_2O_{eq}/m^3 (equivalent to a water soluble alkali content of approximately 2.5kg/m^3) proposed as a safe limit below which A.S.R. is unlikely to occur. Again the experimental results which were used to assess the effectiveness of p.f.a. were gained from specimens containing Beltane opal, in the grain size 150-300μm, as the reactive constituent.

The German work of Sprung and Adabian (67) used p.f.a. to replace part of the fine aggregate content in recognition of the dilution effect involved in cement replacement. Significant expansion results were still obtained.

Notwithstanding the support for a simple dilution process, the majority of opinion considers that the effect of p.f.a. is more fundamental and greater than can be achieved by dilution. Considerable contributions to this effect have been made by Oberholster and Westra (51) and Gaze and Nixon (58) where particular experimental regimes have been devised to test the theory of dilution. Working with a natural aggregate Oberholster and Westra compared the expansion reducing effect of p.f.a. with alkali dilution using a mixture of high and low alkali cements. On the other hand, Gaze and Nixon used a low alkali cement for all mixes but added alkali in the form of potassium sulphate to maintain a constant alkali level even when p.f.a. was used. This latter approach was used in conjunction with Beltane opal in the grain size 150-300µm as the reactive material and included in the mix near to its pessimum level. Earlier work by Nixon and Gaze (60) also concluded that p.f.a. caused a greater reduction in expansion of mortar bars than could be accounted for by alkali dilution, even assuming that the p.f.a. has a zero contribution to the alkali level.

Since the time of some of the earliest work, the quality of the mortar or concrete - as reflected by its physical properties - has been seen to be influential on the amount of damage caused by A.S.R.. Jones and Tarleton (9, 10) found that the porosity of the mix was important, though in somewhat confusing fashion blamed both high and low porosity for a lack of expansion in certain mixes. More recently, since A.S.R. has generally been seen to be caused by hydroxyl ions diffusing towards the reactive aggregate particles, the permeability of the cement paste has become of major interest. This, then, is one mechanism by which p.f.a. can be effective in controlling the reaction, that is by reducing the permeability. The partial replacement of cement by p.f.a. can result in a lower water demand to attain a similar workability (74, 75). In addition, the pozzolanic reaction, which is known to take place between p.f.a. and the calcium hydroxide released during cement hydration (12), can be responsible for a reduction in permeability. This can occur because the products of the reaction hydrate to form C-S-H gel which is additional to that normally produced by the hydrated cement alone. Buttler et al (12, 76) regard this as a means of restoring modern Portland cements to the composition of earlier cements which had a much lower C_3S : C_2S ratio and consequently produced a greater quantity of C.S.H. gel per unit mass of cement. For the cement replacement levels normally used, the amount of C.S.H. gel produced by the p.f.a. reaction is thought to be greater than that lost due to the reduction in the cement content.

Another aspect of the effect of p.f.a. is that the preferential reaction between the p.f.a. particles and the hydroxyl ions is considered to inhibit the reaction between the hydroxyl ions and the reactive silica particles in the aggregate. On the other hand, Hobbs interpreted his own results, obtained with Beltane opal, as showing no suggestion that the hydroxyl ions were depleted by the p.f.a., thus concluding that there was no evidence to suggest a reaction between the hydroxyl ions and the p.f.a. Even with a reaction, however, the depletion of the hydroxyl ion concentration would not be expected since there remains an abundance of the hydroxide materials available to go into solution and maintain the pH level. It may, therefore, be simply the preferential nature of the reaction between the hydroxyl ions and the p.f.a. which assists in inhibiting A.S.R.

One advantage of the pH level being maintained when p.f.a. is used in concrete, is that the passivity effect, known to protect the reinforcement from corrosion, is also maintained.

Whatever the mechanism of its effect, the minimum benefit achieved by the use of p.f.a. must be equivalent to the apparent alkali dilution effect which occurs, assuming that the p.f.a. contributes little to the alkali content of the overall mix. This attitude is reflected in the guidelines on the practical use of p.f.a. (48), produced by the recent working party, which allow for the p.f.a. to be used simply to reduce the amount of alkalis in the mix to below the 3.0kg Na_2O_{eq}/m^3 proposed as a safety limit. Should there be a more fundamental effect over and above dilution, then this should serve to secure a more favourable safety factor against the reaction.

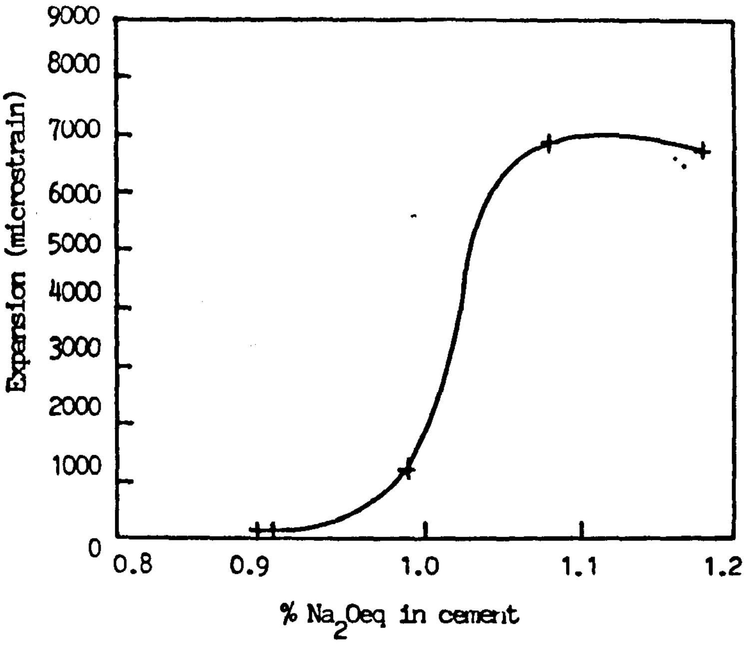


Figure 3.1

Effect of alkali dilution on the expansion of mortar bars with constant Beltane opal concentration - age 4 months.
(after Gaze and Nixon (58))

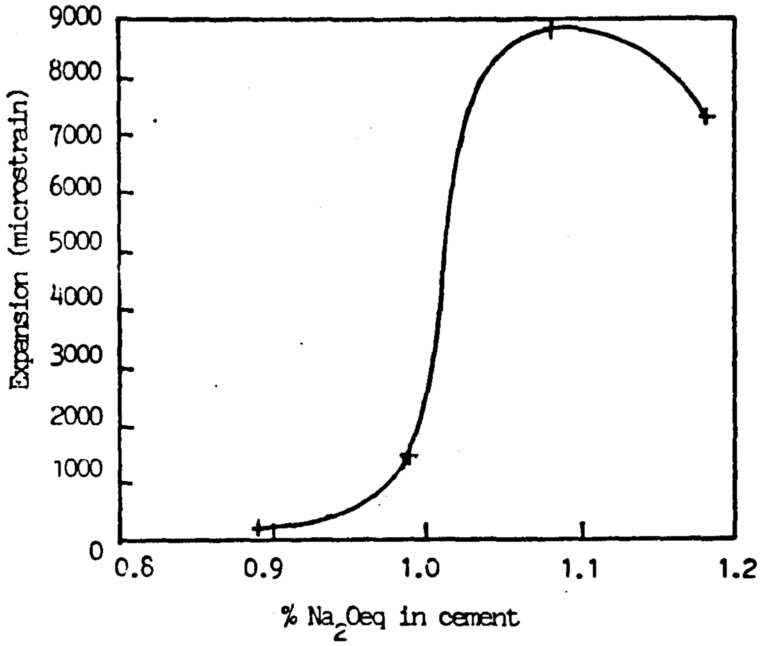


Figure 3.2

Effect of alkali dilution on the expansion of mortar bars with constant Beltane opal concentration - age 10 months.
(after Gaze and Nixon (58))

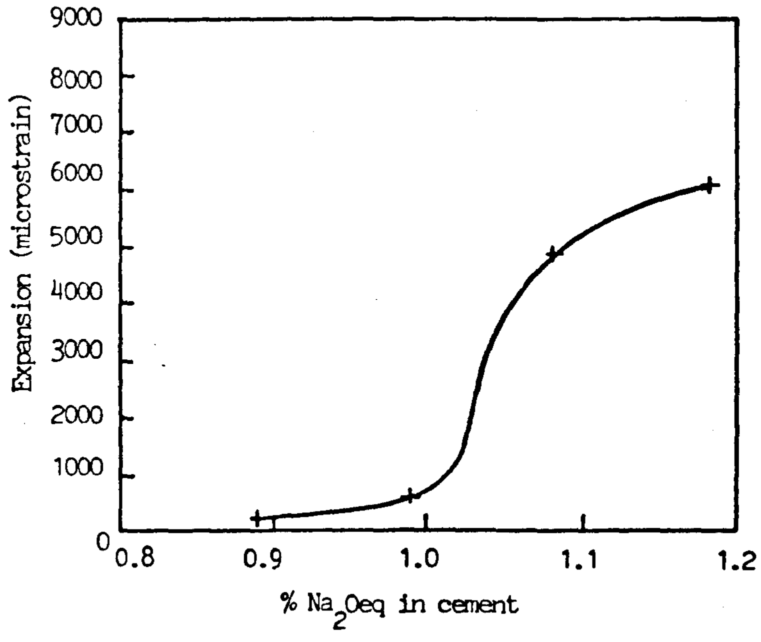


Figure 3.3

Effect of alkali dilution on the expansion of mortar bars with constant Beltane opal concentration - age 3 months.
(after Gaze and Nixon (58))

CHAPTER 4 STATISTICAL METHODS OF EXPERIMENTATION - FACTORIAL ANALYSIS

4.1 STATISTICS IN EXPERIMENTATION

The overall topic of experimentation using statistical methods is discussed in this chapter with reference to the important principles involved and the types of experimental designs available. Detailed attention has been limited to those sections considered to be most suited to the research topic being studied, namely A.S.R. in concrete. Complete accounts of the topic of statistical experimentation are given by, among others, Winer (77), John and Quenouille (78), Cox (79) and Davies (80).

As with any specialised subject, there is a particular vocabulary associated with the use of statistical experimentation and this is explained in some detail in Section 4.1.2 below.

4.1.1 General Introduction

The advantages of statistical methods of experimentation have long been recognised in many diverse fields of interest. In particular such methods have been used extensively for research in agricultural, biological and behavioural science. All these disciplines tend to involve observations which are affected by fluctuations caused by parameters outside the control of the experimenter. Thus, experimental error is quite large in comparison to the effects being investigated and, if conventional methods of experimentation are used, the interpretation of the results can become difficult leading to a low level of confidence in any conclusions which may be drawn. This situation produces the first requirement for a statistical approach which enables many replications of the experiment to be carried out and the experimental error estimated.

Another feature of many fields of investigation and research is the need to include a large number of variables in any experiment, so that the effect of each variable may be established in relation to the performance of the whole experimental unit. The inclusion of a large number of variables in an experiment leads to a vast amount of output being obtained and again the requirement is for statistical methods to assist with interpretation.

Having realised the need for a statistical approach to assist at the interpretation stage it is important also to realise that for a meaningful analysis of experimental results to be possible the whole experiment must have a statistically based design.

Within the field of Civil Engineering there are many areas of interest and topics of research which fall into the category described above. In particular concrete is a classic example since it is a conglomerate material and hence involves a large number of factors in its make-up. Further, each of these factors has its own inherent variation which the experimenter would have great difficulty in controlling.

Indeed, to attempt to control such variations in the constituent materials may well be considered an unacceptable practice since it would constitute suppression of the natural processes which are normally involved, resulting in the research being less closely related to the practical situation.

Traditionally the method of experimentation employed for concrete studies is that of considering each component of the mix separately by varying it in a step-by-step manner over a suitable range whilst holding the other components constant. The components can be either quantitative or qualitative. This method automatically assumes that the effects of the experimental factors are independent and can correspondingly lead to conflicting experimental results if in truth the factors interact with one another. Moreover, the net result of a large research programme can often be a vast amount of information, the interpretation of which is often extremely difficult. This is because the results cannot be condensed into a form which is sufficiently concentrated to give an immediate overall picture of the recorded effects. Contradictory conclusions can therefore arise because of the restrictions inherent in the basic approach to the experimentation.

This is where the Factorial Method of experimental design and analysis becomes advantageous since not only does it allow an overall comparison to be made of the effects of the individual factors themselves but also permits the evaluation of the interactions, if any, between the factors. This is achieved by allowing each factor being studied to vary at each stage of the experiment.

Inevitably certain assumptions must be made so that standard statistical techniques may be applied to the results of the particular experiment. These assumptions concern the nature of the results themselves and they are listed below:-

i) normal distribution of unaccountable variations (or errors) in different measurements of the same effect.

ii) different measurements of the same effect are independent.

iii) the relative sizes of errors manifest in different measurements are unrelated to any treatment factor of the experiment.

The first assumption requires little explanation. The law of normal distribution of errors is well established and widely accepted for measurements taken on concrete specimens since it forms the basis of the most recent method of concrete mix design as explained in "Design of Normal Concrete Mixes" (81).

The second assumption emphasises the fact that when measurements are repeated the process must be carried out on an independent subject rather than simply repeating the same measurement on the original subject.

The final assumption is related to the theory of homogeneity of errors, which enables an overall estimate of experimental error to be made rather than having individual estimates of error for each treatment combination. The case must not arise where larger or smaller errors are associated with certain experimental treatment combinations. Under such circumstances a single estimated value for experimental error would not be valid and any subsequent tests of significance would become meaningless. However, according to the literature (Winer (77)) the method is sufficiently tolerant to allow some departure from this theoretical ideal without complete invalidation of the derived information.

In most types of experimentation, and particularly where a measurement process is involved, there is another type of error which may be manifest due to progressive familiarisation of the experimenter with the procedures involved or an unnoticed link between the experimental treatments and a characteristic of the specimens themselves. This type of error is known as Systematic Error. Serious contamination of the experimental results can arise if suitable steps are not taken to eliminate this error.

Neutralisation of the effects of systematic error is achieved by the process of Randomisation. This method was first devised by Fisher (82) and involves the application of the treatment combinations chosen for inclusion in the experiment in a random order so that any changes in experimental procedures are spread out randomly over the whole extent of the experimental programme. In this way unbiased estimates of the treatment effects may be obtained.

Any programme of experimental research must, then, follow the three logical stages of design, implementation and analysis in order that the fullest amount of information may be extracted from the resources which are being utilised.

4.1.2 Explanation of Terminology

Many of the terms associated with the subject of statistical experimentation have already been used in the foregoing introduction. In this section the meaning of these terms is explained with respect to the type of experiment chosen for the experimental work described in Chapters 5 and 6. In some instances mention will be made of certain statistical parameters and processes which are used in the analysis of the results. These are further explained in Section 4.1.3.

The term FACTOR is interchangeable with the term EXPERIMENTAL VARIABLE and is used to define the individual components incorporated in the make-up of the experimental specimens. A factor can be one of two types, either a TREATMENT FACTOR which is under the direct control of the experimenter or a CLASSIFICATION FACTOR which is not under the direct control of the experimenter but which can be used for classifying or grouping the experimental observations. For example a treatment factor in a experiment studying certain aspects of concrete may be the type of cement or aggregate whereas the age of the specimens at the time of measurement would be a classification factor.

Any experiment which involves the use of only a single factor is known as a UNIFACTOR experiment. Where the make-up of the experimental specimen involves more than one factor the experiment is described as FACTORIAL. Moreover, there are different types of both unifactor and factorial (multifactor) experimental designs, each with their associated methods of results analysis.

In an experiment, each factor or experimental variable can be present in varying stages or LEVELS. These levels can be quantitative such as the proportion of cement in a concrete mix (400g, 500g) or qualitative such as the type of cement (O.P.C., S.R.P.C., H.A.C.)

The DIMENSIONS of a factorial experiment are an expression of the size of the experiment as defined by the number of factors and levels involved. Hence, if an experiment includes four factors each at three levels the dimensions of the experiment are 3 x 3 x 3 x 3, or alternatively, in a special case of symmetry such as this, 3^4.

Within the overall dimensions of a factorial experiment each separate experimental unit is made up from a particular TREATMENT COMBINATION which can be considered as analogous to a recipe. In an experiment where the treatment combinations are repeated, each section of the experiment having a consistent treatment combination is known as a CELL of the experiment.

The purpose of most experiments is to assess the EFFECT which the treatment factors have with respect to the subject being studied. Normally this involves some form of measurement process which monitors any changes in the DEPENDENT VARIABLE, that is the criterion chosen to quantify any variation. It will be seen in Chapter 5 that the dependent variable for the experimental work described in this thesis is the EXPANSION of mortar bar specimens. Effects which are attributable to the treatment factors come in two forms, namely MAIN EFFECTS and INTERACTIONS.

The main effects obtained from a set of experimental results are an expression of the changes in the dependent variable which are attributable to the variations in each of the individual treatment factors. There is a main effect for each level of each treatment factor and its value may be positive or negative. Strictly, then, a main effect is defined as the difference between the mean of all the observations taken on the dependent variable with a particular factor at a particular level and the grand mean of all the observations in the experiment. From this definition it is seen that the main effects of a factor are not simply concerned with the specific levels of a specific factor but depend upon all levels of all the factors involved in the experiment.

Often of more practical usefulness are the differences between the main effects due to the different levels of a factor, which are known as the DIFFERENTIAL MAIN EFFECTS. By definition, a differential main effect is a measure of the extent to which the mean on the dependent variable varies from one level of a factor to the next. Thus it follows that if a factor has a great influence on the dependent variable the differential main effects will be large, whereas for a factor of negligible influence the differential main effects will approach zero.

The interaction effects obtained from a set of experimental results are an expression of the extent to which the changes monitored in the dependent variable from one treatment combination to another cannot be predicted purely from the sum of the corresponding main effects. Interaction can, therefore, be looked upon as an expression of the non-additivity of the main effects or the interdependence of the effects attributable to the various factors. An interaction effect may be derived from any number of factors from two up to the total number of factors in the particular experiment. The term used to define the level of interaction is its ORDER. A FIRST ORDER interaction effect is an interaction between two factors, the order of interaction increasing numerically by one for each additional factor involved. Alternatively, a first order interaction may be described as a 2-factor interaction.

In similar manner to the main effects, the interaction effects can be split up into DIFFERENTIAL INTERACTION EFFECTS. For the particular usage of factorial experimentation that follows, however, such an in depth explanation is not required.

In the practical sense, the requirement of a set of experiments is not so much to establish the numerical value of each main effect and interaction effect, or their differential counterparts, but rather to establish whether or not these effects exist and then to assess their relative overall size and importance. The statistical tool used to achieve this requirement is the analysis of variance, whereby the variance due to each effect can be calculated. For example, the variance which is attributable to the main effects of a particular factor is an overall measure of the extent to which the mean on the dependent variable differs for the various levels of that factor and differs from the grand mean of all the observations. If the variance due to the experimental error inherent in the measurement process is also assessed, then the comparison of these two variances can be used to determine the level of significance of the effect. The various levels of significance for the treatment effects can then be used to compare their relative importance and to show whether or not these effects cause variations which are substantially greater than can be explained by the errors involved in their measurement i.e. than can be explained by pure chance.

4.1.3 Explanation of relevant Statistical Terms and Processes

If the results obtained from an experimental programme are used to estimate the effects caused by the treatment factors involved, then some measure of the reliability of these estimates is required. This requirement is usually satisfied by the STANDARD ERROR of the results which have been used to produce the estimate of the effect. The value of the standard error can then be used to set CONFIDENCE LIMITS which represent extreme values between which the true value of the estimated effect may be expected to fall with a specified degree of confidence.

The confidence limits themselves are found by adding and subtracting a multiple of the standard error to and from the mean value of the estimated effect. The appropriate multiple of the standard error is dependent not only on the degree of confidence required but also on the accuracy with which the standard error has been estimated. It is normal for the accuracy of the standard error to be defined by the DEGREES OF FREEDOM inherent in its estimation, which in turn are set by the number of independent observations used to estimate the standard error. Having established the degrees of freedom involved, the appropriate multiple of the standard error can be obtained from standard statistical tables known as 't' TABLES.

The method used to estimate the standard errors from a set of experimental results is known as the ANALYSIS OF VARIANCE (ANOVA). This process enables a single, but complex, set of computations to partition the variations in a suitable set of experimental observations into components which are attributable to particular causes plus one component which is unattributable to any particular cause. The latter component is the component due to experimental error and is known as the RESIDUAL or ERROR component.

The ANOVA involves calculation of the MEAN SQUARES of the observations which provide unbiased estimates of the VARIANCES, the variance being directly related to the corresponding standard error. Each component of variance which relates to a particular cause may then be used to test the LEVEL OF SIGNIFICANCE of the effect in relation to the total variation in the observations. The test used in the ANOVA is the VARIANCE - RATIO or 'F' TEST. This is carried out by obtaining the ratios between the mean squares that are attributable to the effects under consideration and the residual mean square. The resulting ratios can then be compared with standard tables of the 'F' DISTRIBUTION and the appropriate level of significance obtained. Again, the level of significance is dependent of the degrees of freedom of the two mean squares used to obtain each particular variance ratio.

The level of significance is often expressed in terms of the probability that the effect is caused by pure chance. This is known as the TAIL PROBABILITY. For example, a tail probability of 0.01 would indicate that the variation would only occur by pure chance in 1% of cases. Conversely, in such a case it may be said with 99% confidence that there is a real variation in the value of the dependent variable which is attributable to the cause being examined.

There are no fundamental rules with respect to the cut-off level for significance below which the variation may be considered to be indistinguishable from chance or experimental error. Hence, the decision of what value to use must be made for the individual case. For guidance, however, it is generally accepted that for tail probabilities of under 0.01 the existence of a true effect can be regarded as highly likely. For tail probabilities of between 0.01 and 0.05 the existence of an effect is normally regarded as probable, though under certain circumstances this range is often extended to a tail probability of 0.10. Whatever value is chosen as the cut-off level, it is important to note that there is always a risk, no matter how slight, of either rejecting a genuine effect or failing to reject a chance variation.

4.1.4 Planning an Experimental Programme

The first, and often most difficult, stage of planning an experimental programme is to decide what the experiment is intended to investigate. In most cases, by necessity only a small proportion of the total range of possibilities can be examined leaving the problem of deciding the overall population, which by induction, the sample is intended to represent. The sample must then be carefully chosen so that it is an unbiased representation of the overall population and, therefore, able to accurately predict the population values which are required. In general, larger populations will require a larger experiment to ensure suitable results.

Perfection can only be achieved through an infinite number of observations, since then the true values will be established.

However, by utilising correct methods and efficient experimental designs, reasonable estimates may be obtained from a limited number of observations. Provided that the experiment allows the reproducibility of the results to be assessed, then the reliability of these estimates can be measured.

The next stage of planning is, therefore, to decide upon the required accuracy and then to evaluate a means of achieving this accuracy. Once the coverage of the experiment has been established there are many ways of improving accuracy which fall into two main categories, namely experimental technique and statistical procedures.

Experimental technique can be improved by refining the methods of applying the treatments and measuring the results and also by standardising the experimental conditions, procedures and materials. Often, the standardisation of the experimental material provides the largest obstacle to successful experimentation since it may not be susceptible to control. Under such circumstances it may be possible to form groups of experimental units of near homogeneous quality. The experimental treatments can then be applied in such a manner that most of the treatment effects are still obtainable with only a few being lost because they are restricted to within a particular group.

This, then, is the link between improvements in experimental technique and the use of statistical procedures, since the formation of the groups of experimental units is one of the statistical methods associated with accuracy enhancement. The other main statistical method of improving accuracy is the analysis of covariance which is useful if a series of extra measurements can be taken and used to account for some of the variability in the final measurements. An example may be the unavoidable but measurable differences in treatment subjects at the start of the experiment. It is important, however, that these measurements should not in any way reflect the treatment effects that are being observed.

In the experimental work which follows it will be seen that the choice of a recognised standard test method does much to eliminate extraneous variations in the observations. Close control of testing procedures, environmental conditions and specimen production methods in conjunction with the processing of the experimental materials into standard forms ensures maximum consistency for the experimental specimens and measurements.

4.1.5 The Choice of Experimental Design

With the foregoing planning of Section 4.1.4 in mind the following three main steps must be taken in choosing the most suitable experimental design:-

i) a decision on whether the design is to be of the unifactor or factorial type,

ii) a decision on whether the observations require grouping in order to eliminate any unwanted source or sources of variation in the final measurements and

iii) a decision on whether the number of treatment combinations in a full replication can be fitted into a single block, i.e. whether a 'complete block' design or an 'incomplete block' design is appropriate.

A classification of the main types of design, both unifactor and factorial, is given in the table below:-

<table>
<tr><th></th><th></th><th>UNIFACTOR DESIGNS</th><th>FACTORIAL DESIGNS</th></tr>
<tr><td rowspan="2">COMPLETE BLOCK</td><td>ONE GROUPING</td><td colspan="2">Randomised Blocks (Full factorial replications)</td></tr>
<tr><td>TWO GROUPINGS</td><td colspan="2">Latin Squares</td></tr>
<tr><td rowspan="2">INCOMPLETE BLOCK</td><td>ONE GROUPING</td><td>Balanced Incomplete Blocks
Partially Balanced and Cyclic Designs</td><td>Confounded Designs
Fractional Replication
Split-Plot Designs</td></tr>
<tr><td>TWO GROUPINGS</td><td>Youden Squares
Lattice Squares</td><td>Quasi-Latin Squares</td></tr>
</table>

Since concrete is the topic of research being considered in the present investigation, the unifactor approach to experimentation can immediately be regarded as unsatisfactory for the reasons set out in earlier discussion when the multifactor nature of the material was highlighted. Hence the choice of experimental design rests with those procedures listed under the Factorial Design heading in the above table.

Of the complete block factorial designs the most straightforward type is the Randomised Blocks system since it is probably the easiest to use and is very versatile. Any number of treatment factors and replications can be catered for. The treatment material is firstly stratified into blocks which are, as near as possible, homogeneous. Then, by random application of a full replication of the treatment combinations to each block, the treatment means give rise to unbiased estimates of the treatment effects. The subsequent analysis of variance can therefore be used to obtain the variances due to blocks, treatment factors and residual. The latter may be used to calculate the standard errors of the means and differences between means thus establishing the confidence limits for these statistics.

It is pertinent, here, briefly to consider the format of the experimental work being undertaken. The study of A.S.R. in concrete involves the production of laboratory specimens of concrete or mortar which are made from a combination of the component materials. The treatment factors are, then, the amounts or quality of the component materials. Since the experimental unit is made solely from the treatment materials themselves, and does not involve the application of treatment factors to an existing experimental unit whose make-up is not controllable, the question of homogeneity of the experimental units and stratification into blocks does not arise in this special case. Hence the Factorial Design of the Complete Block - One Grouping type (i.e. Randomised Blocks) is appropriate. This may more conveniently be referred to as simply a Full Factorial Replication, as shown in the table above.

The most frequently used incomplete block designs of the factorial type are Fractional Replication and Confounded Designs, probably due to their relative simplicity. Notwithstanding the above comments, whilst the latter type of experiment involves the use of blocking a brief outline of the associated processes will be included.

Confounding is employed when a factorial experiment becomes very large and would therefore require correspondingly large blocks to be used if a randomised block design were attempted. This, in turn, would increase the residual variation due to the lack of homogeneity of the treatment material within each large block. Confounding enables smaller blocks to be used since each block contains only a proportion of the full replication of treatment combinations. The smaller blocks reduce the residual variation but as a result of the confounding some of the treatment effects are lost since they cannot be separated from the effects of the block differences themselves. That is to say some of the information is 'confounded with blocks'. By careful design only the least valuable information can be confounded thus leaving the most sought after effects free from any block interference.

Fractional replication is closely related to confounding since the treatment combinations used in a fractional factorial design represent the equivalent to one block out of a confounded design which involves the same treatment factors. The fractional factorial method is also similar to confounding in the respect that some of the information which would be produced by a full replication is lost. This is because only a proportion of the total number of the available treatment combinations are actually employed. However, again by careful design, contamination of the most important information can be minimised. Fractional replication causes the variations due to more than one effect to be grouped together in an inseparable way. It is therefore essential that a feature of the experimental design is for the main effects and the most important interactions to be grouped only with the unimportant high order interactions. Groups of inseparable effects are said to be 'aliased' with one another.

The need for a method such as fractional replication arises because, as the dimensions of a factorial experiment are increased by the inclusion of more factors and more levels then the total number of treatment combinations rises rapidly. For example, if 5 factors are to be investigated and each is to be included at 3 different levels than there are $3^5 = 243$ treatment combinations in each replicate.

Confounding does not reduce the total number of treatment combinations to be included but merely splits them up into smaller groups. Fractional replication, however, reduces the number of treatment combinations which are actually carried out. In the example quoted the reduction would be by a factor of $1/3^x$ where 'x' represents the order of the fractional replication.

The method of fractional replication can be effective in one of two ways. The same coverage can be gained from a smaller experiment which requires fewer observations without seriously affecting the most important information or, alternatively, the same number of observations can be used to provide information on a wider field of study.

The design of both types of incomplete block experiments discussed above is controlled by a single parameter or group of parameters selected so that the most important information is retained. These parameters are known as Confounded Interactions or the Defining Contrast for confounded designs and fractional replication respectively.

4.2 DETAILED DISCUSSION OF SELECTED DESIGN

In Section 4.1.5 the two types of experimental design considered to be the most appropriate in this particular instance were found to be Full Factorial Replication and Fractional Replication. The power of the latter, in enabling the number of treatment combinations actually performed to be reduced, has obvious benefits and therefore much consideration was given to the use of this type of design. The purpose of the research, however, was to investigate A.S.R. in concrete using statistical experimentation as a research tool, and not vice-versa. Consequently the overriding decisions were made with A.S.R. as the main priority.

At an early stage, it was decided that each factor included in the experiments should be present at as many levels as practicable and that the number of factors should be confined to those considered most relevant. Therefore, it would have been ideal to carry out an experiment involving a fractional replication of perhaps 4 factors each at 4 levels. However, the power of fractional replication lies in experiments involving a large number of factors each at 2 or 3 levels, when the contamination of the treatment effects can be kept to an acceptable level. In the case of the 4^4 experiment mentioned above, if a 1/4 replicate is attempted, the main effects are aliased with interactions of an unacceptably low order. Since a suitable fractional replication design could not be found, all the experimental work was carried out on a full replication basis.

4.2.1 Notation

To permit the notation to be explained, a two-factor experiment will be taken as an example. The notation for larger experiments is simply an extension of the same system.

It is necessary to consider two frames of reference, these being the overall POPULATION and the EXPERIMENT which is intended to represent the population. For both the population and the experiment the two factors are represented by the symbols A and B. In general all other symbols will refer either to the population when they will be upper case or contain upper case suffices, or to the experiment when they will be lower case or contain lower case suffices. All the required symbols are listed below:-

A , B	the two factors
P , Q	the number of levels of each factor present in the population.
p , q	the number of levels of each factor present in the experiment.
N	the number of possible observations under each cell of the population (often infinite).
n	the number of observations taken under each cell of the experiment.
a_I (I=1, P)	an arbitrary level of factor A when the reference frame is the population.
a_i (i=1, p)	an arbitrary level of factor A when the reference frame is the experiment.
b_J (J=1, Q)	an arbitrary level of factor B when the reference frame is the population.
b_j (j=1, q)	an arbitrary level of factor B when the reference frame is the experiment.
ab_{IJ}	the general treatment combination when the reference frame is the population.
ab_{ij}	the general treatment combination when the reference frame is the experiment.

The dimensions of the experiment are thus p x q with n observations per cell out of an overall population of dimensions P x Q with N possible observations per cell, as defined by the two reference frames of treatment combinations below:-

Field of treatment combinations in the population

	b_1	b_2	- - - -	b_J	- - - -	b_Q
a_1	ab_{11}	ab_{12}	- - - -	ab_{1J}	- - - -	ab_{1Q}
a_2	ab_{21}	ab_{22}	- - - -	ab_{2J}	- - - -	ab_{2Q}
⋮	⋮	⋮		⋮		⋮
a_I	ab_{I1}	ab_{I2}	- - - -	ab_{IJ}	- - - -	ab_{IQ}
⋮	⋮	⋮		⋮		⋮
a_p	ab_{p1}	ab_{p2}	- - - -	ab_{pJ}	- - - -	ab_{pQ}

Field of treatment combinations in the experiment

	b_1	b_2	- - - -	b_j	- - - -	b_q
a_1	ab_{11}	ab_{12}	- - - -	ab_{1j}	- - - -	ab_{1q}
a_2	ab_{21}	ab_{22}	- - - -	ab_{2j}	- - - -	ab_{2q}
⋮	⋮	⋮		⋮		⋮
a_i	ab_{i1}	ab_{i2}	- - - -	ab_{ij}	- - - -	ab_{iq}
⋮	⋮	⋮		⋮		⋮
a_p	ab_{p1}	ab_{p2}	- - - -	ab_{pj}	- - - -	ab_{pq}

It is pertinent to mention here the equivalent short-hand notation which is used in the experimental sections of the research being described. The treatment combination ab_{11} is denoted simply as "00", further examples are listed below for the four-factor experiment actually used:-

0000	$abcd_{1111}$
2012	$abcd_{3123}$

Hence, the levels of factor A run from 0 to (p-1). Whilst this appears to be a complication at present it simplifies certain processes involved in the design and analysis of experiments, particularly by computer, and is the most common notation used. However, the long-hand version is more suited for use in explaining the techniques involved.

In the two different frames of reference above, the symbol ab_{11} may refer to different treatment combinations since the experiment may not include the first level of each factor which occurs in the overall population.

The factors A and B in the experiment can be designated either FIXED or RANDOM depending on the way in which the levels of each factor present in the experiment were selected. The factor A is a random factor if the p levels present in the experiment were selected from the P levels available in the population in a random manner. If, on the other hand, the full number of levels of factor A are included in the experiment, making p equal to P, then factor A becomes a fixed factor. A special case of the latter designation is where the p levels of factor A in the experiment have been selected in a systematic fashion from the P levels available in the population. Under these circumstances the experiment is only representative of the p levels of the factor A in the population, thus reducing P to $P_{effective}$ which is equal to p. In the experimental work described later, the levels of the various factors were selected in a systematic, non-random manner thereby causing them to be designated 'fixed'.

Additional symbols are required with respect to the computations which are carried out on observations obtained; these are listed below:-

X_{IJK}	the general observation under treatment combination a_{IJ} when the frame of reference is the population, where K has the values 1 to N.
X_{ijk}	the general observation under treatment combination a_{ij} when the frame of reference is the experiment, where k has the values 1 to n.
AB_{IJ}	the sum of all the N observations under treatment combination ab_{IJ} in the population.
AB_{ij}	the sum of all the n observations under treatment combination ab_{ij} in the experiment.
μ_{IJ}	the mean value of the N observations under treatment combination ab_{IJ} in the population (i.e. the general cell mean in the population).

$\overline{AB}_{ij}$	the mean value of the n observations under treatment combination ab_{ij} in the experiment (i.e. the general cell mean in the experiment).
$\mu_{I.}$	the average value of the cell means for all cells with factor A at level a_I in the population.
$\overline{A}_i$	the average value of the cell means for all cells with factor A at level a_i in the experiment.
$\mu_{..}$	the grand mean of the dependent variable for all the possible observations under all possible treatment combinations (i.e. in all the cells) of the population.
$\overline{G}$	the grand mean of the dependent variable for all the observations in all the cells of the experiment.

The two reference frames for all observations and calculated means can now be defined.

Field of means in the population

$$
\begin{array}{c|cccccc|c}
 & b_1 & b_2 & \text{- - - -} & b_J & \text{- - - -} & b_Q & \\
\hline
a_1 & \mu_{11} & \mu_{12} & \text{- - - -} & \mu_{1J} & \text{- - - -} & \mu_{1Q} & \mu_{1.} \\
a_2 & \mu_{21} & \mu_{22} & \text{- - - -} & \mu_{2J} & \text{- - - -} & \mu_{2Q} & \mu_{2.} \\
\vdots & \vdots & \vdots & & \vdots & & \vdots & \vdots \\
a_I & \mu_{I1} & \mu_{I2} & \text{- - - -} & \mu_{IJ} & \text{- - - -} & \mu_{IQ} & \mu_{I.} \\
\vdots & \vdots & \vdots & & \vdots & & \vdots & \vdots \\
a_P & \mu_{P1} & \mu_{P2} & \text{- - - -} & \mu_{PJ} & \text{- - - -} & \mu_{PQ} & \mu_{P.} \\
\hline
 & \mu_{.1} & \mu_{.2} & \text{- - - -} & \mu_{.J} & \text{- - - -} & \mu_{.Q} & \mu_{..}
\end{array}
$$

where
$$\mu_{IJ} = \frac{\sum_{K} X_{IJK}}{N} = \frac{AB_{IJ}}{N} \qquad (4.1)$$

$$\mu_{I.} = \frac{\sum_{J} \mu_{IJ}}{Q} = \frac{\sum\sum_{JK} X_{IJK}}{NQ} \qquad (4.2)$$

$$\mu_{.J} = \frac{\sum_{I} \mu_{IJ}}{P} = \frac{\sum\sum_{IK} X_{IJK}}{NP} \qquad (4.3)$$

$$\mu_{..} = \frac{\sum_{I} \mu_{I.}}{P} = \frac{\sum_{J} \mu_{.J}}{Q} = \frac{\sum\sum\sum_{IJK} X_{IJK}}{NPQ} \qquad (4.4)$$

Field of means in the experiment

	b_1	b_2	----	b_j	----	b_q	
a_1	$\overline{AB}_{11}$	$\overline{AB}_{12}$	----	$\overline{AB}_{1j}$	----	$\overline{AB}_{1q}$	$\overline{A}_1$
a_2	$\overline{AB}_{21}$	$\overline{AB}_{22}$	----	$\overline{AB}_{2j}$	----	$\overline{AB}_{2q}$	$\overline{A}_2$
$\vdots$	$\vdots$	$\vdots$		$\vdots$		$\vdots$	$\vdots$
a_i	$\overline{AB}_{i1}$	$\overline{AB}_{i2}$	----	$\overline{AB}_{ij}$	----	$\overline{AB}_{iq}$	$\overline{A}_i$
$\vdots$	$\vdots$	$\vdots$		$\vdots$		$\vdots$	$\vdots$
a_p	$\overline{AB}_{p1}$	$\overline{AB}_{p2}$	----	$\overline{AB}_{pj}$	----	$\overline{AB}_{pq}$	$\overline{A}_p$
	$\overline{B}_1$	$\overline{B}_2$	----	$\overline{B}_j$	----	$\overline{B}_q$	$\overline{G}$

where
$$\overline{AB}_{ij} = \frac{\sum_{k} X_{ijk}}{n} = \frac{AB_{ij}}{n} \qquad (4.5)$$

$$\overline{A}_i = \frac{\sum_{j} \overline{AB}_{ij}}{q} = \frac{\sum\sum_{jk} X_{ijk}}{nq} = \frac{A_i}{n} \qquad (4.6)$$

$$\overline{B}_j = \frac{\sum_{i} \overline{AB}_{ij}}{p} = \frac{\sum\sum_{ik} X_{ijk}}{np} = \frac{B_j}{n} \qquad (4.7)$$

$$\overline{G} = \frac{\sum_{i} \overline{A}_i}{p} = \frac{\sum_{j} \overline{B}_j}{q} = \frac{\sum\sum\sum_{ijk} X_{ijk}}{npq} = \frac{G}{npq} \qquad (4.8)$$

Hence, with regard to the symbols for the experiment only, any symbol such as A_i (i.e. where the symbol is not "capped") refers to a summation for all observations which include the particular factor or factors denoted at the level shown and any symbol such as $\overline{A}_i$ refers to the corresponding average value.

4.2.2 Definition of Main Effects, Interaction Effects and their Variances

The terms Main Effect and Interaction Effect have already been explained in Section 4.1.2. The basic definitions can now be translated into symbol form, based on the notation of the previous section. Further, it has also been explained that the statistical tool which is used to quantify the relative size of the treatment effects, which are attributable to the various experimental factors, is the variance due to these effects. Thus, the variance due to each type of effect must also be defined.

Considering the population frame of reference the main effect of factor A at level a_I is defined as

$$\alpha_I = \mu_{I.} - \mu_{..} \qquad (4.9)$$

which is the difference between the mean on the dependent variable for all observations with factor A at level a_I and the grand mean of all possible observations.

Similarly, the main effect of factor A at level $a_{I'}$ is defined as

$$\alpha_{I'} = \mu_{I'.} - \mu_{..} \qquad (4.10)$$

Now, the differential main effect between these two levels of factor A is the difference between the two main effects

$$\alpha_I - \alpha_{I'} = \mu_{I.} - \mu_{I'.} \tag{4.11}$$

This is of more practical use since in the general case differential main effects are always estimable from an experiment whereas the main effects themselves might not be.

Similar definitions hold for factor B using the symbol β to represent the main effects.

By definition, the variance due to the main effects of factor A is

$$\sigma_\alpha^2 = \frac{\sum_I (\mu_{I.} - \mu_{..})^2}{P-1}$$

$$= \frac{\sum_I \alpha_I^2}{P-1} \tag{4.12}$$

$$= \frac{\sum_I (\alpha_I - \alpha_{I'})^2}{P(P-1)} \qquad \text{(for all different pairs of } I, I' \text{ with } I<I')$$

Similarly, the variance due to the main effects of factor B is defined as

$$\sigma_\beta^2 = \frac{\sum_J (\mu_{.J} - \mu_{..})^2}{Q-1}$$

$$= \frac{\sum_J \beta_J^2}{Q-1} \tag{4.13}$$

$$= \frac{\sum_J (\beta_J - \beta_{J'})^2}{Q(Q-1)} \qquad \text{(for all different pairs of } J, J' \text{ with } J<J')$$

The variances due to the main effects, σ_α^2 and σ_β^2, are overall measures of the extent to which the mean on the dependent variable differs according to the changes between the various levels of the factors. For example, in the case where σ_α^2 has a low value approaching zero then the differential main effects of factor A are shown to be negligible, thus indicating the lack of an effect due to factor A. Conversely, where σ_α^2 is found to have a large value, this implies that one or more of the differential main effects of factor A are large, indicating the presence of an effect due to factor A.

It is worthy of note that the definitions of the main effects and the differential main effects include the grand mean, $\mu_{..}$, and hence involve all the measurements taken in the experiment. These effects are, therefore, not simply defined in terms of the individual factor being considered.

An Interaction Effect has already been described as a measure of the interdependency of the effect of one factor on another or an expression of the non-additivity of the main effects. If we consider factor A at level a_I and factor B at level b_J then the interaction between them is designated by $\alpha\beta_{IJ}$ and the relevant mean on the dependent variable is μ_{IJ}. The value of this mean can be written as follows

$$\mu_{IJ} = \mu_{..} + \alpha_I + \beta_J + \alpha\beta_I$$

$$\text{hence} \quad \alpha\beta_{IJ} = \mu_{IJ} - (\alpha_I + \beta_J + \mu_{..}) \qquad (4.14)$$

using the definitions of α_I and β_J this becomes

$$\alpha\beta_{IJ} = \mu_{IJ} - \mu_{I.} - \mu_{.J} + \mu_{..} \qquad (4.15)$$

It can be shown that the summation of the interaction effects over all values of either I or J produces a value of zero.

In the same way that the main effects were expressed in terms of differential main effects, the interaction effects can also be expressed as differential interaction effects. These are simply the differences between pairs of interaction effects. However, the differential interaction effects can be of two types, namely compound and simple. The compound type involves two differential main effects in addition to the difference between the relevant cell means, whereas a simple differential main effect only involves a single differential main effect. The general example of each type is given below

Compound differential interaction effect

$$\alpha\beta_{IJ} - \alpha\beta_{I'J'} = \mu_{IJ} - \mu_{I'J'} - (\alpha_I - \alpha_{I'}) - (\beta_J - \beta_{J'})$$

$$= \mu_{IJ} - \mu_{I'J'} - \mu_{I.} + \mu_{I'.} - \mu_{.J} + \mu_{.J'} \qquad (4.16)$$

Simple differential interaction effect

$$\alpha\beta_{IJ} - \alpha\beta_{IJ'} = \mu_{IJ} - \mu_{IJ'} - (\beta_J - \beta_{J'})$$

$$= \mu_{IJ} - \mu_{IJ'} - \mu_{.J} + \mu_{.J'} \qquad (4.17)$$

The variance due to the interaction effects of factors A and B is defined as

$$\sigma_{\alpha\beta}^2 = \frac{\sum\limits_{I}\sum\limits_{J} (\alpha\beta_{IJ})^2}{(P-1)(Q-1)} \qquad (4.18)$$

$$= \frac{\sum\limits_{I}\sum\limits_{J} (\mu_{IJ} - \mu_{I.} - \mu_{.J} + \mu_{..})^2}{(P-1)(Q-1)}$$

The final variance which requires definition is that due to experimental error, that is the variance which cannot be attributed to any of the treatment effects. This is required to allow comparisons to be made with the variances which are due to the treatment effects in order to establish whether these variances are significantly greater than can be explained by experimental error (i.e. chance variations).

Within any cell of the population, that is under any treatment combination, the variance of the N possible measurements on the dependent variable is called the within-cell variance and is defined as

$$\sigma_{IJ}^2 = \frac{\sum\limits_{K} (X_{IJK} - \mu_{IJ})^2}{N - 1} \qquad (4.19)$$

Since the assumption of homogeneity of error variance has been made then the within-cell variance for the population is constant, hence

$$\sigma_{IJ}^2 = \sigma_{\varepsilon}^2 \text{ (for all I,J)} \qquad (4.20)$$

4.2.3 Definition of Experimental Estimates of Treatment Effects, Error and their Variances

It has already been stated that the case of current interest is one where the treatment factors are designated 'fixed'. The population is therefore restricted to the factors and levels systematically chosen for inclusion in the experiment. Hence, the definitions of the population effects and variances in the foregoing section can be re-written using lower case symbols i,j,p and q as direct substitutions for their upper case counterparts. The revised population variances due to the attributable effects and experimental error are listed below

Main effects of factor A

$$\sigma_\alpha^2 = \frac{\sum_i (\mu_{i.} - \mu_{..})^2}{p - 1} \tag{4.21}$$

Main effects of factor B

$$\sigma_\beta^2 = \frac{\sum_j (\mu_{.j} - \mu_{..})^2}{q - 1} \tag{4.22}$$

Interaction effects

$$\sigma_{\alpha\beta}^2 = \frac{\sum\sum_{ij}(\mu_{ij} - \mu_{i.} - \mu_{.j} + \mu_{..})^2}{(p-1)\ (q-1)} \tag{4.23}$$

Within cell (experimental error)

$$\sigma_{ij}^2 = \frac{\sum_K (X_{ijK} - \mu_{ij})^2}{N - 1} \tag{4.24}$$

$$= \sigma_\varepsilon^2 \text{ (for all i,j)}$$

The general form of these variances is a SUM OF SQUARES divided by the DEGREES OF FREEDOM inherent in the summation.

It still remains that the experiment will only include a sample of n observations out of the possible N observations under each treatment combination. Considering, firstly, experimental error, under these circumstances the within cell variance for the cell ab_{ij} is given by

$$S_{ij}^2 = \frac{\sum_k (X_{ijk} - \overline{AB}_{ij})^2}{n - 1} \qquad (4.25)$$

which is an estimate of σ_ε^2. A better estimate of σ_ε^2 is obtained by averaging the value of the within cell variance over all the cells of the experiment, since it is assumed that error variance is homogeneous. This statistic is known as the pooled error variance (S^2_{pooled}) or the within cell mean square ($MS_{w.cell}$) and is an unbiased estimate of σ_ε^2.

$$MS_{w.cell} = S^2_{pooled} = \frac{\sum\sum_{ij} S_{ij}^2}{pq}$$

$$\text{i.e.} \quad \frac{\sum\sum\sum_{ijk} (X_{ijk} - \overline{AB}_{ij})^2}{pq(n-1)} \qquad (4.26)$$

In the special case of experimental designs which do not have more than one observation under each treatment combination (i.e. one observation per cell) the error variance is designated the error mean square (MS error) and is often taken to be the mean square due to the highest order interaction in the analysis of variance.

The variances obtained from the experiment which are due to the attributable effects are also known as mean squares and for the two factor experiment of n observations per cell these are defined as

Mean Square due to the main effects of factor A

$$MS_a = \frac{SS_a}{df} = \frac{nq\sum_i (\overline{A}_i - \overline{G})^2}{p-1} \qquad (4.27)$$

Mean Square due to the main effects of factor B

$$MS_b = \frac{SS_b}{df} = \frac{np\sum_j (\overline{B}_j - \overline{G})^2}{q-1} \qquad (4.28)$$

Mean Square due to the interaction effects

$$MS_{ab} = \frac{SS_{ab}}{df} = \frac{n\sum_{ij}\sum (\overline{AB}_{ij} - \overline{A}_i - \overline{B}_j + \overline{G})^2}{(p-1)(q-1)} \qquad (4.29)$$

It can be seen that these mean squares are again a sum of squares divided by the degrees of freedom but that the sum of squares incorporates a multiplier. This multiplier represents the number of observations used to obtain the lowest order mean in the summation term, that is the multipliers nq, np and n represent the number of observations in the means $\overline{A}_i$, $\overline{B}_j$ and $\overline{AB}_{ij}$ respectively. Apart from this additional component, the mean squares take the same form as the expressions for the population variances but with the means from the experimental observations substituted for the corresponding population values.

The need for the multipliers is derived from the properties of the general linear model, applied to the experiment, in which it is assumed that the variations caused by the experimental error and each of the treatment effects are independent of each other. Under these circumstances, the multipliers ensure that the expected value of each mean square contains the error variance term multiplied by a coefficient of unity, as shown below

$$MS_a \quad : \quad \text{expected value} = \sigma_\varepsilon^2 + nq\sigma_\alpha^2 \tag{4.30}$$

$$MS_b \quad : \quad \text{expected value} = \sigma_\varepsilon^2 + np\sigma_\alpha^2 \tag{4.31}$$

$$MS_{ab} \quad : \quad \text{expected value} = \sigma_\varepsilon^2 + n\ \sigma_{\alpha\beta}^2 \tag{4.32}$$

$$MS_{error} \quad : \quad \text{expected value} = \sigma_\varepsilon^2 \tag{4.33}$$

The multipliers, therefore, aid interpretation since in a situation where no treatment effects exist, the values of all the mean squares above are virtually equal. This is because under such circumstances σ_α^2, σ_β^2 and $\sigma_{\alpha\beta}^2$ approach zero and hence, each mean square represents an independent estimate of the error variance σ_ε^2. Conversely, if the effects attributable to the treatment factors and their interaction are real then MS_a, MS_b and MS_{ab} should be substantially greater than MS_{error} since σ_α^2, σ_β^2 and $\sigma_{\alpha\beta}^2$ should have significant values.

The relative sizes of the mean squares attributable to the treatment effects and the mean square due to experimental error are, then, the mechanism by which a judgement can be made to determine whether the variations can be explained by pure chance or whether they indicate that the experimental variables have a real effect on the dependent variable. This is where the variance ratio or 'F'-test is employed to obtain the level of significance of each effect. The 'F' statistic obtained from each ratio of mean squares is compared with tabulated information of the 'F'-distribution in accordance with the degrees of freedom inherent in both the numerator and the denominator of the ratio. In this way the probability that the variation is due to pure chance, i.e. the tail probability, is assessed. Whilst the presence of an effect can never be absolutely proven, the lower the tail probability the greater the likelihood that a real effect exists.

The 'F'-test, then, shows the significance level of the attributable effects. For those effects which are shown to be significant the actual effect itself can be estimated from the experimental results. To obtain the best available estimate the general linear model and its assumed constraints are required, thus

$$X_{ijk} = \mu + \alpha_i + \beta_j + \alpha\beta_{ij} + \varepsilon_{ijk} \qquad (4.34)$$

with the constraints that

$$\sum_i \alpha_i = 0; \quad \sum_j \beta_j = 0; \quad \sum_i \alpha\beta_{ij} = 0; \quad \sum_j \alpha\beta_{ij} = 0$$

For the particular 'fixed' factor experiment under consideration, a set of normal equations can be obtained using the principle of least squares, whereby the sum of the squares of the residual (error) is minimised. The solution of these equations leads directly to the following least squares estimates of the required population values:-

Best unbiased estimate of $\mu_{..}$ $= \overline{G}$ (4.35)

Best unbiased estimate of α_i $= \overline{A}_i - \overline{G}$ (4.36)

Best unbiased estimate of β_j $= \overline{B}_j - \overline{G}$ (4.37)

Best unbiased estimate of $\alpha\beta_{ij}$ $= \overline{AB}_{ij} - \overline{A}_i - \overline{B}_j + G$ (4.38)

The above equations represent the best unbiased estimates, available from the experiment, which are linear in X_{ijk} and yield minimum standard errors.

CHAPTER 5 DETAILS OF THE INVESTIGATION - PHYSICAL

5.1 FRAMEWORK OF EXPERIMENTAL WORK

5.1.1 Standard Test Methods

Wherever possible the experimental work was conducted according to published standard test methods. In the absence of any appropriate British Standard test for the assessment of the level of alkali-silica reactivity in cement-aggregate combinations, the standard test methods of the American Society of Testing and Materials were selected.

The relevant standards were ASTM C227-71 (re-approved 1976) (18) entitled "Potential alkali reactivity of cement-aggregate combinations (mortar bar method)" and ASTM C441-69 (re-approved 1975) (24) entitled "Effectiveness of mineral admixtures in preventing excessive expansion of concrete due to the alkali-aggregate reaction". Both these standard test methods were current at the time of conception of the experimental proposals. However, they have subsequently been revised as shown in the "List of References" at the end of this thesis.

Due to the complex nature of the current work, in comparison with the purpose for which these standards were designed, some adaptation of these test methods was required to allow for the inclusion of all the aspects of the A.S.R. phenomenon under investigation. In particular, modifications were necessary to allow for greater flexibility in certain of the parameters in the mix design which are normally held constant throughout the tests. In addition certain decisions had to be made where the two standards gave conflicting instructions.

Subsidiary information was required regarding the classification and properties of the constituents of the mortar mix. In general, this information was obtained using British Standard techniques. All cements were analysed for the relevant chemical and physical properties in accordance with BS 4550: Part 2: 1970 (83) and BS 4550: Part 3: 1978 (84) respectively. For the former standard the Draft Amendment Slip No. 1 dated October 1979 (85) was used.

The pulverised fuel ash was tested for compliance with BS 3892: Part 1: 1965 (86) and was supplied under an Agrement Board certificate No. 81/841 (87) thus ensuring consistent quality.

All aggregate material was tested according to the methods of BS 812: Part 2: 1975 (88) "Methods of sampling and testing of mineral aggregates, sands and fillers - Physical Properties".

5.1.2. Laboratory Conditions, Equipment and Procedures

The laboratory facilities required for the production, storage and monitoring of the expansion of mortar bars according to the aforementioned methods comprise three separate rooms or cabinets. Each room represents a different controlled environment, the details of which are given in Table 5.1.

Plates 5.1, 5.2 and 5.3 show the Moulding/Measurement Room, Curing Cabinet and Storage Room which were set up in The University laboratory and were used for the main Series 2 experiments. The temperature condition in the Moulding/Measurement Room was maintained by a Rootes Tempair air conditioning unit capable of either heating or cooling the room to a preset level. This unit was in operation continuously throughout the periods of experimentation. The relative humidity requirement was provided by intermittent boosting from an automatically controlled electric spray humidifier which operated only when the room was in use. Both these units are shown in Plate 5.4.

The temperature of the Curing Cabinet was indirectly controlled by the Tempair unit since the cabinet was kept in the Moulding/Measurement Room. Also, as the cabinet was both watertight and airtight, and contained standing water, the relative humidity was maintained at its required high level. Temperature control in the Storage Room was provided by two thermostatically controlled convector heaters. The high required temperature and fineness of control were helped by the extremely high level of thermal insulation inherent in the structure of the room.

The earlier Series 1 work was conducted using the facilities already established for the purpose at Probe Laboratories Ltd, Watford, England.

To enable the 25mm x 25mm x 250mm (gauge length) specimens to be produced, special moulds were fabricated in the University workshop (Plate 5.5). The design was such that the gauge studs were supported in the assembled moulds so that they were encastre in the mortar bar. On removal of the bar from the mould the square gauge stud supporting plates could be unscrewed from the specimen, exposing the slightly projecting head of the gauge stud. The gauge length over which the expansions were calculated was measured between the internal ends of the gauge studs, as shown in Fig 5.1.

In order to comply with the requirements of the ASTM standard method of preparation of mortar bars the mortar mixer must have certain operational characteristics; these are detailed in Table 5.2. The Hobart C.E. 100 mixer (Plate 5.4) is suitable for the purpose and was used in the preparation of all the test specimens.

Consistency of the mortar mix was tested with the flow table apparatus shown in Plate 5.6. The flow of the mix under a given input of energy was measured as a percentage increase in diameter of a standard compacted truncated cone of the fresh mortar. In ASTM C227-71 the flow test is included as a compliance clause to ensure constant consistency of the mortars, the results being required to fall within a specified band. The Series 1 experiments followed this procedure. In Series 2, however, where two distinct free water contents were used, the flow test was conducted purely to obtain extra information on the properties of the mortar mixes.

Three specimens were cast from each mortar mix. After initial curing and subsequent removal from the moulds, the groups of bars were stored in special airtight containers (Plate 5.7). These containers were lined with blotting paper to enhance evaporation of the water from the reservoir in the bottom of the container, thus maintaining the required high level of relative humidity inside the container. The mortar bars were supported above the surface of the water with no weight on the gauge studs. Each container was covered at the top with a piece of plastic sheeting, the airtight seal being provided by an adhesive strip and heavy duty rubber bands.

The procedure used for monitoring any length changes in the mortar bars did not involve measurement of the physical length of the bars. The length over which the expansion takes place is known as the gauge length and is the distance between the inner ends of the two gauge studs. At each measurement age the length of the specimen was compared with the length of a reference bar which is made from Invar. The length comparisons were made using the device shown in Plate 5.8 which is known as a comparator. Due to the low value of coefficient of linear expansion for Invar, the length of the reference bar remains virtually constant over the permissible temperature range of the Measurement Room. Hence, the change in the difference between the reference bar reading and the specimen reading defines any change in length of the specimen. Expansions were calculated in relation to the initial length measurements taken at age 24 hours, immediately after demoulding.

Within each Series of experiments the mortar mixes were prepared in random order to satisfy the requirements of

Factorial Analysis. The full sequence of events involved in the preparation, storage and testing of the specimens is discussed separately under the sections on each set of experiments. The chosen ages of measurement were adhered to within ±2 hours up to and including age 2 months and ±1 day thereafter.

In the second series of experiments some of the mortar mixes were repeated in order to produce 2" cube specimens. The cubes were produced in groups of 8, for use in compressive strength tests. Again special gang moulds were fabricated for the purpose of casting the cubes, these are shown in Plate 5.9. Compaction of the mortar in the cube moulds was carried out in a manner similar to that for the mortar bar specimens so that similar mortar densities were achieved.

5.1.3 Analytical Procedures

The raw experimental data obtained as explained in the previous section required conversion to produce expansion figures for each mortar mix. The formula for these computations is given below:-

$$E = \frac{(L_X - R_X) - (L_R - R_R)}{250} \times 10^{-6} \mu\varepsilon$$

where E = expansion
250 = specimen gauge length
L_X = length reading for mortar bar at time "x"

L_R = length reading for mortar bar at start of test

R_x = length reading for reference bar at time "x"

R_R = length reading for reference bar at start of test

Hence an expansion expressed in microstrain was calculated for each mortar bar at each chosen age of measurement, "x". Since each mortar mix is represented by three mortar bars the average of each set was then obtained.

As a precautionary measure against spurious results from individual bars, the repeatability of each set of measurements was computed and compared with the repeatability compliance clauses of the relevant ASTM standard test methods. The repeatability of any set of measurements is assessed by calculating the deviation of each individual value from the mean of the set.

In addition to the tabulated numerical output of expansion data, extensive graphical output was obtained in the form of expansion plotted against either time or one of the various treatment factors inherent in the make-up of the experiments. Graphs were assembled as both single and multiple plots, the latter to aid direct comparison of treatment effects.

It has already been stated that one of the main advantages of a statistically based method of experimentation is the suitability of the results for statistical interpretation. The technique known as the "Analysis of Variance" (ANOVA) was used to evaluate the significance of the treatment effects enabling them to be ranked in order of importance. This process also evaluates the significance of the interaction effects which shows whether the treatment factors are independent or whether they interact making their effects interdependent and not simply additive.

The repetitive nature of each stage of the computational procedures discussed above make them ideally suited to computerisation. Hence a complete set of computer programs was written to enable the experimental data to be converted directly into the three forms of output required. The various stages of this process are shown diagramatically in the form of a flow chart in Fig 5.2. The statistical part of the computations was carried out using a standard program from the Biomedical Computer Programs package (89) available on the Honeywell system at The City University.

5.2 SERIES 1 EXPERIMENTS

5.2.1 Experimental Treatment Combinations

The Series 1 experiments comprised a 3^3 Full Factorial set which has been discussed in an earlier report (90). Listed below are the criteria chosen for investigation of their effect on A.S.R.

Factor A - cement alkali content
Factor B - percentage replacement of cement by p.f.a. (refer to Figure 5.3)
Factor C - reactive aggregate content expressed as a percentage by weight of the overall aggregate

Each Factor was considered at three different levels, hence forming the 27 different mixes of the 3^3 Full Factorial design. The levels of Factors B and C could be controlled directly and hence set at any chosen value, whereas the levels of Factor A were predetermined by the commercially produced cements unless extra alkali was used an an admixture. This latter possibility was rejected since it was considered to increase further the complexity of the mix and, in consequence, would make it more difficult to interpret the results.

The replacement of cement by p.f.a. under Factor B was carried out by keeping the volume of cementitious material constant and equal to the volume of Portland cement in the mixes free of p.f.a.. In addition, for a 15% replacement level of cement by p.f.a. the cementitious mixture was 15% p.f.a. and 85% Portland cement by mass. It therefore follows that, because of the difference in the relative densities of p.f.a. and Portland cement the total mass of the cementitious material in a blended mixture was less than the mass of cement in an equivalent mix free of p.f.a.. This replacement process is fully explained in Figure 5.3.

Factor C, the reactive aggregate content, is simply expressed as the percentage reactive aggregate by weight of the total dry aggregate, the overall weight of dry aggregate remaining constant for each mix.

A three digit mix reference number is used to describe each of the mixes in the Series 1 section of the study. The digits in the mix reference numbers denote the level at which each experimental Factor is present, in the order cement - p.f.a. - aggregate (i.e. Factors A - B - C as listed above). When Pyrex glass is present in the mix, the mix reference number includes the letter 'P' as a postscript. Full details of the experimental Factors and their respective levels used in Series 1 are shown in Table 5.3. An example of a mix reference number from this table is given below:-

121P - this denotes a mix containing a medium alkali cement with 40% replacement by p.f.a. and an aggregate comprising 50% sand and 50% Pyrex glass.

In addition to Table 5.3, full details of the batch quantities of each mix, the accepted flow reading, the estimated compacted wet density of the mortar bars and the calculated proportions of the mortar bars expressed in kilogrammes per cubic metre of fully compacted wet mortar, are given in Tables A.1 and A.2 of Appendix A.

5.2.2 Materials

It was stated in the previous section that the available levels of Factor A were restricted by the cements which were commercially available. Since one of the reasons underlying the inception of this research was the occurrence of A.S.R. in the UK, the choice was further restricted to cements produced in the UK. The three cements selected for use were known to have a suitable range of alkali contents from data published shortly before the specimens were prepared. (69)

Representative samples of the cements used in the experiments were analysed in order to determine their alkali contents according to the methods of BS 4550 (84, 85). The values obtained from the analytical results are given in Table 5.4 where the content of the individual alkalis Potassium Oxide and Sodium Oxide are stated separately, together with the widely used combined figure expressed as total equivalent Sodium Oxide (Na_2O_{eq}). All figures are quoted as percentages by weight of total cement. The relevant physical properties of the cements are also given in Table 5.4.

Again recently published work (69) was consulted in making the choice of which p.f.a. to use. One of the salient points emerging from this work was that whilst different sources of p.f.a. have a variety of physical and chemical properties they all have a similar effect on A.S.R. when used as a cement replacement material. The choice of which p.f.a. to use was, therefore, quite arbitrary. The physical properties of the selected material are listed in Table 5.4.

For the control mixes with zero reactive aggregate content and also for the non-reactive proportion of the other mixes, crushed flint gravel from a quarry in the Thames Valley region of Essex was used. Earlier work (9,10) had shown this aggregated to be suitable for the purpose. The reactive component was crushed Pyrex glass as specified in ASTM C441-69 (24). Both types of aggregate were crushed and graded into the five size fractions shown in Table 5.5. The crushed material was recombined at the mix batching stage, equal proportions from each size fraction were used to produce the grading shown in Table 5.5 and listed in ASTM C441-69 (24). Both aggregate materials were batched in an oven dry condition. Table 5.4 shows the relevant physical properties of the two aggregate materials.

5.2.3 Experimental Procedures

Preparation of the test specimens followed the stages shown in Figure 5.4 which come directly from ASTM C227-71 (18), with the flow test being used to standardise the consistency of the mortar mix. Due to limitations imposed by the quantity of material available some mixes which marginally failed the repeatability rejection test were necessarily accepted, as shown by certain values in the results section appertaining to Series 1.

After the initial curing period of 24 ±2 hours, the specimens were removed from the moulds and an initial reference measurement taken. Immediately afterwards the gauge studs were greased to prevent rusting and the specimens were then sealed into their storage containers. All containers were then placed in the Storage Room for the high temperature conditioning.

Further measurements were taken at the ages listed in Table 5.6 to enable the expansion, if any, to be calculated. Prior to any measurement the specimens were allowed to acclimatise to the conditions in the Measurement Room for a minimum of 16 hours (Figure 5.4). In addition, the water in the Storage Containers was replaced by fresh tap water each time the specimens were returned to their container after measurement.

At the end of the original 4 month test period a visual examination was conducted on each specimen to assess the condition of the bar on the basis of the criteria listed below.

(i) state of compaction of mortar
(ii) extent of warping
(iii) extent of surface exudation
(iv) surface appearance (mottled, stained, cracked etc)

After the visual examination the specimens were returned to their Storage Containers and replaced in the Storage Room. On completion of all the visual examinations the Storage Containers were removed from the Storage Room and stored under ambient laboratory conditions (approximately 20°C) until the 14 month expansion measurements were taken. Thereafter storage was continued at ambient laboratory temperature with the specimens sealed into polythene bags. Expansion measurements at age 18 months were taken for the high alkali cement bars only.

5.3 SERIES 2 EXPERIMENTS

5.3.1 Experimental Treatment Combinations

Due to the importance of the factors chosen for investigation in the preliminary experiments of Series 1, the same three factors were again chosen for inclusion in Series 2. However, with the exception of Factor A, i.e. the cement alkali content, the number of levels of each factor was increased to four and different increments used. In addition, a fourth factor was incorporated into Series 2, Factor D, the free water content of the mix. By definition the free water content is the amount of water available in the mortar or concrete over and above that which is absorbed by the aggregate in reaching the saturated surface dry condition.

Factor D was introduced at two levels, nominally low and high. For those mixes which did not contain p.f.a., free water/cement ratios of 0.40 and 0.55 were used for the low and high free water contents respectively. For mixes which did contain p.f.a., however, the free water/cementitious ratios varied slightly from the above values due to the differences in the relative densities of the cements and the p.f.a. and also the manner in which cement replacement was conducted (refer to Section 5.2.1 and Figure 5.3).

The number of levels of each of the four factors described above was restricted to the chosen value to enable all the treatment combinations to be included within an experimental framework of practicable dimensions. The 3 x 4 x 4 x 2 factorial set included 96 different mixes, details of which are shown in Table 5.7. In Tables B.1 and B.2 of Appendix B information on the mixes is expanded to give the batch quantities, the value of flow obtained, the estimated compacted wet density and the calculated mix proportions of the mortar bars expressed in kilogrammes per cubic metre of fully compacted wet mortar.

Unlike Series 1, the experimental work of Series 2 was not simply restricted to the chosen factorial set but was extended to include mortar bar tests on extra mixes using Pyrex glass as the reactive aggregate. These tests focus their attention on particular aspects of the overall investigation and the mixes are designated the "EP" mixes. Additionally, some mortar bar tests were carried out on mortars using Beltane opal as the reactive aggregate, designated the "BO" mixes. For subsidiary information, compressive strength tests on 2" mortar cube type specimens were also included in the experimental program.

The EP mixes, which are detailed in Table 5.7 and Tables B.3 and B.4 of Appendix B, were designed to investigate further the specific effect on A.S.R. of the four individual factors of Series 2. Two of the factors were simply considered at extra levels thus extending the coverage beyond the framework of the factorial set. However, the remaining two factors, namely cement alkali content and p.f.a. content, were examined from a different perspective, as explained below.

Given that the aggregate being used is reactive, or potentially so, it has been documented (7, 21) that the criterion to be used to avoid the possibility of A.S.R. is the alkali content of the mix, expressed as Na_2O_{eq} in kilogrammes per cubic metre of compacted wet concrete. To investigate this hypothesis, four mixes were designed with differing alkali contents based on the amount of high alkali cement used in the mix rather than the use of cements with different alkali contents. These mixes were designed such that the alkali contents of the compacted mixes were similar to those obtained using the different cements of the 3 x 4 x 4 x 2 factorial set and extended to contents below the 3kg Na_2O_{eq}/m^3 recommended by some authorities as the maximum safe limit (7, 21, 57)

The second batch of EP mixes were based on the use of p.f.a. to combat the reaction. In the main experiments the p.f.a. was used as a partial replacement for the cement content of the reactive mix. It has been suggested (57, 61, 67) that the process involved in such usage may be simply due to the dilution of the amount of alkali available for reaction as a consequence of the reduction in the cement:aggregate ratio. Therefore, four mixes were designed using the p.f.a. as an admixture to the main constituents of the most highly reactive non-p.f.a. mix from the 3 x 4 x 4 x 2 factorial set. Whilst this could still be seen as a reduction in the concentration of alkali derived from the cement expressed as a proportion of the total mix, the effective ratio of alkali from the cement:reactive silica from the aggregate remained unaltered.

The third and fourth factors for individual consideration were the reactive aggregate content and the free water content respectively. Both were considered at levels not included in the main factorial programme. In the work already described in Series 2 Pyrex glass contents of 0%, 33.3%, 66.6% and 100% were considered. In anticipation of the low contents required for significant expansions with Beltane opal as the reactive constituent, four comparative mixes with low contents of Pyrex glass were designed. Likewise four different free water contents were investigated extending both extremes of the range chosen for the Series 2 factorial set.

All the EP mixes were designed to be potentially highly reactive. The three factors not under consideration in any mix were maintained at their most reactive level.

The BO mixes were included primarily for comparison with their Pyrex glass counterparts, in particular where the success, or otherwise, of p.f.a. used as a cement replacement material to reduce expansion caused by A.S.R. is concerned. Table 5.7 and Tables B.5 and B.6 of Appendix B define the selected mixes. The low proportions of Beltane opal were included to investigate the region of an expected pessimum content which leads to the maximum expansion and is widely discussed elsewhere (21, 45, 53, 92). In addition, mixes containing high proportions of 33.3%, 66.6% and 100% Beltane opal were examined since, although such high contents are not normally used with this material, it was considered desirable to investigate the implied theory (45, 92) that above the pessimum content the expansion tends to zero with increasing Beltane opal content.

To supplement the information emanating from the Series 2 factorial set of expansion results, mortar test cubes (2") were produced for all the mixes containing the high alkali cement. The mix proportions were as detailed in Table B.2 of Appendix B, using batch quantities to enable 8 cubes to be made from each mix. A check was made to ensure that the flow value obtained for each of these mixes was similar to that recorded for the corresponding mix used to make the mortar bar specimens.

Again a system of mix reference numbers is used to describe each of the mortars used in the whole of the Series 2 section of the study. The four digits denote the level at which each experimental factor is present, in the order cement - p.f.a. - aggregate - free water (i.e. the Factors A - B - C - D). The letter following the mix reference number denotes the presence and type of reactive aggregate used, 'P' representing Pyrex glass and 'B' representing Beltane opal. An example of a mix reference number is given below:-

2321P - this denotes a mix containing high alkali cement with 45% replacement by p.f.a., an aggregate comprising 33.3% sand and 66.6% Pyrex glass and using the higher free water content.

The corresponding mix to 2321P incorporating Beltane opal as the reactive component would be denoted by the mix reference number 2321B.

5.3.2. Materials

As far as possible, the materials used for the Series 2 experiments were the same as those used in the earlier experimental work of Series 1. The low and medium alkali cements were identical except for the normal variations expected from any single producer. The high alkali cement, however, had to be different since that used for Series 1 was no longer available because of changes made in the manufacturing process at the cement works concerned. In consequence a replacement UK cement was selected which from previously published work (69) was known to be associated with a high level of reactivity. Table 5.8 shows the relevant physical and chemical properties of the three cements used.

P.f.a. was used from the same source as that used for Series 1 and was, therefore, subject only to minor variations. The chemical and physical properties of this material are given in Table 5.8.

Both the Pyrex glass and the flint gravel aggregates were obtained from sources identical to those used for the Series 1 experiments. However, due to the increased quantities required for Series 2, a different process was used for crushing these materials into the correct grading sizes. Moreover, this produced a variation in the particle shape of the crushed material. The Beltane opal was obtained through Professor Sidney Diamond of Purdue University, Indiana and originated from the Beltane quarry in California. This material was crushed and graded to the same size fractions used for the other types of aggregate, as shown in Table 5.5. The physical and chemical properties of all three aggregates used in Series 2 are shown in Table 5.8.

5.3.3 Experimental Procedures

For the preparation of the mortar bars of Series 2 procedures identical to those used for Series 1 were followed up to the stage where the flow test was carried out. Since the fourth factor, Factor D - free water content, was included it was no longer possible to comply with the standard consistency clause of ASTM C227-71. Hence, the flow test was incorporated not as a rejection criterion but purely as a parameter to monitor the mix consistency achieved. Reference to Figure 5.4 highlights this difference in the Series 1 and Series 2 procedures.

The storage and measurement procedures adopted were identical to those for the early part of Series 1 in most respects. However, the ages of measurement of the specimens were changed, this being necessary due to the extra number of mixes involved. The ages of measurement are listed in Table 5.9. For reasons explained in Chapter 7, Details of the Investigation - Chemical, after each measurement process the level of water in the Storage Containers was replenished but not replaced.

As a replacement for the lengthy visual inspection procedure, at the end of the main test period of 6 months photographs were taken of the most expansive mixes and also of those which exhibited the greatest amounts of surficial exudation.

Mixing of the mortar for the 2" test cubes followed stages identical to those in the mortar bar procedure. 8 cubes were cast from each mix, compaction techniques being similar to those for the mortar bars.

Figure 5.5 shows that after the initial 24 ± 2 hours curing time 2 cubes were placed in a standard curing tank, submerged in water at 20°C. The remainder were wrapped in a cling film, sealed into polythene bags and placed in the Storage Room at 37.8°C. The water cured cubes were tested at age 28 days to give a "standard" compressive strength value. The other cubes were tested in pairs at ages 14 days, 2 months and 6 months during which time the A.S.R. was progressing.

Before compression testing the "dry" cured cubes were soaked overnight during their cooling down period and all cubes were weighed in water and in air so that the saturated density could be computed. The testing procedure for determining compressive strength was based on British Standard test method BS 4551: 1980 (93) using a constant rate of loading equal to 11.6kN/minute (i.e. in the range 0.03 to 0.10 N/(mm^2s).

TABLE 5.1 DETAILS OF ENVIRONMENTALLY CONTROLLED ROOMS AND CABINETS

NAME/REFERENCE	TEMPERATURE LIMITS	RELATIVE HUMIDITY LIMITS
Moulding/Measurement Room	23.0°C ± 1.7°C	50% minimum
Curing Cabinet	23.0°C ± 1.7°C	95% minimum
Storage Room	37.8°C ± 1.7°C	-

TABLE 5.2 CHARACTERISTICS OF MIXER

SPEED SETTING	PADDLE ROTATION SPEED	PLANETARY MOTION
Slow	140 ± 5 r.p.m.	62 r.p.m.
Medium	285 ± 10 r.p.m.	125 r.p.m.

TABLE 5.3 EXPLANATION OF MIX REFERENCE NUMBERS - SERIES 1

FACTOR LEVEL NUMBER	FACTOR A - CEMENT	FACTOR B - REPLACEMENT OF CEMENT USING PFA	FACTOR C - AGGREGATE
0	Low alkali (SRPC)*	0%	100%S-0%P
1	Medium alkali (OPC)	20%	50%S-50%P
2	High alkali (OPC)	40%	0%S-100%P

Note:- S = sand
P = Pyrex glass (Reactive Aggregate)

* to BS 4027: 1980 (91)

TABLE 5.4 PROPERTIES OF MATERIALS - SERIES 1

(i) CEMENTS

ALKALI LEVEL	CEMENT TYPE	ACID SOLUBLE ALKALI CONTENT (%) *			SPECIFIC GRAVITY++
		Na_2O	K_2O	Na_2O_{eq} +	
0-LOW	SRPC	0.20	0.44	0.49	3.15
1-MEDIUM	OPC	0.30	0.79	0.82	3.06
2-HIGH	OPC	0.21	1.75	1.36	3.14

(ii) PULVERISED FUEL ASH

CHEMICAL/PHYSICAL PROPERTY		MEASURED VALUE
PARTICLE SIZE	Residue on 150μm sieve	0.6%
	Residue on 45μm sieve	8.0%
CARBON CONTENT	Loss on ignition (LOI)	3.6%
SPECIFIC GRAVITY	Using Kerosine ++	2.31
	Using distilled water	2.35
WATER SOLUBLE ALKALIS	Na_2O	0.14%
	K_2O	0.06%
	Na_2O_{eq} +	0.18%
TOTAL ALKALIS **	Na_2O	1.83%
	K_2O	3.52%
	Na_2O_{eq} +	4.15%

TABLE 5.4 continued

(iii) AGGREGATES		MEASURED VALUE	
CHEMICAL/PHYSICAL PROPERTY		SAND	PYREX GLASS
RELATIVE DENSITY ***	Oven dry	2.51	2.24
	Saturated surface dry	2.56	2.24
	Apparent	2.65	2.24
WATER ABSORPTION COEFFICIENT ***		2.0%	0.0%
TOTAL ALKALIS**	Na_2O	0.07%	4.13%
	K_2O	0.07%	0.06%
	Na_2O_{eq} +	0.12%	4.17%

Note:- * = test conducted to BS 4550: Part 2: 1970 (83, 85)
** = hydrofluoric (H.F.) extraction
*** = test conducted to BS 812: Part 2: 1975 (88)
+ = $Na_2O_{eq}\% = Na_2O\% + 0.658K_2O\%$
++ = test conducted to BS 4550: Part 3: 1978 (84)

TABLE 5.5 GRADING OF AGGREGATES

SIEVE SIZE		.PERCENTAGE BY WEIGHT
PASSING	RETAINED ON	
4.75mm	2.36mm	20%
2.36mm	1.18mm	20%
1.18mm	600μm	20%
600μm	300μm	20%
300μm	150μm	20%

TABLE 5.6 AGE OF SPECIMENS AT MEASUREMENT AND STORAGE REGIME - SERIES 1

SPECIMEN AGES AT MEASUREMENT	STORAGE CONDITIONS	PRE-MEASUREMENT ACCLIMATISATION
24 hours ± 2 hours	Hot Room	16-20 hrs in Measurement Room
14 days ± 2 hours	Hot Room	16-20 hrs in Measurement Room
28 days ± 2 hours	Hot Room	16-20 hrs in Measurement Room
2 months ± 2 hours	Hot Room	16-20 hrs in Measurement Room
4 months ± 1 day	Hot Room	16-20 hrs in Measurement Room
14 months *	Ambient Room Temp.	16-20 hrs in Measurement Room
18 months *	Ambient Room Temp.	16-20 hrs in Measurement Room

Note:- Ambient room temperature 20°C
* = approximate age only
** = specimens stored in sealed plastic bag having been removed from the normal Storage Containers at the previous measurement age.

TABLE 5.7 EXPLANATION OF MIX REFERENCE NUMBERS - SERIES 2

(i) 3x4x4x2 FACTORIAL SET

FACTOR LEVEL NUMBER	FACTOR A - CEMENT	FACTOR B - REPLACEMENT OF CEMENT USING P.F.A.	FACTOR C - AGGREGATE	FACTOR D - FREE WATER CONTENT *
0	Low alkali (SRPC) **	0%	100%S - 0%P	Low (0.40)
1	Medium alkali (OPC)	15%	67%S - 33%P	High (0.55)
2	High alkali (OPC)	30%	33%S - 67%P	-
3	-	45%	0%S - 100%P	-

(ii) E.P. MIXES

FACTOR LEVEL NUMBER	FACTOR A - REDUCTION IN CEMENT CONTENT +	FACTOR B - P.F.A. CONTENT AS AN ADMIXTURE	FACTOR C- AGGREGATE	FACTOR D - FREE WATER CONTENT *
0	-	0%	-	-
1	-	-	-	High (0.55)
2	0%	-	-	V.Low (0.35)
3	70%	-	0%S-100%P	Low/Medium (0.45)
4	63%	10%	96%S-4%P	Medium/High (0.50)
5	54%	20%	92%S-8%P	V.High (0.60)
6	37%	30%	88%S-12%P	
7	-	40%	84%S-16%P	

TABLE 5.7 continued

(iii) B.O. MIXES

FACTOR LEVEL NUMBER	FACTOR A - CEMENT	FACTOR B - REPLACEMENT OF CEMENT USING P.F.A.	FACTOR C - AGGREGATE	FACTOR D - FREE WATER CONTENT *
0	-	0%	-	-
1	-	-	67%S - 33%B	High (0.55)
2	High alkali (OPC)	-	33%S - 67%B	-
3	-	45%	0%S - 100%B	-
4	-	-	96%S - 4%B	-
5	-	-	92%S - 8%B	-
6	-	-	88%S - 12%B	-
7	-	-	84%S - 16%B	-

* = figures in brackets represent free w/c ratio for control mixes containing no p.f.a.
** = to BS 4027: 1980 (91)
\+ = all cement high alkali (OPC)
S = Sand
P = Pyrex Glass
B = Beltane Opal

TABLE 5.8 PROPERTIES OF MATERIALS - SERIES 2

(i) CEMENTS

ALKALI LEVEL	CEMENT TYPE	ACID SOLUBLE ALKALI CONTENT (%) *			SPECIFIC GRAVITY
		Na_2O	K_2O	Na_2O_{eq} +	
0-LOW	SRPC	0.19	0.41	0.46	3.13
1-MEDIUM	OPC	0.30	0.64	0.72	3.07
2-HIGH	OPC	0.40	1.17	1.17	3.03

(ii) PULVERISED FUEL ASH

CHEMICAL/PHYSICAL PROPERTY		MEASURED VALUE
PARTICLE SIZE	Residue on 45μm sieve	8.7%
CARBON CONTENT	Loss on ignition (LOI)	4.7%
SPECIFIC GRAVITY	Using distilled water	2.33
WATER SOLUBLE ALKALIS	Na_2O	0.13%
	K_2O	0.07%
	Na_2O_{eq} +	0.18%
TOTAL ALKALIS ++	Na_2O	1.32%
	K_2O	2.36%
	Na_2O_{eq} +	2.88%
ACID SOLUBLE ALKALIS*	Na_2O	0.37%
	K_2O	0.71%
	Na_2O_{eq} +	0.84%

TABLE 5.8 continued

(iii) AGGREGATES

CHEMICAL/PHYSICAL PROPERTY		MEASURED VALUE		
		SAND	PYREX GLASS	BELTANE OPAL
RELATIVE DENSITY ***	Oven dry	2.56	2.23	2.02
	Saturated surface dry	2.60	2.23	2.08
	Apparent	2.66	2.23	2.16
WATER ABSORPTION COEFFICIENT ***		1.4%	0.0%	3.2%
TOTAL ALKALIS **	Na_2O	0.06%	3.77%	0.11%
	K_2O	0.10%	0.47%	0.13%
	Na_2O_{eq} +	0.12%	4.09%	0.20%
ACID SOLUBLE ALKALIS *	Na_2O	0.02%	0.11%	0.02%
	K_2O	0.02%	0.03%	0.02%
	Na_2O_{eq} +	0.03%	0.13%	0.03%

Note:- * = test conducted to BS 4550: Part 2: 1970 (83, 85)
** = hydrofluoric (H.F.) extraction
*** = test conducted to BS 812: Part 2: 1975 (88)
+ = $Na_2O_{eq}\% = Na_2O\% + 0.658K_2O\%$
++ = test conducted to BS 4550: Part 3: 1978 (84)

TABLE 5.9 AGE OF SPECIMENS AT MEASUREMENT AND STORAGE REGIME - SERIES 2

SPECIMEN AGES AT MEASUREMENT	STORAGE CONDITIONS	PRE-MEASUREMENT ACCLIMATISATION
24 hours ± 2 hours	Hot Room	16-20 hrs in Measurement Room
14 days ± 2 hours	Hot Room	16-20 hrs in Measurement Room
2 months ± 2 hours	Hot Room	16-20 hrs in Measurement Room
6 months ± 1 day	Hot Room	16-20 hrs in Measurement Room
12 months ± 1 week	Hot Room	16-20 hrs in Measurement Room

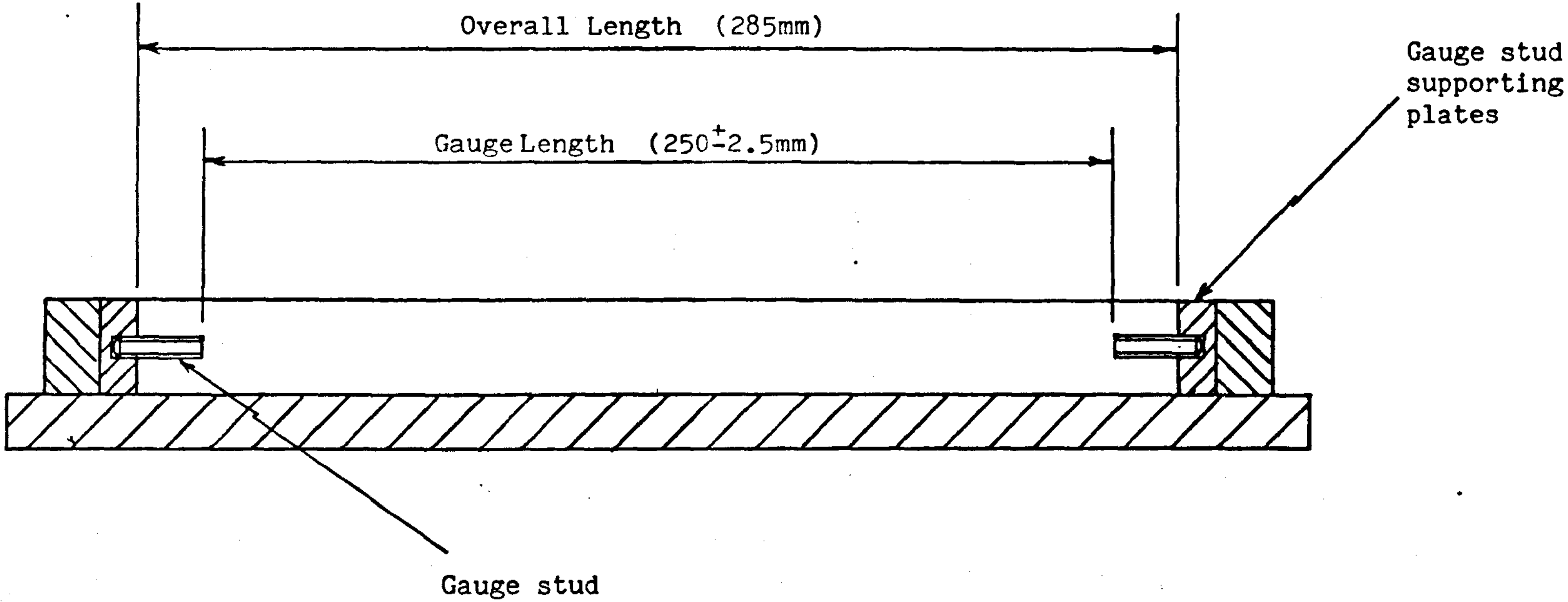


Figure 5.1 Cross-sectional diagram showing construction of mould for fabricating mortar bar specimens.

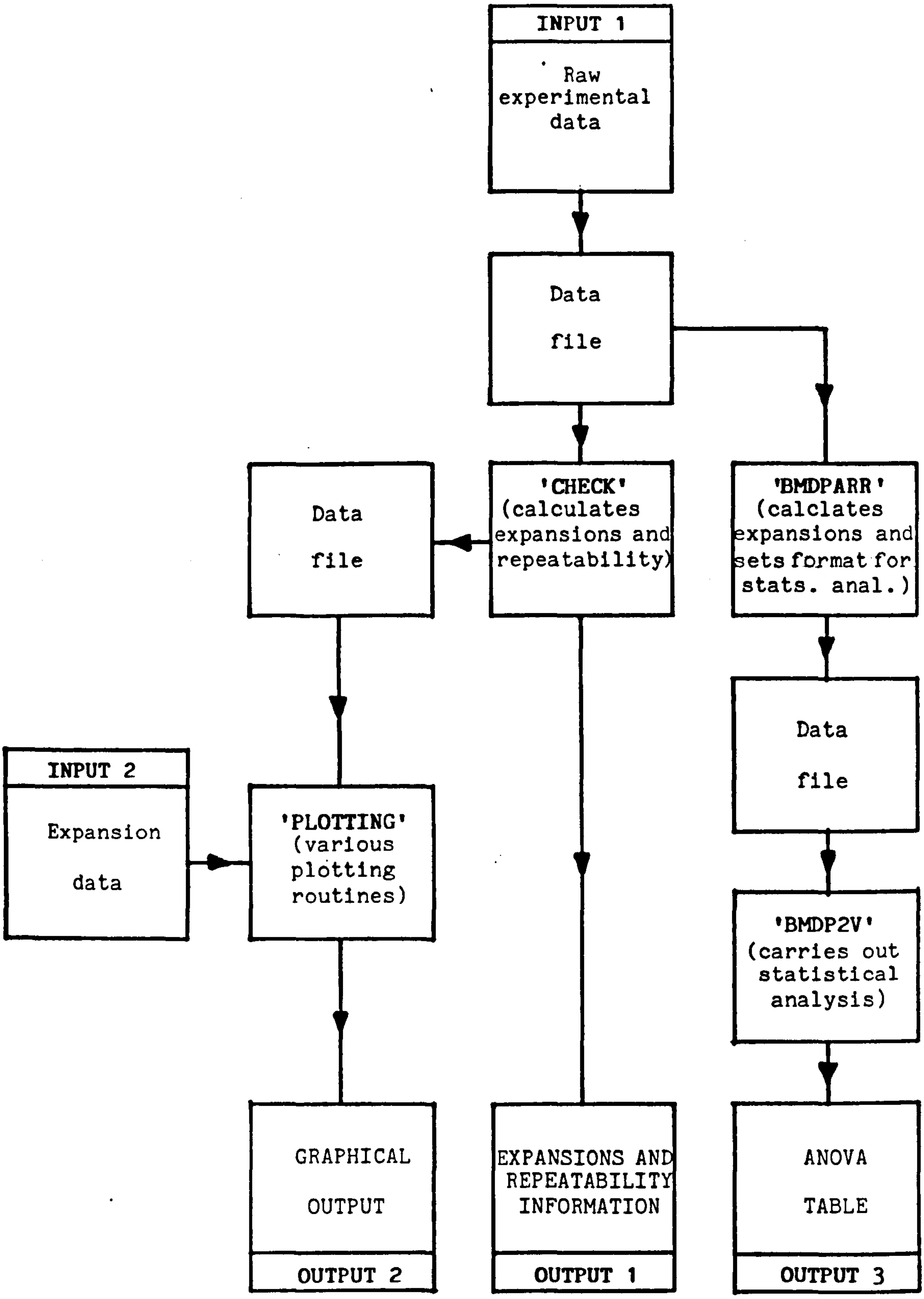


Figure 5.2 **Flow chart for computer analysis of expansion data**

FIGURE 5.3 EXPLANATION OF PROCEDURE USED TO CALCULATE BATCH QUANTITIES FOR MIXES CONTAINING P.F.A. AS A PARTIAL REPLACEMENT FOR THE CEMENT CONTENT

Let:- W_c = weight of cement per batch for non-p.f.a. mixes.

W_b = weight of combined cementitious material per batch.

R = percentage replacement parameter (by mass).

S = specific gravity of p.f.a.

Assume: specific gravity of cements = 3.15.

NON-P.F.A. MIXES

All the cementitious material was cement, hence $W_b = W_c$.

P.F.A. MIXES

The cementitious material was a mixture of cement and p.f.a. where R% by mass was p.f.a. and (100-R)% by mass was cement. In calculating the value of W_b a further restraint was applied such that the volume of the cementitious material remained constant at the value set by the volume of the cement in the non-p.f.a. mixes. It follows that the combined weight of the cementitious material in a p.f.a. mix was not equal to W_c, unless S = 3.15.

Hence, $$W_b = W_c \times \frac{100}{[100-R + R(3.15/S)]}$$

Note:- For the Series 2 materials where S = 2.33, the ASTM C441 regime of replacing 25% of the cement by an equal volume of p.f.a. would be equivalent to substituting a value of R = 19.78 in the above equation.

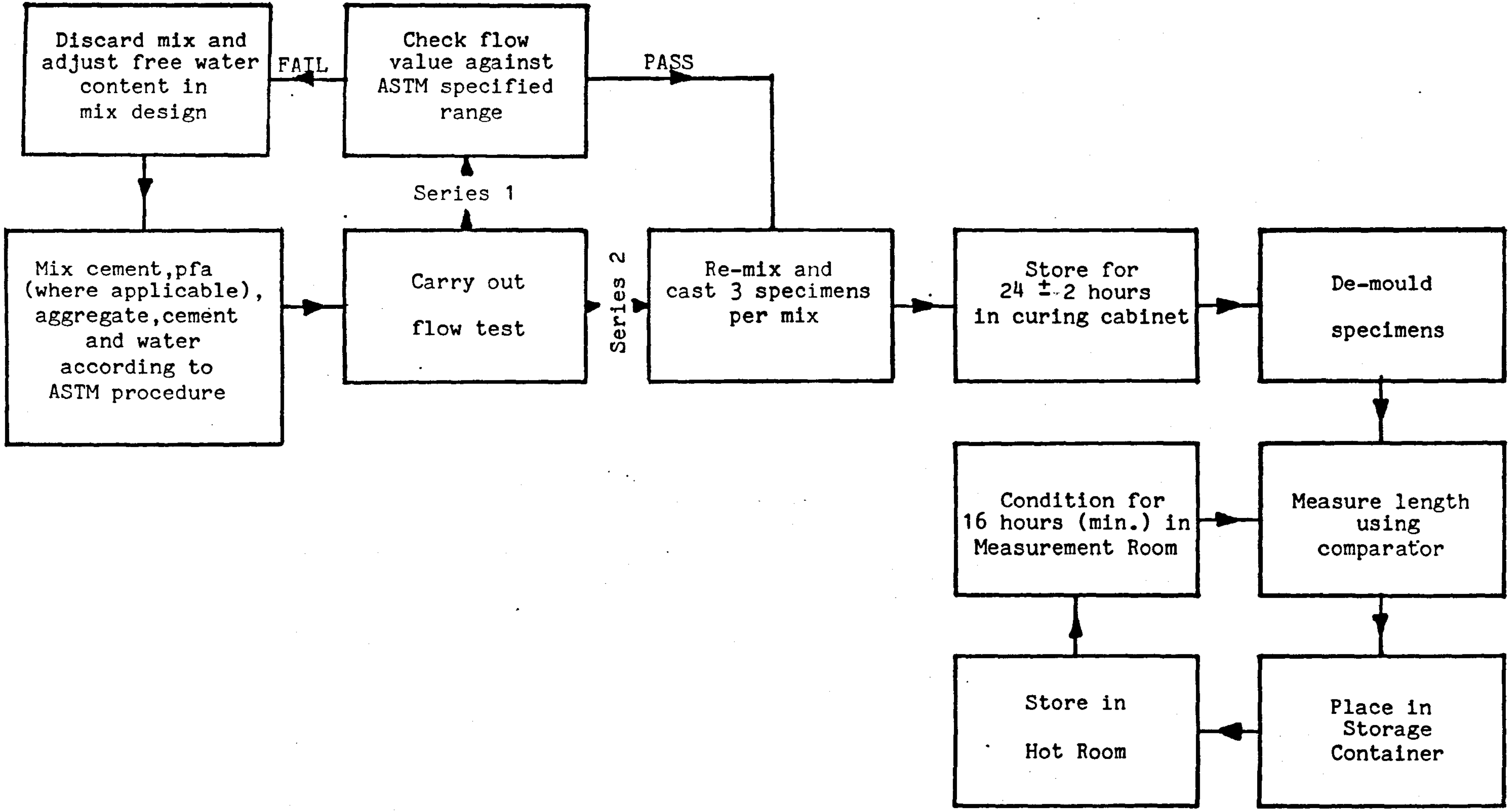


Figure 5.4 Flow chart for preparation,storage and measurement of mortar bars,Series 1 & 2

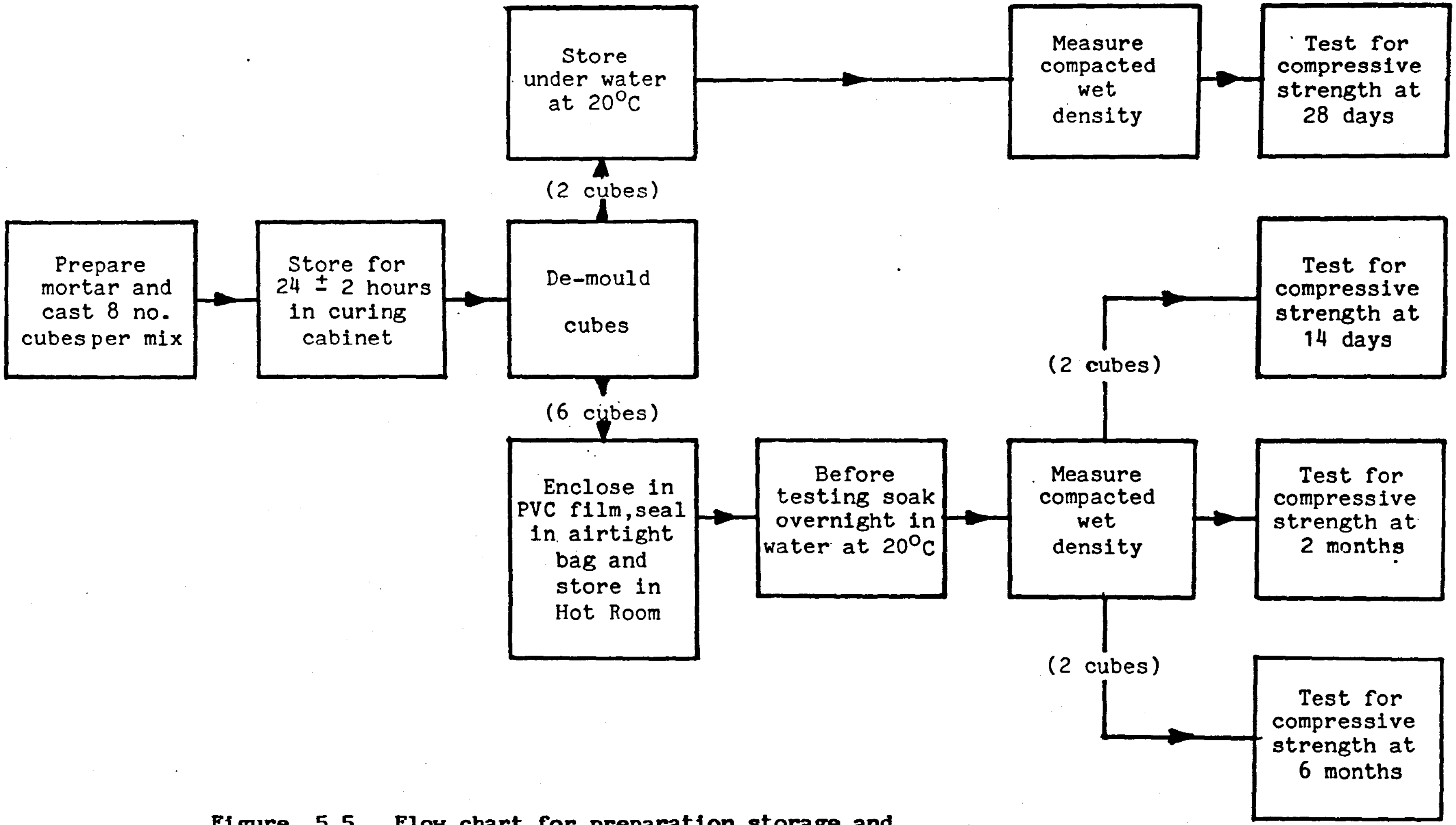


Figure 5.5 Flow chart for preparation, storage and testing of 2" cubes

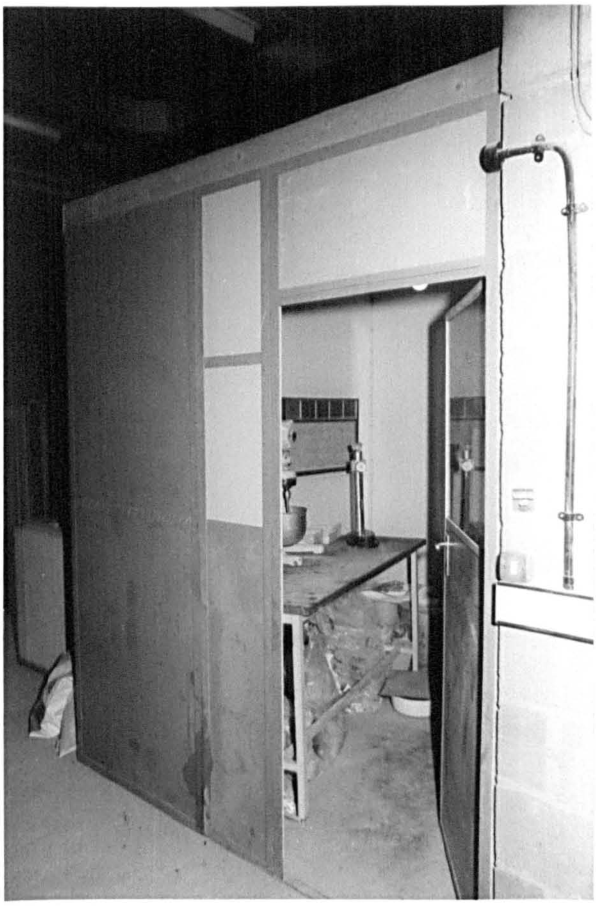


Plate 5.1
Environmentally controlled moulding/measurement room

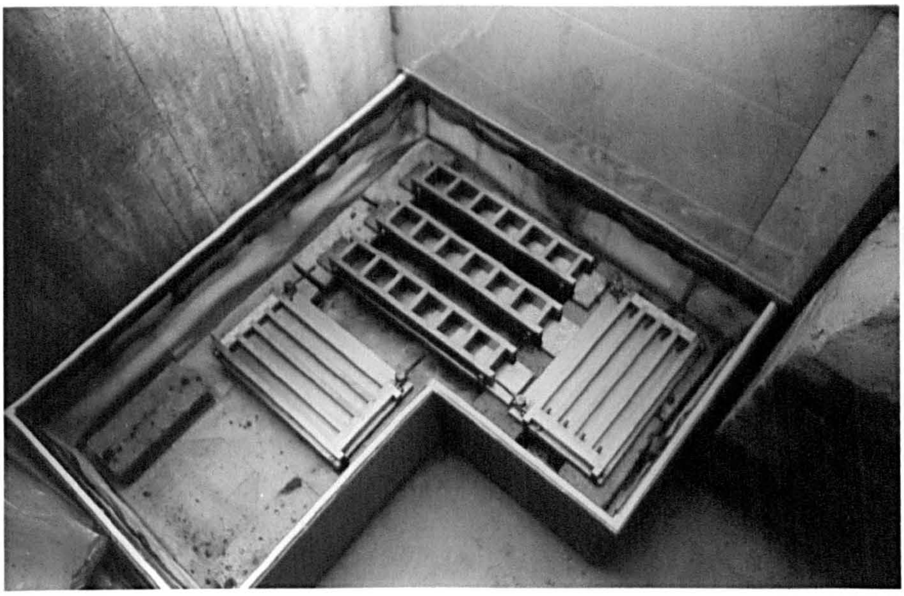


Plate 5.2 Curing cabinet with specimen moulds in place.

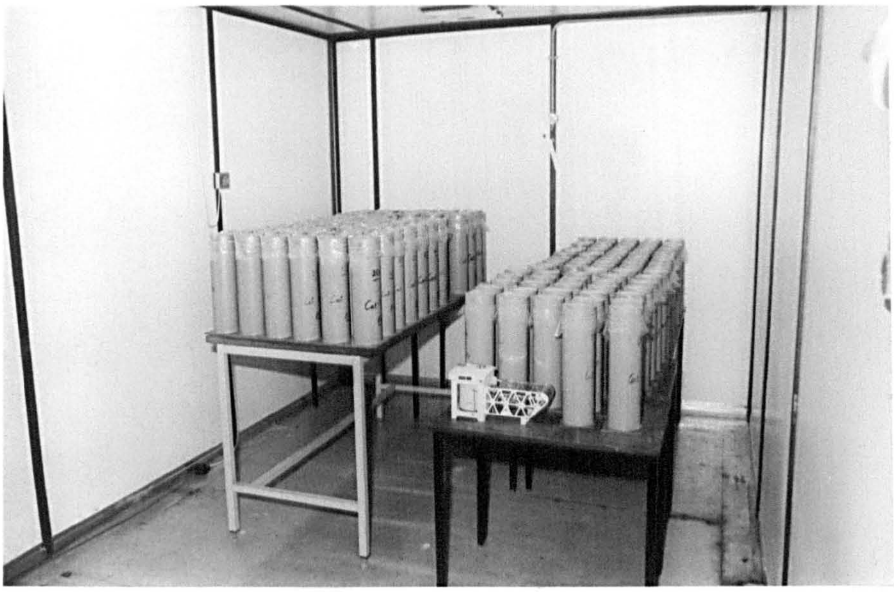


Plate 5.3 Temperature controlled storage room with storage containers in place.

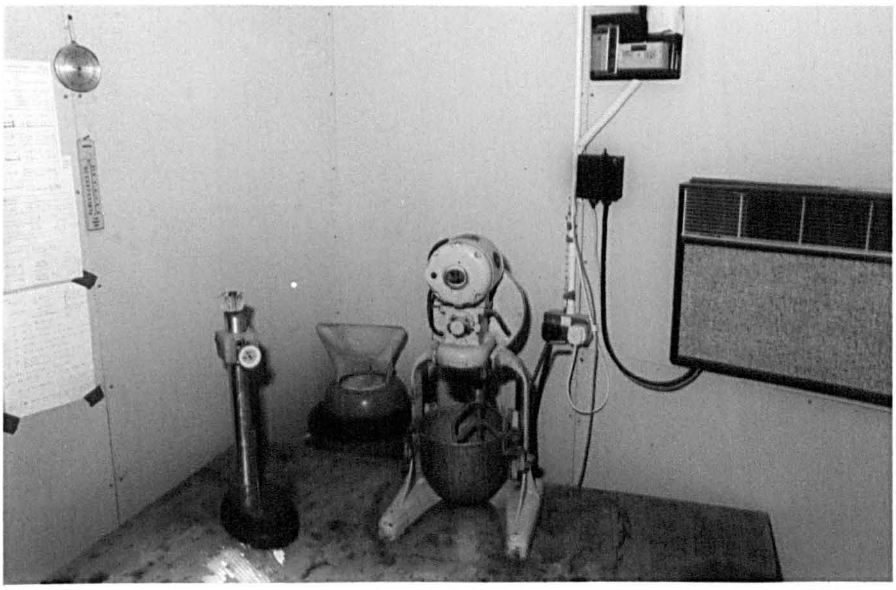


Plate 5.4 Inside view of moulding/measurement room, showing Hobart mixer, length comparator, heater/refridgeration unit and humidifier.

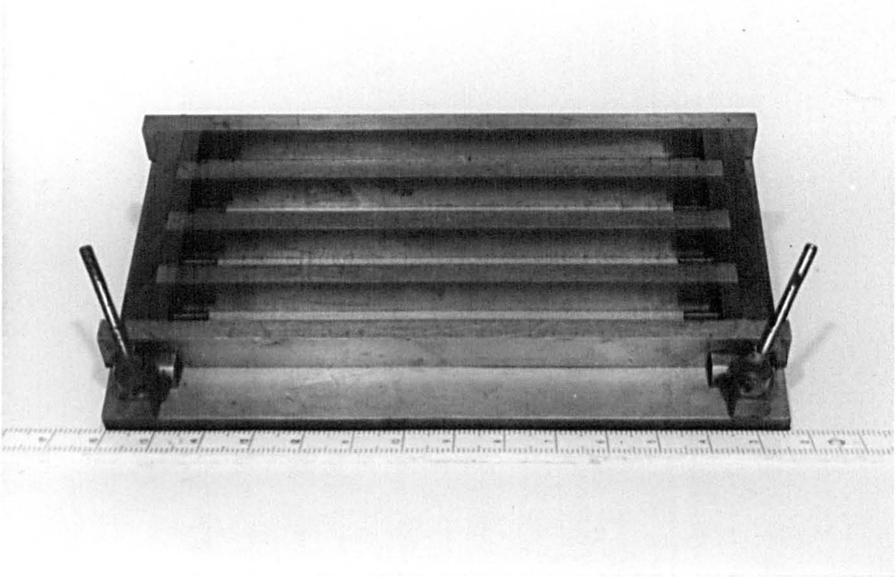


Plate 5.5 Gang mould capable of making four mortar bar specimens with encastre gauge studs.

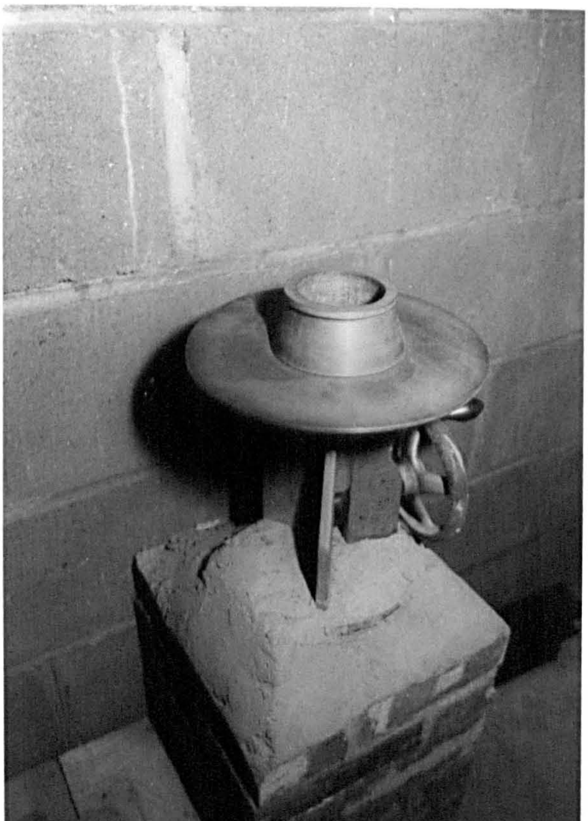


Plate 5.6
Flow Table

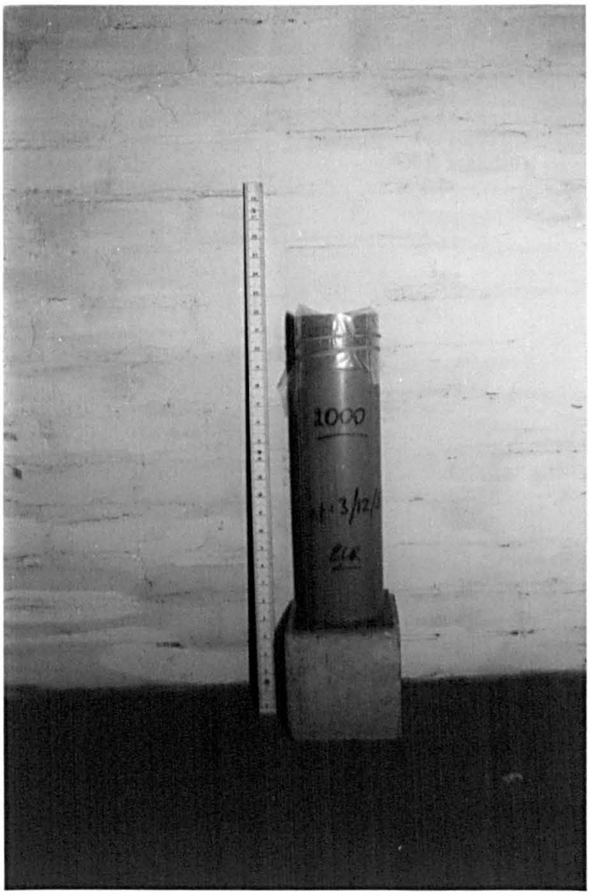


Plate 5.7
Typical storage container

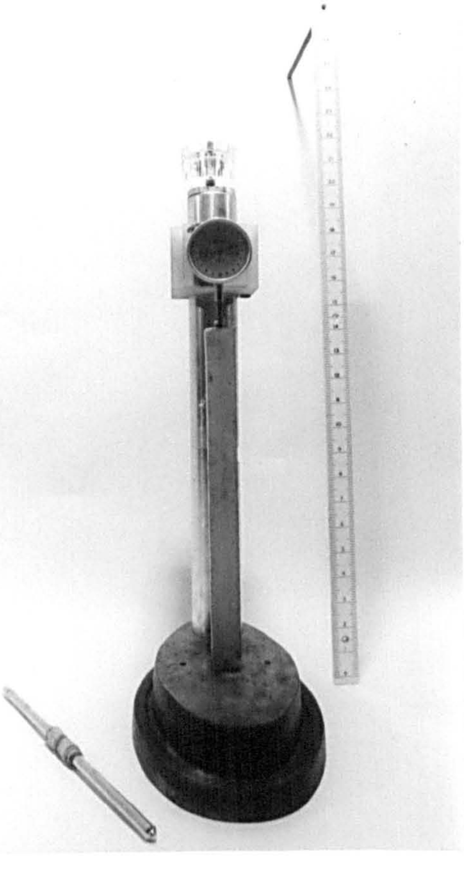


Plate 5.8
Comparator with mortar bar specimen in place

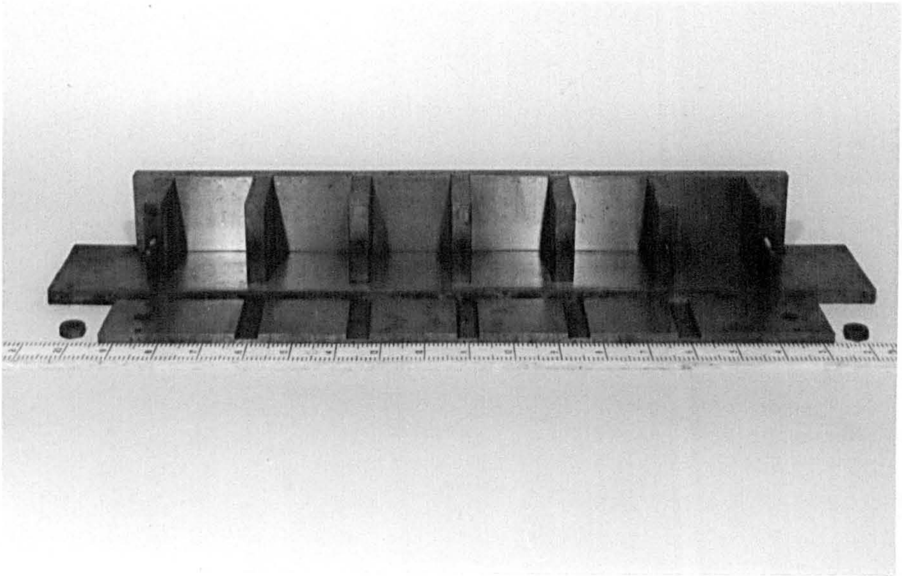


Plate 5.9 Gang mould capable of making six cube specimens (2").

CHAPTER 6 PRESENTATION AND DISCUSSION OF EXPERIMENTAL RESULTS - PHYSICAL

6.1 SERIES 1 MORTAR BAR EXPANSION TESTS (Mixes 000 to 222P)

The average expansions for each of the 27 mortar mixes of the Series 1 experiments are listed in Table C.1 of Appendix C for each of the 5 measurement ages (6 measurement ages in the case of the high alkali cement bars). These data are expressed in microstrain ($\mu\varepsilon$) computed from the measured length changes and the nominal gauge length of 250mm. An explanation of the system of mix reference numbers is given in Chapter 5, Section 5.2.1.

In Table C.1 the level of reactivity of each mix is given a designation which is based on the limit of 'harmful' reactivity of 1000$\mu\varepsilon$ at 6 months (or 500$\mu\varepsilon$ at 3 months when no 6 month results are available) stated in ASTM C33, which is referred to directly by ASTM C227. The limiting value in this case was taken to be 670 $\mu\varepsilon$ at 4 months which is a linear interpolation between the two figures previously quoted. It must be emphasised that the intended purpose of the ASTM mortar bar test is to examine particular cement/aggregate combinations which are proposed for use in constructional concrete. ASTM C227 was not originally designed to include the use of p.f.a. nor synthetic aggregates such as Pyrex glass. Hence, the designation of the mixes herein as either 'harmful' or 'non-harmful' is purely for the purpose of comparison.

Full details of the repeatability computations which were carried out on the expansion measurements are presented in Table C.2 of Appendix C. To maintain the completeness of the experimental results some measured values which fell marginally outside the ASTM repeatability tolerance limits were of necessity retained. In Table C.1 any set of results falling into this category is identified.

The expansion results for each individual mortar mix are also presented graphically in Appendix C (Figures C.1 to C.27). The discrete points plotted on these graphs at the appropriate measurement ages are joined using linear interpolation, since the relatively few data points make curve-fitting inappropriate. Under a different experimental regime continuous monitoring could be used to produce a smooth curve.

Information arising from the visual examination of the mortar bars subsequent to the 4 month expansion measurements is presented in Table C.3 of Appendix C. Certain bars were found to have warped by more than an obviously negligible amount. Subsequent calculations, however, proved that, within the bounds of the accuracy of the expansion measurements, even the maximum noted warping was not sufficient to significantly affect the expansion values.

6.1.1 Expansion of Bars in relation to Time

Reference to the tabulated and graphical results in Appendix C shows that the most rapid expansion occurred during the first two months after casting. Generally, there was no significant increase in expansion beyond four months up to the end of the monitoring period (e.g. mixes 201 to 202P). Notable exceptions to this generalisation are listed below:-

Mix Reference Numbers	012P
	022P
	112P
	122P
	211P
	212P
	222P

In particular mixes 012P, 112P, 211P and 212P showed considerable increases in expansion up to the measurement at age 14 months. Beyond this age a further measurement, at age 18 months, was made only on those bars containing the high alkali cement (200 to 222P) by which stage the expansion of mixes 211P and 212P appeared to have terminated. It should, however, be mentioned that subsequent to the measurements taken at 4 months the specimens were stored under different conditions, as explained in Chapter 5, Section 5.2.3. In addition further changes were made after the measurements at 14 months. Despite these changes in storage regime, mix reference 222P continued to show a slow but steady increase in expansion, even up to the measurement taken at 18 months.

From the mix references listed above it seems likely that the expansion characteristics which relate to time are also linked to the experimental factor B, that is the use of p.f.a.. This link is discussed in detail in the section on p.f.a. below.

6.1.2 Expansion of Bars in relation to Pyrex Glass Content (expressed as a percentage of the total aggregate)

Within the results from the Series 1 experiments all possible comparisons show that the trend is for expansion to increase with increasing Pyrex glass content. This is shown in Figures 6.1, 6.2 and 6.3 for the low, medium and high alkali cement mixes respectively (vid. mixes 000 to 002P, 100 to 102P and 200 to 202P). The three mixes 000, 100 and 200, which do not contain any reactive aggregate, may be considered to be controls and effectively represent the 'zero' level for expansion.

In Figure 6.4 it is shown that the above trend increases in prominence as the alkali content of the cement increases, and that in each case the maximum expansion is attained when 100% Pyrex glass aggregate is used. There is no evidence from the results to suggest a pessimum level for Pyrex glass of less than 100%. However, the use of only two levels of Pyrex glass addition does not permit this possibility to be completely eliminated. Reference should also be made to the results of the Series 2 experiments where additional levels of Pyrex glass addition were included.

6.1.3 Expansion of Bars in relation to the Alkali Content of the Cement (expressed as equivalent Na_2O, as a percentage of the cement content by weight)

For the most highly reactive mixes, that is those containing 100% Pyrex glass aggregate, Figure 6.5 clearly shows that increasing the level of alkali in the cement results in an increase in the observed expansion (vid. mixes 002P, 102P and 202P).

As regards the mixes containing only 50% Pyrex glass aggregate, whilst the overall magnitude of the observed expansions is reduced, the effect of the level of cement alkalis is consistent with that for the 100% Pyrex glass specimens. This is shown in Figure 6.6 for mixes 001P, 101P and 201P.

Reference to the tabulated results for the non-Pyrex glass mixes 000, 100 and 200 shows that the three cements produce similar results, which is to be expected for a non-reactive aggregate. These results represent, therefore, the 'zero' level of expansion any variation in length not being attributable to A.S.R..

6.1.4 Expansion of Bars containing P.F.A. as a Partial Replacement for the Cement Content

A marked reduction in the measured expansions of bars containing Pyrex glass was noted when p.f.a. was introduced as a partial replacement for the cement content. Up to the measurement at age 4 months this effect is consistent for mixes containing either 50% or 100% Pyrex glass in the aggregate and also for all three types of cement (vid. mixes 001P, 011P, 021P; 002P, 012P, 022P; 101P, 111P, 121P; 102P, 112P, 122P; 201P, 211P, 221P; 202P, 212P, 222P). For the lowest alkali cement only, this trend is broken by the results taken at 14 months since the expansion of mix reference 012P exceeds the corresponding expansion for mix reference 002P, as shown in Figure 6.7. In the same Figure it is also shown that the observed expansion for mix reference 022P remains substantially below the values for either of the other two compared mixes. The trend for reduction in expansion by use of p.f.a. continues up to the 14 month readings for all the other relevant groups of mixes. This positive effect is demonstrated in various ways in Figures 6.8 to 6.10.

The data in Table C.1 indicate an apparent change with time in the effectiveness of p.f.a. as an expansion inhibitor. Comparisons show that the p.f.a. reduces expansion by a diminishing degree as time progresses. The rate of expansion for mixes containing p.f.a. tends to decline with time more slowly than the corresponding non-p.f.a. mix. This effect is more clearly shown in Figures 6.7, 6.8 and 6.11 to 6.13 where, at the later measurement ages, the graphs for the p.f.a. mixes are seen to begin converging with the associated graph for the non-p.f.a. mix.

Further evidence of this time-dependent feature can be gained by expressing the expansion of the p.f.a. mixes as a percentage of the corresponding non-p.f.a. mix, as in the data of Table 6.1. At early ages, up to the 4 month measurements in certain cases, the p.f.a. reduces the expansion to a percentage of the corresponding non-p.f.a. mix which decreases with time. Beyond this initial stage, however, with further increases in time, there is a recovery in the percentage expansion value, indicating the convergence seen in the graphs.

For all three cement types this effect is much more pronounced for the mixes which contain a 20% p.f.a. replacement relative to those containing the higher 40% replacement.

6.1.5 Statistical Interpretation of Expansion Data

The results presented in Sections 6.1.1 to 6.1.4 give clear indications of the level of involvement of the three experimental factors in the expansion of mortar bars due to A.S.R.. In statistical terminology, the main effects of the experimental factors are highly significant and, therefore, cause strong trends to be evident in the expansion results. Additional information can be sought from the statistical manipulation of the basic expansion figures since the design of the set of experiments was carried out on a statistical basis.

It was explained in Section 5.1.2 of Chapter 5 that the bulk of the statistical analysis was carried out using a standard statistical package program from a suite of commercially available computer programs (89). Later, however, a set of results from the Series 2 experiments will be used to carry out a long-hand version of the computations in order to illustrate the procedure involved in the Analysis of Variance (ANOVA). Output from the computer for the five main measurement ages of Series 1 (14 days, 28 days, 2 months, 4 months and 14 months) are given in Tables 6.2 to 6.6 together with a summary of the tail probabilities in Table 6.7.

The ANOVA tables confirm the high level of significance of the main effects of the three experimental factors. These derived data are also indicative of a fairly strong interaction between the experimental factors A and B, namely between the effect of cement alkali content and the use of p.f.a.. If 10% is taken to be the cut-off level for significance, below which the variations are considered to be indistinguishable from any experimental error or uncontrollable variation, then the interactions involving the reactive aggregate content (namely AC and BC in the ANOVA tables) can be regarded as negligible.

The model used to perform this ANOVA incorporates the second order interaction (ABC) as the estimate of experimental error, because the data comprise only one result per cell of the experiment. The variations which are due to the insignificant interactions may be removed from the ANOVA by pooling them with the experimental error term. This would form the basis of an alternative model and thus enable further and better interpretation of the data. It is not permissible, however, to remove the variations due to particular interactions whilst retaining other variations due to interactions of the same order. Hence, to remove the two insignificant first order interactions AC and BC from the ANOVA table, the remaining first order interaction AB must also be removed.

The ANOVA tables corresponding to this revised experimental model are given in Tables 6.8 to 6.12 with a summary of the Tail Probabilities in Table 6.13. It can be seen in these tables that the main effects continue to show a high level of significance. This indicates that the main effects are an order of magnitude more important than even the most significant interaction.

When the variance of the 14 month measurements is analysed according to the original model, the main effects of the Experimental Factors A, B and C are shown to be significant at the 99.1%, 97.7% and 98.9% levels respectively. The most highly significant interaction, between Factors A and B, yields a value of 92.8%. Using the revised model, the significance of levels of the main effects are reduced to 97.8%, 94.2% and 97.4% and thus all remain highly significant in comparison to the new error term.

The ANOVA shows that within the ranges of the experimental factors chosen for inclusion in this set of experiments, the alkali content of the cement has the greatest influence on observed expansion. The remaining two main effects have marginally lesser influence, but even the least effect, which is produced by the use of p.f.a., is highly significant in determination of the extent of expansion due to A.S.R..

6.2 SERIES 2 MORTAR BAR EXPANSION TESTS - 3x4x4x2 FACTORIAL SET (Mixes 0000 to 2331P)

The expansions of the 96 mortar mixes which comprise the 3 X 4 X 4 X 2 factorial section of Series 2 were measured at four ages (14 days, 2 months, 6 months and 12 months). The results from these measurements are contained in Table D.1 of

Appendix D, expansions being quoted in microstrain ($\mu\varepsilon$). The system of mix reference numbers is explained in Chapter 5, Section 5.3.1.

Table D.1 also contains an assessment of the level of reactivity of each mix which is again based on the limit of 'harmful' reactivity of 1000$\mu\varepsilon$ at 6 months given in ASTM C33 and referred to directly by ASTM C227. The mixes are designated either harmful or non-harmful. However, it must be reiterated that this ASTM designation is included herein purely for the purpose of comparison of one mix relative to the next.

Graphical representations of the expansion results for each individual mortar mix are found in Figures D.1 to D.96 of Appendix D where the discrete data points are again joined by straight lines.

In continuation of the experimental regime adopted in Series 1, a record was kept of the outcome of the repeatability checks required by the ASTM standard test methods. Output from the associated computer calculations for each of the four measurement ages is also contained in Appendix D (Table D.2).

Typical examples of the photographs taken of the mortar bars after the 6 month expansion measurements are shown in Plates 6.1 to 6.11. The first group of photographs, Plates 6.1 to 6.6 demonstrate the increase in surficial exudation and deposits which occurred when Pyrex glass was included in a mortar containing the high alkali cement. In Plates 6.7 to

6.9 these phenomena are seen to reduce as p.f.a. is used to partially replace the cement content of the mix. For comparison, the remaining photographs, Plates 6.10 and 6.11, show the surface condition of the most highly expansive mix containing the low alkali cement and the control mix containing the high alkali cement with maximum replacement by p.f.a..

6.2.1 Expansion of Bars in relation to Time

The time-based trends established from the Series 1 results are also evident in those from Series 2, the most rapid expansion occurring at early ages. To the extent that it can be judged from the few data points available, by plotting expansion versus time graphs, the most usual overall form of this relationship is a rapid rise in the rate of expansion very soon after casting followed by a decay type curve thereafter. This suggests that the expansion tends to a finite limit, a different limit applying for each individual mortar mix.

In particular it is seen that the bulk of the ultimate expansion, as monitored by this test procedure, was manifest within the first 2 months after casting. After 2 months there was, in most cases, little further expansion up to the final measurements taken in this series of tests at an age of 12 months (e.g. Mixes 2000 to 2030P and 2001 to 2031P). In a manner similar to Series 1, not all the mixes followed this generalisation. Those which had significantly different expansion/time characteristics are listed below:-

0131P	*
0231P	x
1121P	*
1131P	*
1221P	x
1231P	*
1331P	x
2021P	*
2111P	*
2121P	*
2131P	*
2221P	*
2231P	*
2321P	x
2331P	x

where * denotes expansion at 6 months 'harmful'
x denotes expansion at 6 months 'non-harmful'

All the above mixes showed a significant increase in expansion beyond the measurement at 2 months. This can be looked upon as evidence that the attenuation of their expansion rates with time is considerably lower than normal. The link between this characteristic and the presence of p.f.a. in the mix is quite prominent, 2021P being the only exception to this observation in the list of mixes.

It is also apparent that the list contains exclusively mixes which incorporate the higher free water content. This point is discussed at length later when a possible connection between the use of p.f.a. and the free water content of the mix is established.

6.2.2 Expansion of Bars in relation to Pyrex Glass Content (expressed as a percentage of the total aggregate content)

Significant expansions were only observed for mixes containing Pyrex glass. Any mixes without Pyrex glass can, therefore, be treated as experimental controls and used to quantify the base level of expansion unrelated to A.S.R..

The overall trend was for expansion to increase with increasing Pyrex glass content, approaching a maximum expansion for mixes containing an aggregate comprising 100% of the reactive material (vid. mixes 0000 to 0030P, 0001 to 0031P; 1000 to 1030P, 1001 to 1031P; 2001 to 2031P). This is shown in Figures 6.14 to 6.18 where expansion is plotted against age of measurement for each set of compared mixes grouped together on a single set of axes. Further representations of this effect are included in the plots of expansion versus Pyrex glass content which form Figures 6.19 and 6.20. Similar trends are mirrored in the corresponding groups of mixes which include p.f.a. to the various levels, though the absolute values of expansion are dissimilar.

In contrast, the group of mixes omitted from the above comments, namely 2000 to 2030P, shows an indication of a pessimum content for Pyrex glass of less than 100%. In Figure 6.21 it can be seen that the measured expansion for mix 2020P is greater than that for mix 2030P at all measurement ages after 14 days. For the expansion measurements taken at

6 months, Figure 6.22 shows this pessimum effect more clearly, the indications being that maximum expansion would be obtained with a Pyrex glass content in the latter half of the range 33.3% to 100%. The use of p.f.a. with this group of mixes takes the expansions back into step with the generalisation that maximum expansion occurs with mixes containing 100% Pyrex glass aggregate. This may indicate that either mix 2020P or mix 2030P yielded a spurious result. Conversely, the general form of the expansion versus Pyrex glass content curves, shown in Figures 6.19 and 6.20, could be construed to indicate the possibility of a peak expansion for a Pyrex glass content below 100% and in the range 66.6% to 100%.

6.2.3 Expansion of Bars in relation to the Alkali Content of the Cement (expressed as equivalent Na_2O, as a percentage of the cement content by weight)

For the mortars containing 100% Pyrex glass aggregate, the results show that, within the bounds of alkali content set by the cements chosen for the experiments, cements with higher alkali contents promote larger expansions. The expansion against age of measurement graphs for the groups of mixes 0030P/1030P/2030P and 0031P/1031P/2031P, shown in Figures 6.23 and 6.24 respectively, demonstrate this effect for both the chosen free water contents. Figures 6.25 to 6.27 indicate further that neither reducing the proportion of Pyrex glass in the aggregate to either 66.6% or 33.3% nor the introduction of p.f.a. to any of its three levels, caused any noticeable change to the observation.

For the expansion measurements taken at age 6 months, consider the average expansion for all the potentially reactive mixes (i.e. those containing any level of Pyrex glass) which involve the high alkali cement, to represent 100%. The corresponding figures for the medium and low alkali cements can be shown to be 48% and 18% respectively. This clearly expresses the dominant effect of the alkali content of the cement over all the levels of the other factors involved in the experimental set of mortars.

6.2.4 Expansion of Bars containing P.F.A. as a Partial Replacement for the Cement Content

Comparisons similar to those used for the Series 1 results confirm that p.f.a. reduces the expansion of mortar bars which contain Pyrex glass. In particular, Figure 6.28 shows the magnitude of this effect at age 6 months for bars containing 100% Pyrex glass aggregate and the higher free water content. The expansion reducing effect caused by a p.f.a. replacement level of 45% used with the high alkali cement over the full range of Pyrex glass contents is shown in Figure 6.29.

It has been discussed previously that there may be a change in the characteristics of the expansion versus age of measurement graphs brought about by the use of p.f.a.. This trend is again evident in the Series 2 results. Figures 6.30 and 6.31 reveal a tendency for the rate of expansion to decline more slowly with age when p.f.a. is incorporated into the mix. It can be seen from these two figures that, in general, at later ages the curves for the p.f.a. mixes begin to converge with the curve for the appropriate non-p.f.a. mix. In most cases, however, the reduction in expansion attributable to the use of p.f.a. remains extremely significant.

The expansions of the p.f.a. mixes expressed as percentages of the related non-p.f.a. mixes form the basis of Table 6.14. For the high free water content mixes this percentage figure generally falls to a minimum at the 2 month measurement and then shows a recovery over the later measurements, thus substantiating the Series 1 results. However, the corresponding Series 2 figures for the lower free water content mixes do not follow this trend but rather yield a sensibly steady percentage for all measurement ages. This is again suggestive of a strong link between the long term expansion reducing properties of p.f.a. and the free water content of the mix, which has already been referred to in Section 6.2.1.

In addition to the above, the Series 2 factorial set of experiments show p.f.a. to be more effective in reducing the expansions of mixes containing the higher free water content. This is clearly demonstrated by the graphs in Figures 6.32 and 6.33 where expansion is plotted against p.f.a. content. Note the intersection of the curves for the high and low free water content mixes. Such is the difference in the effectiveness of the p.f.a., that the normal condition of the higher free water content being associated with higher expansions is completely reversed at high replacement levels.

A comparison of the magnitude of this effect for measurements taken at 2 months (Figure 6.32), 6 months (Figure 6.34) and 12 months (Figure 6.35) shows that it tends to diminish with time, as shown by the progressive movement of the intersection point on these graphs towards greater age intervals. This change with time might be expected from the earlier discussion concerning the expansion rates of p.f.a. mixes. It is apparent, therefore, that the effectiveness of p.f.a. as an expansion inhibitor is more stable with the lower free water content mixes which, under most circumstances, represent better quality mortars.

The decline in the effectiveness of p.f.a. with time for the high free water content mixes is emphasised by the results from the mixes listed in Section 6.2.1. From this list it is clear that the mixes which continue to exhibit significant rates of expansion beyond the 2 month measurements are exclusively those with the higher free water content. In addition, only one is a mix which does not contain p.f.a. This same list also comprises mainly mixes where the expansion remains above the ASTM 'harmful' limit (at age 6 months) despite the use of p.f.a. Conversely, this would suggest that where the reduction in expansion due to the use of p.f.a. is sufficient for the 'harmful' limit not to be exceeded the effect is reasonably stable with time and, therefore, likely to remain active.

Notwithstanding the foregoing, for reactive mixes within the Series 2 factorial set, p.f.a. was never found to increase expansion to a level above that for the corresponding non-p.f.a. mix at any of the measurement ages within the monitoring period of 12 months.

6.2.5 Expansion of Bars in relation to Free Water Content

Extensive discussion on the effect of the free water content of the mix has already been included in the earlier sections due to its involvement in the explanation of other effects.

Within the range considered, the effect of the free water content of the mix on the level of observed expansion of the mortar bars is shown to be rather less outstanding than the main effects of the other experimental factors. Generally, it is apparent that larger expansions are obtained from mixes containing the higher free water level, as shown in Figures 6.36 and 6.37. This generalised observation was sometimes reversed for mixes containing p.f.a.

For the non-p.f.a. mixes which contain significant amounts of Pyrex glass, thus making them expansive, there is an occasional tendency for the early expansions of the lower free water content mixes to marginally exceed their higher free water content counterparts (Figures 6.38 and 6.39). At later ages the figures show that this situation is reversed to bring the relative expansions into line with the norm referred to above. It is therefore apparent that the onset of rapid expansion can be delayed in mixes containing a high free water content.

The link between the effect of free water content and the effectiveness of p.f.a. as an expansion inhibitor, which can cause a change in the relationship between the expansions of high and low free water content mixes, is fully discussed in the preceding section.

6.2.6 Statistical Interpretation of Expansion Data

For the initial statistical analysis of the Series 2 factorial expansion results, the experimental model includes all the main effects, first order interactions and second order interactions. The single third order interaction (ABCD) is removed from the analysis to provide an estimate of the experimental error.

The ANOVA tables for this model are set out in Tables 6.15 to 6.18 for the four measurement ages at which the expansions of the mortar bars were measured (14 days, 2 months, 6 months and 12 months). The Tail Probabilities for all the measurement ages are summarised in Table 6.19. In addition to these computer derived ANOVA tables, Appendix H contains a fully explained hand-calculated set of computations for the 6 month expansion results of Series 2.

Tables 6.15 to 6.19 show that most of the effects, both main effects and interaction effects, produce high levels of significance. However, the Tail Probabilities of the second order interactions, in particular ABD and ACD, are generally an order of magnitude higher than any of the other values. On a relative basis, therefore, these effects are not as important. Correspondingly, Tables 6.20 to 6.23 contain the results of a second ANOVA based on a new experimental model which excludes the second order interactions in addition to the third order interaction, the variations due to these five effects being pooled together in the experimental error term. A summary of the Tail Probabilities from the new ANOVA tables is given in Table 6.24.

The statistics from the new model show clear confirmation of the high significance level of the main effects of the three experimental factors which are a repetition of the Series 1 experiments, namely the cement alkali content, the p.f.a. replacement level and the reactive aggregate content. From the ANOVA table for the 14 day results, however, the fourth factor, the free water content, is shown to have a low level of significance and, therefore, to have a negligible effect on expansion. At the later measurement ages this situation is dramatically altered, as shown by the rise in significance level to a value comparable with those for the other three main effects.

It is apparent, then, that the statistical analysis of the main effects produces the same interpretation as the observations derived directly from the expansion data. The high level of significance obtained for the main effects of the first three experimental factors shows that each has a strong influence on the observed expansion. The initial temporary stage of insignificance for the main effect of the free water content, followed by a rapid rise to a very high level of significance thereafter, confirms the general observation that increasing the free water content from its lower value to its higher value increases expansion. The early low significance level indicates a time dependent aspect in the main effect due to this factor.

Regarding the interactions between the experimental factors, the ANOVA tables show that most of the first order interactions have a high level of significance, only the interaction AD between the cement alkali content and the free water content shows a relatively large Tail Probability, indicating that this effect is comparatively weak. Even in the revised model only the AD interaction has a tail probability greater than 10% indicating that this is the only insignificant first order interaction.

The high significance value for the BD interaction between the use of p.f.a. and the free water content is confirmation of the observed link between these two factors, where p.f.a. was seen to be more effective in reducing the expansion of reactive mixes incorporating the higher free water content. The high significance of the remaining first order interactions AB, AC, BC and CD is an expression of the non-additivity of effects caused by the individual factors themselves.

For example, considering the 12 month expansion results, the use of p.f.a. at the 45% replacement level (for mixes containing 100% Pyrex glass aggregate and the high free water content) produces a reduction in expansion over the non-p.f.a. mix of 2659 $\mu\varepsilon$, 4000$\mu\varepsilon$ and 5984$\mu\varepsilon$ for the low, medium and high alkali cement mixes respectively. The high level of significance of the AB interaction is an expression of the differences between these and the other figures inherent in its calculation. If there was no interaction, all three differences would be similar.

The importance of the information which indicates the existence of strong interactions, is that it shows the mechanism of the reaction to be complex and that the effects of individual parameters cannot be used to forecast the expansion of bars incorporating combinations of the parameters.

Similar treatment of the least significant interaction (AD) between the cement alkali content and the free water content shows that the sum of the effects caused by these factors taken individually is more nearly equivalent to the combined effect. Again considering the 12 month expansion results, the increase in free water content from the lower level to the higher level (for mixes containing 0% p.f.a. and 100% Pyrex glass aggregate) results in expansion increases of 1448$\mu\varepsilon$, 1522$\mu\varepsilon$ and 2928$\mu\varepsilon$ for the low, medium and high alkali cements respectively. In particular, for the low and medium alkali cements these figures are quite similar indicating the weaker nature of the interaction.

6.3 SERIES 2 MORTAR BAR EXPANSION TESTS - 'EXTRA PYREX' SET (Mixes 2032P to 2035P, 2041P to 2071P, 2431P to 2731P and 3031P to 6031P)

The mortar bars of the 'Extra Pyrex' extension to the coverage of the Series 2 factorial set of mixes were produced, conditioned and monitored in a manner identical to that for all the other Series 2 mortar bars. The expansion results are set out in Table E.1 of Appendix E, together with a record of the repeatability compliance checks in Table E.2. These numerical expansion data are also translated into individual plots of expansion versus age of measurement which form Figures E.1 to E.16, also in Appendix E.

Unlike their counterparts from the factorial sets of Series 1 and 2, the expansion results from the EP mixes are not suitable for any advanced statistical manipulation.

The set of 16 mixes fall into 4 groups, each of which examines a particular aspect of the alkali silica reaction that is linked to the Series 2 factorial set of experiments. Full details of the mixes can be found in Chapter 5, Section 5.3. Each aspect of the reaction is considered separately and, when possible, independently of any changes to the other factors involved in the make-up of the mortar mixes. It is important to consider, however, that these additional experiments are not able to take account of any interaction between the experimental factors.

The following discussion takes each group of 4 mixes in turn, any trends found in the results being related back to the appropriate foregoing results of Series 1 and 2. Photographic records of certain mortar bars from this set of mixes are contained in Plates 6.12 to 6.16.

6.3.1 Expansion Bars with varying Free Water Contents (Mixes 2032P to 2035P)

The four mixes were designed to examine the effect of variations in the free water content on the expansion of mortars containing 100% reactive aggregate and no p.f.a. They are in addition to the two related mixes included in the Series 2 factorial set, namely 2030P and 2031P. Since p.f.a. has not been used in these mixes it is both possible and more meaningful to refer to the free water contents in terms of the free water/cement ratios (w/c) which are listed below:-

Mix Reference Number	Free w/c ratio
2032P	0.30
2030P *	0.40
2033P	0.45
2034P	0.50
2031P *	0.55
2035P	0.60

(* denotes mix from Series 2 factorial set)

Figure 6.40 shows a combined plot of expansion versus age of measurement for the mixes. Close examination of the curves at the initial measurement age of 14 days reveals further evidence of the tendency for the early expansions of high free water content mixes to be temporarily supressed, this was discussed in Section 6.2.5.

For the later ages it can be seen that within the lower range of free water contents the trend is for higher expansions to be promoted by increases in the amount of free water, again referring back to the earlier discussion in Section 6.2.5. However, Figure 6.40 demonstrates that, above a free w/c ratio of 0.55, a further increase in the free water content yields a reduction in expansion. It is therefore apparent that a pessimum value of free w/c ratio exists, which for these mixes lies between 0.50 and 0.60. This pessimum effect is more clearly shown in Figure 6.41 and 6.42 where expansion is plotted versus free w/c ratio for the 6 month and 12 month results respectively.

6.3.2 Expansion of Bars with Low Pyrex Glass Contents (Mixes 2041P to 2071P)

For the four levels of Pyrex glass content used in the Series 2 full factorial set it has been shown that generally 100% Pyrex glass is associated with the largest expansion and thus represents the pessimum content. Further, successive reductions in the range 100%, 66.6% and 33.3% were each shown to cause reduced expansion. The results of the four mixes 2041P to 2071P show that this trend is continued for Pyrex glass contents of 16%, 12%, 8% and 4% incorporated into mixes using the high alkali cement, no p.f.a. and the higher free water content from the main set. Figure 6.43 shows the plot of expansion versus Pyrex glass content, including the supplementary points, demonstrating the lack of a possible irregularity in the curve at low Pyrex glass contents.

6.3.3 Expansion of Bars containing P.F.A. used as an Admixture (Mixes 2431P to 2731P)

In contrast to the use of p.f.a. as a partial replacement for the cement content, this group of four mixes incorporated p.f.a. as a direct addition to the most highly reactive non-p.f.a. mix from the Series 2 main set of experiments, namely mix 2031P. Thus, the effect of p.f.a. addition may be compared with the effect of cement replacement in the range of mixes 2031P to 2331P.

Reference to the results in Table E.1 and Figures E.9 to E.12 of Appendix E shows that for additions of 10% to 40% (by weight of cement), p.f.a. used as an admixture has a beneficial effect as an expansion inhibitor. The comparison of the two methods of utilizing p.f.a. is illustrated in Figure 6.44 which also shows further evidence of the progressive reductions in expansion in the group of mixes 2431P to 2731P. It is apparent that the direct addition of p.f.a. to a reactive mortar mix is not as effective in terms of expansion reduction as a similar percentage cement replacement. On a relative basis, a 30% addition is shown to be similar in effectiveness to a 15% replacement. Whilst this difference is obviously significant it does not detract from the beneficial effect that p.f.a. produces.

The success of p.f.a. addition in reducing the expansion of a highly reactive mix indicates that when it is used as a partial replacement of the cement content it does not simply act as a diluent of the cement alkali/reactive silica ratio thereby reducing expansion. Rather there is evidence of a more fundamental effect which is still active when a direct addition of p.f.a. is used, the cement alkali/reactive silica (from the aggregate) ratio thus remaining constant. Two possibilities remain for the mechanism of this effect. It may be purely physical, with the p.f.a. acting as a filler to occupy the voids, interstices and capillaries within the mortar matrix and thus obstructing the expansive nature of the A.S.R.. Alternatively, the mechanism may be chemical with the p.f.a. acting as a chemical inhibitor. This problem will be investigated further in the Chemical Investigation section described in Chapters 7 and 8.

6.3.4 Expansion of Bars containing Reduced Quantities of High Alkali Cement (Mixes 3031P to 6031P)

The range of alkali contents in this group of four mixes, produced by using different quantities of the highest alkali cement, is comparable with the range produced by the three different cements used in the Series 2 factorial set (that is in mixes 0031P, 1031P and 2031P). Below is a list of the calculated alkali contents of the various mixes which are given in kilogrammes equivalent sodium oxide per cubic metre (kg Na_2O_{eq}/m^3) of fully compacted wet mortar for alkalis derived from the cement only.

Mix Reference Number	Alkali Content (kg Na_2O_{eq}/m^3)
0031P *	2.42
1031P *	3.76

2031P *	6.10
3031P	2.68
4031P	3.23
5031P	3.74
6031P	4.64

(* denotes mix from Series 2 factorial set)

The effect of the alkali content of the cement itself on the expansion of mortar bars has already been discussed in Section 6.1.3 for Series 1 mixes and Section 6.2.3 for Series 2 mixes. This effect can now be reviewed in comparison with the expansions of the new group of mixes. Reference back to Figure 6.24 gives an indication of the variation in expansion derived from the different cements of mixes 0031P, 1031P, 2031P. Similarly, Figure 6.45 shows the variation in expansion derived from the different levels of high alkali cement used in mixes 3031P to 6031P. Comparison of these two sets of graphs, shows that reducing the amount of alkalis available from the cement by lowering the cement content of the mix has a far greater effect on expansion than reducing the mix alkali level to a similar value by using a fixed amount of lower alkali cement. This effect may be due in full or in part to an increased porosity and void space corresponding to the mortars with reduced cement contents.

6.4 SERIES 2 MORTAR BAR EXPANSION TESTS - 'BELTANE OPAL' SET (Mixes 2001 to 2371B)

The results of the mortar bar expansion tests for mixes incorporating Beltane opal as the reactive aggregate are set out in Appendix F in tabulated (Table F1) and graphical (Figures F1 to F16) form. Repeatablity compliance checks were also conducted, the results of which comprise Table F2.

It is immediately apparent from the results that the expansions of the Beltane opal mixes are much lower that their Pyrex glass counterparts, the difference being in the region of a factor of ten. Much greater expansions have been achieved with the use of Beltane opal (45) under different experimental regimes involving the incorporation of only the most reactive size fraction and different storage temperatures.

Despite the much lower expansion values, Plates 6.17 to 6.22 show that the Beltane opal mixes still exhibit substantial amounts of surficial exudation and staining. This is particularly true for the mix 2041B (Plate 6.17) which produced the maximum expansion.

6.4.1 Expansion of Bars in relation to Time

The expansion versus time graphs take a similar form to the comparable graphs for mortars containing Pyrex glass, notwithstanding the scale change of the expansion itself. In general the bulk of the expansion again occurs during the first 2 months after casting. However, in every case the Beltane opal bars show an increase in expansion between the ages 2 months and 6 months, although in certain instances this increase is fairly nominal. Beyond 6 months and up to the final expansion measurements at 12 months, the mixes listed below show a further increase which, in all cases, is fairly nominal.

2011B

2021B

2031B

2341B

6.4.2 Expansion of Bars in relation to Beltane Opal Content

The mixes 2001 and 2301, which contain purely non-reactive sand as aggregate, represent the datum level against which all expansions from the Beltane opal mixes may be compared. From the results in Appendix F, it can be concluded that, in all the cases included in this set of experiments, the inclusion of Beltane opal into the mix produces a greater expansion value than the datum level. However, the form of the graph showing expansion versus Beltane opal content (e.g. Figure 6.46, for the 6 month results) is quite different from the corresponding curve for Pyrex glass (cf Figure 6.46 with Figure 6.43).

There is clear evidence of a strong pessimum content lying between 0% and 8% Beltane opal content by weight of total aggregate. The most likely position for this pessimum value is around 4% Beltane opal, since this is the point of maximum expansion in these experiments. This observation is in agreement with the published findings of other research workers, even when the Beltane opal has been included in the mortar only in its most reactive particle size range of 150 to

300μm (21).

With further increases in Beltane opal content, above the pessimum value, the expansion is seen to fall off rapidly to a minimum in the region of 12% to 33%. Beyond this region the expansion rises again, towards a secondary maximum at 100% Beltane opal content. In the literature (21, 45) it has been implied that above the pessimum content the expansion of mortars containing Beltane opal tends towards zero, becoming negligible beyond a content of approximately 20% by weight of total aggregate. Notwithstanding the fact that the information in the literature is again related to the different experimental regime referred to earlier, these two different forms of the relationship between expansion and Beltane opal content would appear to be irreconcilable.

6.4.3 Expansion of Bars containing P.F.A. as a Partial Replacement for the Cement Content

It has been stated previously (94) that at the pessimum Beltane opal content of 4% the use of p.f.a. at a high cement replacement level of 45% produces a marked reduction in the observed expansion (see Figure 6.47). At all other Beltane opal concentrations, after the initial expansion measurements taken at 14 days, the use of p.f.a. yields increased expansion (e.g. Figure 6.48). The increases are small in comparison with the decrease observed at the pessimum Beltane opal content and also relative to the comparable Pyrex glass mixes. (cf. mixes 2011P - 2311P, 2021P - 2321P and 2031P - 2331P with mixes 2011B - 2311B, 2021B - 2321B and 2031B - 2331B respectively). For the expansions observed at ages 6 months and 12 months respectively, Figures 6.49 and 6.50 show the overall effect of using p.f.a. with Beltane opal reactive mixes.

There is some evidence contained in the results from the Beltane opal mixes to suggest that p.f.a. produces an initial delay in the onset of rapid expansion. This is similar to the observation made for the Pyrex glass mixes. In Table 6.25 the expansions of the Beltane opal mixes which contain p.f.a. are expressed as a percentage of their non-p.f.a. counterparts. At the pessimum Beltane opal concentration of 4%, where the p.f.a. has a similar effect to that observed when it is used with Pyrex glass, the trend with time for the percentage expansion value is for it to diminish to a minimum at age 2 months followed by a recovery up to the final measurement taken at age 12 months. This is in complete agreement with the trend observed for the Pyrex glass mixes.

At all other Beltane opal concentrations, where the use of p.f.a. causes increased expansion, the situation is reversed, since the percentage expansion values of Table 6.25 now rise to a maximum at age 2 months and thereafter tend to diminish. Thus in the case of the Beltane opal mixes it is the non-p.f.a. mixes which show the greater tendency to continue expanding at later ages except where the pessimum Beltane opal concentration is used. This is the converse of the observation for the Pyrex glass mixes.

6.5 SERIES 2 COMPRESSION TESTS ON 2" MORTAR CUBES (Mixes 2000 to 2331P)

The main purpose of the mortar cube tests was to gain some insight into the level of deterioration of the mortar in comparison with the measured linear expansion of the equivalent mortar bar. Whilst the microcracking associated with expansion will have its greatest effect on the tensile strength of the mortar, if the disruption is serious one would also expect the homogeneity of the mortar structure to be affected, thus causing a reduction in compressive strength,

The compressive strength of each mix (mixes containing high alkali cement only) was measured at the three earliest ages selected for expansion measurement in the mortar bars, namely 14 days, 2 months and 6 months. Ideally the cube specimens for these tests would have been stored under identical conditions to those for the corresponding mortar bars. However, due to practical limitations regarding the facilities available at the time the work commenced, this was not possible. Insufficient storage containers of the type where specimens are able to be supported over a reservoir of water were available. The alternative method of storage which was selected, whereby the damp cubes were wrapped in polythene film, was considered to be the most suitable method that was available. The procedure assumed that the moisture sealed within the polythene film would be sufficient to allow the expansive reaction to proceed uninhibited, as suggested by Diamond et al (49) who carried out expansion tests on specimens sealed in butyl rubber jackets.

For comparison, a further set of compression tests was carried out on mortar cubes cured and stored under the normal conditions employed for standard compression testing of mortar cubes as defined in BS 4551: 1980 (93).

The compression test results are given in Table G.1 of Appendix G. Each figure in the table is an average value obtained from the results of two individual cubes. Also shown in Table G.1 is the average measured saturated density of each set of 8 cubes, which in most cases compares favourably with the calculated wet density given in Table B.1 of Appendix B.

6.5.1 Variations in Strength apparently unrelated to A.S.R.

Before considering the variations in measured strength in relation to expansion caused by A.S.R., it is pertinent to consider those variations in strength which exist and appear unrelated to this phenomenon.

From the results of Table G.1 it can be seen that for the 'wet' cured cubes tested at 28 days, there is a trend for the use of p.f.a. to reduce the measured strength, particularly with the percentage cement replacements of 30% and 45%. This is true for both levels of free water content and all levels of Pyrex glass content, for example, comparing the 28 day strengths of the pairs of mixes 2000/2300P, 2030P/2330P and 2011P/2211P. In the last two pairs the strength is reduced by the use of p.f.a. despite the reduction in the expansive reaction which p.f.a. is known to achieve (see expansion results in Table D.1 of Appendix D).

With respect to the 'dry' cured cubes, the effects are similar for the three sets of results taken at 14 days, 2 months and 6 months. The use of p.f.a. at cement replacement levels of 15% and 30% causes a progressive increase in measured strength for the non-reactive mixes (i.e. those containing 100% non-reactive aggregate). For the 45% cement replacement level, however, the measured strength falls off again but remains above the strength of the corresponding non-p.f.a. mix. This trend is fairly consistent for both levels of free water content.

It is generally accepted that the partial replacement of the cement content by the use of p.f.a. can cause early strength reductions in comparison with the equivalent non-p.f.a. mix. At later ages, this early strength loss is generally followed by the production of higher strengths from the p.f.a. mixes. The results in Table G.1 indicate that for the 'dry' (and hot) cured cubes the measured strengths of the non-reactive p.f.a. mixes exceeds the value of their non-p.f.a. counterparts even at the earliest measurement age of 14 days and for the highest replacement level of 45%.

The reduction in the 28 day strength of the 'wet' cured cubes, brought about by the inclusion of p.f.a., is then indicative that the pozzolanic properties of the p.f.a. have not had sufficient time to allow them to compensate for the reduced cement content. This effect is normal and totally expected when consideration is given to the method used for the cement replacement calculation. However, the strength results from the 'dry' cured cubes show that the high storage temperature more than compensates for this effect, enabling the mixes incorporating p.f.a. to produce the higher strengths.

6.5.2 Variations in Strength in relation to Mortar Bar Expansion

Considering firstly the 'wet' cured specimens, the compressive strengths at 28 days, as shown in Table G.1, indicate a trend for measured strength to decrease with increasing Pyrex glass content and hence increasing expansion. This effect is much more evident for mixes containing no p.f.a. or containing p.f.a. at the lowest cement replacement level of 15% but appears to be consistent for both levels of free water content used in the experiments.

At the higher levels of cement replacement of 30% and 45% there are two possible factors which may contribute to the reduction in the effect of Pyrex glass content on measured strength. In Section 6.2.4 the expansion has been shown to be greatly reduced by the use of 30% and 45% p.f.a., even for the highest level of Pyrex glass content. The effect on strength relative to Pyrex glass content might, therefore, also be expected to be reduced. In addition, the tendency for p.f.a. itself to reduce the 28 day strength of 'wet' cured mortar may mask the effect due to the Pyrex glass content.

Incorporated within any effect on strength, which is attributed to expansion and disruption caused by the inclusion of Pyrex glass into the mix, is the effect due to the strength of this material itself. The Pyrex glass and the crushed sand are unlikely to produce mortars of identical strength even if used with a very low alkali cement and hence in the absence of serious expansion. Since no testing was carried out to pursue this particular point, apportionment of the effect into these two components is not possible.

The results obtained from the compression tests carried out on the 'dry' cured mortar cubes do not exhibit such a well defined trend in relation to Pyrex glass content. For the higher free water content mixes there is no recognisable trend whatsoever. In many cases for the lower free water content mixes, any decrease in strength due to increasing Pyrex glass content only occurs over the highest part of the Pyrex glass range (66.6% and 100%). In particular it is notable that for the non-p.f.a. mixes the results obtained at 2 months and 6 months show the minimum strength to be obtained from the mix containing 0% Pyrex glass. This is indicative that any trend for strength to be reduced by increases in Pyrex glass content cannot be simply dismissed as being caused by the Pyrex glass being a weaker and less physically suitable aggregate than the crushed flint gravel (sand). At higher levels of p.f.a. inclusion, however, the successive increases in the amount of Pyrex glass used in the mix do show a slight trend towards strength reductions.

Notwithstanding the possible degradation effect of using mortars containing p.f.a. under water, the differences in the trends of strength relative to the Pyrex glass content which are exhibited at each level of cement replacement for the two curing regimes, would seem to indicate that the 'dry' method does not allow the system to behave in the expected manner. It therefore appears that there is insufficient moisture for the cubes to respond in the same way as the mortar bars respond to the high humidity environment established in the storage containers. This observation complicates any attempt to relate variations in strength measurements to the measured expansions from the mortar bars.

No satisfactory explanation has been found for the trend in strengths of 'dry' cured cubes to show an increase with time up to age 2 months and then to fall off again at the 6 month measurement. This effect is consistent at all levels of cement replacement, all levels of Pyrex glass content and for both free water contents.

TABLE 6.1 EXPANSION OF MIXES INCORPORATING P.F.A. EXPRESSED AS A PERCENTAGE OF THE EXPANSION OF THE CORRESPONDING NON-P.F.A. MIX - SERIES 1

Source of Expansion Ratio	Age of Measurement					
	14 days	28 days	2 months	4 months	14 months	18 months
$\frac{212P}{202P} \times 100\%$	29.4%	21.6%	22.2%	24.8%	31.8%	31.5%
$\frac{222P}{202P} \times 100\%$	18.7%	12.5%	12.0%	11.7%	13.4%	13.9%
$\frac{211P}{201P} \times 100\%$	24.1%	17.0%	16.3%	19.5%	29.4%	28.9%
$\frac{221P}{201P} \times 100\%$	14.0%	9.3%	8.8%	9.3%	12.1%	11.1%
$\frac{112P}{102P} \times 100\%$	45.4%	34.5%	34.7%	35.8%	44.7%	-
$\frac{122P}{102P} \times 100\%$	19.9%	15.4%	15.1%	15.1%	18.1%	-
$\frac{111P}{101P} \times 100\%$	48.6%	35.2%	33.3%	35.3%	50.8%	-
$\frac{121P}{101P} \times 100\%$	30.9%	27.9%	24.4%	26.7%	39.8%	-
$\frac{012P}{002P} \times 100\%$	80.0%	58.7%	49.1%	72.3%	134.9%	-
$\frac{022P}{002P} \times 100\%$	82.4%	44.3%	32.9%	33.5%	43.7%	-
$\frac{011P}{001P} \times 100\%$	78.7%	63.2%	65.8%	69.5%	101.6%	-
$\frac{021P}{001P} \times 100\%$	90.7%	50.5%	39.2%	33.3%	91.3%	-
$\frac{102P}{202P} \times 100\%$	34.3%	30.9%	30.8%	31.7%	32.7%	-
$\frac{002P}{202P} \times 100\%$	4.9%	5.6%	8.3%	9.1%	10.4%	-
$\frac{201P}{202P} \times 100\%$	52.4%	53.8%	57.8%	58.0%	58.0%	-
$\frac{101P}{102P} \times 100\%$	21.6%	20.4%	22.1%	22.4%	22.7%	-
$\frac{001P}{002P} \times 100\%$	6.5%	5.7%	4.3%	5.3%	6.1%	-

```
PAGE   6     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  1-ST
DEPENDENT VARIABLE - EXPN1

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE      EFFECT           SUM OF     DEGREES OF       MEAN          F       TAIL
                                  SQUARES      FREEDOM        SQUARE                 PROB.

      MEAN                    4528046.25926       1      4528046.25926     31.00    0.0005
      CEMENT        A         2885750.51852       2      1442875.25926      9.88    0.0069
      PFA           B         1900414.74074       2       950207.37037      6.51    0.0210
      AGGRGATE      C         2504864.29630       2      1252432.14815      8.58    0.0102
      CP            AB        1915140.81481       4       478785.20370      3.28    0.0718
      CA            AC        1686039.25926       4       421509.81481      2.89    0.0944
      PA            BC        1236086.37037       4       309021.59259      2.12    0.1704
  1   ERROR                   1168390.74074       8       146048.84259
```

TABLE 6.2 ANOVA TABLE FOR SERIES 1 - 14 DAY EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE   7     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  2-ND
DEPENDENT VARIABLE - EXPN2

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE       EFFECT            SUM OF     DEGREES OF       MEAN            F        TAIL
                                     SQUARES     FREEDOM        SQUARE                    PROB.

      MEAN                        8941831.25926     1     3941831.25926        20.31    0.0020
      CEMENT         A            6515265.40741     2     3257632.70370         7.40    0.0152
      PFA            B            5809643.18519     2     2904821.59259         6.60    0.0203
      AGGRGATE       C            5263914.96296     2     2631957.48148         5.98    0.0258
      CP             AB           5368130.81481     4     1467032.70370         3.33    0.0692
      CA             AC           3712506.37037     4      928126.59259         2.11    0.1714
      PA             BC           3777579.25926     4      944394.81481         2.15    0.1663
  1   ERROR                       3521546.74074     8      440193.34259
```

TABLE 6.3 ANOVA TABLE FOR SERIES 1 - 28 DAY EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE    8     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  3-RD
DEPENDENT VARIABLE - EXPN3

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE      EFFECT           SUM OF     DEGREES OF      MEAN            F       TAIL
                                   SQUARES     FREEDOM        SQUARE                  PROB.

      MEAN                     11292093.37037      1    11292093.37037      21.41    0.0017
      CEMENT        A           8255159.18519      2     4127579.59259       7.83    0.0131
      PFA           B           7698240.07407      2     3849120.03704       7.30    0.0157
      AGGRGATE      C           6752450.29630      2     3376225.14815       6.40    0.0219
      CP            AB          7405751.70370      4     1851437.92593       3.51    0.0616
      CA            AC          4252666.81481      4     1063166.70370       2.02    0.1850
      PA            BC          4715206.59259      4     1178801.64815       2.24    0.1547
  1   ERROR                     4218724.96296      8      527340.62037
```

TABLE 6.4 ANOVA TABLE FOR SERIES 1 - 2 MONTH EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE   9     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  4-TH
DEPENDENT VARIABLE - EXPN4

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE        EFFECT               SUM OF      DEGREES OF        MEAN           F        TAIL
                                         SQUARES      FREEDOM          SQUARE                  PROB.

      MEAN                           13666136.33333       1    13666136.33333       23.52    0.0013
      CEMENT          A               9196957.55556       2     4598478.77778        7.91    0.0127
      PFA             B               8352134.88889       2     4176067.44444        7.19    0.0163
      AGGRGATE        C               7926888.66667       2     3963444.33333        6.82    0.0187
      CP              AB              7861949.55556       4     1965487.38889        3.38    0.0670
      CA              AC              4664914.44444       4     1166228.61111        2.01    0.1864
      PA              BC              5228066.44444       4     1307016.61111        2.25    0.1530
  1   ERROR                           4648543.11111       8      581067.88889
```

TABLE 6.5   ANOVA TABLE FOR SERIES 1 - 4 MONTH EXPANSION
RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE  10     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  5-TH
DEPENDENT VARIABLE - EXPN5

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE       EFFECT            SUM OF      DEGREES OF       MEAN            F        TAIL
                                     SQUARES      FREEDOM        SQUARE                    PROB.

      MEAN                      18388426.81481       1     18388426.81481       32.33     0.0005
      CEMENT         A          10105080.51852       2      5052540.25926        8.88     0.0093
      PFA            B           7178158.74074       2      3589079.37037        6.31     0.0226
      AGGRGATE       C           9586148.96296       2      4793074.48148        8.43     0.0107
      CP             AB          7449262.59259       4      1862315.64815        3.27     0.0720
      CA             AC          4643323.70370       4      1162080.92593        2.04     0.1809
      PA             BC          5149073.48148       4      1287268.37037        2.26     0.1513
 1    ERROR                      4549675.18512       8       568709.39815

NUMBER OF INTEGER WORDS OF STORAGE USED IN PRECEDING    PROBLEM    3148
CPU TIME USED      11.580 SECONDS
```

TABLE 6.6 ANOVA TABLE FOR SERIES 1 - 14 MONTH EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

TABLE 6.7 SUMMARY OF TAIL PROBABILITIES - SERIES 1, ORIGINAL EXPERIMENTAL MODEL

	Source of Variation	Age of Measurement				
		14 days	28 days	2 months	4 months	14 months
MAIN EFFECTS	Cement - A	0.0069	0.0152	0.0131	0.0127	0.0093
	P.f.a. - B	0.0210	0.0203	0.0157	0.0163	0.0226
	Aggregate - C	0.0102	0.0258	0.0219	0.0187	0.0107
1st ORDER INTERACTONS	AB	0.0718	0.0692	0.0616	0.0670	0.0720
	AC	0.0944	0.1714	0.1850	0.1864	0.1809
	BC	0.1704	0.1663	0.1547	0.1530	0.1513

```
PAGE   6     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  1-ST
DEPENDENT VARIABLE - EXPN1

      SOURCE       EFFECT         SUM OF      DEGREES OF      MEAN          F      TAIL
                                 SQUARES       FREEDOM       SQUARE                PROB.

      MEAN                   4528046.25926       1     4528046.25926     15.08   0.0009
      CEMENT         A       2385750.51852       2     1442875.25926      4.81   0.0198
      PFA            B       1900414.74074       2      950207.37037      3.16   0.0640
      AGGRGATE       C       2504864.29630       2     1252432.14815      4.17   0.0306
 1    ERROR                  6005657.18519      20      300282.85926
```

TABLE 6.8 ANOVA TABLE FOR SERIES 1 - 14 DAY EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL

```
PAGE   7     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  2-ND
DEPENDENT VARIABLE - EXPN2

      SOURCE        EFFECT            SUM OF      DEGREES OF       MEAN              F        TAIL
                                      SQUARES      FREEDOM        SQUARE                      PROB.

      MEAN                         8941831.25926       1      8941831.25926        10.59     0.0040
      CEMENT          A            6515265.40741       2      3257632.70370         3.86     0.0382
      PFA             B            5809643.18519       2      2904821.59259         3.44     0.0519
      AGGRGATE        C            5263914.96296       2      2631957.48148         3.12     0.0662
  1   ERROR                       16879763.18519      20       843988.15926
```

TABLE 6.9 **ANOVA TABLE FOR SERIES 1 - 28 DAY EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL**

```
PAGE    8      ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  3-RD
DEPENDENT VARIABLE - EXPN3

       SOURCE        EFFECT              SUM OF     DEGREES OF       MEAN             F        TAIL
                                         SQUARES     FREEDOM         SQUARE                     PROB.

       MEAN                          11292093.37037       1    11292093.37037      10.97     0.0035
       CEMENT          A              8255159.18519       2     4127579.59259       4.01     0.0344
       PFA             B              7698240.07407       2     3849120.03704       3.74     0.0417
       AGGRGATE        C              6752450.29630       2     3376225.14815       3.28     0.0587
  1    ERROR                         20592350.07407      20     1029617.50370
```

TABLE 6.10   ANOVA TABLE FOR SERIES 1 - 2 MONTH EXPANSION RESULTS, REVISED EXPERIMENTAL MODEL

```
PAGE    9      ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  4-TH
DEPENDENT VARIABLE - EXPN4

      SOURCE         EFFECT             SUM OF       DEGREES OF       MEAN            F         TAIL
                                        SQUARES       FREEDOM         SQUARE                     PROB.

      MEAN                        13666136.33333        1    13666136.33333        12.20     0.0023
      CEMENT            A          9196957.55556        2     4598478.77778         4.11     0.0321
      PFA               B          8352134.88889        2     4176067.44444         3.73     0.0421
      AGGRGATE          C          7926888.66667        2     3963444.33333         3.54     0.0483
  1   ERROR                       22403473.55556       20     1120173.67778
```

TABLE 6.11   ANOVA TABLE FOR SERIES 1 - 4 MONTH EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL

```
PAGE  10     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 1,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  5-TH
DEPENDENT VARIABLE - EXPN5

       SOURCE      EFFECT            SUM OF     DEGREES OF       MEAN           F        TAIL
                                     SQUARES     FREEDOM         SQUARE                  PROB.

       MEAN                     18388426.81481       1   18388426.81481      16.87     0.0005
       CEMENT        A          10105080.51852       2    5052540.25926       4.64     0.0222
       PFA           B           7178158.74074       2    3589079.37037       3.29     0.0580
       AGGRGATE      C           9586148.96296       2    4793074.48148       4.40     0.0261
  1    ERROR                    21796334.96296      20    1089816.74815

NUMBER OF INTEGER WORDS OF STORAGE USED IN PRECEDING    PROBLEM    2762
CPU TIME USED      12.054 SECONDS
```

TABLE 6.12 **ANOVA TABLE FOR SERIES 1 - 14 MONTH EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL**

TABLE 6.13 SUMMARY OF TAIL PROBABILITIES - SERIES 1, REVISED EXPERIMENTAL MODEL

	Source of Variation	Age of Measurement				
		14 days	28 days	2 months	4 months	14 months
MAIN EFFECTS	Cement - A	0.0198	0.0382	0.0344	0.0321	0.0222
	P.f.a. - B	0.0640	0.0519	0.0417	0.0421	0.0580
	Aggregate - C	0.0306	0.0662	0.0587	0.0483	0.0261

TABLE 6.14 EXPANSION OF MIXES INCORPORATING P.F.A. EXPRESSED AS A PERCENTAGE OF THE EXPANSION OF THE CORRESPONDING NON-P.F.A. MIX - SERIES 2

Source of Expansion Ratio	Age of Measurement			
	14 days	2 months	6 months	12 months
$\frac{2131P}{2031P} \times 100\%$	46.2%	43.1%	46.1%	48.0%
$\frac{2231P}{2031P} \times 100\%$	24.0%	21.6%	26.9%	30.7%
$\frac{2331P}{2031P} \times 100\%$	12.7%	10.1%	11.5%	12.6%
$\frac{2130P}{2030P} \times 100\%$	47.8%	48.7%	49.5%	49.4%
$\frac{2230P}{2030P} \times 100\%$	38.8%	39.2%	40.0%	39.6%
$\frac{2330P}{2030P} \times 100\%$	27.0%	27.2%	27.7%	27.7%
$\frac{2121P}{2021P} \times 100\%$	51.5%	46.8%	55.0%	55.6%
$\frac{2221P}{2021P} \times 100\%$	20.6%	14.5%	18.0%	21.9%
$\frac{2321P}{2021P} \times 100\%$	10.9%	7.5%	8.4%	9.1%
$\frac{2120P}{2020P} \times 100\%$	37.9%	35.4%	36.1%	35.6%
$\frac{2220P}{2020P} \times 100\%$	24.8%	22.5%	23.5%	23.4%
$\frac{2320P}{2020P} \times 100\%$	14.5%	13.1%	13.9%	13.9%
$\frac{2111P}{2011P} \times 100\%$	38.8%	33.1%	56.8%	61.6%
$\frac{2211P}{2011P} \times 100\%$	23.0%	13.9%	16.1%	17.2%
$\frac{2311P}{2011P} \times 100\%$	14.3%	8.1%	10.1%	9.9%
$\frac{2110P}{2010P} \times 100\%$	40.9%	30.6%	30.0%	29.9%
$\frac{2210P}{2010P} \times 100\%$	22.9%	17.2%	18.6%	19.1%
$\frac{2310P}{2010P} \times 100\%$	19.2%	15.3%	16.6%	16.8%

TABLE 6.14 - continued

Source of Expansion Ratio	Age of Measurement			
	14 days	2 months	6 months	12 months
$\frac{1131P}{1031P} \times 100\%$	40.2%	51.4%	60.4%	62.5%
$\frac{1231P}{1031P} \times 100\%$	19.0%	18.4%	28.2%	34.6%
$\frac{1331P}{1031P} \times 100\%$	7.3%	6.0%	8.0%	8.7%
$\frac{1130P}{1030P} \times 100\%$	41.7%	42.3%	42.2%	41.5%
$\frac{1230P}{1030P} \times 100\%$	27.5%	27.4%	28.2%	28.3%
$\frac{1330P}{1030P} \times 100\%$	14.7%	14.4%	15.1%	15.5%
$\frac{1121P}{1021P} \times 100\%$	37.6%	42.3%	66.1%	74.4%
$\frac{1221P}{1021P} \times 100\%$	9.8%	6.8%	10.7%	14.3%
$\frac{1321P}{1021P} \times 100\%$	8.1%	5.1%	6.6%	6.9%
$\frac{1120P}{1020P} \times 100\%$	32.6%	29.9%	29.5%	28.7%
$\frac{1220P}{1020P} \times 100\%$	14.7%	14.6%	16.4%	15.5%
$\frac{1320P}{1020P} \times 100\%$	11.9%	12.3%	13.6%	13.8%
$\frac{1111P}{1011P} \times 100\%$	98.6%	66.3%	72.3%	76.6%
$\frac{1211P}{1011P} \times 100\%$	53.2%	33.3%	45.8%	44.1%
$\frac{1311P}{1011P} \times 100\%$	48.9%	37.7%	45.4%	46.0%
$\frac{1110P}{1010P} \times 100\%$	52.4%	46.7%	49.2%	40.6%
$\frac{1210P}{1010P} \times 100\%$	32.2%	37.1%	41.2%	41.3%
$\frac{1310P}{1010P} \times 100\%$	44.2%	36.8%	44.1%	49.8%

TABLE 6.14 continued

Source of Expansion Ratio	Age of Measurement			
	14 days	2 months	6 months	12 months
$\frac{0131P}{0031P} \times 100\%$	79.3%	40.8%	71.8%	79.3%
$\frac{0231P}{0031P} \times 100\%$	60.6%	8.3%	10.5%	20.8%
$\frac{0331P}{0031P} \times 100\%$	49.7%	6.1%	5.3%	5.9%
$\frac{0130P}{0030P} \times 100\%$	31.0%	24.8%	28.0%	27.3%
$\frac{0230P}{0030P} \times 100\%$	16.3%	11.3%	12.6%	12.8%
$\frac{0330P}{0030P} \times 100\%$	14.8%	9.4%	10.3%	9.5%
$\frac{0121P}{0021P} \times 100\%$	107.8%	58.7%	100.0%	133.8%
$\frac{0221P}{0021P} \times 100\%$	75.0%	26.0%	29.3%	36.3%
$\frac{0321P}{0021P} \times 100\%$	135.9%	97.1%	116.2%	130.0%
$\frac{0120P}{0020P} \times 100\%$	60.6%	43.6%	54.1%	63.3%
$\frac{0220P}{0020P} \times 100\%$	75.8%	45.6%	34.9%	34.5%
$\frac{0320P}{0020P} \times 100\%$	77.8%	45.6%	45.9%	43.9%
$\frac{0111P}{0011P} \times 100\%$	118.3%	89.4%	96.7%	83.1%
$\frac{0211P}{0011P} \times 100\%$	112.7%	70.6%	85.7%	48.2%
$\frac{0311P}{0011P} \times 100\%$	108.5%	67.1%	86.8%	71.1%
$\frac{0110P}{0010P} \times 100\%$	119.6%	86.8%	80.0%	159.5%
$\frac{0210P}{0010P} \times 100\%$	107.1%	58.8%	78.8%	56.8%
$\frac{0310P}{0010P} \times 100\%$	76.8%	39.7%	38.8%	43.2%

```
PAGE  11     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  1-ST
DEPENDENT VARIABLE - EXPN1

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE       EFFECT          SUM OF      DEGREES OF       MEAN            F       TAIL
                                   SQUARES      FREEDOM         SQUARE                  PROB.

      MEAN                      29810646.00000     1    29810646.00000     960.76     0.
      CEMENT        A           14726965.75000     2     7363482.87500     237.32     0.
      PFA           B           13830909.41667     3     4610303.13889     148.58     0.
      AGGRGATE      C           17587286.25000     3     5862428.75000     188.94     0.
      FREEWCON      D               3601.50000     1        3601.50000       0.12     0.7373
      CP            AB           7356014.08333     6     1226002.34722      39.51     0.
      CA            AC           8576315.25000     6     1429385.87500      46.07     0.
      PA            BC           9908740.66667     9     1100971.18519      35.48     0.
      CF            AD             49015.75000     2       24507.87500       0.79     0.4690
      PF            BD            112360.41667     3       37453.47222       1.21     0.3356
      AF            CD              5524.75000     3        1841.58333       0.06     0.9804
      CPA           ABC          4267579.58333    18      237087.75463       7.64     0.0000
      CPF           ABD           247996.58333     6       41332.76389       1.33     0.2938
      CAF           ACD           146804.75000     6       24467.45833       0.79     0.5904
      PAF           BCD           169100.33333     9       18788.92593       0.61     0.7766
  1   ERROR                       558506.91667    18       31028.16204
```

TABLE 6.15 **ANOVA TABLE FOR SERIES 2 - 14 DAY EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL**

```
PAGE   12      ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  2-ND
DEPENDENT VARIABLE - EXPN2

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE      EFFECT          SUM OF      DEGREES OF      MEAN          F       TAIL
                                  SQUARES      FREEDOM       SQUARE                 PROB.

      MEAN                   58041930.37500      1     58041930.37500    1087.09   0.
      CEMENT        A        24836603.25000      2     12418301.62500     232.59   0.
      PFA           B        34737197.70833      3     11579065.90278     216.87   0.
      AGGRGATE      C        32479480.70833      3     10826493.56944     202.77   0.
      FREEWCON      D         2161200.16667      1      2161200.16667      40.48   0.0000
      CP            AB       14002278.91667      6      2333713.15278      43.71   0.
      CA            AC       11384729.41667      6      1897454.90278      35.54   0.0000
      PA            BC       21075653.37500      9      2341739.31944      43.86   0.
      CF            AD         895392.58333      2       447696.29167       8.39   0.0027
      PF            BD        2859195.58333      3       953065.19444      17.85   0.0000
      AF            CD        1335904.91667      3       445301.63889       8.34   0.0011
      CPA           ABC       7494882.75000     18       416382.37500       7.80   0.0000
      CPF           ABD       1490965.91667      6       248494.31944       4.65   0.0050
      CAF           ACD        561957.08333      6        93659.51389       1.75   0.1657
      PAF           BCD       2129863.33333      9       236651.48148       4.43   0.0035
  1   ERROR                    961053.41667     18        53391.85648
```

TABLE 6.16 ANOVA TABLE FOR SERIES 2 - 2 MONTH EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE  13     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  3-RD
DEPENDENT VARIABLE - EXPN3

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

        SOURCE        EFFECT            SUM OF     DEGREES OF        MEAN          F          TAIL
                                       SQUARES      FREEDOM         SQUARE                   PROB.

        MEAN                       73138196.34375      1     73138196.34375     1013.43     0.
        CEMENT          A          26559549.18750      2     13279774.59375      184.01     0.
        PFA             B          35999823.36458      3     11999941.12153      166.28     0.
        AGGRGATE        C          40076651.03125      3     13358883.67708      185.11     0.
        FREEWCON        D           4734372.51042      1      4734372.51042       65.60     0.0000
        CP              AB         12996523.47917      6      2166087.24653       30.01     0.0000
        CA              AC         11286771.56250      6      1881128.59375       26.07     0.0000
        PA              BC         22609453.76042      9      2512161.52894       34.81     0.
        CF              AD          1038167.14583      2       519083.57292        7.19     0.0051
        PF              BD          3957056.86458      3      1319018.95486       18.28     0.0000
        AF              CD          2995050.86453      3       998350.28819       13.83     0.0001
        CPA             ABC         7602630.77083     18       422368.37616        5.85     0.0002
        CPF             ABD         1202315.35417      6       200385.89236        2.78     0.0432
        CAF             ACD          965096.60417      6       160849.43403        2.23     0.0877
        PAF             BCD         2868146.92708      9       318682.99190        4.42     0.0036
  1     ERROR                       1299043.22917     18        72169.06829
```

TABLE 6.17   ANOVA TABLE FOR SERIES 2 - 6 MONTH EXPANSION
RESULTS,ORIGINAL EXPERIMENTAL MODEL

```
PAGE  14     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  4-TH
DEPENDENT VARIABLE - EXPN4

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

        SOURCE      EFFECT          SUM OF    DEGREES OF       MEAN          F       TAIL
                                    SQUARES    FREEDOM        SQUARE                 PROB.

        MEAN                    75710880.37500      1    75710880.37500    1024.95   0.
        CEMENT        A         27783643.75000      2    13891821.87500     188.06   0.
        PFA           B         34374940.70833      3    11458313.56944     155.12   0.
        AGGRGATE      C         43350486.87500      3    14450162.29167     195.62   0.
        FREEWCON      D          5880600.00000      1     5880600.00000      79.61   0.0000
        CP            AB        12923146.16667      6     2153857.69444      29.16   0.0000
        CA            AC        11765218.00000      6     1960869.66667      26.55   0.0000
        PA            BC        22054269.70833      9     2450474.41204      33.17   0.
        CF            AD         1127856.25000      2      563928.12500       7.63   0.0040
        PF            BD         3919656.08333      3     1306552.02778      17.69   0.0000
        AF            CD         3811106.25000      3     1270368.75000      17.20   0.0000
        CPA           ABC        7695761.41667     18      427542.30093       5.79   0.0003
        CPF           ABD        1158825.16667      6      193137.52778       2.61   0.0531
        CAF           ACD        1232082.00000      6      205347.00000       2.78   0.0430
        PAF           BCD        2603877.33333      9      289319.70370       3.92   0.0066
   1    ERROR                    1329627.91667     18       73868.21759

NUMBER OF INTEGER WORDS OF STORAGE USED IN PRECEDING    PROBLEM   13448
CPU TIME USED      54.618 SECONDS
```

TABLE 6.18 ANOVA TABLE FOR SERIES 2 - 12 MONTH EXPANSION RESULTS,ORIGINAL EXPERIMENTAL MODEL

TABLE 6.19 SUMMARY OF TAIL PROBABILITIES - SERIES 2, ORIGINAL EXPERIMENTAL MODEL

	Source of Variation	Age of Measurement			
		14 days	2 months	6 months	12 months
MAIN EFFECTS	Cement - A	0.0000	0.0000	0.0000	0.0000
	P.f.a. - B	0.0000	0.0000	0.0000	0.0000
	Aggregate - C	0.0000	0.0000	0.0000	0.0000
	Free Water - D	0.7373	0.0000	0.0000	0.0000
1st ORDER INTERACTIONS	AB	0.0000	0.0000	0.0000	0.0000
	AC	0.0000	0.0000	0.0000	0.0000
	BC	0.0000	0.0000	0.0000	0.0000
	AD	0.4690	0.0027	0.0051	0.0040
	BD	0.3356	0.0000	0.0000	0.0000
	CD	0.9804	0.0011	0.0001	0.0000
2nd ORDER INTERACTIONS	ABC	0.0000	0.0000	0.0002	0.0003
	ABD	0.2938	0.0050	0.0432	0.0531
	ACD	0.5904	0.1657	0.0877	0.0430
	BCD	0.7766	0.0035	0.0036	0.0066

```
PAGE  11      ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  1-ST
DEPENDENT VARIABLE - EXPN1

       SOURCE       EFFECT            SUM OF      DEGREES OF       MEAN             F        TAIL
                                      SQUARES      FREEDOM        SQUARE                     PROB.

       MEAN                       29810646.00000       1    29810646.00000     315.25    0.
       CEMENT         A           14726965.75000       2     7363482.87500      77.87    0.
       PFA            B           13830909.41667       3     4610303.13889      48.75    0.
       AGGRGATE       C           17587286.25000       3     5862428.75000      62.00    0.
       FREEWCON       D               3601.50000       1        3601.50000       0.04    0.8460
       CP             AB           7356014.08333       6     1226002.34722      12.97    0.
       CA             AC           8576315.25000       6     1429385.87500      15.12    0.
       PA             BC           9908740.66667       9     1100971.18519      11.64    0.
       CF             AD             49015.75000       2       24507.87500       0.26    0.7726
       PF             BD            112360.41667       3       37453.47222       0.40    0.7563
       AF             CD              5524.75000       3        1841.58333       0.02    0.9963
 1     ERROR                       5389988.16667      57       94561.19591
```

TABLE 6.20 ANOVA TABLE FOR SERIES 2 - 14 DAY EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL

```
PAGE  12     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  2-ND
DEPENDENT VARIABLE - EXPN2

        SOURCE        EFFECT             SUM OF       DEGREES OF        MEAN             F        TAIL
                                         SQUARES       FREEDOM          SQUARE                    PROB.

        MEAN                         58041930.37500       1    53041930.37500        261.77    0.
        CEMENT          A            24836603.25000       2    12418301.62500         56.01    0.
        PFA             B            34737197.70833       3    11579065.90278         52.22    0.
        AGGRGATE        C            32479480.70833       3    10826493.56944         48.83    0.
        FREEWCON        D             2161200.16667       1     2161200.16667          9.75    0.0028
        CP              AB           14002278.91667       6     2333713.15278         10.52    0.0000
        CA              AC           11384729.41667       6     1897454.90278          8.56    0.0000
        PA              BC           21075653.87500       9     2341739.31944         10.56    0.
        CF              AD             895392.58333       2      447696.29167          2.02    0.1422
        PF              BD            2859195.58333       3      953065.19444          4.30    0.0084
        AF              CD            1335904.91667       3      445301.63889          2.01    0.1230
 1      ERROR                        12638722.50000      57      221731.97368
```

TABLE 6.21 ANOVA TABLE FOR SERIES 2 - 2 MONTH EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL

```
PAGE  13     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  3-RD
DEPENDENT VARIABLE - EXPN3

       SOURCE       EFFECT             SUM OF      DEGREES OF        MEAN          F       TAIL
                                       SQUARES      FREEDOM         SQUARE                 PROB.

       MEAN                       73138196.34375       1     73138196.34375      299.12    0.
       CEMENT        A            26559549.18750       2     13279774.59375       54.31    0.
       PFA           B            35999823.36458       3     11999941.12153       49.08    0.
       AGGRGATE      C            40076651.03125       3     13358883.67708       54.63    0.
       FREEWCON      D             4734372.51042       1      4734372.51042       19.36    0.0000
       CP            AB           12996523.47917       6      2166087.24653        8.86    0.0000
       CA            AC           11286771.56250       6      1881128.59375        7.69    0.0000
       PA            BC           22609453.76042       9      2512161.52894       10.27    0.
       CF            AD            1038167.14583       2       519083.57292        2.12    0.1290
       PF            BD            3957056.86458       3      1319018.95486        5.39    0.0024
       AF            CD            2995050.86458       3       998350.28819        4.08    0.0107
  1    ERROR                      13937232.88542      57       244512.85764                  .
```

TABLE 6.22 ANOVA TABLE FOR SERIES 2 - 6 MONTH EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL

```
PAGE  14     ASTM RESULTS ANALYSIS,F-TESTS,MICROSTRAIN  EXPANSION SERIES 2,REPEAT.MEAS.
ANALYSIS OF VARIANCE FOR  4-TH
DEPENDENT VARIABLE - EXPN4

       SOURCE       EFFECT            SUM OF      DEGREES OF       MEAN            F        TAIL
                                     SQUARES      FREEDOM         SQUARE                    PROB.

       MEAN                    75710880.37500        1    75710880.37500      307.81     0.
       CEMENT        A         27783643.75000        2    13891821.87500       56.48     0.
       PFA           B         34374940.70833        3    11458313.56944       46.58     0.
       AGGRGATE      C         43350486.87500        3    14450162.29167       58.75     0.
       FREEWCON      D          5880600.00000        1     5880600.00000       23.91     0.0000
       CP            AB        12923146.16667        6     2153857.69444        8.76     0.0000
       CA            AC        11765218.00000        6     1960869.66667        7.97     0.0000
       PA            BC        22054269.70833        9     2450474.41204        9.96     0.0000
       CF            AD         1127856.25000        2      563928.12500        2.29     0.1102
       PF            BD         3919656.03333        3     1306552.02778        5.31     0.0027
       AF            CD         3811106.25000        3     1270368.75000        5.16     0.0032
  1    ERROR                   14020173.83333       57      245967.96199                    .

NUMBER OF INTEGER WORDS OF STORAGE USED IN PRECEDING    PROBLEM    8348
CPU TIME USED      44.955 SECONDS
```

TABLE 6.23 **ANOVA TABLE FOR SERIES 2 - 12 MONTH EXPANSION RESULTS,REVISED EXPERIMENTAL MODEL**

TABLE 6.24 SUMMARY OF TAIL PROBABILITIES - SERIES 2, REVISED EXPERIMENTAL MODEL

	Source of Variation	Age of Measurement			
		14 days	2 months	6 months	12 months
MAIN EFFECTS	Cement - A	0.0000	0.0000	0.0000	0.0000
	P.f.a. - B	0.0000	0.0000	0.0000	0.0000
	Aggregate - C	0.0000	0.0000	0.0000	0.0000
	Free Water - D	0.8460	0.0028	0.0000	0.0000
1st ORDER INTERACTIONS	AB	0.0000	0.0000	0.0000	0.0000
	AC	0.0000	0.0000	0.0000	0.0000
	BC	0.0000	0.0000	0.0000	0.0000
	AD	0.7726	0.1422	0.1290	0.1102
	BD	0.7563	0.0084	0.0024	0.0027
	CD	0.9963	0.1230	0.0107	0.0032

TABLE 6.25 EXPANSION OF MIXES INCORPORATING P.F.A. EXPRESSED AS A PERCENTAGE OF THE EXPANSION OF THE CORRESPONDING NON-P.F.A. MIX - BELTANE OPAL MIXES

Source of Expansion Ratio	Age of Measurement			
	14 days	2 months	6 months	12 months
$\frac{2311B}{2011B} \times 100\%$	127.3%	171.2%	159.8%	153.5%
$\frac{2321B}{2021B} \times 100\%$	151.2%	170.3%	156.0%	137.3%
$\frac{2331B}{2031B} \times 100\%$	133.3%	156.6%	141.0%	130.3%
$\frac{2341B}{2041B} \times 100\%$	46.8%	44.4%	47.4%	50.8%
$\frac{2351B}{2051B} \times 100\%$	84.0%	118.0%	123.5%	124.0%
$\frac{2361B}{2061B} \times 100\%$	97.3%	147.4%	150.3%	160.3%
$\frac{2371B}{2071B} \times 100\%$	84.4%	159.3%	164.7%	143.6%

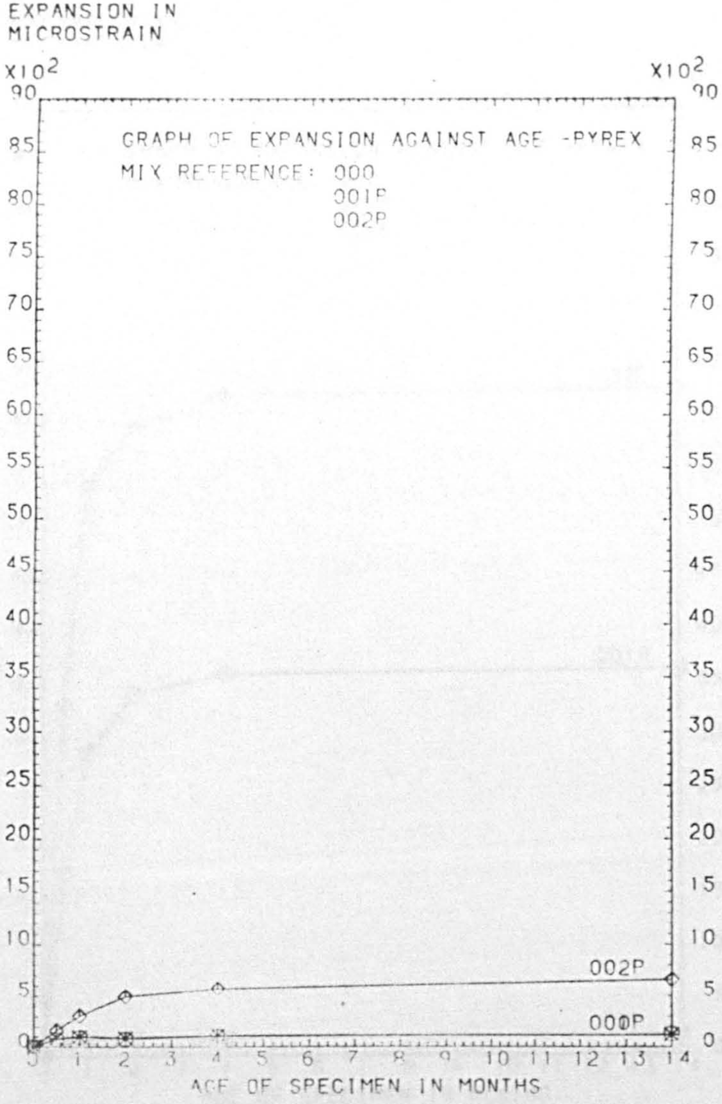


Figure 6.1

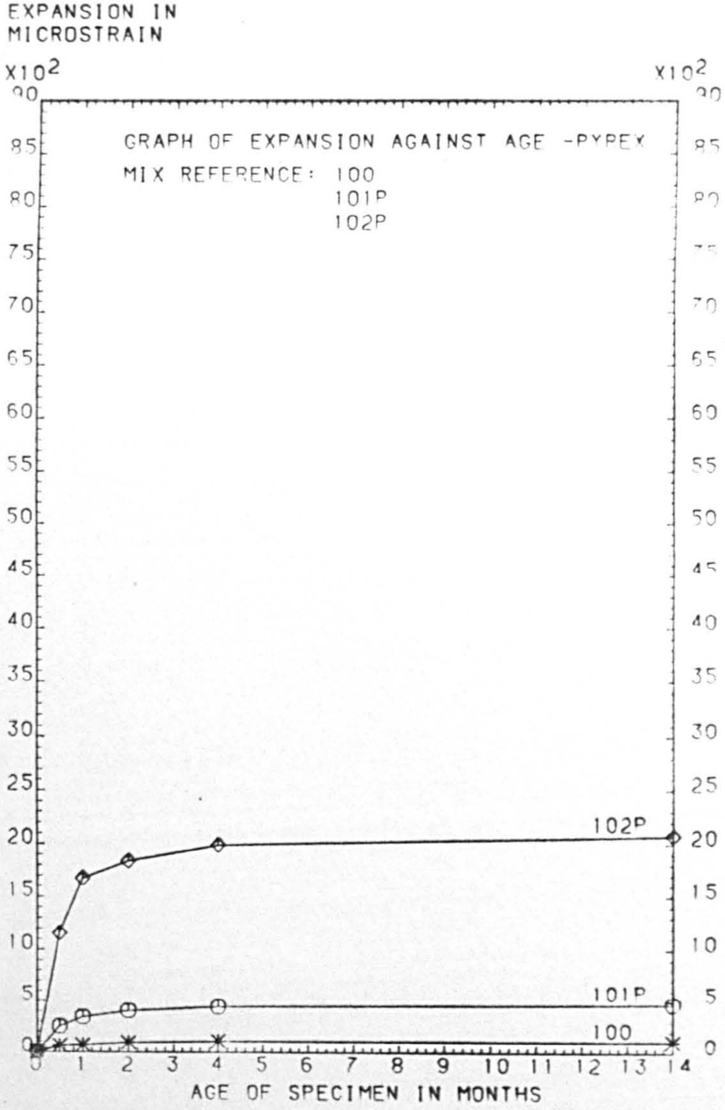


Figure 6.2

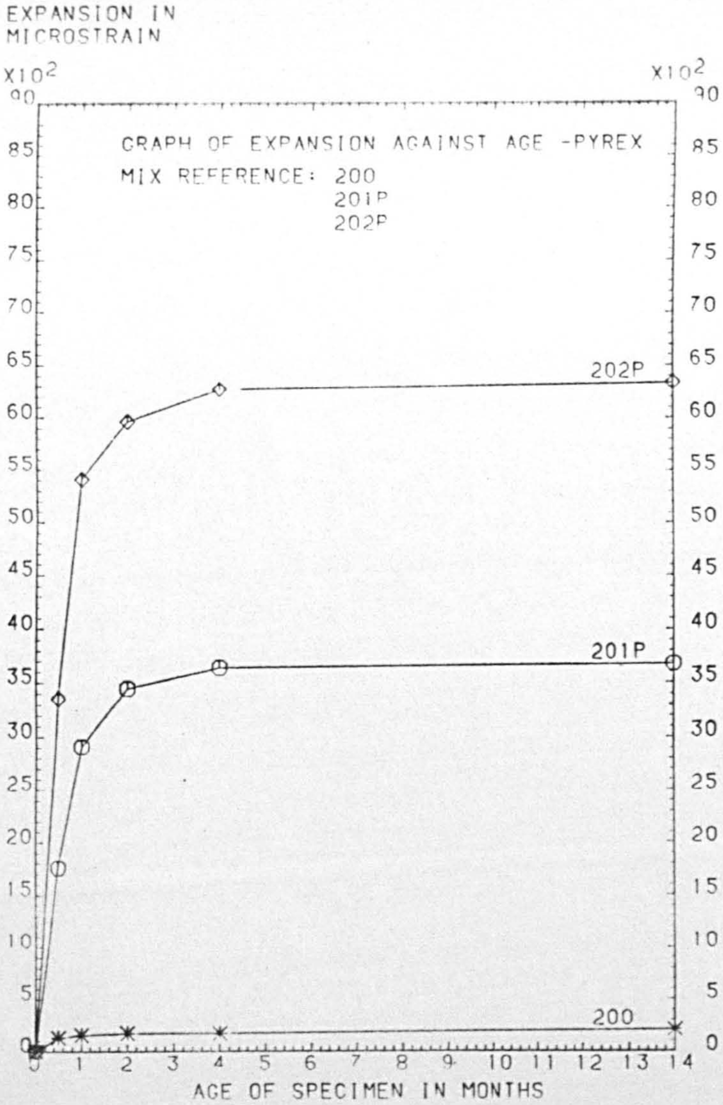

EXPANSION IN MICROSTRAIN
X10^2
X10^2
GRAPH OF EXPANSION AGAINST AGE -PYREX
MIX REFERENCE: 200
201P
202P
202P
201P
200
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
AGE OF SPECIMEN IN MONTHS

Figure 6.3

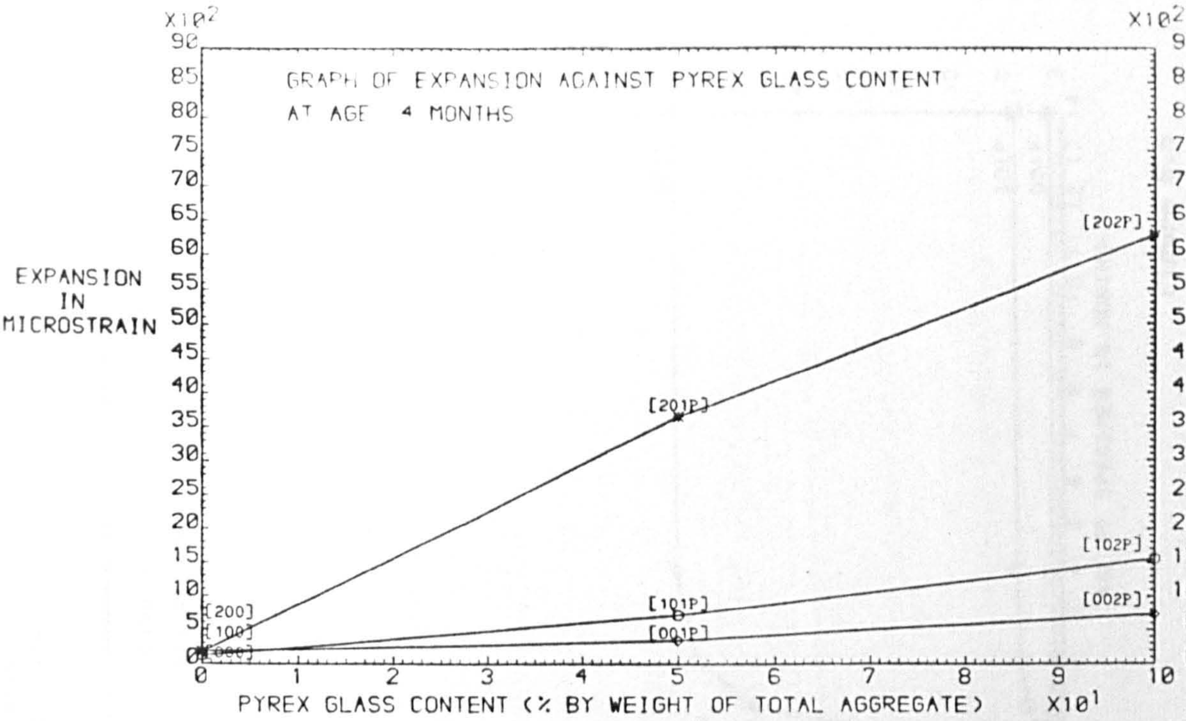

GRAPH OF EXPANSION AGAINST PYREX GLASS CONTENT
AT AGE 4 MONTHS
EXPANSION IN MICROSTRAIN
X10^2
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
[200]
[100]
[000]
[201P]
[101P]
[001P]
[202P]
[102P]
[002P]
0
1
2
3
4
5
6
7
8
9
10
PYREX GLASS CONTENT (% BY WEIGHT OF TOTAL AGGREGATE)
X10^1

Figure 6.4

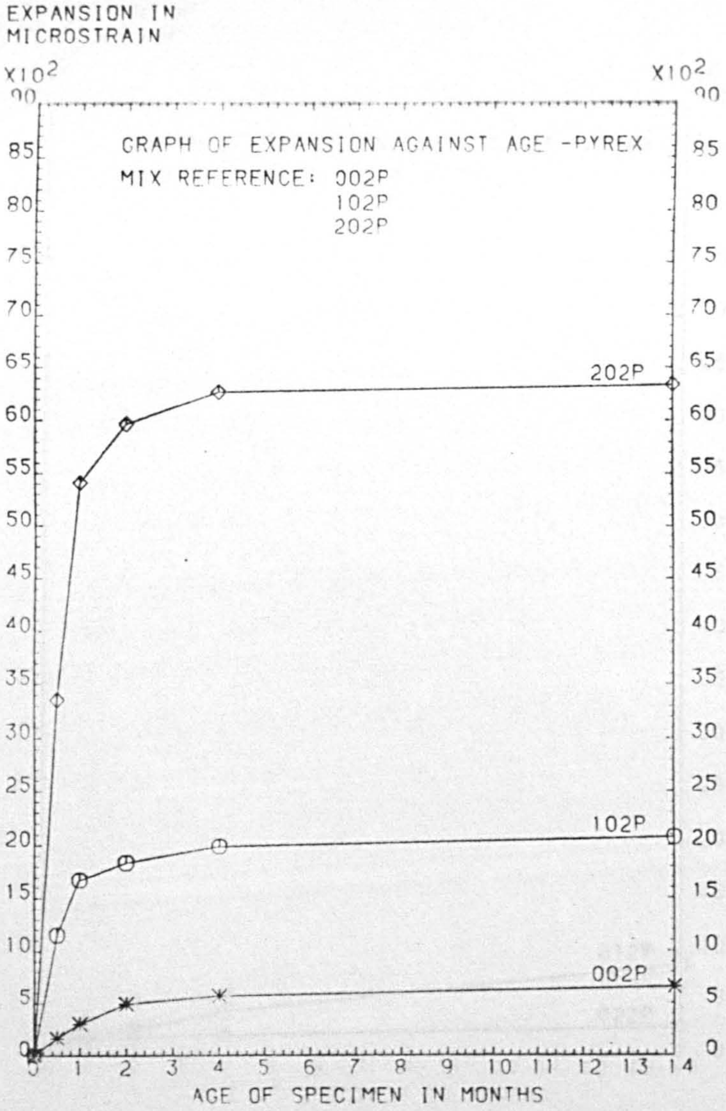


Figure 6.5

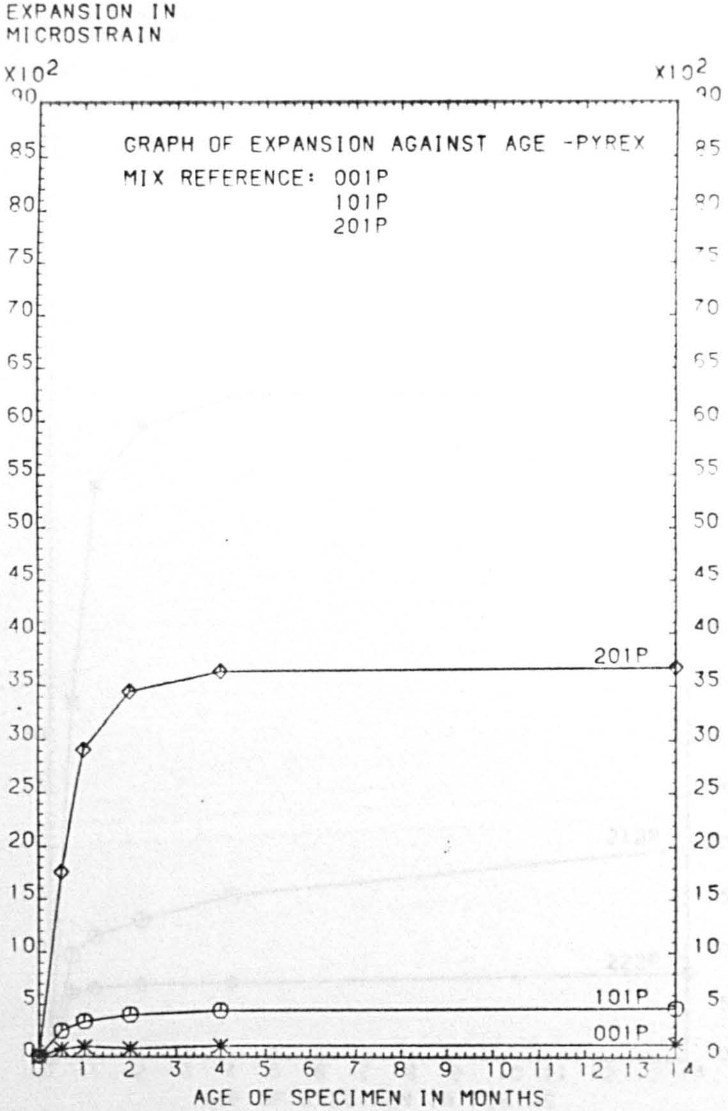


Figure 6.6

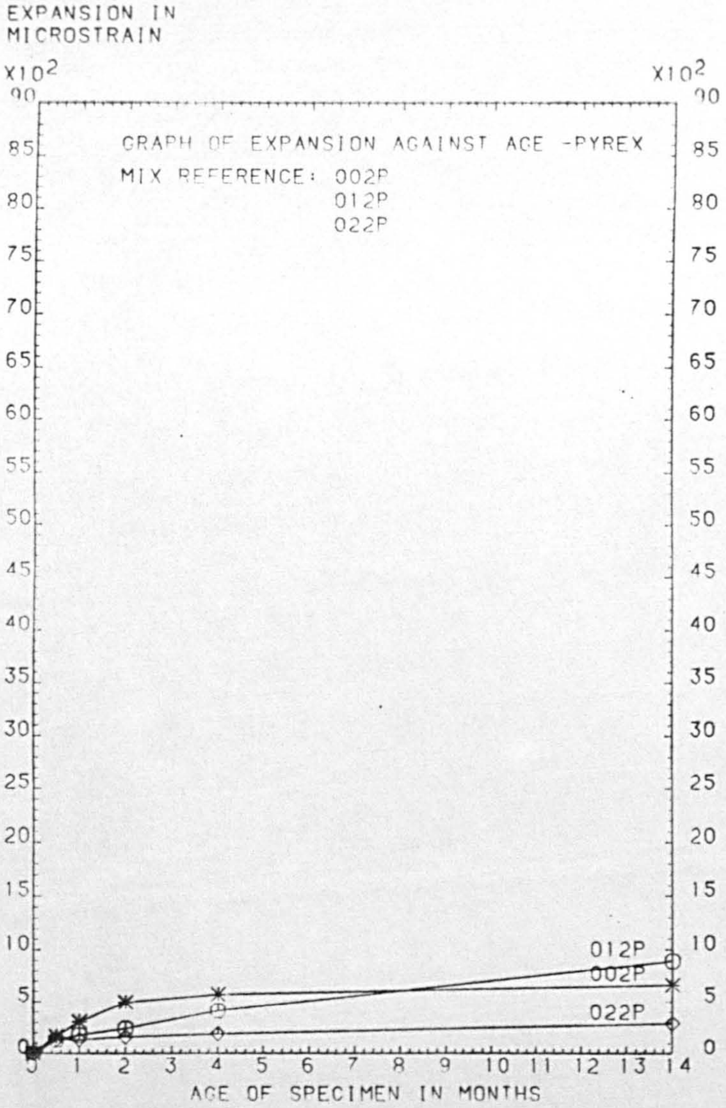


Figure 6.7

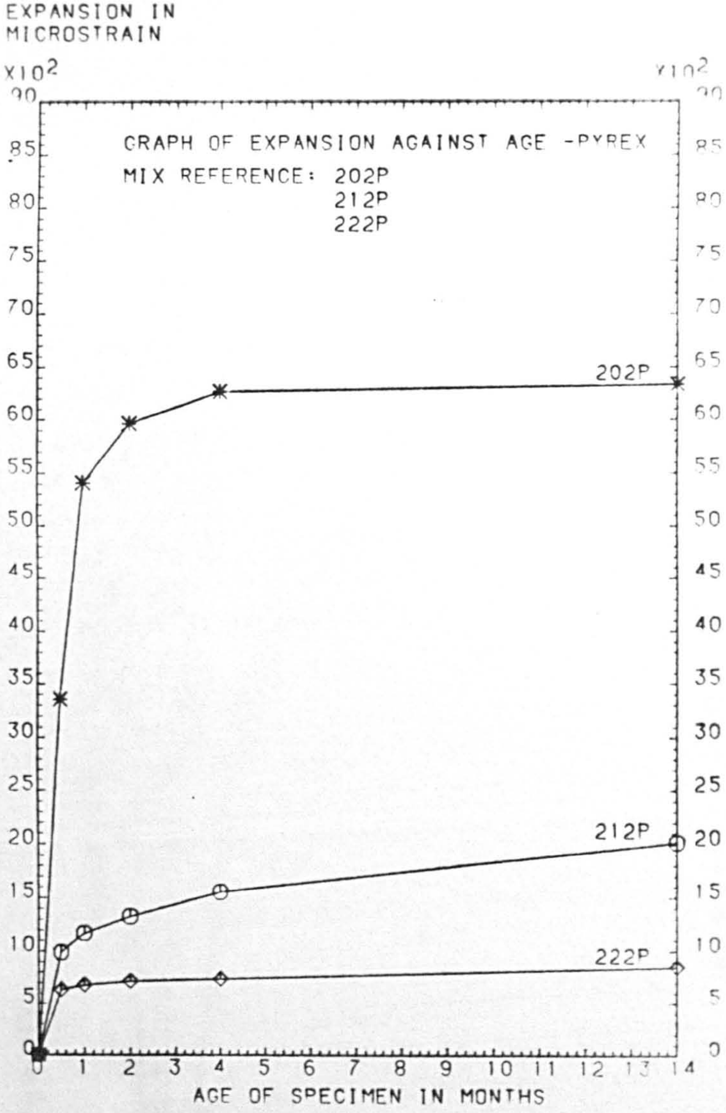


Figure 6.8

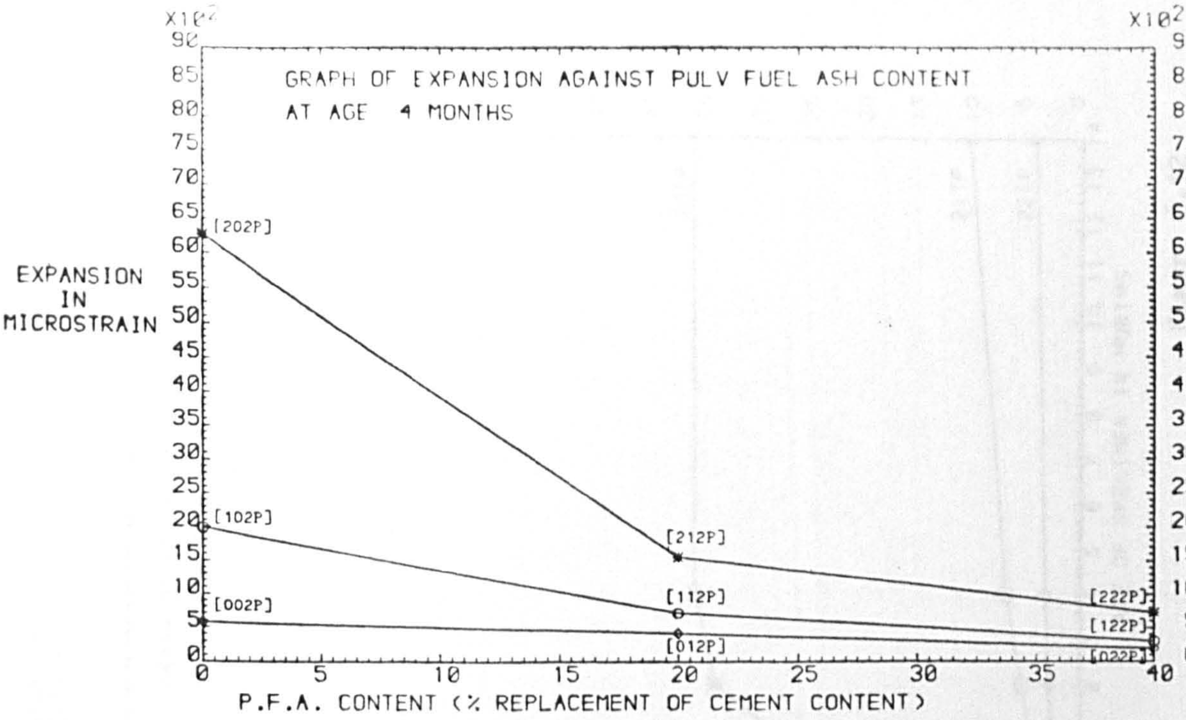


Figure 6.9

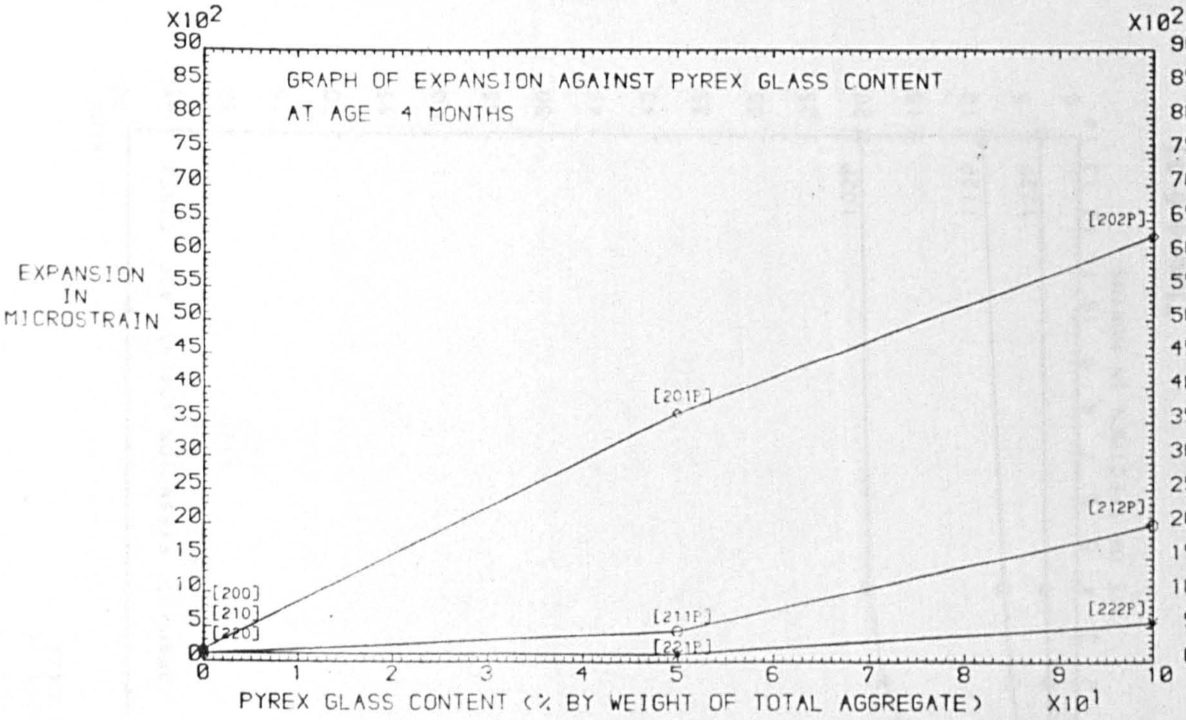


Figure 6.10

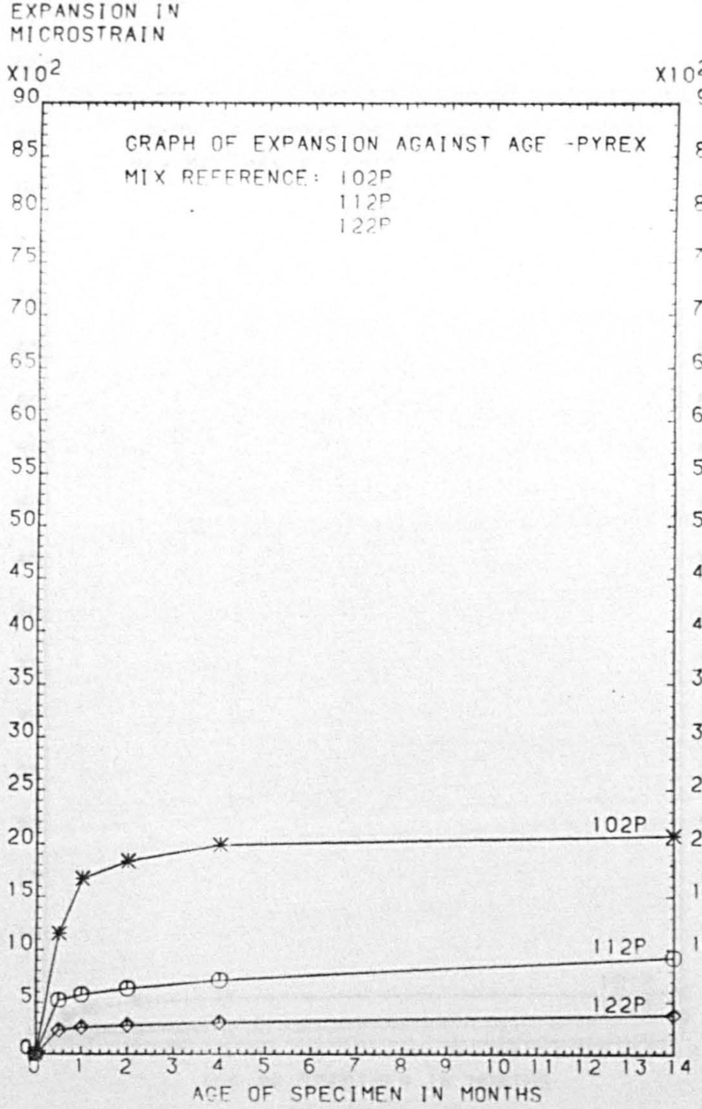


Figure 6.11

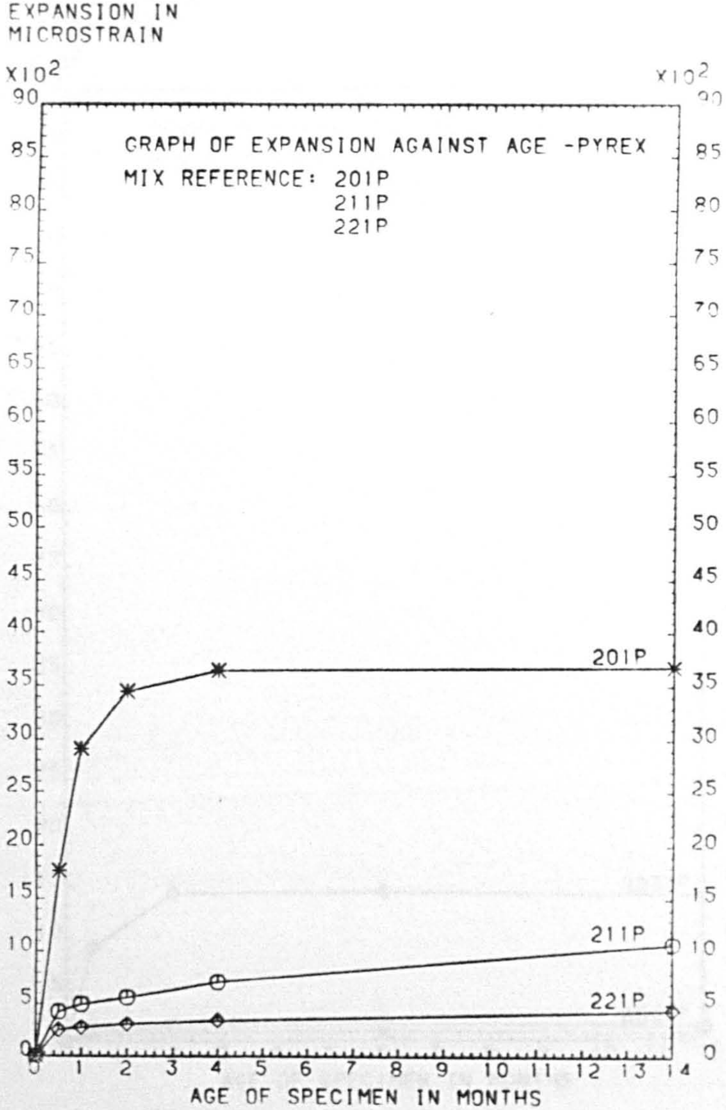


Figure 6.12

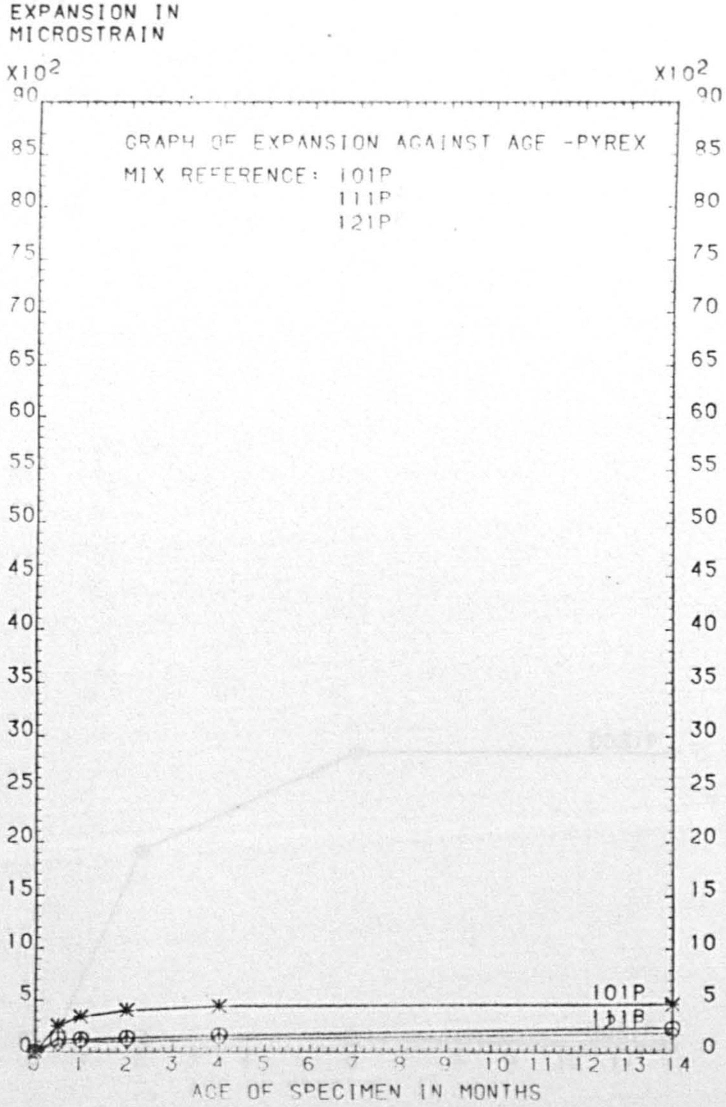


Figure 6.13

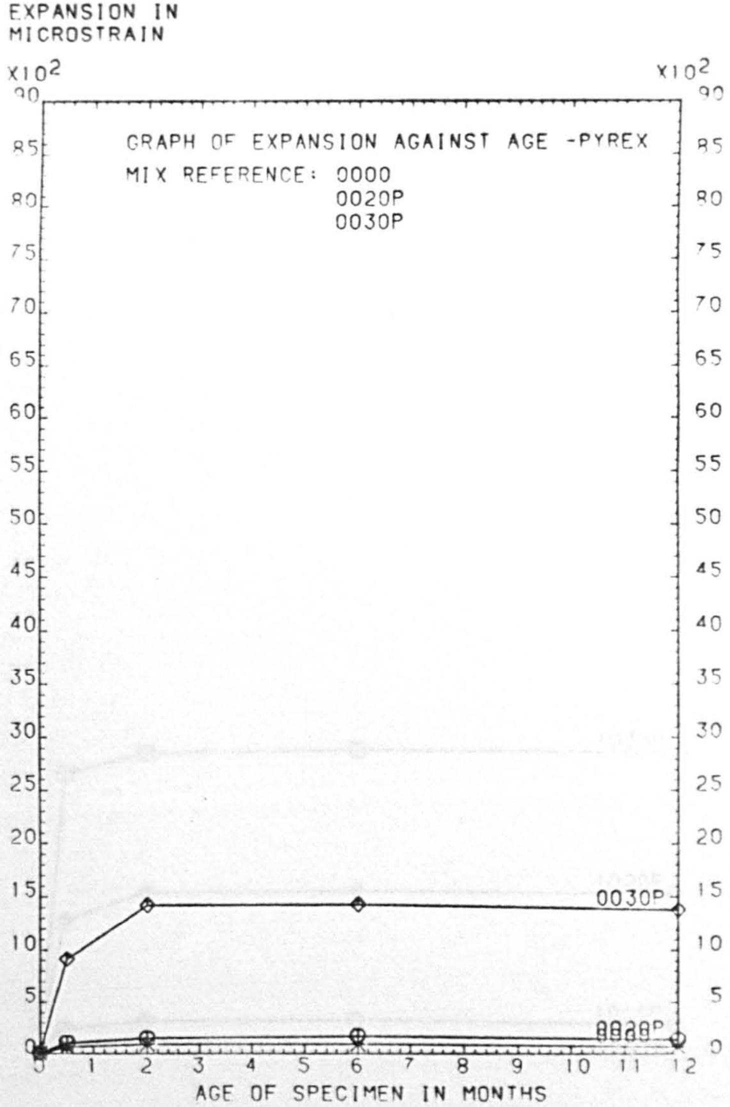


Figure 6.14

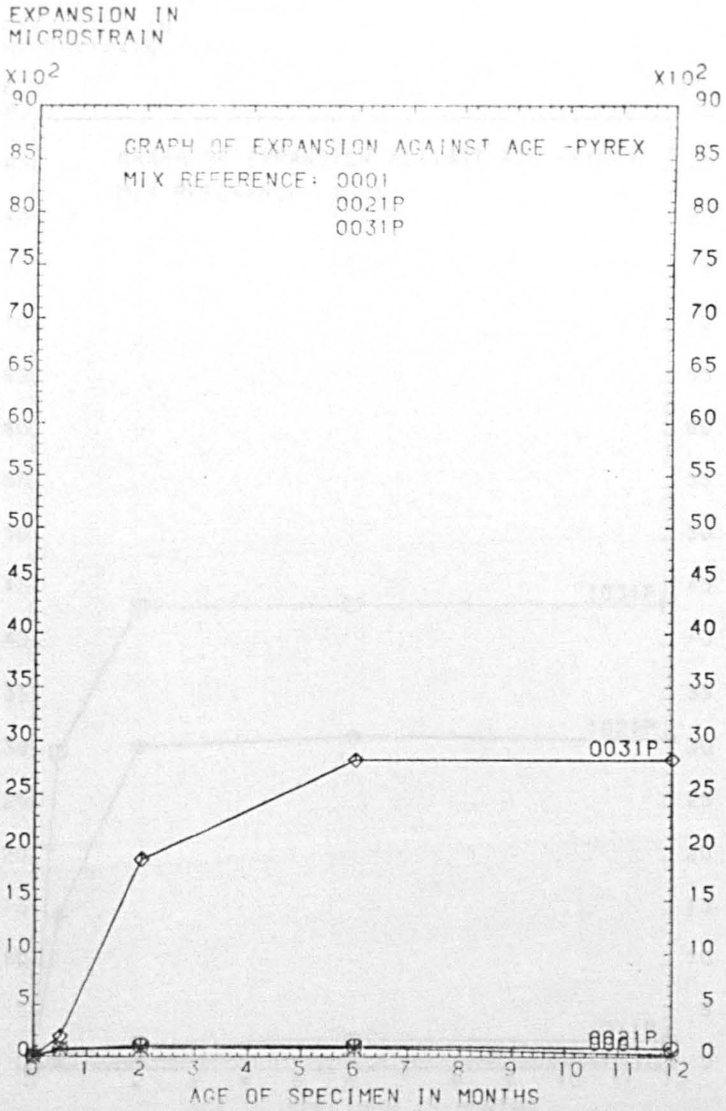


Figure 6.15

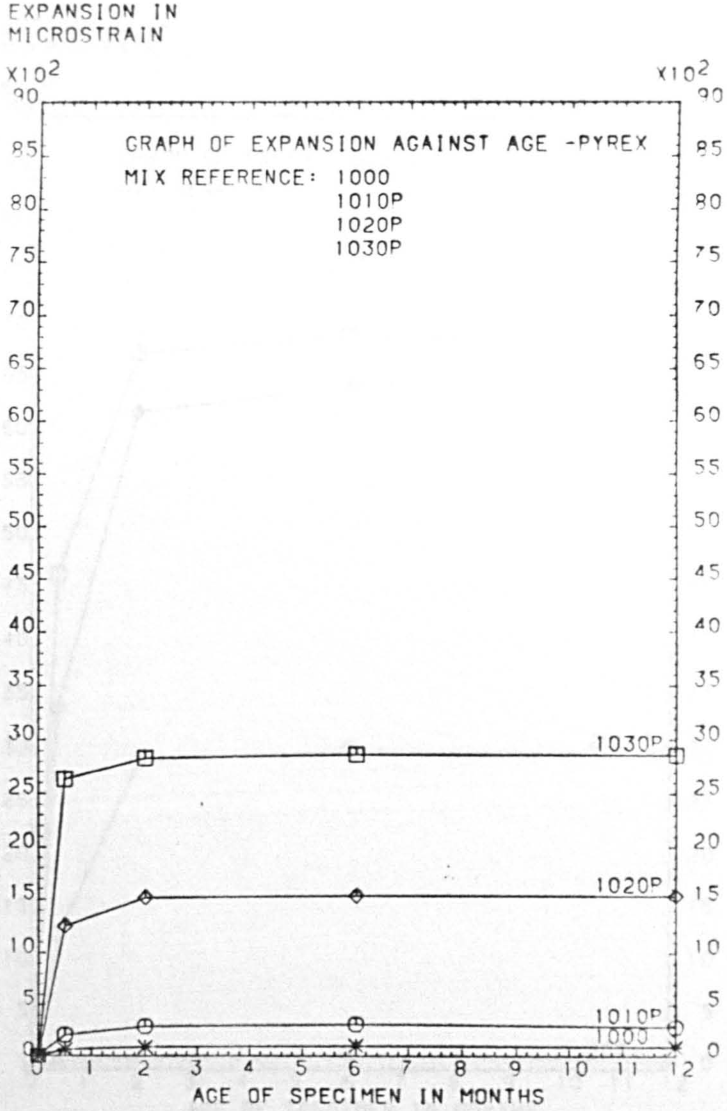


Figure 6.16

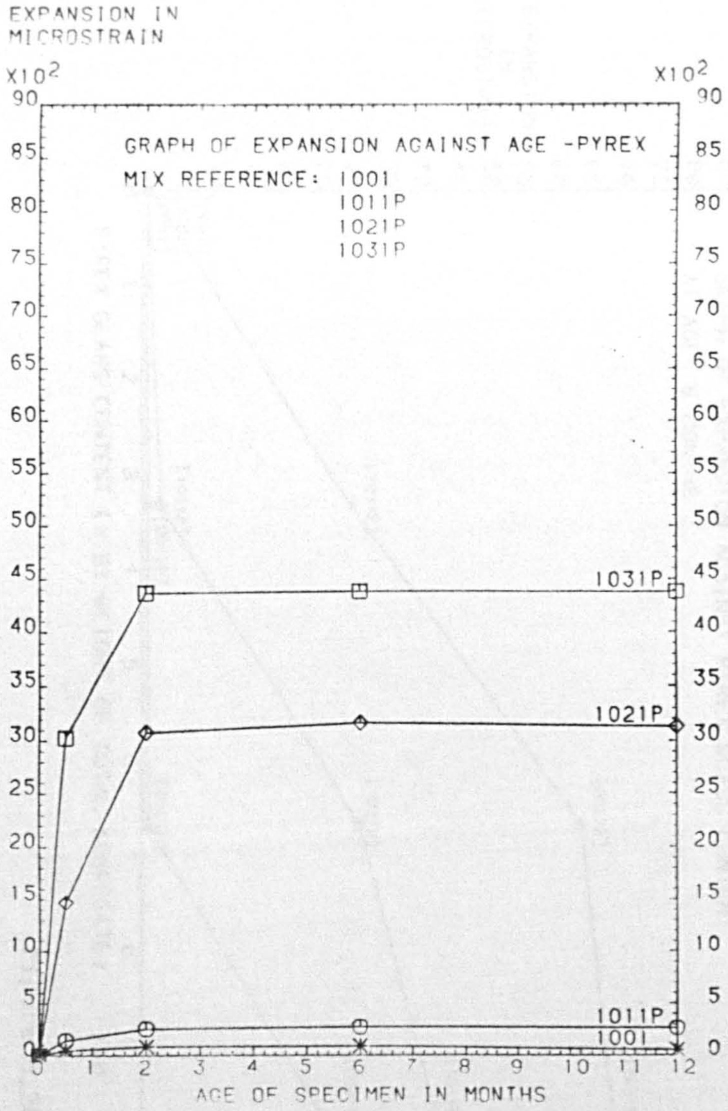


Figure 6.17

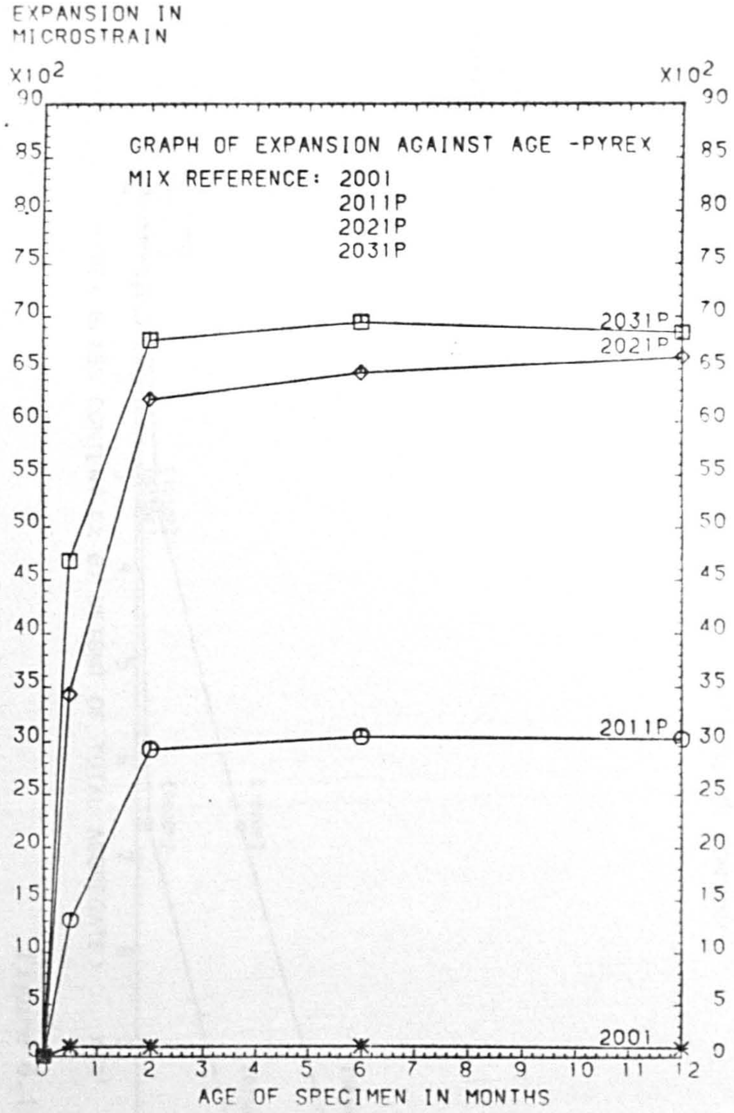


Figure 6.18

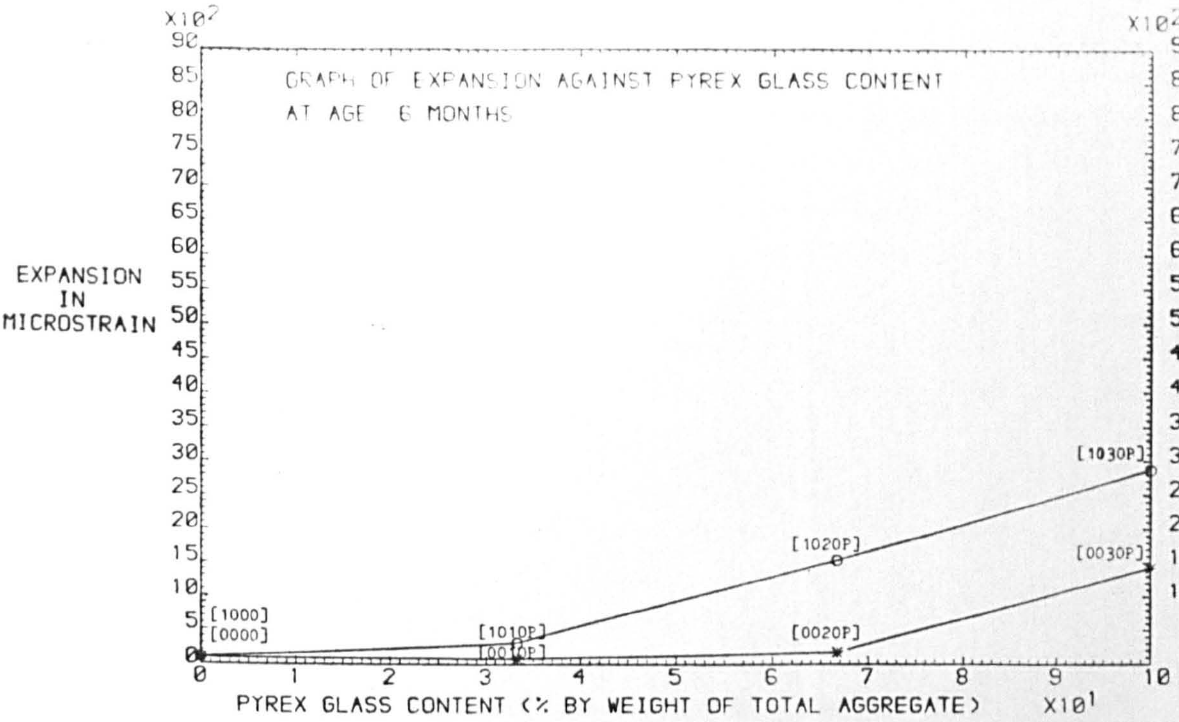


Figure 6.19

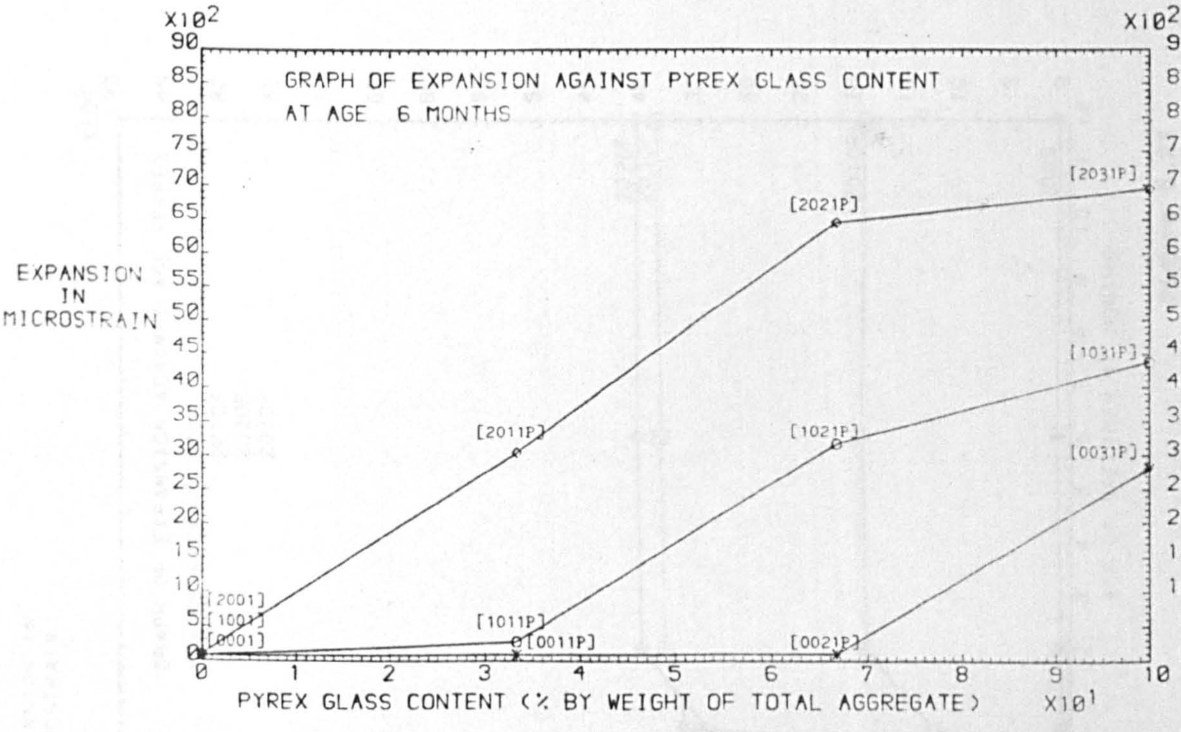
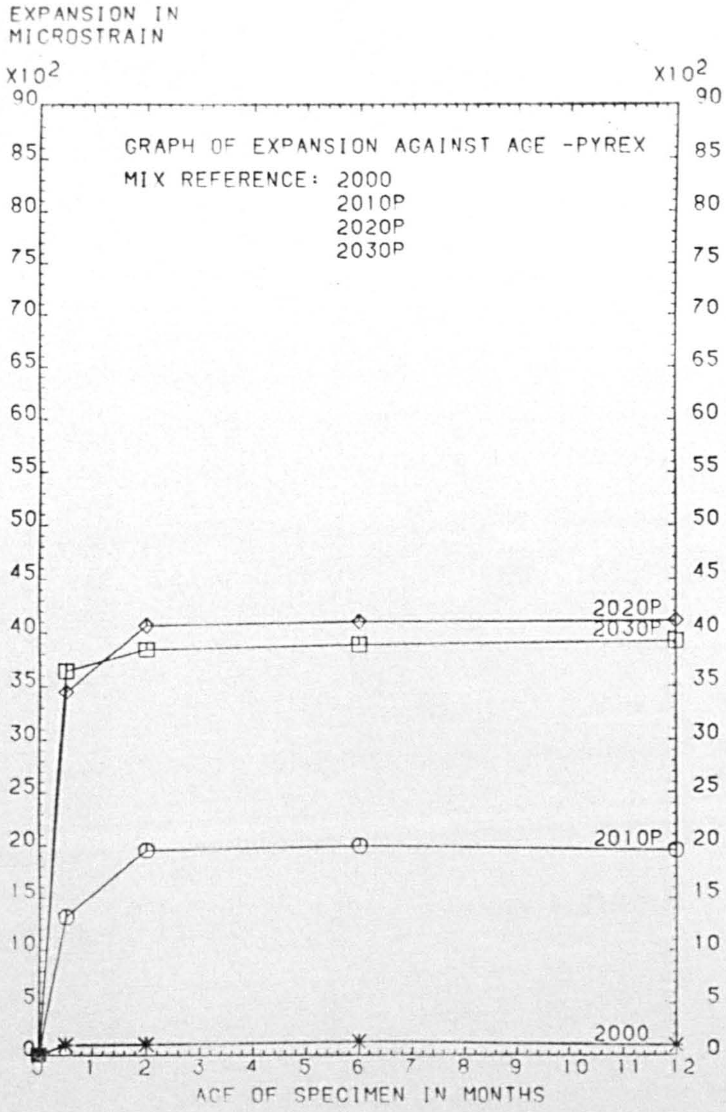


Figure 6.20


EXPANSION IN MICROSTRAIN
X10^2
X10^2
GRAPH OF EXPANSION AGAINST AGE -PYREX
MIX REFERENCE: 2000
2010P
2020P
2030P
2020P
2030P
2010P
2000
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
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11
12
AGE OF SPECIMEN IN MONTHS

Figure 6.21

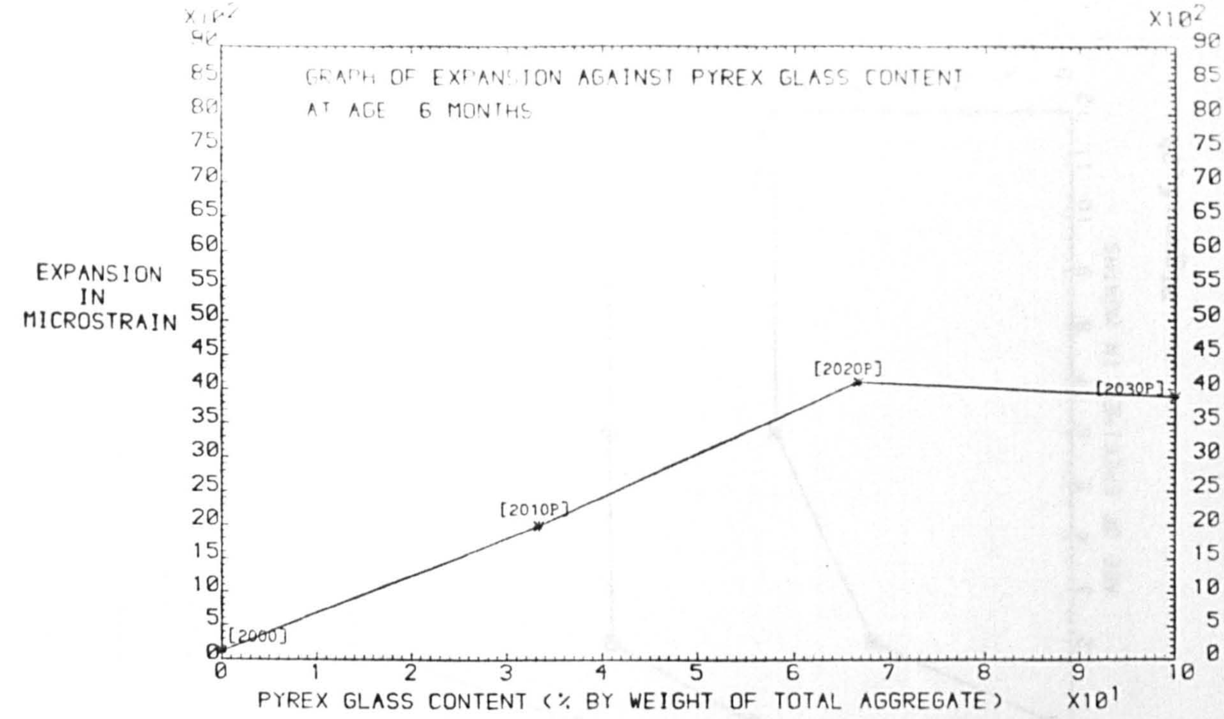

GRAPH OF EXPANSION AGAINST PYREX GLASS CONTENT
AT AGE 6 MONTHS
EXPANSION IN MICROSTRAIN
X10²
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
[2000]
[2010P]
[2020P]
[2030P]
0
1
2
3
4
5
6
7
8
9
10
PYREX GLASS CONTENT (% BY WEIGHT OF TOTAL AGGREGATE)
X10¹

Figure 6.22

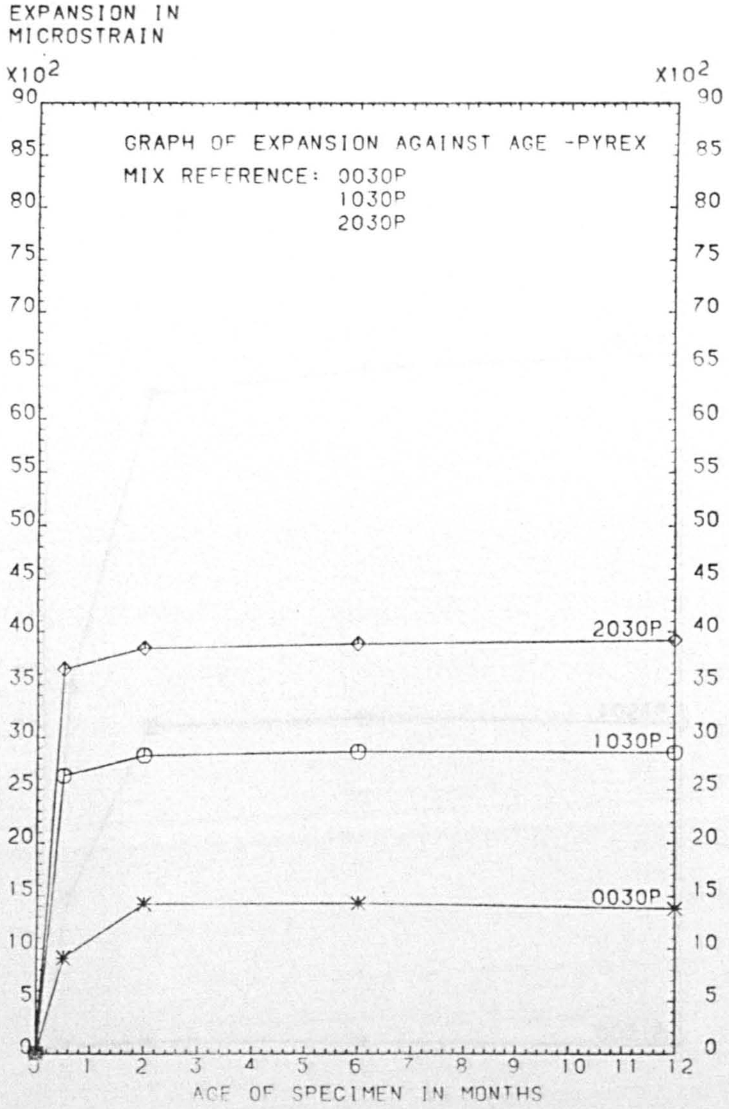


Figure 6.23

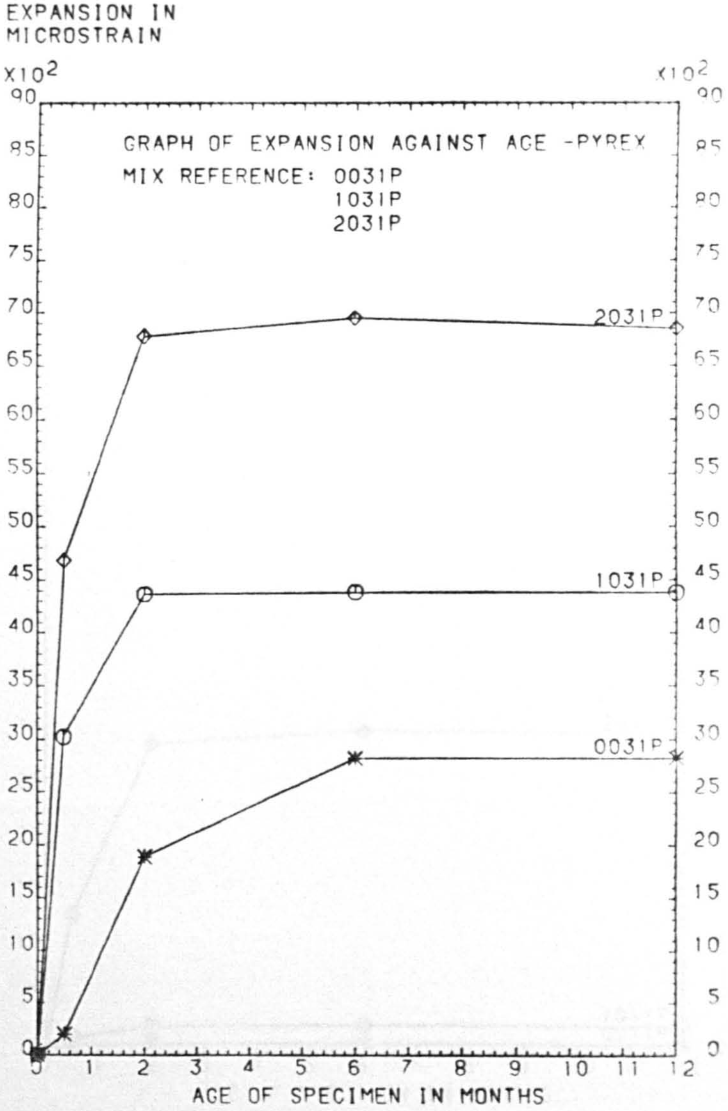


Figure 6.24

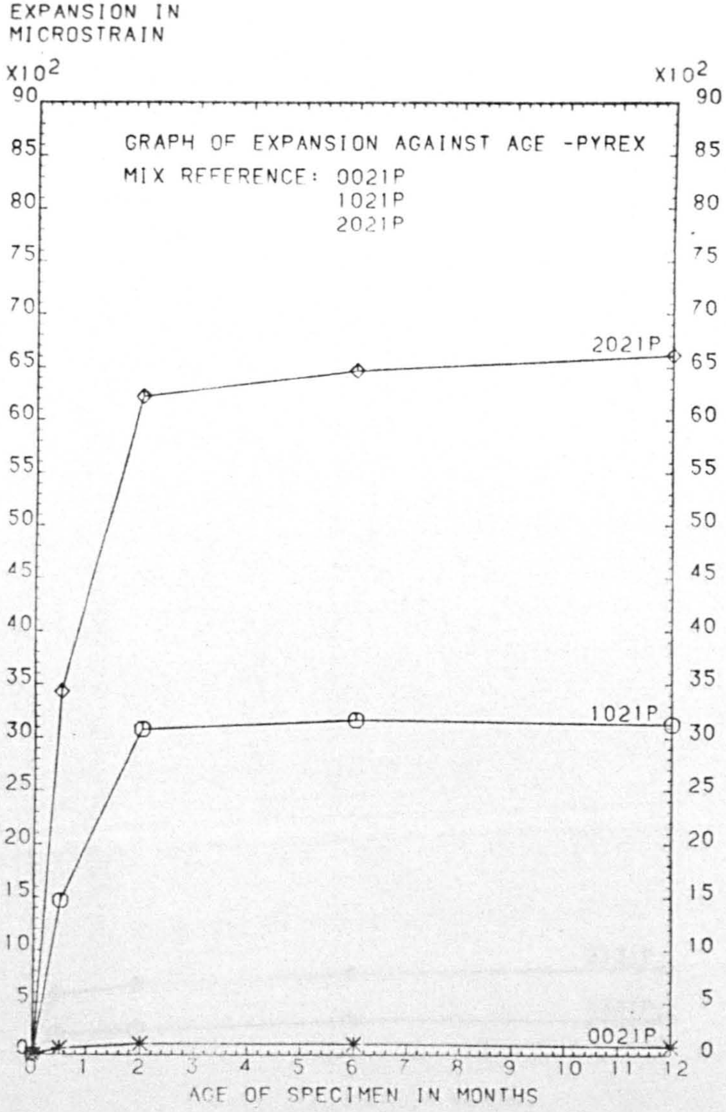


Figure 6.25

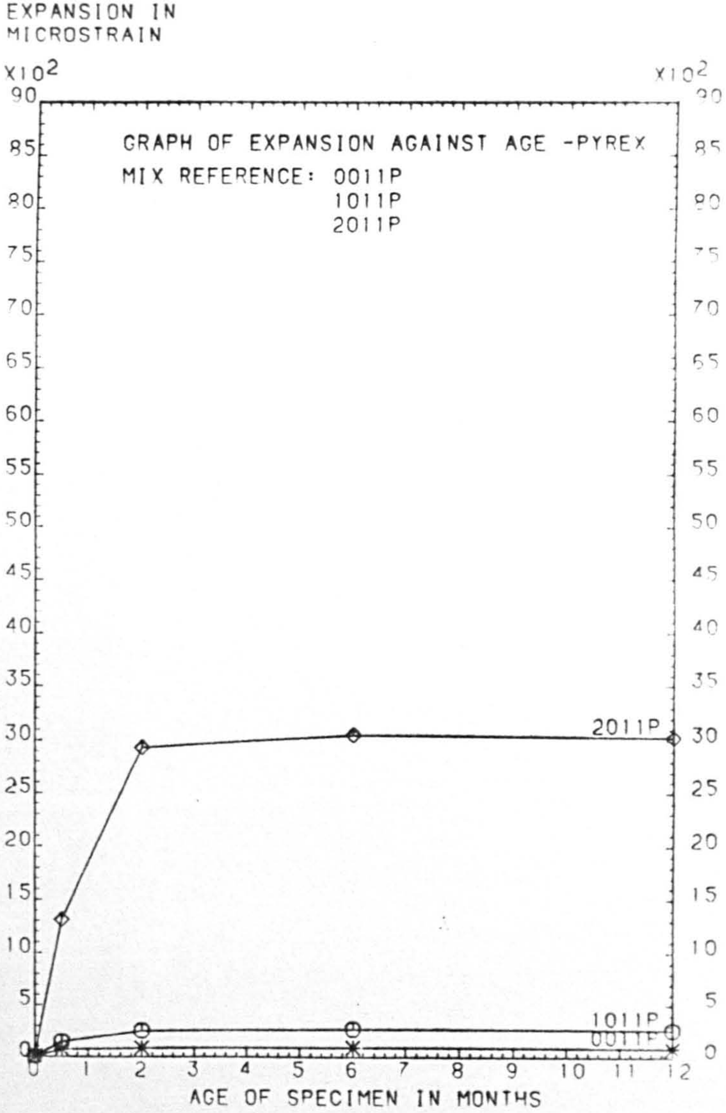
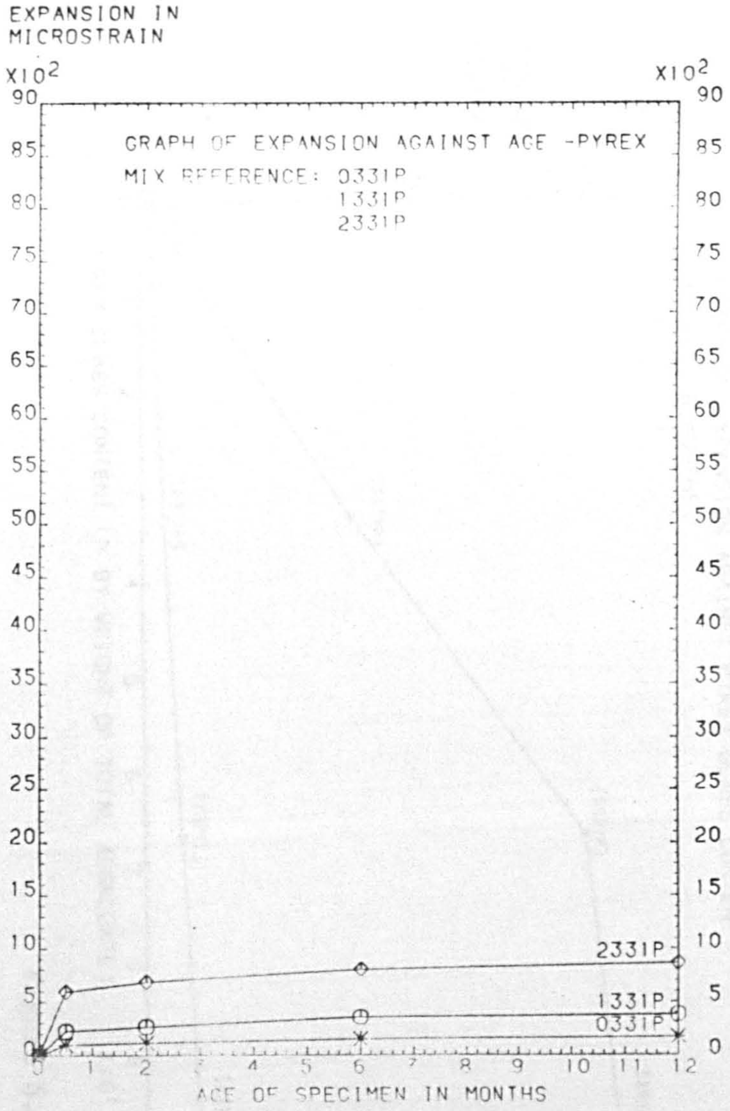


Figure 6.26


EXPANSION IN MICROSTRAIN
X10^2
X10^2
GRAPH OF EXPANSION AGAINST AGE -PYREX
MIX REFERENCE: 0331P
1331P
2331P
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
2331P
1331P
0331P
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11
12
AGE OF SPECIMEN IN MONTHS

Figure 6.27

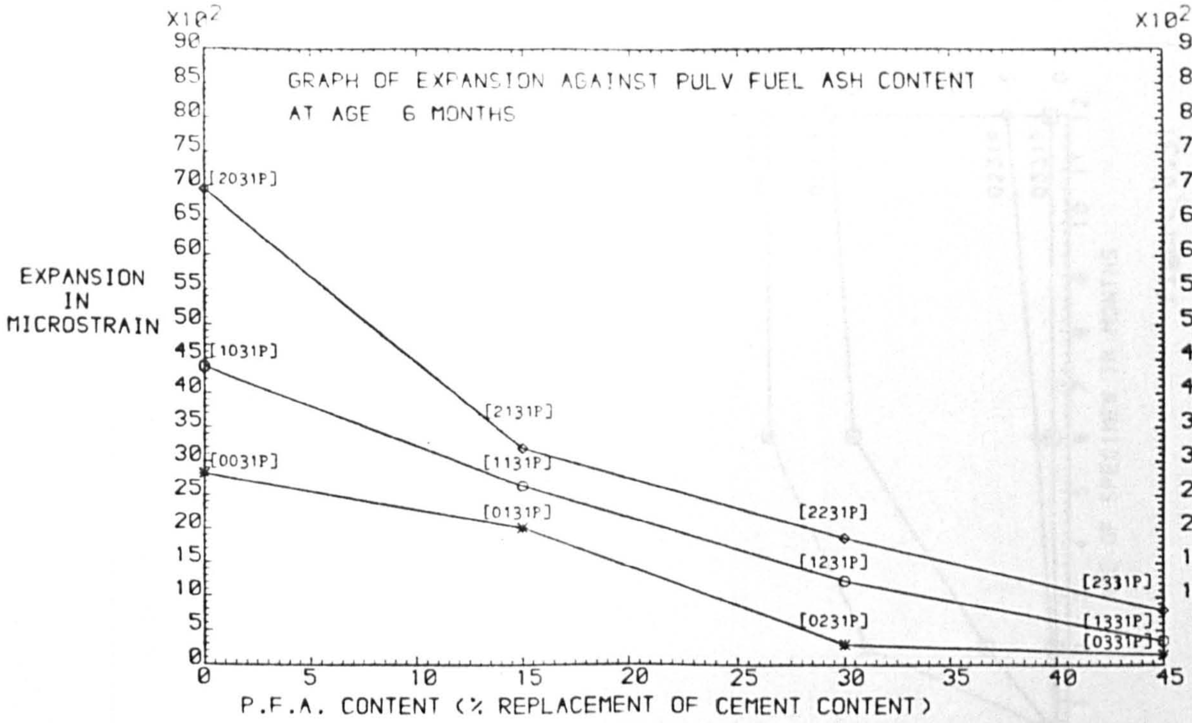
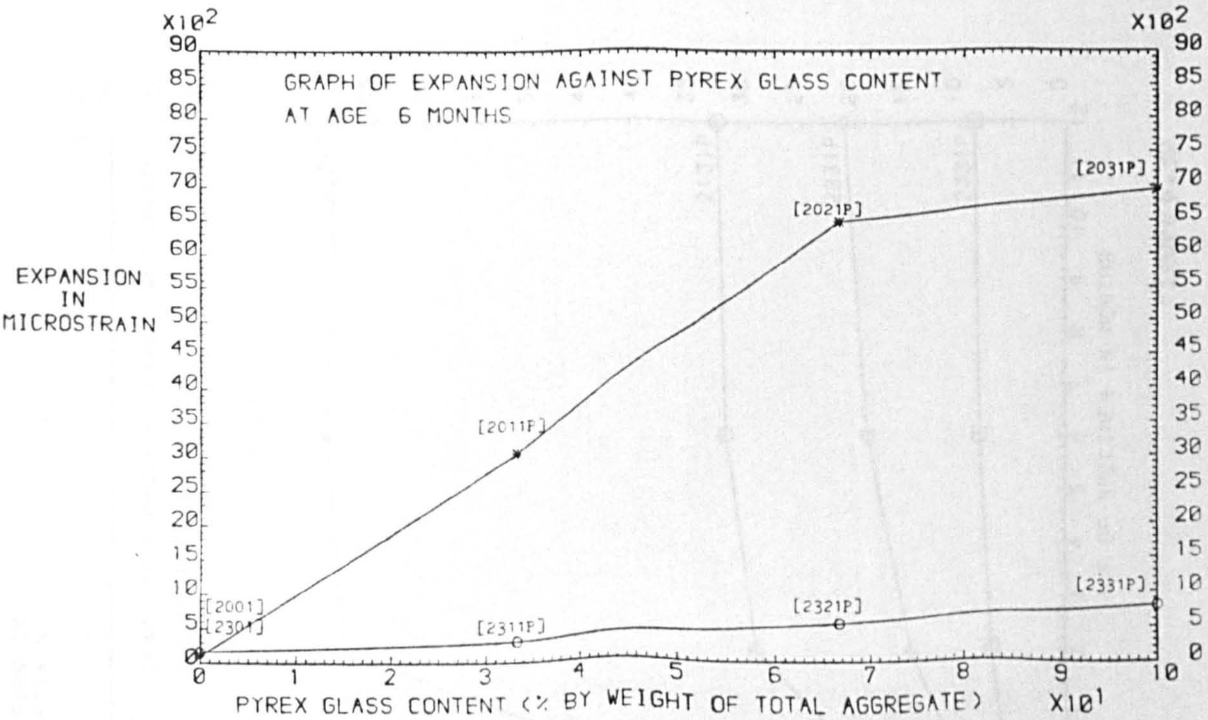


Figure 6.28



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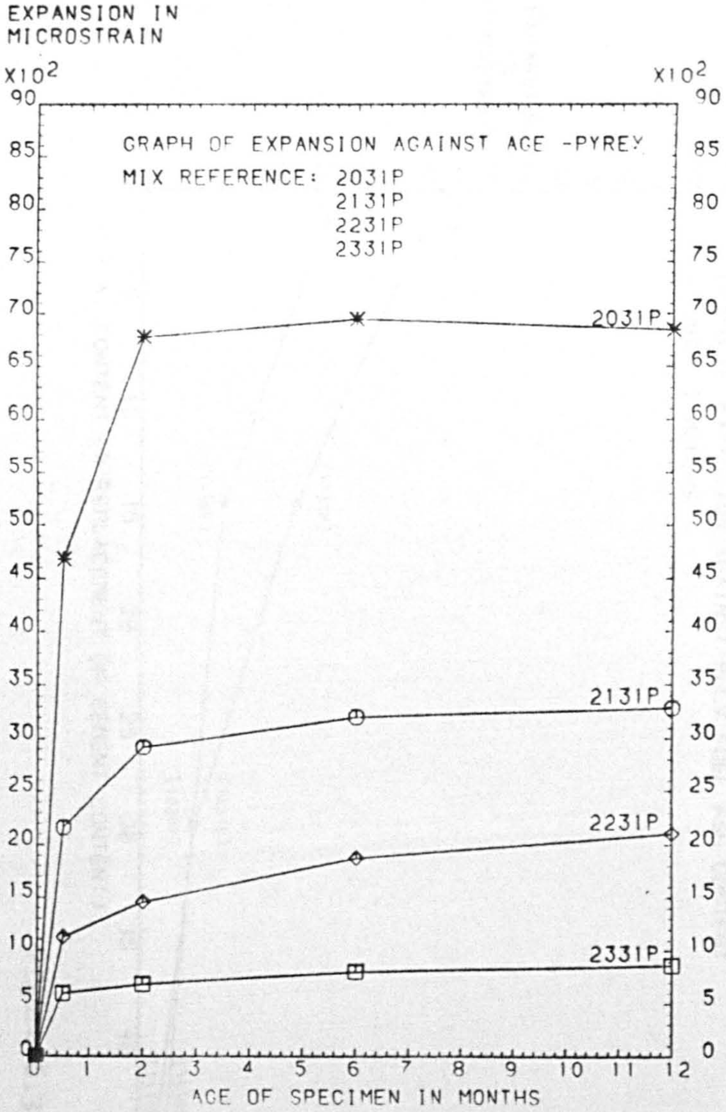


Figure 6.30

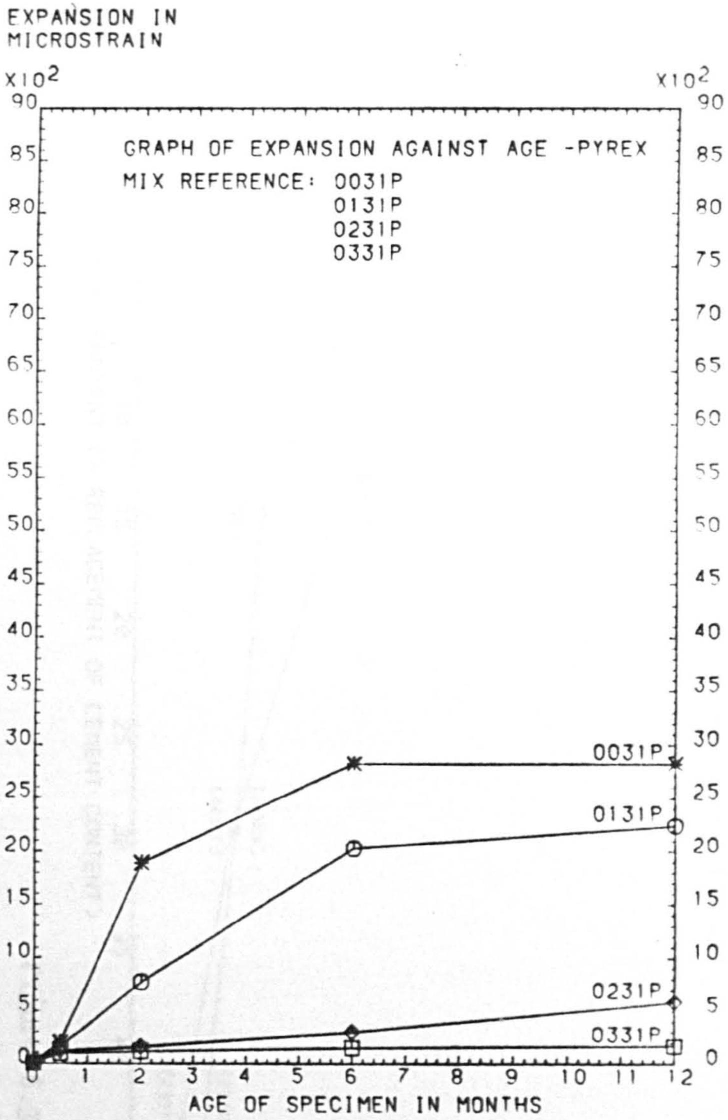


Figure 6.31

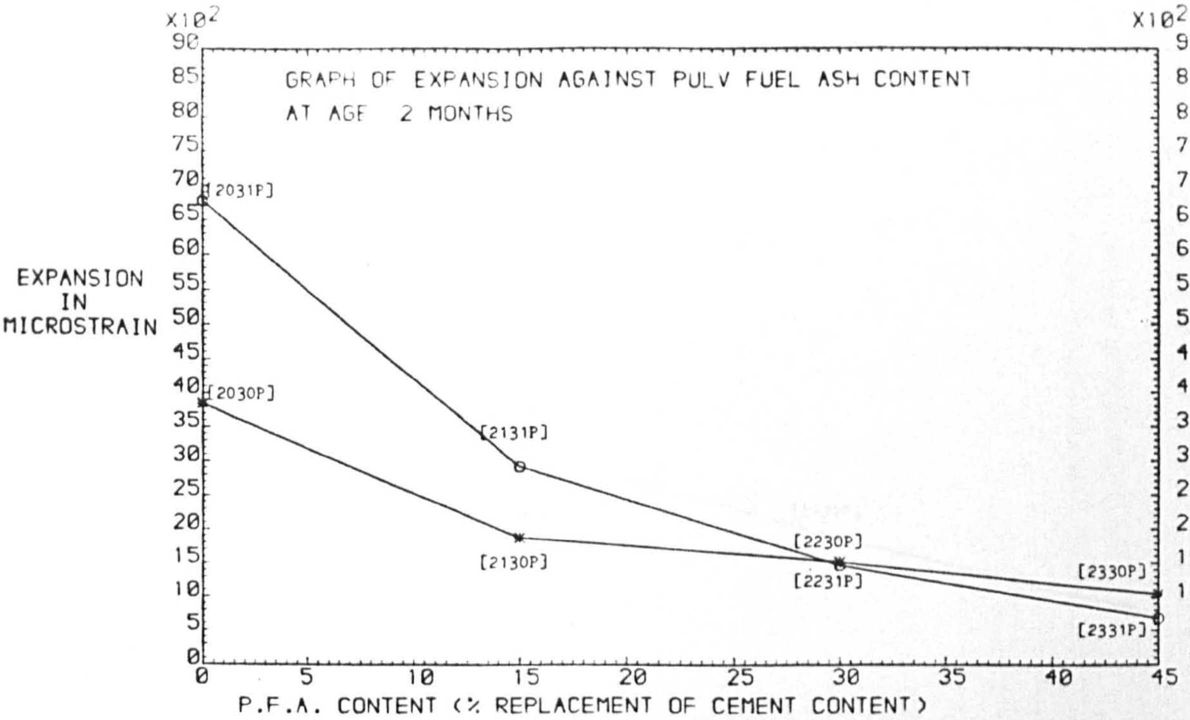


Figure 6.32

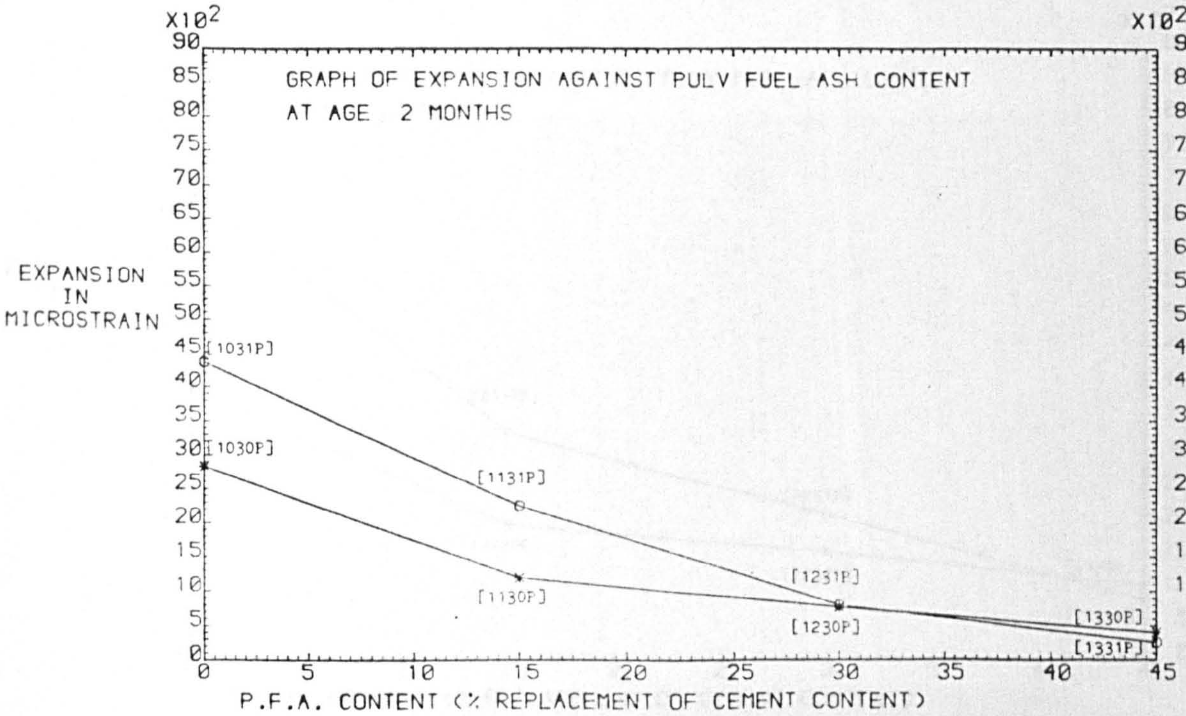


Figure 6.33

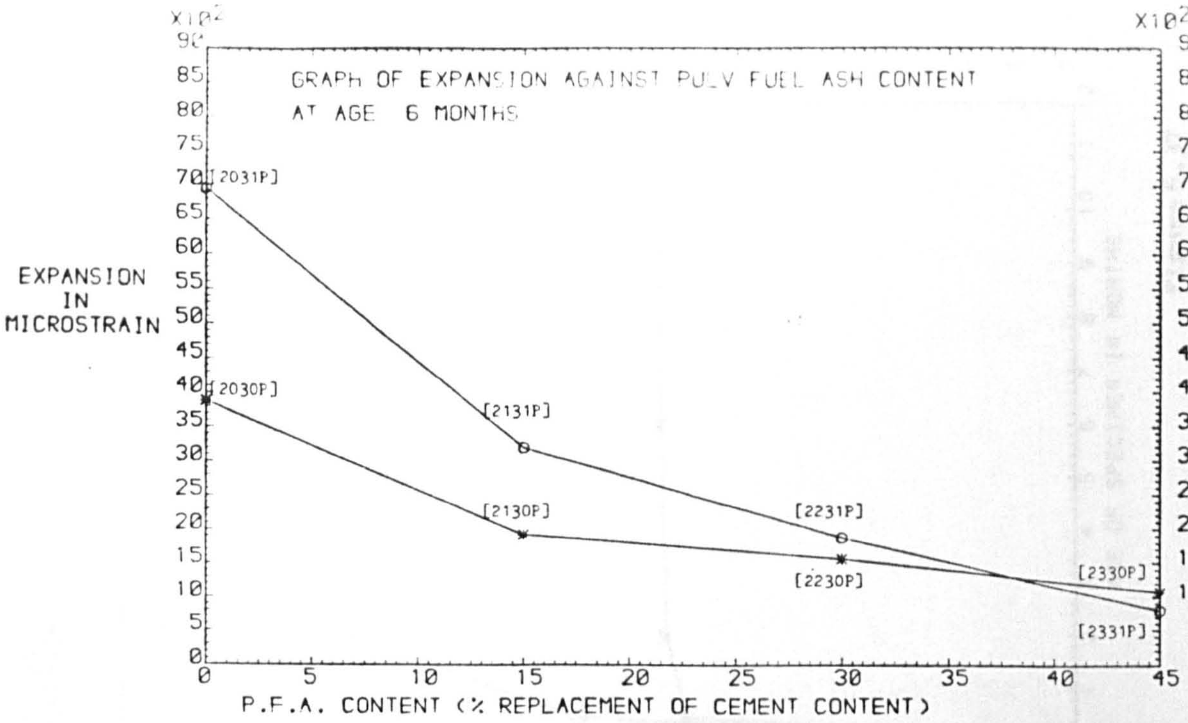


Figure 6.34

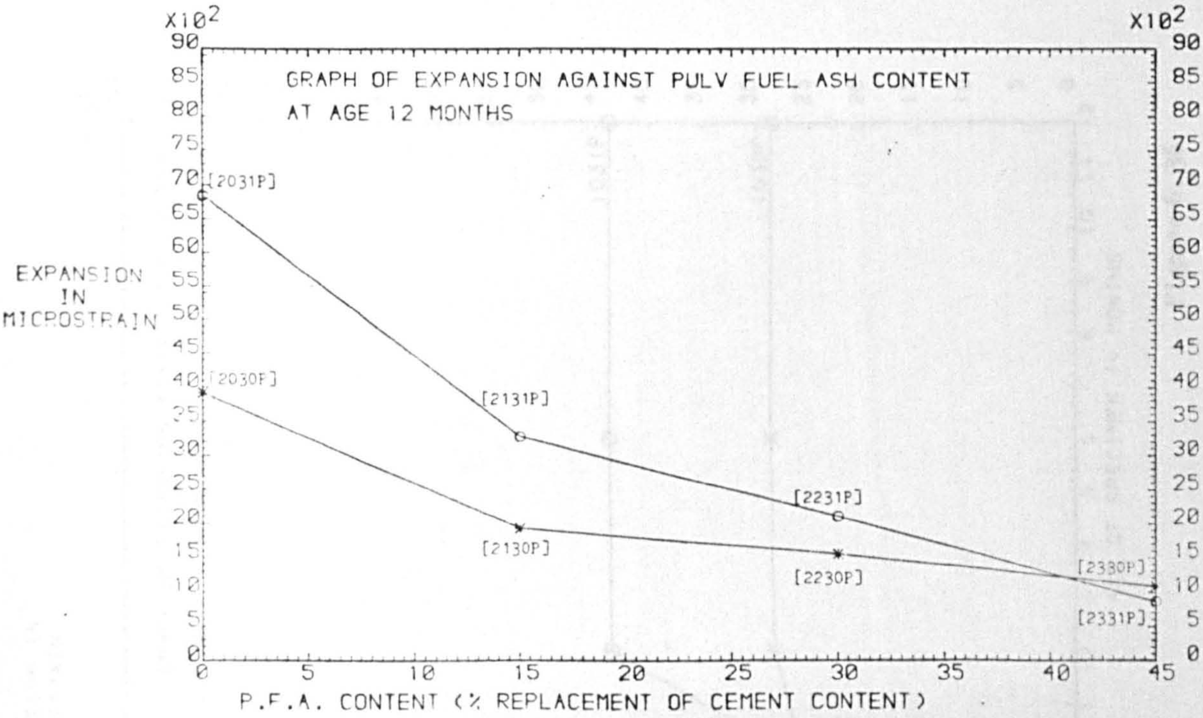


Figure 6.35

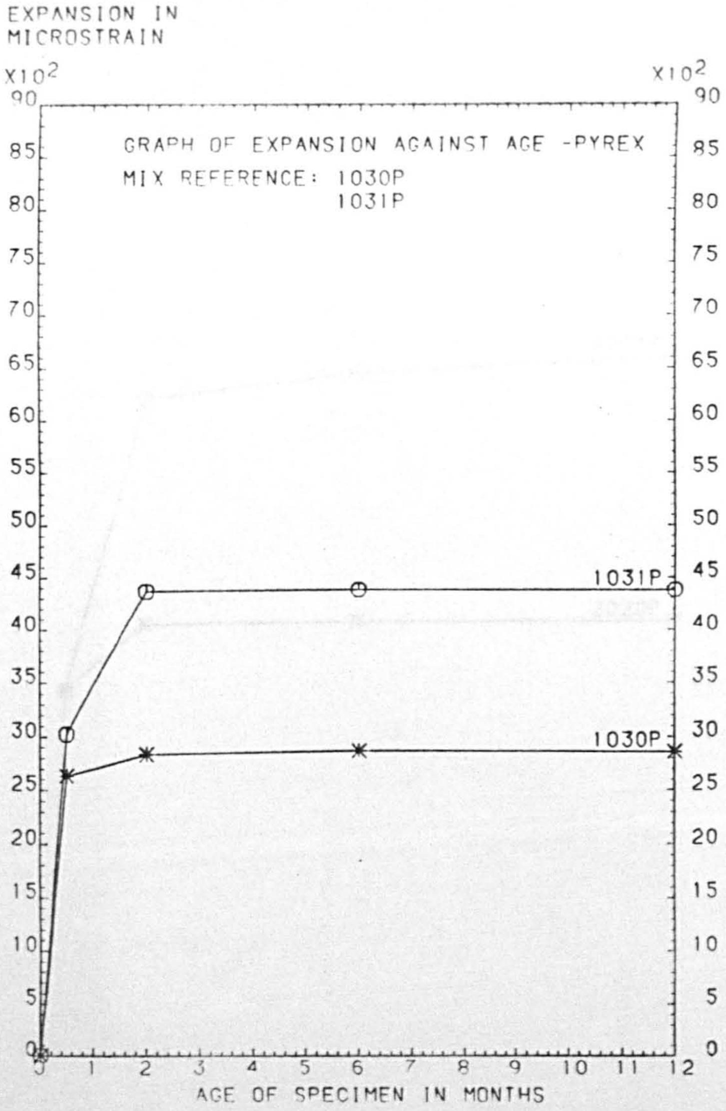


Figure 6.36

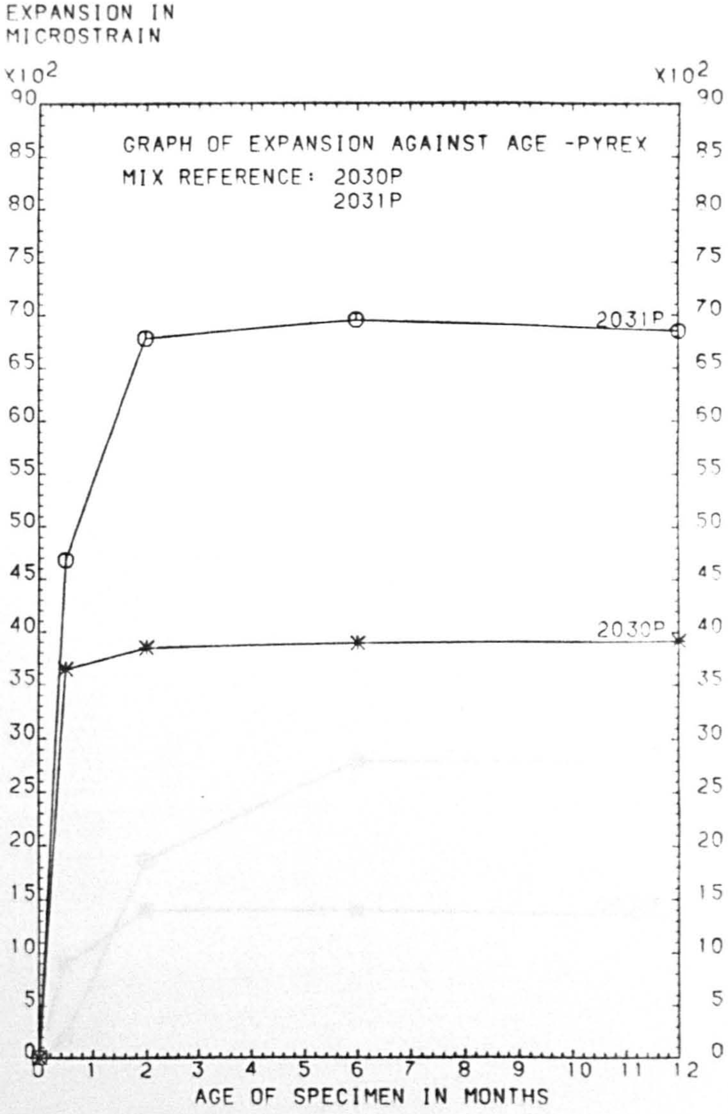


Figure 6.37

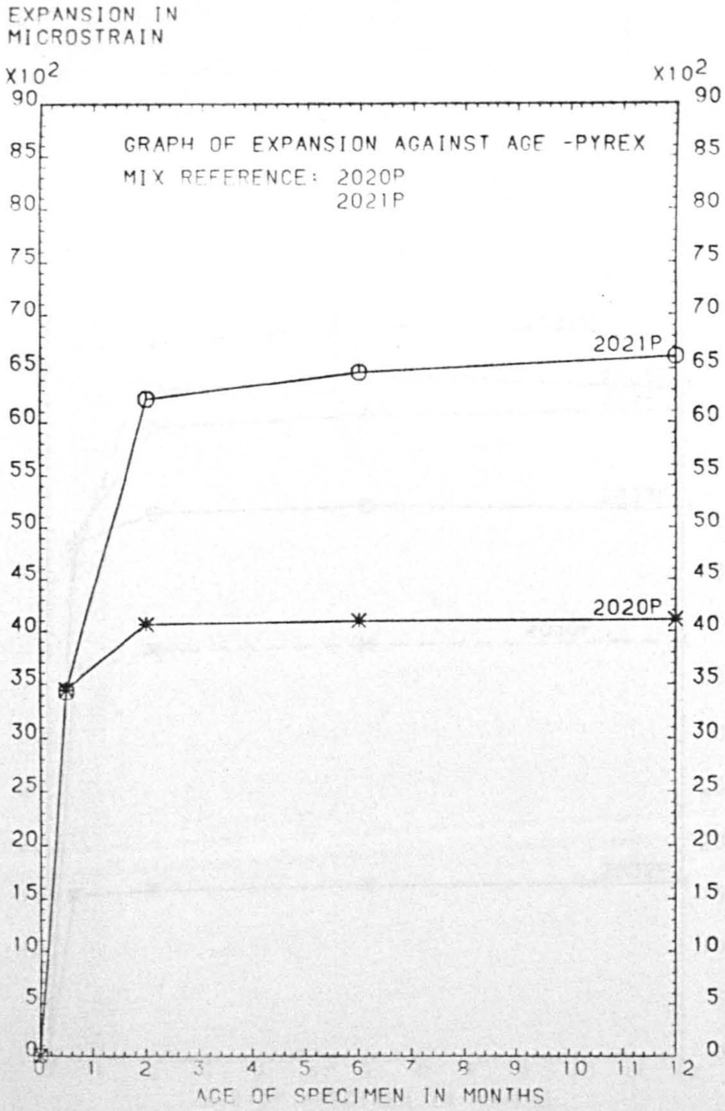


Figure 6.38

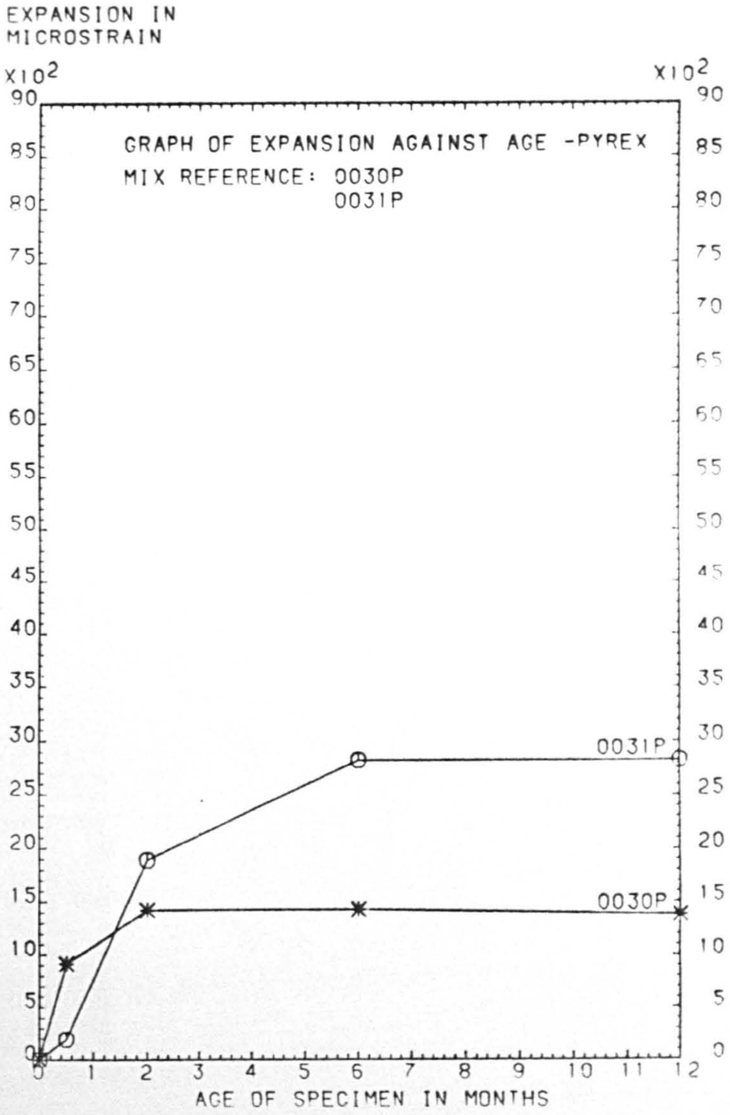


Figure 6.39

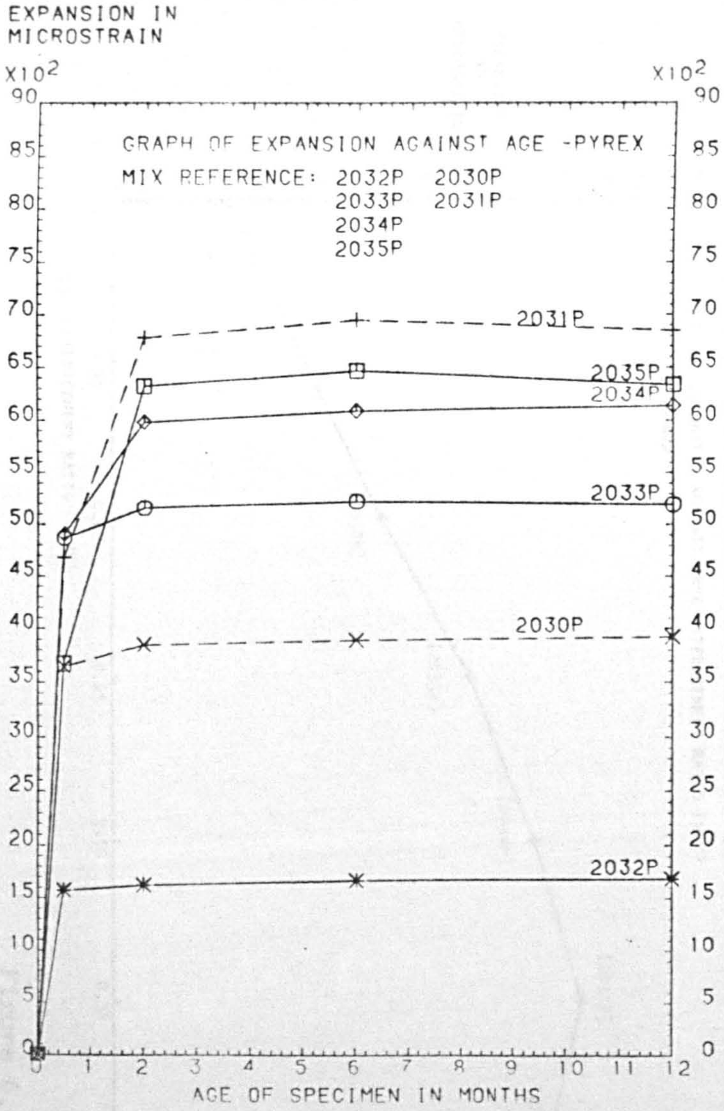

EXPANSION IN MICROSTRAIN
X10²
GRAPH OF EXPANSION AGAINST AGE -PYREX
MIX REFERENCE: 2032P 2030P
2033P 2031P
2034P
2035P
2031P
2035P
2034P
2033P
2030P
2032P
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
0 1 2 3 4 5 6 7 8 9 10 11 12
AGE OF SPECIMEN IN MONTHS

Figure 6.40

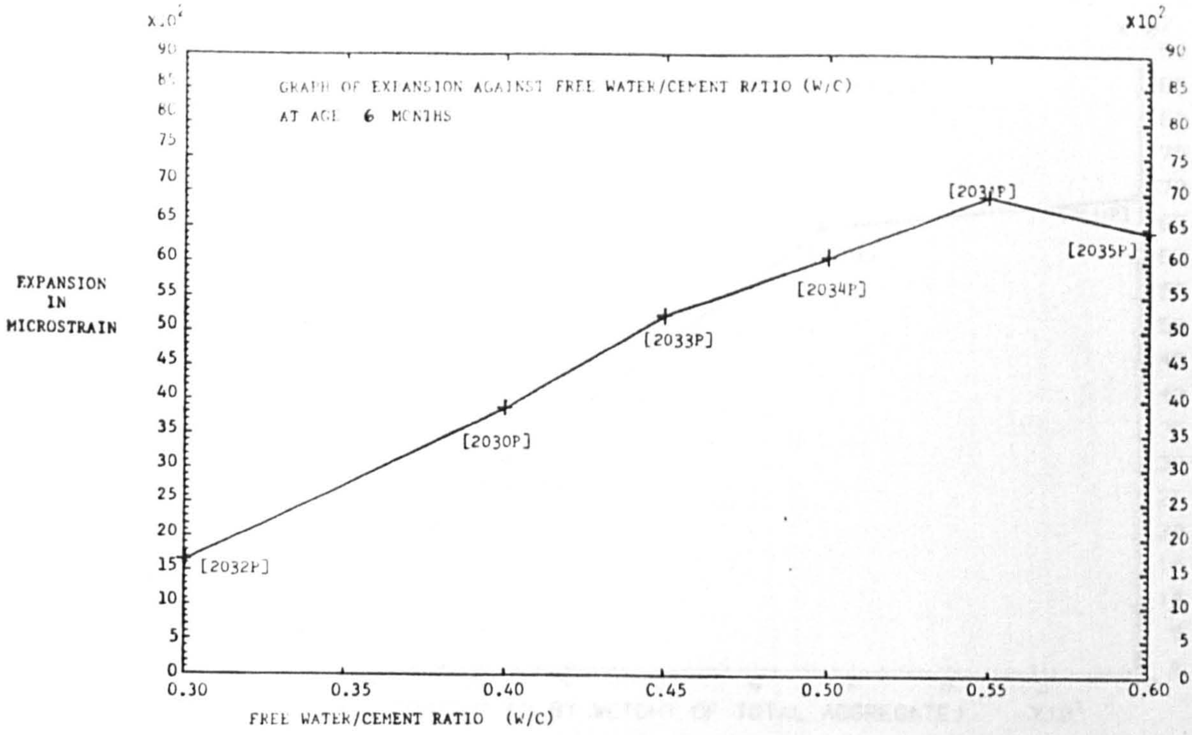


Figure 6.41

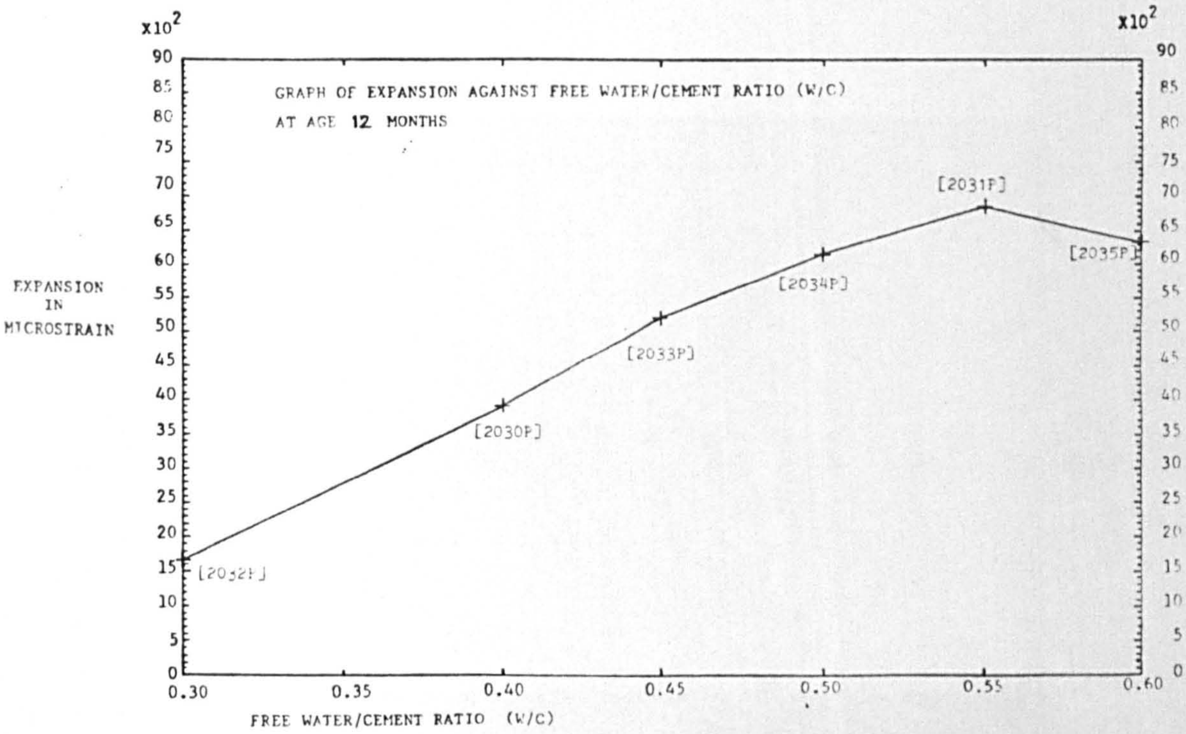


Figure 6.42

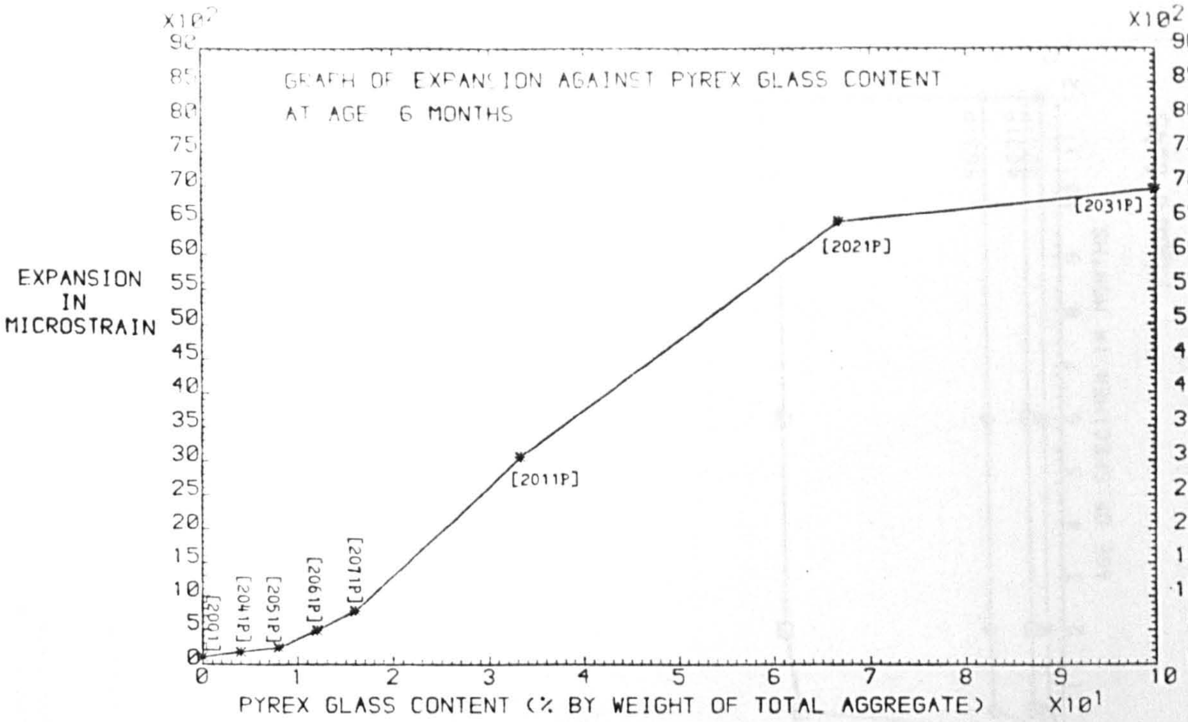

GRAPH OF EXPANSION AGAINST PYREX GLASS CONTENT
AT AGE 6 MONTHS
EXPANSION IN MICROSTRAIN
X10^2
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
PYREX GLASS CONTENT (% BY WEIGHT OF TOTAL AGGREGATE)
X10^1
0
1
2
3
4
5
6
7
8
9
10
[2001]
[2041P]
[2051P]
[2061P]
[2071P]
[2011P]
[2021P]
[2031P]

Figure 6.43

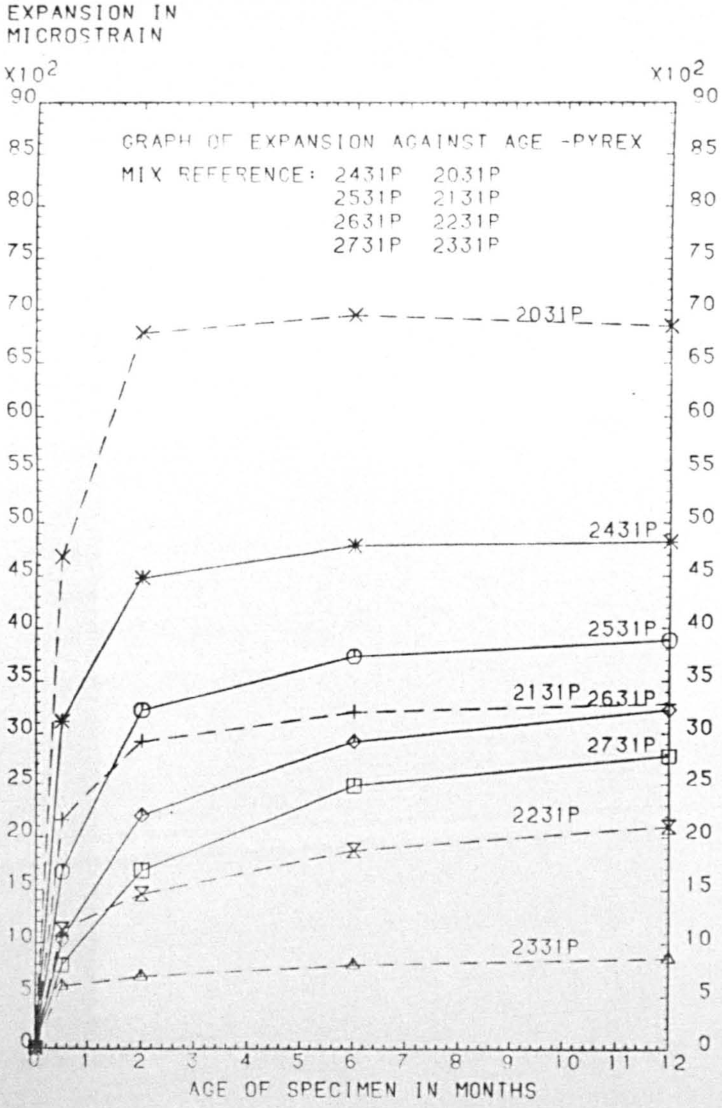


Figure 6.44

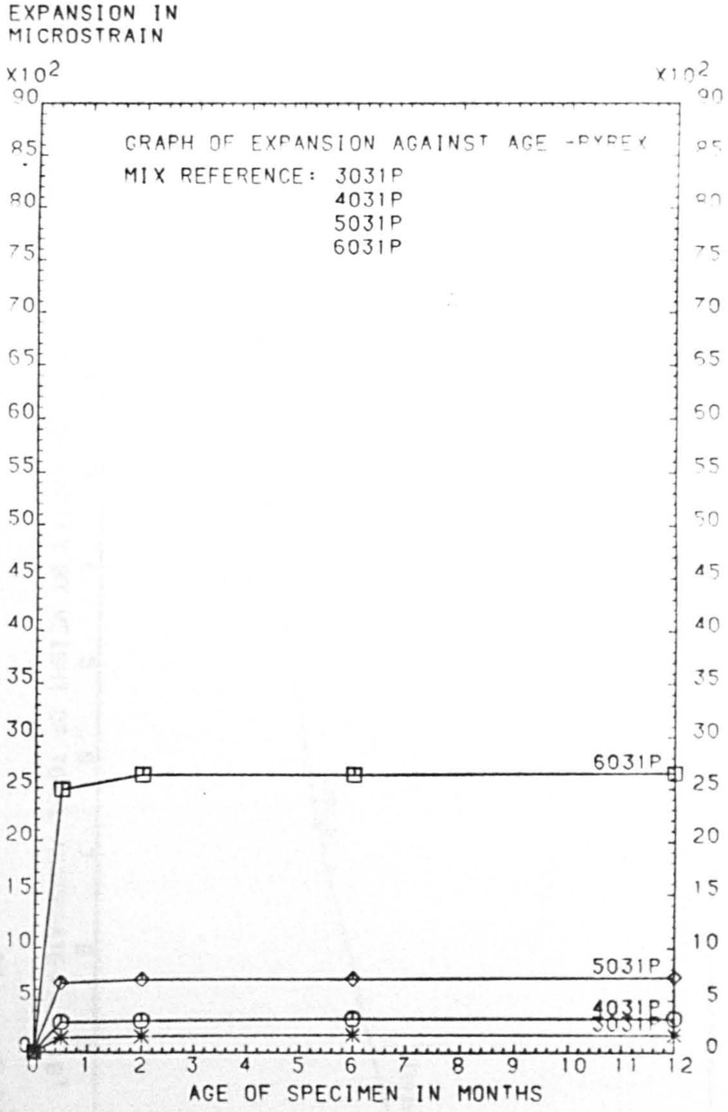


Figure 6.45

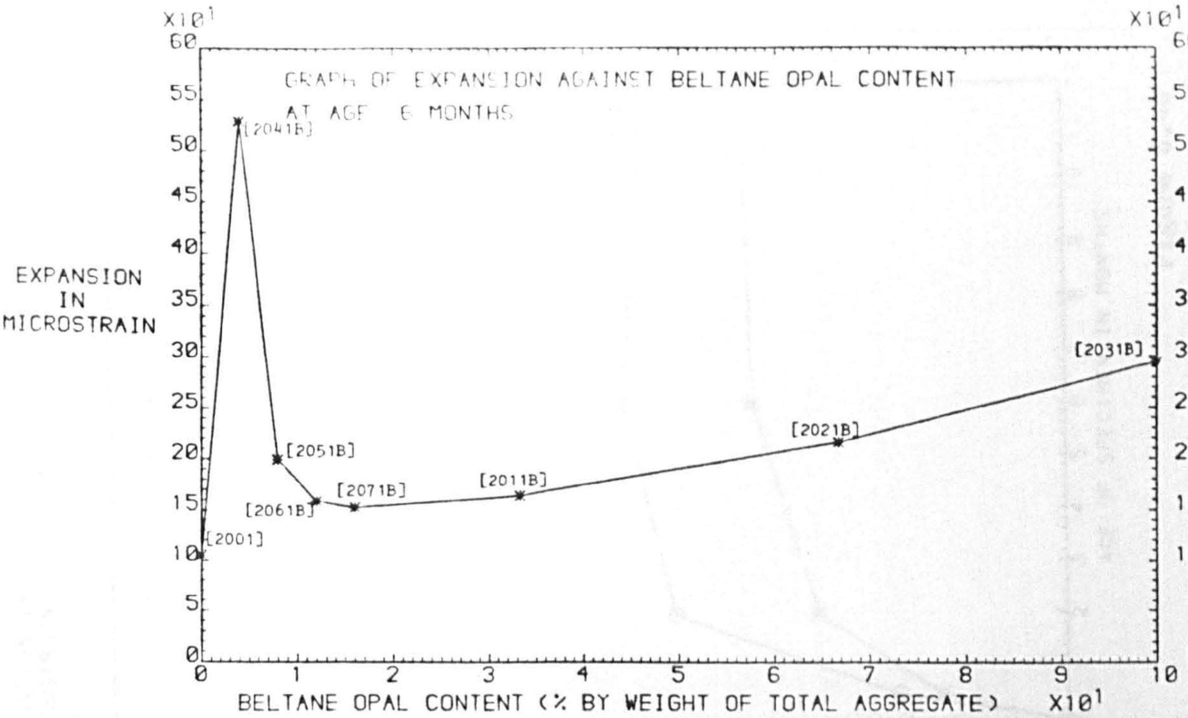

GRAPH OF EXPANSION AGAINST BELTANE OPAL CONTENT
AT AGE 6 MONTHS
EXPANSION IN MICROSTRAIN
X10¹
X10¹
60
55
50
45
40
35
30
25
20
15
10
5
0
[2041B]
[2051B]
[2061B]
[2071B]
[2011B]
[2021B]
[2031B]
[2001]
0
1
2
3
4
5
6
7
8
9
10
BELTANE OPAL CONTENT (% BY WEIGHT OF TOTAL AGGREGATE) X10¹

Figure 6.46

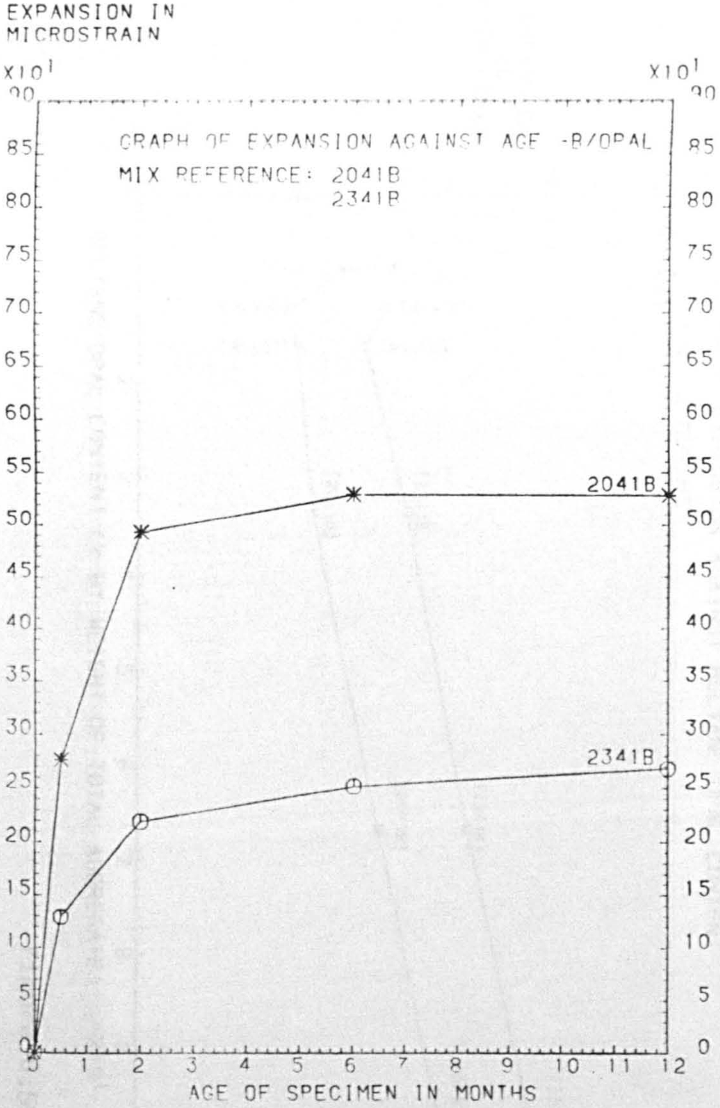


Figure 6.47

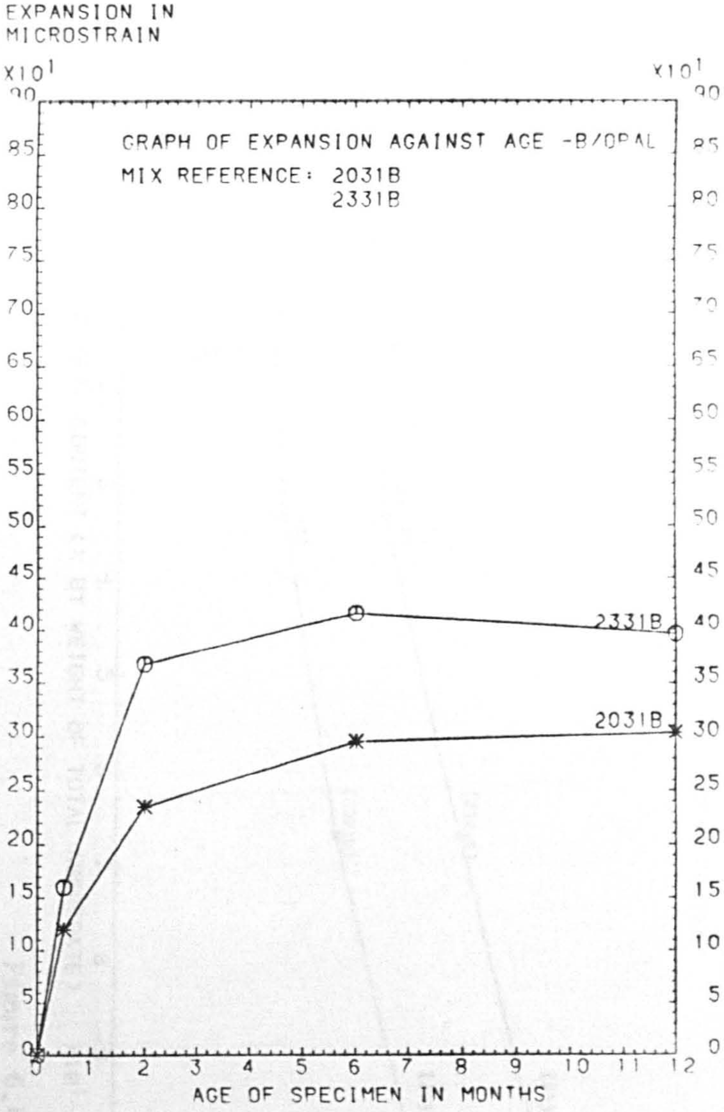


Figure 6.48

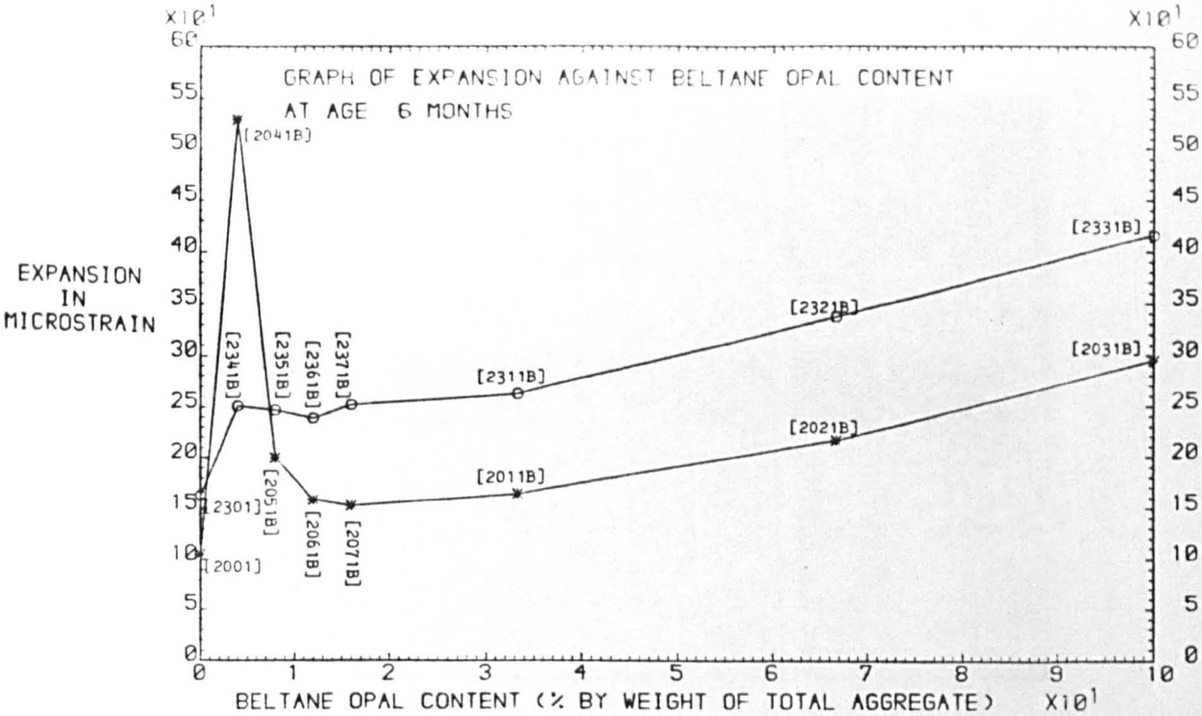


Figure 6.49

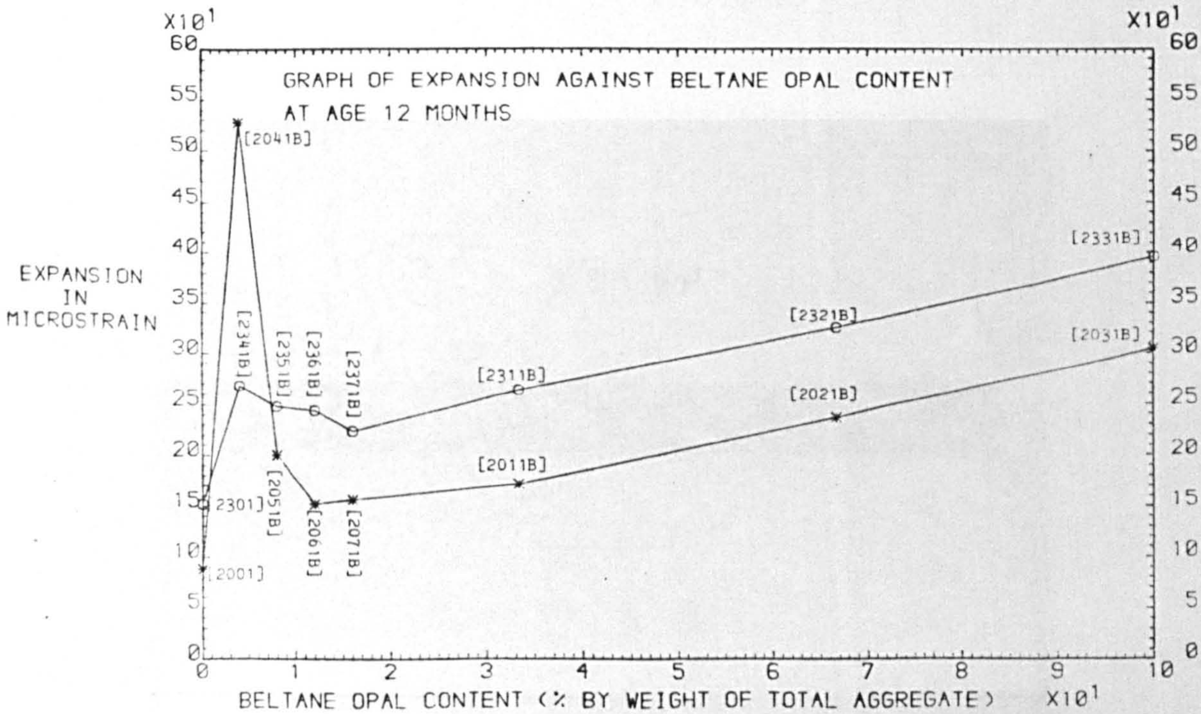


Figure 6.50

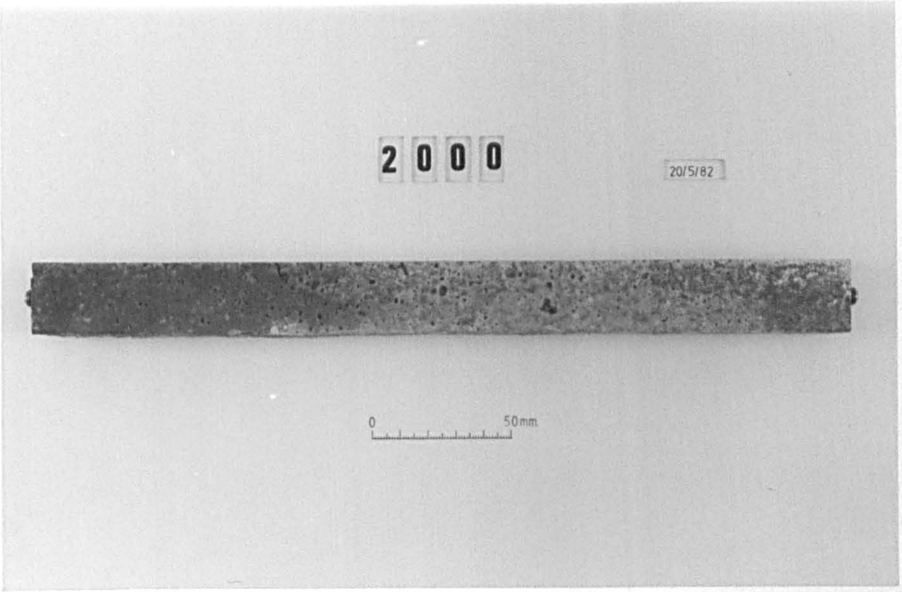


Plate 6.1 Mix Ref. 2000, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

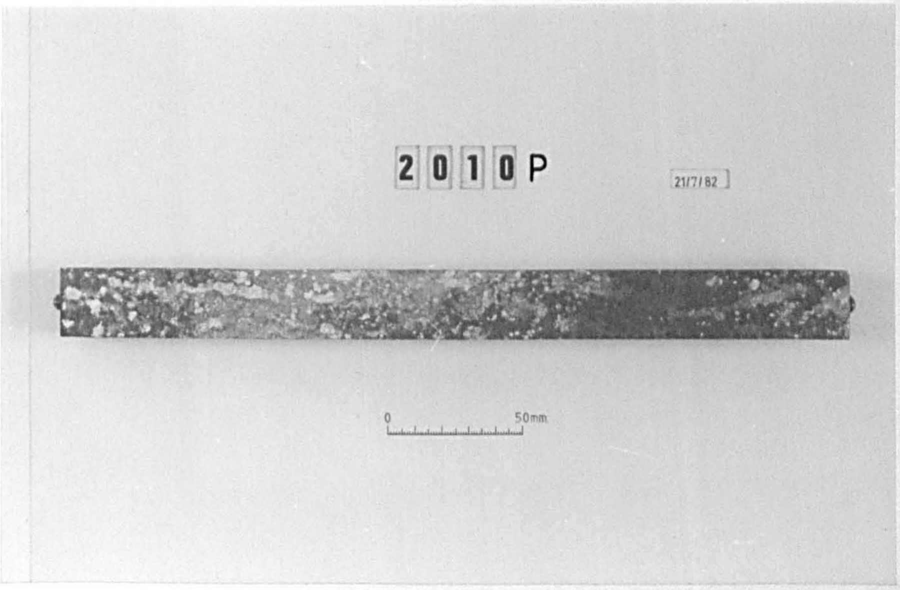


Plate 6.2 Mix Ref. 2010P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

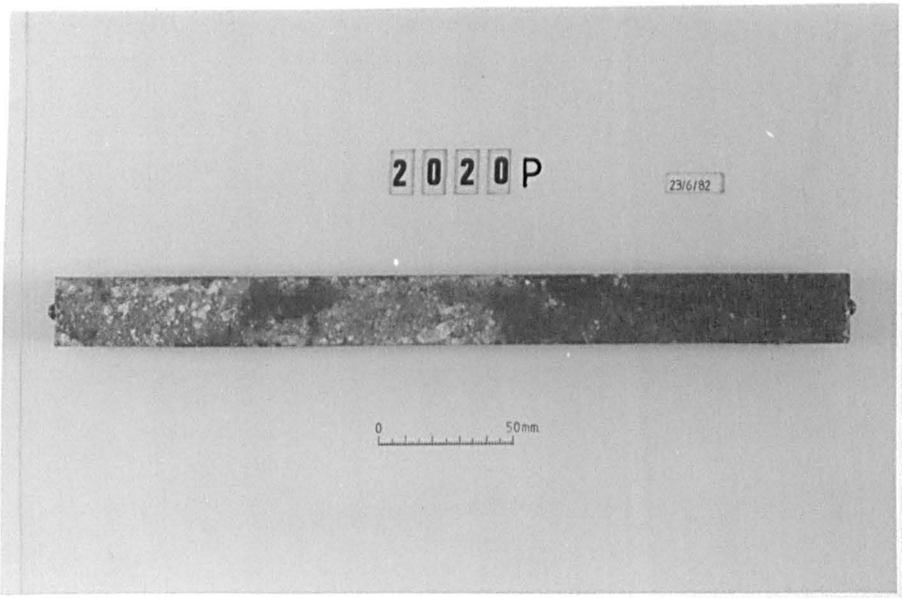


Plate 6.3 Mix Ref. 2020P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

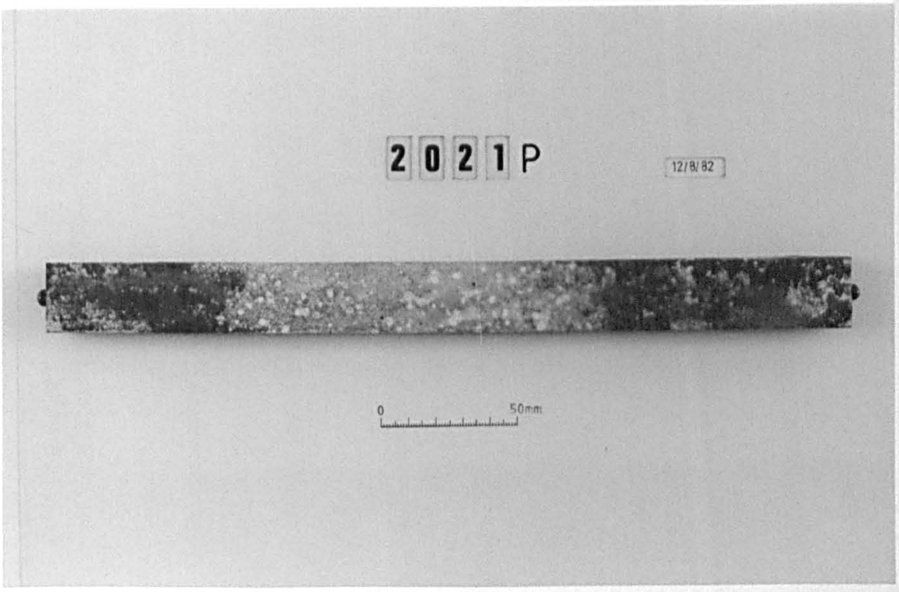


Plate 6.4 Mix Ref. 2021P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

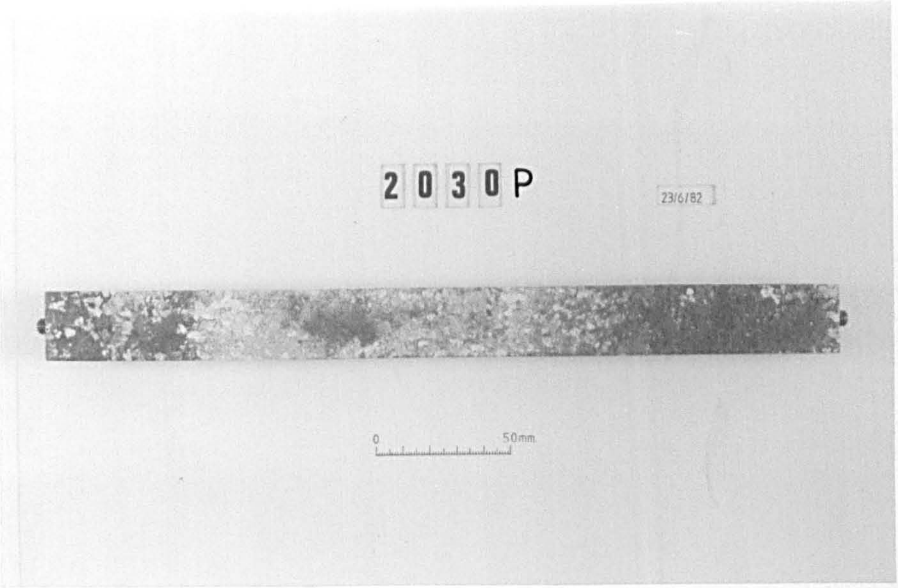


Plate 6.5 Mix Ref. 2030P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

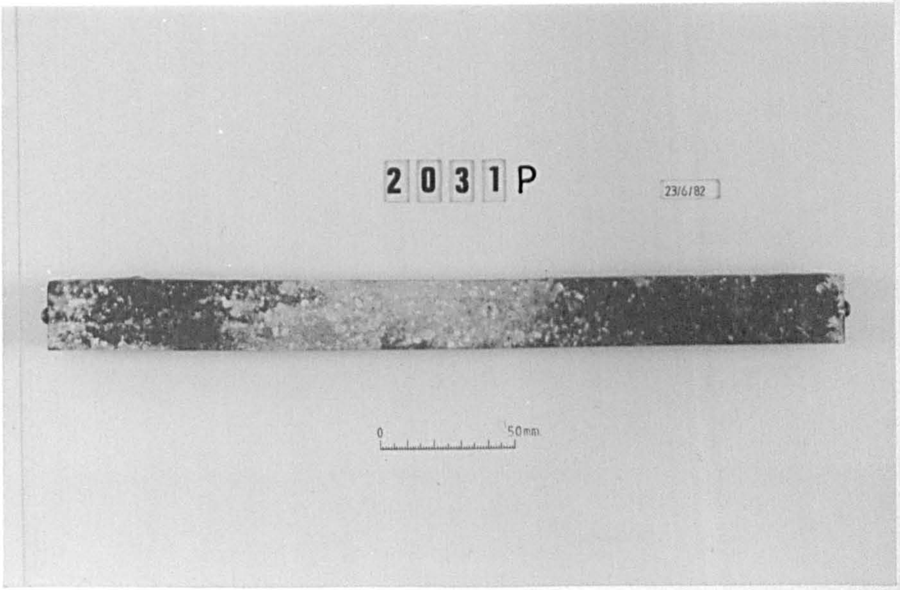


Plate 6.6 Mix Ref. 2031P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

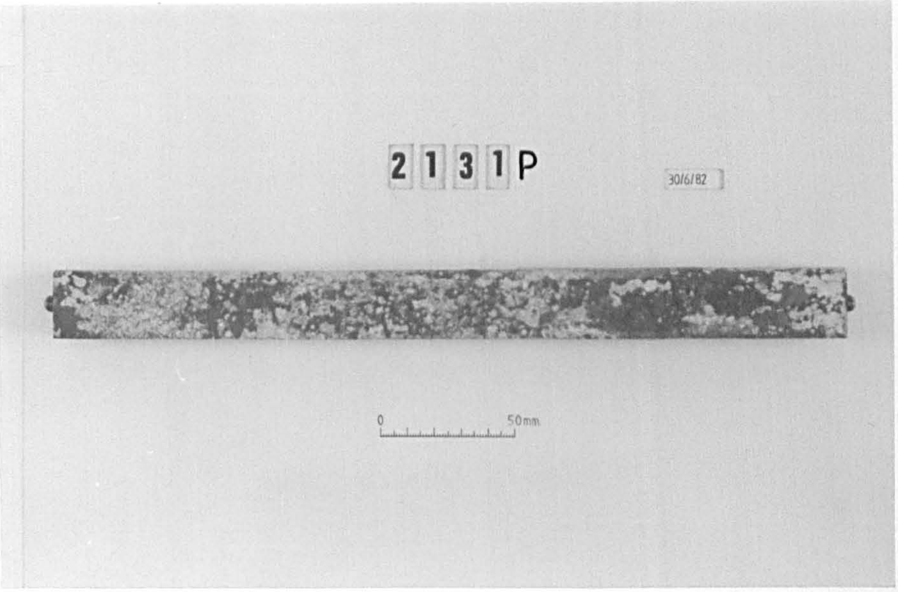


Plate 6.7 Mix Ref. 2131P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

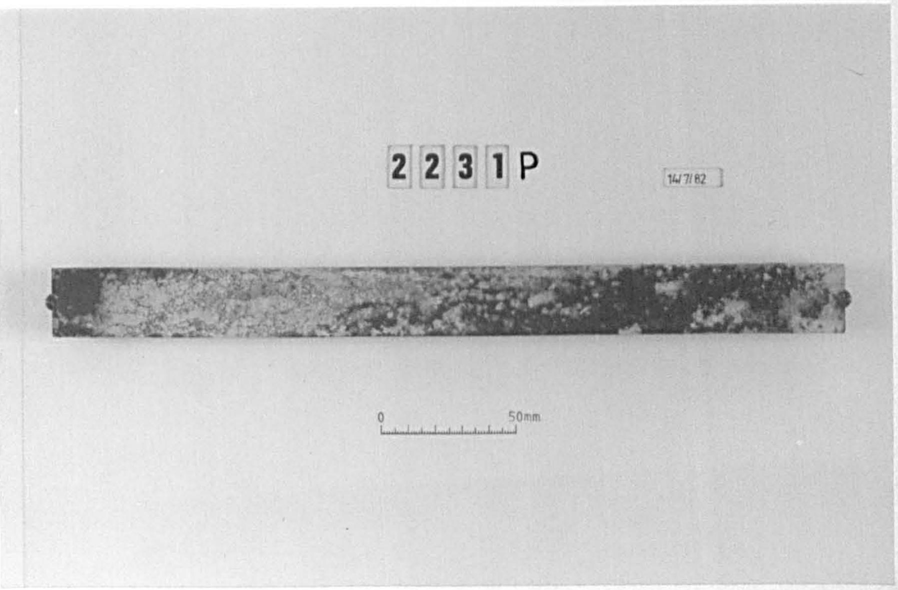


Plate 6.8 Mix Ref. 2231P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

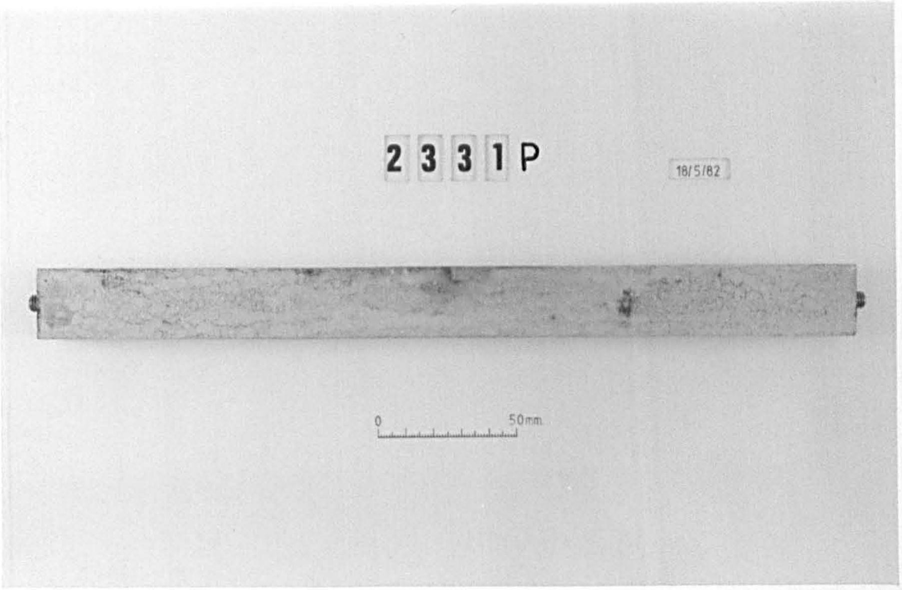


Plate 6.9 Mix Ref. 2331P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

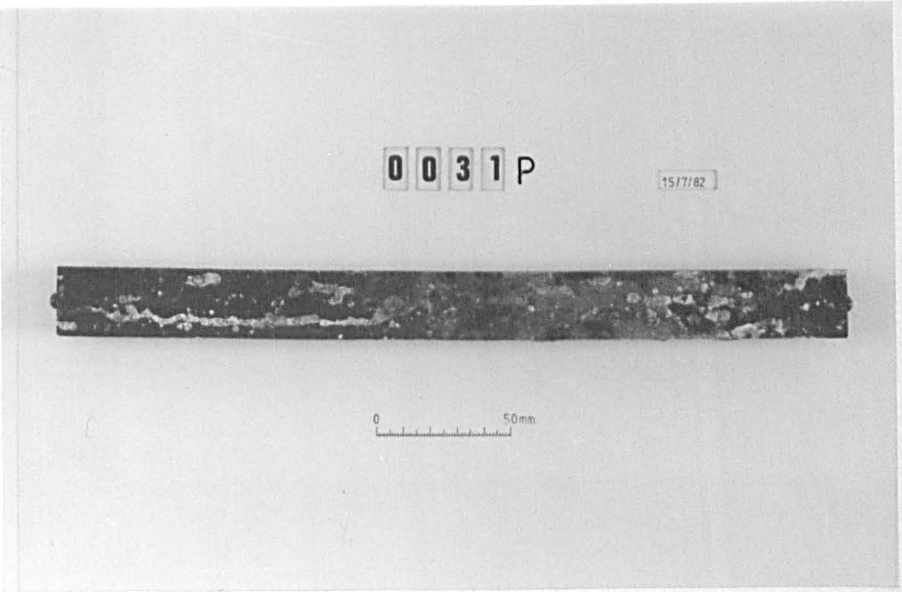


Plate 6.10 Mix Ref. 0031P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

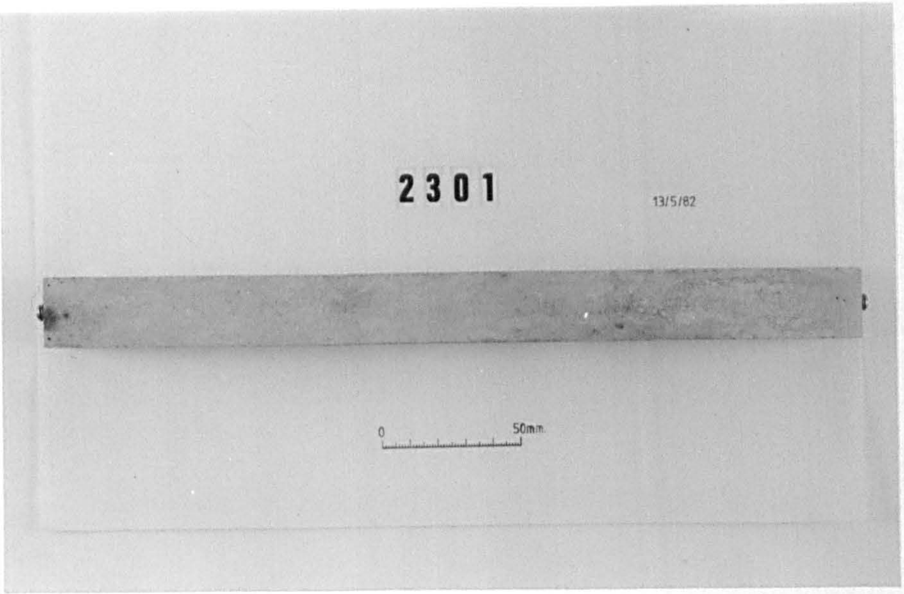


Plate 6.11 Mix Ref. 2301, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

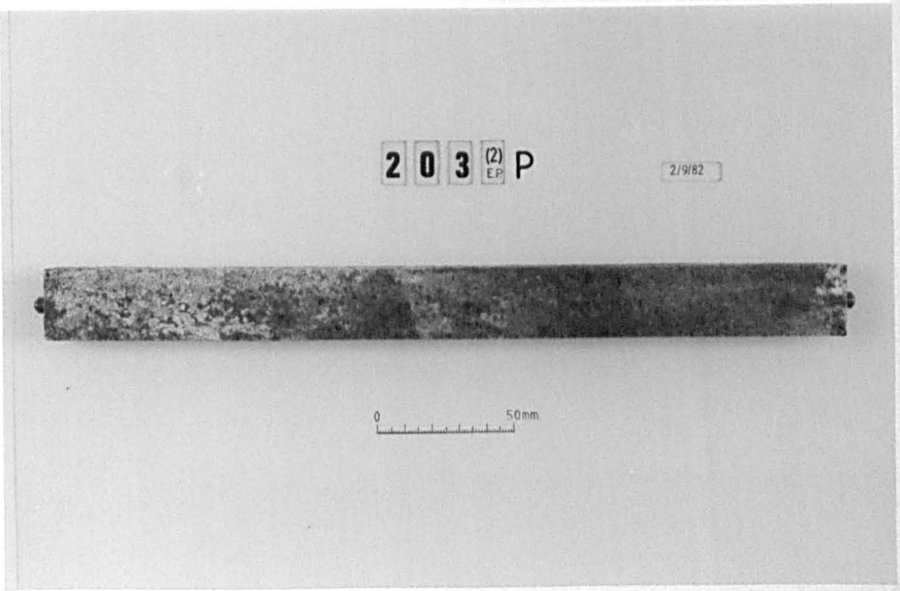


Plate 6.12 Mix Ref. 2032P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

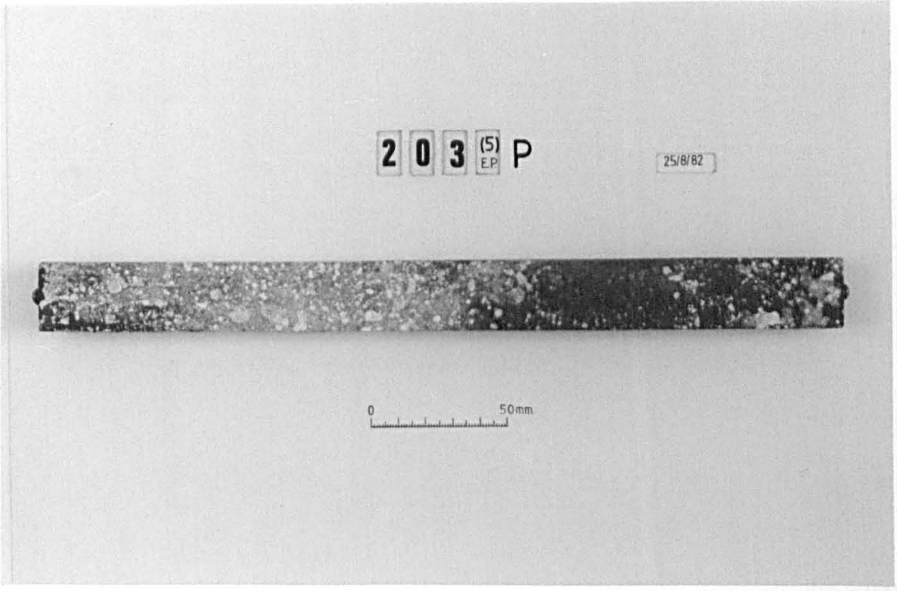


Plate 6.13 Mix Ref. 2035P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

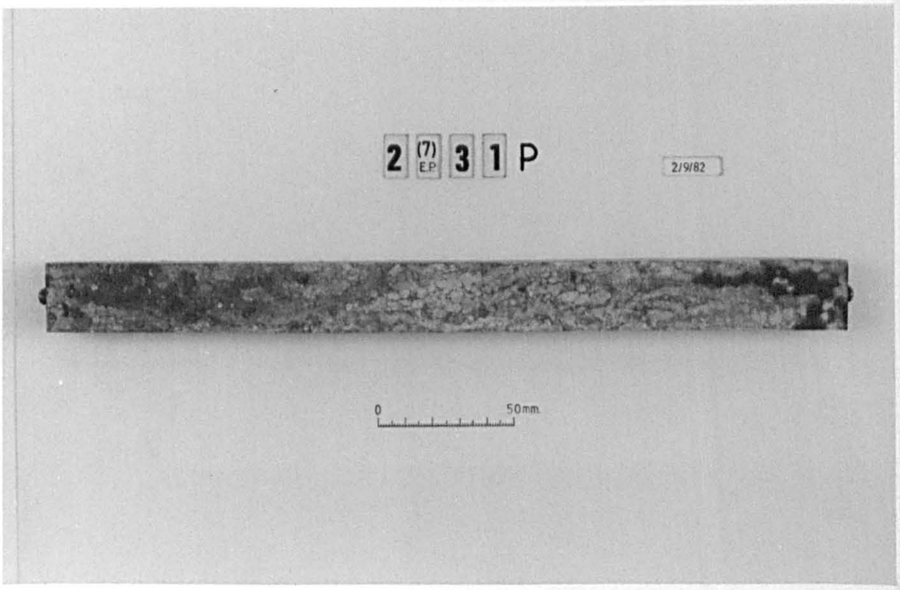


Plate 6.14 Mix Ref. 2731P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

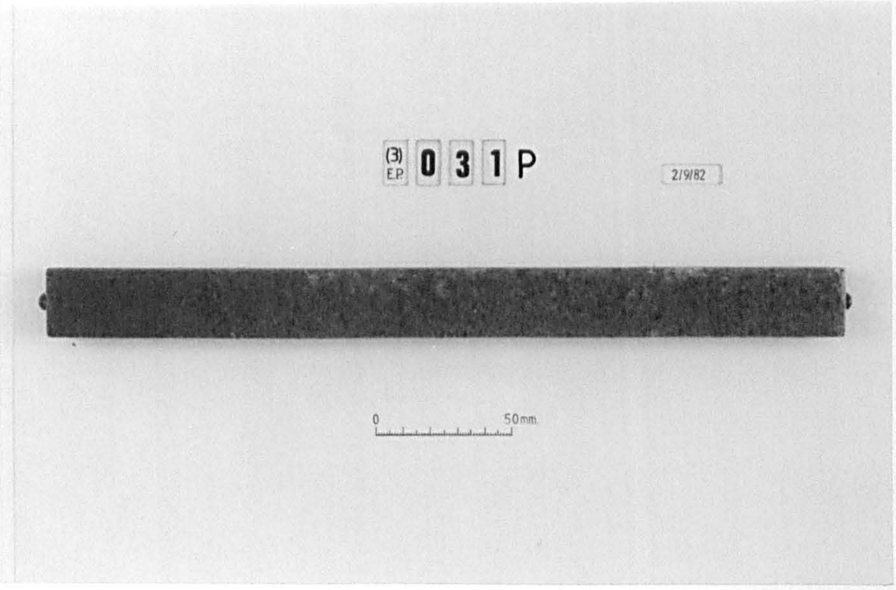


Plate 6.15 Mix Ref. 3031P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

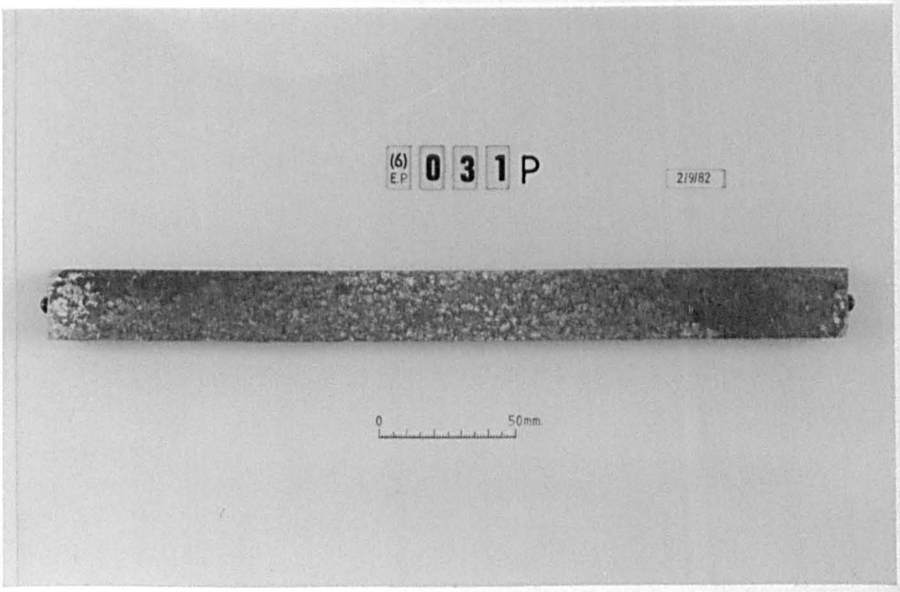


Plate 6.16 Mix Ref. 6031P, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

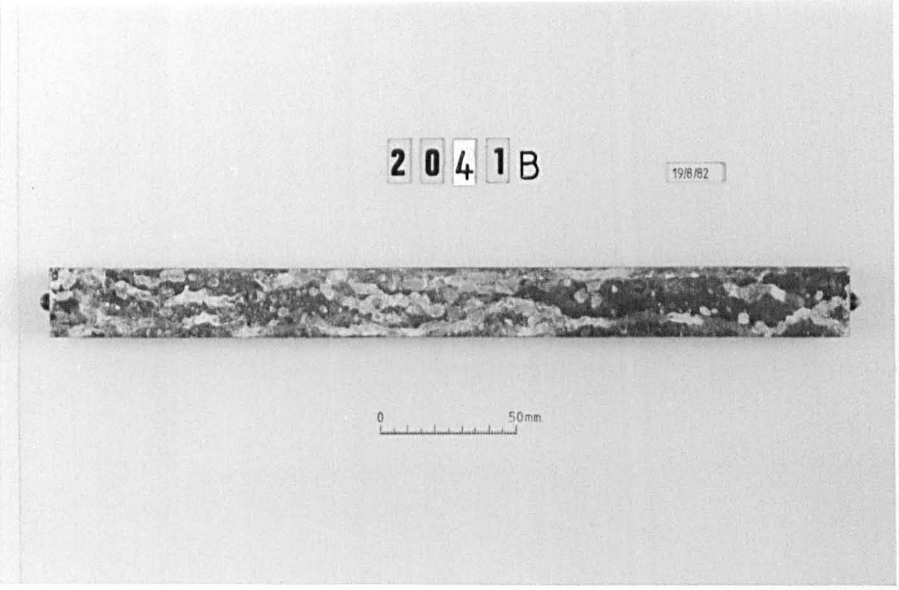


Plate 6.17 Mix Ref. 2041B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

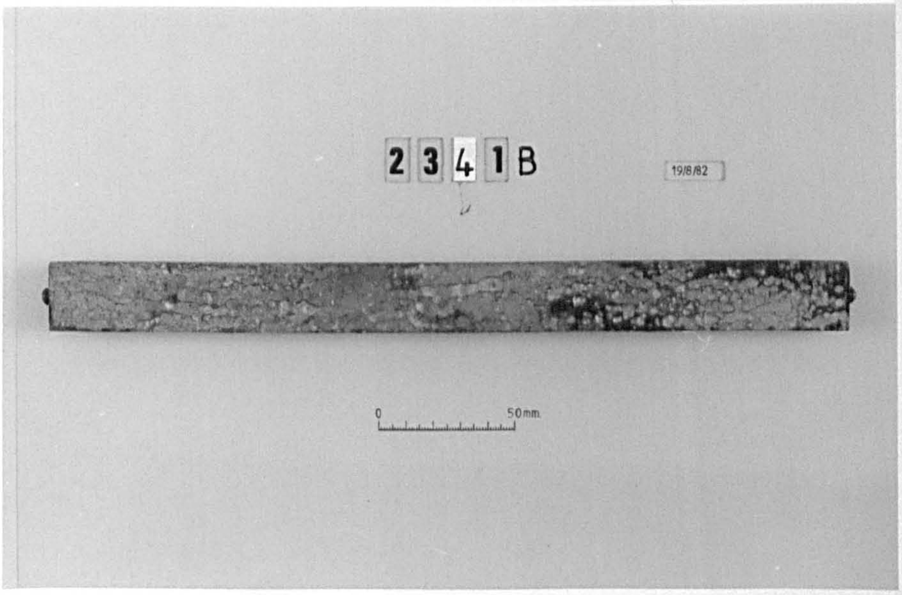


Plate 6.18 Mix Ref. 2341B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

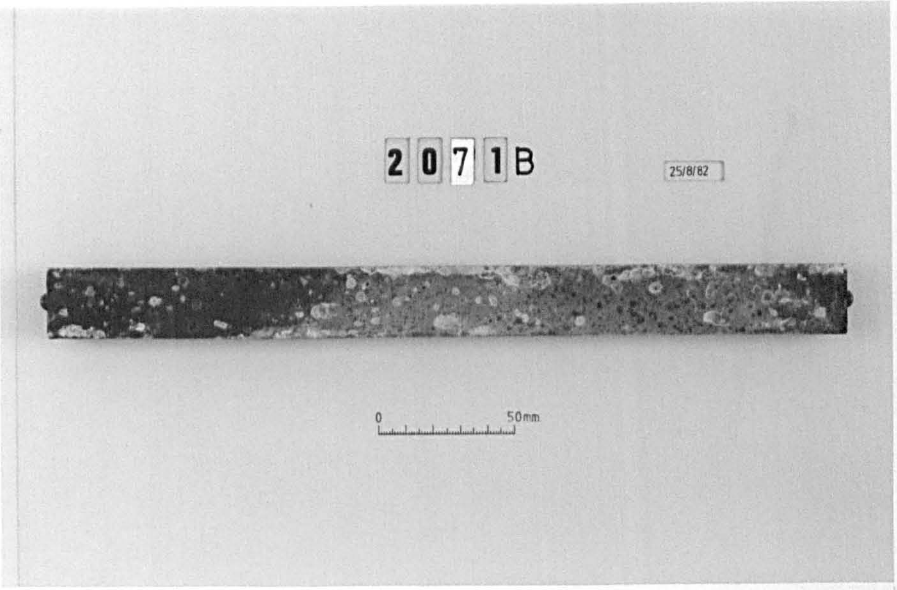


Plate 6.19 Mix Ref. 2071B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

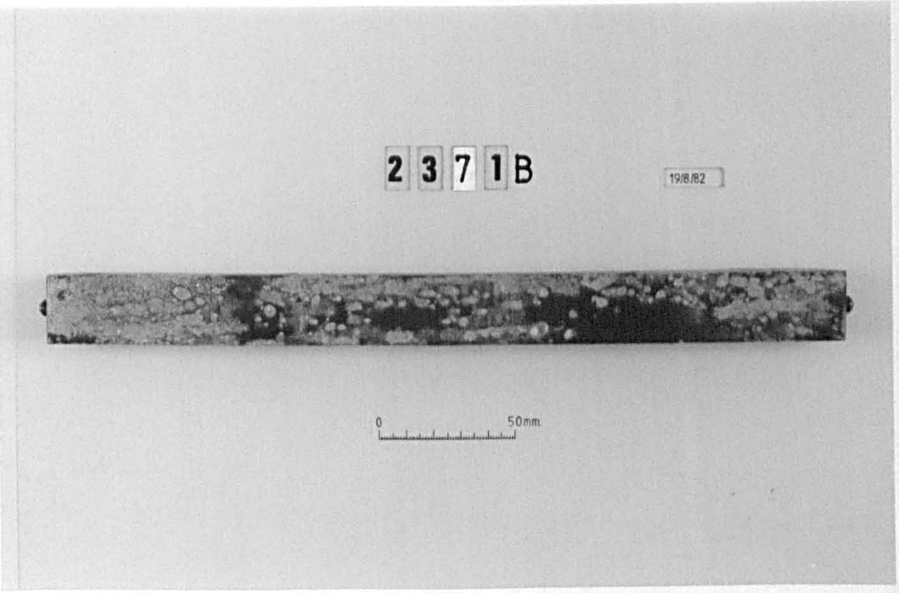


Plate 6.20 Mix Ref. 2371B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

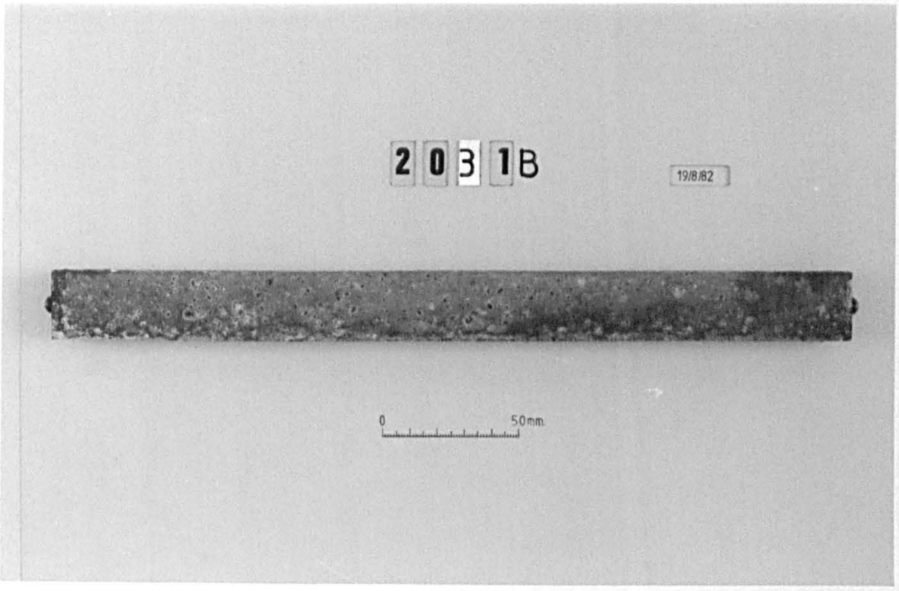


Plate 6.21 Mix Ref. 2031B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

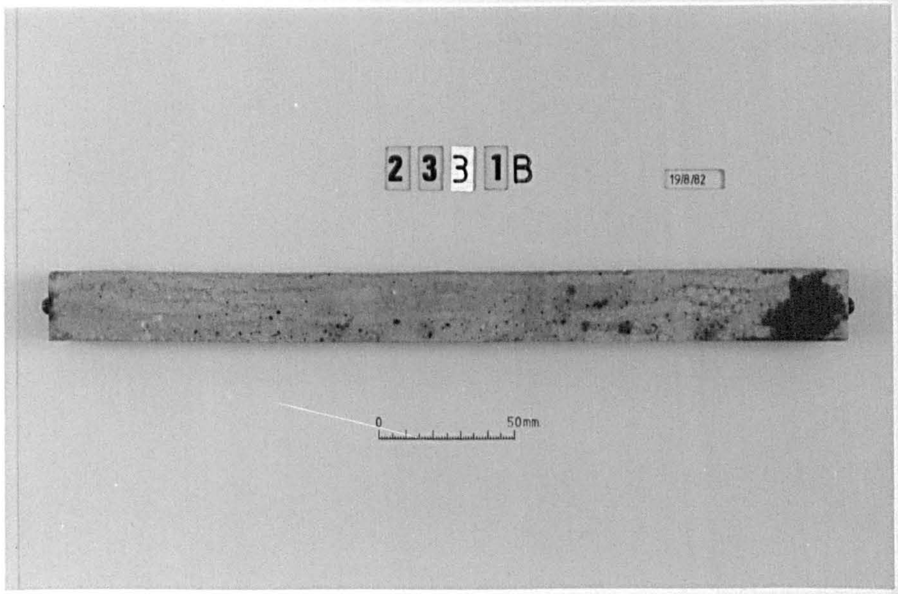


Plate 6.22 Mix Ref. 2331B, showing the surface condition of a typical mortar bar subsequent to the expansion measurement taken at age 6 months.

CHAPTER 7 DETAILS OF THE INVESTIGATION - CHEMICAL

7.1 INTRODUCTION TO CHEMICAL INVESTIGATION

In the preceding chapter mention was made of the apparent controversy surrounding the use of p.f.a. to inhibit expansion due to A.S.R.. The group of mixes 2431P to 2731P from the "Extra Pyrex" set produced expansion results which go part way towards dispelling the opinion that the introduction of p.f.a. acts to simply dilute the amount of alkalis available for reaction. This led to the suggestion, in the discussion section of Chapter 6, that the results are indicative of a more fundamental reaction taking place, which involves the p.f.a. and thus inhibits the normal course of A.S.R.

In addition, at the time of the experimental stages of this research, there was a growing weight of opinion gathering behind the hypothesis that the important criterion to consider, when designing a concrete mix using an aggregate known to be either reactive or suspect, was the alkali content of the mix expressed in kilogrammes of equivalent sodium oxide per cubic metre of wet concrete (kg Na_2O_{eq}/m^3). A figure of 3.0kg Na_2O_{eq}/m^3 had been expressed as a safe limit for the acid soluble alkali content, below which the deleterious reaction would not occur.

The success of p.f.a. in reducing expansions in the physical tests, together with the two subjects of debate listed above, provided ample stimulus for the chemical investigation which can be seen as an extension to the scope of the original research. The physical information in respect of mortar bar expansions, gained from the standard ASTM test methods, was already supplemented by certain chemical properties of the constituent materials, in particular their equivalent sodium oxide alkali contents. As a logical extension to this supplementary information a particular analytical approach was developed by which further information could be gained concerning the chemical aspects of the reaction, including the contribution made by p.f.a..

The mortar bars which had been used in the Series 2 expansion tests were themselves subjected to chemical analysis of their acid soluble alkali contents. The method of analysis was based on the BS 4550 method used in the analysis of cements. This work was done in order to establish the effect, if any, that p.f.a. has on the alkali balance within the mortar system. In addition, the water from the reservoir at the bottom of each of the mortar bar storage containers was also analysed for its alkali content. This established any tendency for leaching to take place and thereby completed the overall picture.

When used together, these two values of alkali content allow comparisons to be drawn between the alkalis previously found to exist in the constituent materials and the alkalis now found to be liberated in the mortar bars and their storage environment.

In this introductory section it is, perhaps, pertinent to mention that the inception of this chemical stage of the research came towards the completion of the physical expansion tests. Consequently the whole experimental regime was not designed with any chemical stage in mind. Moreover, the various stages of the analytical work were each conceived from the outcome of the previous stage. As a result of this step-by-step decision process, the modus operandi of the complete chemical investigation may not appear to be the most suitable approach. Indeed, if the work were to be repeated or continued, with the benefit of hindsight and foreknowledge, more rigorous and closely controlled methods would undoubtedly be adopted.

7.2 EXPERIMENTAL PROCEDURES

7.2.1 Samples for Chemical Analysis

The chemical tests were carried out on samples obtained from the mortar mixes of the Series 2 set of experiments only. A single mortar bar was removed from each storage container leaving two bars available for any future expansion measurements. In addition, the liquid at the bottom of the storage containers was removed for analysis and replaced with fresh water.

Removal of the mortar bar and the liquid sample was not always carried out simultaneously. Further, the age of the specimens varied considerably when the chemical analyses were carried out. Full details of the age of the specimens when the samples for chemical analysis were removed from the storage container can be found incorporated into the tabulation of the analytical results of Chapter 8. However, to summarise, for the mortars containing the high alkali cement (including the 'EP' and 'BO' mixes) the bar for chemical analysis was removed from the storage container immediately after the expansion measurement at 6 months. The chemical analysis was carried out as soon as practicable thereafter. The liquid analyses corresponding to these mortars followed approximately 9 months later. As regards the remaining mortars of the Series 2 experiments, namely those containing the low and medium alkali cements, both the bar and liquid specimens for chemical analysis were removed from the storage containers together, but at a much later stage in the overall experimental programme. The ages of the specimens in the latter group varied between approximately 16 months and 27 months when the chemical analyses were underataken.

7.2.2 Analytical Methods

Following the same ideals as the expansion tests, the chemical analyses were carried out, wherever possible, using recognised standard test methods. To this end the dilute hydrochloric acid soluble alkali contents of the mortar bars were determined using the method described in BS 4550: Part 2: 1970.

The liquids from the bottom of the storage containers were simply diluted to a suitable concentration and analysed for their sodium and potassium contents using a flame photometer. The total volume of liquid in each storage container was measured approximately during the sampling procedure using a conventional measuring cylinder. A more accurate method of volume determination was considered inappropriate because of the possible errors already existing in the form of spillages during expansion monitoring and the residue of liquid left trapped in the blotting paper lining to the storage containers.

7.3 DISCUSSION OF THE EXPERIMENTAL STAGES AND DECISIONS

The considerations behind the development of the need for the chemical investigations have already been outlined above, where the stage-by-stage approach to the experimentation was also mentioned. A more detailed discussion of the stages in the experimentation and the basis of the decision making process is now presented.

In the first instance the chemical tests were devised simply to assess the level of alkalis in the mortar bar and facilitate comparison of the results from mixes containing p.f.a. and those free from p.f.a. respectively. Highly reactive mixes were considered to be of prime importance and hence the tests were initially restricted to the mortars containing the high alkali cement. The decision to undertake this first set of chemical analyses was taken prior to the expansion measurements at 6 months. Hence, the most convenient procedure to adopt was to remove the bar for chemical analysis during the 6 month expansion test.

The results from this first stage of the chemical tests were found to contain some unexpected inconsistencies which, as will be seen in the following chapter, concerned an imbalance in the total amount of alkalis in the bars which contained no Pyrex glass or p.f.a.. That is to say, the total amount of alkalis in a mix such as 2000 did not add up to the alkalis supplied to the mortar by way of the consistuent materials.

To complete the picture it was therefore decided that the liquid in the reservoir at the bottom of the storage containers should be analysed since this appeared to be the only remaining source for the missing alkalis. Any alkalis found in this liquid are indicative of the amount of leaching taking place during the expansion tests. It was possible to carry out this stage of the chemical tests only because the liquid in the storage containers had not been changed during the earlier stages of the expansion tests.

On completion of the two-stage chemical testing carried out on the specimens from mixes containing the high alkali cement, the results were found to exhibit certain trends. It was therefore considered appropriate to carry out the same tests on the mortars containing the low and medium alkali cements to investigate whether similar trends existed. Unavoidably the ages of the mortar specimens had by this stage increased significantly beyond the age of 6 months when the bars containing the high alkali cement were analysed. However, for the final set of mortars it was possible to remove both the mortar bar and the liquid samples for chemical analysis at the same time.

CHAPTER 8 PRESENTATION AND DISCUSSION OF EXPERIMENTAL RESULTS - CHEMICAL

8.1 ANALYTICAL RESULTS

The full results from the chemical analysis of the alkali contents of the mortar bars and the liquid from the bottom of their storage containers are presented in Tables J.1 to J.3 of Appendix J. These data are from the Series 2 experiments, the three tables containing the results for the 3 x 4 x 4 x 2 factorial set, the 'E.P.' mixes and the 'B.O.' mixes respectively.

Tables J.1 to J.3 also give details of the age of the specimens when the sample for chemical analysis was removed from the storage container.

The alkali contents are given in terms of the individual sodium and potassium oxides in addition to a combined figure of total equivalent sodium oxide. For ease of comparison the alkali contents of both the bars and the liquids are expressed as percentages with respect to the mass of the mortar bar with which they are associated. The total equivalent sodium oxide figure is also expressed in kilogrammes per cubic metre based on the actual measured density of the bar which was analysed. The measured densities are quoted in the results tables and may be compared with the calculated values given in Tables B.1 to B.3 of Appendix B.

Using the relevant data from Tables J.1 to J.3, Tables 8.1 to

8.3 combine the results of the chemical analyses of the bars and liquids with information gained from the anlaysis of the individual mix constituents. In this way the acid soluble alkali content of the bars and liquids can be compared with acid soluble alkalis provided by the mix constituents in their original form.

8.2 THE EFFECT OF P.F.A. ON THE ALKALI LEVELS

8.2.1 Mixes containing no Reactive Aggregate

Considering firstly the mixes containing the highest alkali cement with no p.f.a. substitution, namely Mix Refs. 2000 and 2001, the appropriate results from Table 8.1 show that both the mortar bars and the liquid yielded a considerable amount of alkalis. The combination of these two values, bar plus liquid, represents the amount of alkali found in the overall system. This is shown in Table 8.1 where it is compared with the combined acid soluble alkali content of the individual mix constituents.

On substitution of 15% of the cement content by p.f.a., as in Mix Refs. 2100 and 2101, the amount of alkali in the overall system increased, despite the acid soluble alkali content of the p.f.a. being considerably less than the equivalent figure for the cement. This rise in alkali content continued for substitutions of 30% and 45% p.f.a. in Mix Refs. 2200, 2300, 2201 and 2301. For the mixes containing p.f.a. there is a considerable imbalance between the amount of alkali found in the overall system and the combined acid soluble alkali content of the individual mix constituents.

The general increase in the alkali content of the overall system was not the only observed effect attributable to the inclusion of p.f.a. in the mix. Reference to the individual alkali contents of the bars and the liquids for the same series of mixes shows that whilst the alkali content of the bars was increased when p.f.a. was used, the alkali content of the liquids was reduced. Thus, p.f.a. was responsible for a reduction in the amount of alkali being leached from the bars during the A.S.T.M. mortar bar test.

The relationships between the amount of alkali in the bars, the amount of alkali leached from the bars and the level of p.f.a. substitution are shown in Figure 8.1 for the two groups of mixes 2000 to 2300 and 2001 to 2301. The curves show that the relationships are regular but non-linear.

Before considering whether parallel effects attributable to the use of p.f.a. are shown for the mixes containing the medium and low alkali cements, it is perhaps pertinent to examine the effects which the three different Portland cements themselves had on the various alkali levels. This can be seen in Figures 8.2 to 8.5.

For mixes containing no p.f.a. and no reactive aggregate, there was a clear and steady increase in both the alkali content of the bar and the alkali content of the liquid corresponding to the change from low, through medium, to high alkali cement. This is shown in Figure 8.2 for the groups of mixes 0000 to 2000 and 0001 to 2001. Figure 8.2 also shows evidence that the increase in the alkali content of the specimens is proportional to the change in the alkali content of the cement.

When 45% p.f.a. was substituted into the cementitious material, Figure 8.3 shows that the rise in alkali content of the bars continued to be proportional to the alkali content of the cement (mixes 0300 to 2300 and 0301 to 2301). The gradient of the increase, however, is shown to be reduced since there was less cement in the mix. Unlike the mixes containing no p.f.a., Figure 8.3 shows that the amount of alkali being leached from the bars and into the liquid was unaffected by the cement alkali content when 45% p.f.a. was included. The value remained at the base level set by the lowest alkali cement.

For mixes containing 100% Pyrex glass but no p.f.a., the alkalis in the bar again increased with increasing cement alkali content. However, Figure 8.4 shows that the relationship does not take a linearly proportional form for the two groups of mixes 0030P to 2030P and 0031P to 2031P. This figure also shows that when Pyrex glass was present in the mix the cement alkali level did not cause any significant changes to the amount of alkalis leached from the bars.

The inclusion of both p.f.a. and Pyrex glass in the mix resulted in the alkali content of the bars being proportional to the level of alkalis in the cement with little evidence of the type of cement having any effect on the amount of alkalis leached from the bars. These comments relate to mixes 0330P to 2330P and 0331P to 2331P as shown in Figure 8.5.

The effect of p.f.a. on the alkali content of mixes containing the medium and low alkali cements can now be examined to investigate whether the response is similar to that found for the high alkali cement.

With respect to the amount of alkali in the overall system and the amount of alkali in the bars, the results for the mortars containing the medium and low alkali cements show parallel trends to those noted for the high alkali cement. That is for the groups of mixes 1000 to 1300, 1001 to 1301, 0000 to 0300 and 0001 to 0301 both the amount of alkali in the overall system and the amount of alkali in the bar increase with increasing p.f.a. substitution. This is shown in Figures 8.6 and 8.7 for the medium and low alkali cements respectively.

The results for the mixes containing the low alkali cement, however, did not show any significant tendency for the level of p.f.a. substitution to reduce the amount of alkali leached from the bars. The amount of alkali in the liquids remained sensibly constant over the range of p.f.a. substitution employed. This lack of an effect is probably due to the low level of alkali leached from bars containing the low alkali cement even in the absence of p.f.a., thus offering little potential for the p.f.a. to further reduce leaching.

When the medium alkali cement was used, the effect of p.f.a. on the amount of alkali leached from the bars was for a slight reduction with increasing p.f.a. substitution. It is noteworthy that the graphs of the amount of alkali in the liquid against p.f.a. substitution for the three different cements, Figures 8.1, 8.6 and 8.7, all tend towards similar minimum values of alkali content at 45% p.f.a. substitution.

To summarise, when used as a partial replacement of the cement content, p.f.a. was found to be associated with an increase in the alkali content of the overall mortar bar system for specimens stored in a closed environment as in the A.S.T.M. mortar bar test. This trend for increasing alkali content was also noted for the amount of alkali in the mortar bars considered alone. Both these effects were similar for mixes containing the high, medium and low alkali cements. With respect to the amount of alkali leached from the mortar bars, the p.f.a. was found to cause a reduction in the amount of alkali contained in the liquid at the bottom of the storage containers. This effect occurred provided that the alkali content of the cement was high enough to allow sufficient alkali to be leached from the bars containing no p.f.a.. The latter effect is consistent with the trends noted in the various alkali contents in relation to the alkali content of the cement employed.

8.2.2 Mixes containing Pyrex Glass

In general, the results show evidence of an increase in the alkali contents of the mortar bars containing p.f.a in comparison to the corresponding bars in which Portland cement was the only cementitious component. Using the same comparison, the results exhibit some evidence of a small decrease in the amount of alkali leached from the bars as the level of p.f.a. substitution was increased. The latter effect showed a tendency to be more prominent for the lower Pyrex glass concentrations. The combination of these two effects, for mixes containing Pyrex glass, is that, generally, the amount of alkali in the overall system was found to increase when p.f.a. was used as a partial replacement of the cement content. These comparisons are shown in Figures 8.8 to 8.12, in which the effects with different concentrations of Pyrex glass and for the three different Portland cements are examined.

The relationships between the amount of alkali in the bars, the amount of alkali leached from the bars and the level of p.f.a. substitution are not as clearly defined for these mixes containing Pyrex glass. This is probably due to the more complex system which develops within a reactive mortar and the effect which Pyrex glass itself has on the alkali levels.

Figures 8.13 to 8.15 examine the effect produced by the Pyrex glass. There is evidence that the amount of alkali present in the bars was greater when Pyrex glass was included in the mix, in comparison to the corresponding mix containing no Pyrex glass. For the medium and low alkali cements the relationship between the alkali content of the bar and the amount of Pyrex in the mix was found to be fairly linear (Figures 8.14 and 8.15). There is also evidence that the amount of alkali leached from the bars was reduced when Pyrex glass was included in the mix. This effect was found to be more clearly defined for the high and medium alkali cements where a linear relationship is evident. The combined effect is that the Pyrex glass was responsible for an increase in the amount of alkali in the overall system.

These effects, observed with respect to Pyrex glass, bear a marked resemblance to those previously discussed for p.f.a. substitution in mixes containing no reactive aggregate. Thus, the two materials must act in competition when both are included in the mix. This is no doubt an over simplification of the true situation, but may explain the reduced effect which p.f.a. had on the various alkali contents with mixes containing Pyrex glass.

For the mixes containing the high alkali cement, Pyrex glass concentrations of 66.6% and 100% are shown in Figure 8.13 to reduce the level of alkali found in the liquid from the storage tank to a value approaching that which would be anticipated with 45% p.f.a. substituted into a mix containing no reactive aggregate. Thus, with such a mix, the p.f.a. has no further contribution to make in reducing the amount of alkalis leached from the bars.

Similarly, the observed increase in the alkali content of the bars when Pyrex glass was included in the mix, may explain the reduced effect which p.f.a. had on the alkali content of bars containing Pyrex glass, when compared with the parallel effect for bars devoid of reactive aggregate. The p.f.a. and the Pyrex glass compete for the same reactant, namely the hydroxyl ion, the Pyrex glass reacting to a much reduced extent as indicated by the large decrease in the observed expansion. Consequently, some of the alkali which would normally be released by the Pyrex glass was retained. This would have offset some of the alkali released by the p.f.a., thus making it appear that the p.f.a. was having a reduced effect.

8.2.3 Mixes containing Beltane Opal

Comparison of the numerical results in Table 8.3 again shows that there was a considerable increase in the amount of alkali present in the mortar bars when p.f.a. was used. There was not, however, any significant evidence that p.f.a. produced a reduction in the amount of alkali leached out of the bars, no trend being evident in either direction. These effects are shown in Figure 8.16.

The lack of a p.f.a. derived effect on the amount of alkali leached out of the bar may be explained by the contribution made by the Beltane opal itself in this respect. In Figure 8.17 it can be seen that the Beltane opal produced a very marked reduction in the amount of alkali leached out of the bars. The extremely low alkali levels which remained in the liquid, even with a Beltane opal concentration of only 4%, offered little scope for p.f.a. to produce a further reduction.

8.2.4 Statistical Interpretation of Chemical Analysis Data

The chemical analysis results from the main 3 x 4 x 4 x 2 factorial set of mixes are suitable for the same type of statistical treatment as was applied to the corresponding expansion measurements. To this end, analysis of variance computations were carried out on the alkali contents of the bars, the alkali contents of the liquids and the overall alkali content of the mortar bar/liquid system. The resulting ANOVA tables are presented in Tables 8.4 to 8.6.

The ANOVA tables are summarised in Table 8.7 using symbols to denote the significance level of the individual effects and interactions.

The summary shows that the four treatment factors employed in the experiments have highly significant main effects on all three alkali contents. This confirms the earlier discussion which highlighted the contribution made by the cement alkali content, the p.f.a. substitution level and the Pyrex glass concentration towards the alkali concentration in the experimental specimens.

It is also apparent from Table 8.7 that there is considerable interaction between the various treatment factors, the free water content being the notable exception. The lack of significant interactions with free water content probably indicates that the main effect attributable to this factor are not chemical effects but are simply caused by the physical changes in the density and permeability of the specimens associated with the different free water contents.

The first order interactions between the other three treatment factors, all of which have significant levels in excess of

90%, show that to a certain extent the effects of these factors are interdependent. This may have been anticipated for the interactions AB and AC which involve the cement alkali content, since this remains the major source of alkali in the specimens. It is reasonable to assume that the effect which p.f.a. had on the alkali content of the specimens would be partially dependent on the alkali content of the cement for which it was being substituted. Thus the interaction AB should be significant. It is also reasonable to assume that if Pyrex glass was to have an effect on the alkali content of the specimen, then the extent of the effect would depend on the alkali content of the corresponding Pyrex free mix. This is largely controlled by the alkali content of the cement and thus suggests a significant AC interaction.

The high significance of the BC interaction between the level of p.f.a. substitution and the Pyrex glass concentration cannot be explained in these terms. The occurrence of this interaction is thought to substantiate the description given in Section 8.2.2. Here the effect which p.f.a. had on the alkali concentration was said to be partially offset in mixes containing Pyrex glass because of a reduction in the effect which the Pyrex glass had on the alkali concentration in the absence of p.f.a. This is further evidence that the mechanism by which p.f.a. helps to inhibit the expansive A.S.R. occurring between the Pyrex glass and the OH^- is one of reaction and not simple dilution.

8.3 THE EXTENT OF THE P.F.A. REACTION

The published results of experimental work carried out on pastes of p.f.a. and calcium hydroxide (12) have shown that there is a close correspondence between the rates of dissolution of the p.f.a. and the liberation of alkali metal ions into solution. It has been suggested (94) that this behaviour also occurs in mortars which contain p.f.a., the alkali metal ions being released as the pozzolanic reaction proceeds. The results discussed in Section 8.2 further indicate that this is the case, and hence the results of the alkali analyses may provide a means of estimating the extent to which the p.f.a. has reacted.

For the mixes containing no reactive aggregate, calculations to estimate the percentage reaction of the p.f.a. are presented in two different forms in Tables 8.8 and 8.9. In the former, Table 8.8, the percentage reaction is calculated having made no allowance for the original acid soluble content of either the aggregate material or the p.f.a.. This is the technique that was employed in producing the information presented in an earlier publication (94). However, the results from the chemical analysis carried out on samples of the p.f.a. and the sand, which are given in Table 5.8, show there to be significant quantities of acid soluble alkalis in both these constituent materials. This may be relevant to the percentage reaction calculations, which are therefore repeated in Table 8.9 making full allowance for the original alkali content of both materials.

The results from the calculations show that the two computational procedures produce considerably differing values. However, both sets of figures show that there is a very high level of reaction. Considering the group of mixes 2000 to 2300, the appropriate results from Table 8.8 show that the reaction of the p.f.a. is estimated to be 94% at a replacement level of 15%, 88% at a replacement level of 30% and 84% at a replacement level of 45%. In contrast, the equivalent data from Table 8.9 shows the level of reaction of the p.f.a. to be 69%, 70% and 68% at replacement levels of 15%, 30% and 45% respectively. Thus, when the original acid soluble alkali contents of all the constituent materials are taken into consideration the estimated percentage reaction of the p.f.a. remains fairly steady at around 70% for all replacement levels up to the maximum used of 45%.

The true percentage reaction value is likely to fall within the range denoted by the two extreme values given above, the remainder of the figures obtained in Tables 8.8 and 8.9 being generally similar to those discussed in detail. The question of whether or not an allowance should be made for the original acid soluble alkali content of all the mix components has, at present, no clear cut answer. It is apparent, however, that the alkali content of the sand must be allowed for otherwise the p.f.a. is being considered to have 'released' the alkali coming from this source in addition to that actually emanating from itself. Again using the group of mixes 2000 to 2300, if an allowance is made for the sand only then the percentage reaction figures become 78% or 79% for all three cement replacement levels. Thus, it seems likely that for cement replacement levels of up to 45% a fairly constant level of reaction can be expected.

With respect to making an allowance for the original acid soluble alkali content of the p.f.a. itself, there is no evidence available from the current experiments to tip the balance either way. However, it is possible to surmise that the part of the p.f.a. which holds the acid soluble alkalis will be the most easily broken down component of its particle structure and, hence, the section most likely to react first. This hypothesis would suggest that the originally acid soluble alkalis should be retained in the calculations.

The estimation of the extent of the p.f.a. reaction is only intended to provide a pictorial representation of the actual situation which develops in the mortar bars. The values produced do indicate the considerable reaction which the p.f.a. undergoes, but they should be considered in conjunction with the possible inaccuracies inherent in the experimental procedures which are highlighted in the following Section 8.4.

8.4 DISCUSSION

It is important to reiterate at this point in the discussion the step-by-step approach which was adopted for the chemical analysis stage of the current investigation. To a certain extent, as mentioned in Chapter 7, the mortar bars were analysed at differing ages, and in some instances the liquid from the bottom of the storage container was not sampled at the same time as the bar for analysis was removed from the container. Moreover, as the experiments were not originally designed with the chemical analysis stage included, there was no check kept on the spillage of water from the storage containers during the expansion monitoring process and the amount of alkali retained by the blotting paper lining to the storage containers was not measured. An extra degree of uncertainty, therefore, exists in the measured amount of alkali leached from the bars and into the liquid.

Notwithstanding these reservations, in Section 8.2 above, the results from the chemical analyses indicate that where an effect was observed, the substitution of p.f.a. for part of the cement content consistently increased the alkali level in the mortar bar in comparison to the corresponding mix containing only Portland cement as the cementitious material. In addition, the p.f.a. was also found to decrease the amount of alkali leached from the bar, the combined effect being an increase in the amount of alkali in the overall system.

Despite this observation the results presented in Chapter 6 showed that for all Pyrex glass concentrations and for Beltane opal at the pessimum concentration, the use of p.f.a. caused a reduction in the observed expansion.

This further substantiates the opinion that p.f.a. does not act as a simple diluent of the alkali level and thereby contribute towards expansion reduction in reactive mixes. This being the case a more fundamental reaction process must be the mechanism of the p.f.a. effect. The calculations to estimate the extent of this reaction of the p.f.a. produced an indication that a minimum reaction level of 68% may be expected for cement replacement levels of up to 45%. This figure was produced by the most conservative method of calculation discussed in Section 8.3.

These observations also undermine the recent stipulation that the alkali content of the mortar or concrete, expressed in kilogrammes per cubic metre, is the criterion to use when judging whether a mix containing a suspect aggregate is potentially reactive (21, 48). As previously mentioned, a safe limit of 3.0kg Na_2O_{eq}/m^3 has been suggested for the acid soluble alkali content for concretes. The results of this investigation show that mixes containing a highly reactive material such as Pyrex glass can contain alkalis considerably in excess of this amount and yet remain below the expansion limit used to denote a 'reactive' mix in the A.S.T.M. mortar bar test. Conversely, mix 0031P exceeded the 12 month safe expansion limit but contained an alkali level of only 2.42kg Na_2O_{eq}/m^3.

Thus, it is clear that the suggested theoretical limit of 3.0kg Na_2O_{eq}/m^3 for a safe alkali level is not satisfactory. Further, when p.f.a. is included in the mix, the expansion observed is apparently unrelated to the alkali level of the mix since it has been shown that expansion decreases despite an increased alkali level. It would therefore appear that the alkali content of the mix in kg/m^3 is not a suitable criterion on which to base safety limits for minimising the risk of A.S.R.

TABLE 8.1 COMPARISON OF RESULTS FROM THE CHEMICAL ANALYSIS OF THE ALKALI CONTENT OF THE MORTAR BARS AND LIQUIDS WITH THE ALKALIS PROVIDED BY THE MIX CONSTITUENTS

SERIES 2, 3 x 4 x 4 x 2 FACTORIAL SET OF MIXES

MIX REF. NO.	ALKALI CONTENT OF SPECIMENS (kg Na_2O_{eq}/m^3)			ALKALI CONTENT FROM MIX CONSTITUENTS * (kg Na_2O_{eq}/m^3)				
	BAR	LIQUID*	BAR + LIQUID	CEMENT	P.F.A.	SAND	PYREX GLASS	Σ CONSTITUENTS
0000	1.83	0.39	2.22	2.84	-	0.42	-	3.26
0001	1.56	0.51	2.07	2.59	-	0.38	-	2.97
0010P	2.39	0.73	3.12	2.76	-	0.27	0.59	3.62
0011P	1.92	0.91	2.83	2.53	-	0.25	0.54	3.32
0020P	3.41	0.88	4.29	2.69	-	0.13	1.14	3.96
0021P	1.98	1.29	3.27	2.47	-	0.12	1.05	3.64
0030P	3.28	0.62	3.90	2.62	-	-	1.67	4.29
0031P	3.19	0.73	3.92	2.42	-	-	1.54	3.96
0100	3.98	0.71	4.69	2.29	0.74	0.42	-	3.45
0101	3.76	0.81	4.57	2.09	0.68	0.38	-	3.15
0110P	4.53	0.76	5.29	2.23	0.72	0.27	0.59	3.81
0111P	4.03	0.98	5.01	2.04	0.66	0.25	0.54	3.49
0120P	4.84	0.87	5.71	2.17	0.71	0.13	1.14	4.15
0121P	4.01	1.12	5.13	1.99	0.65	0.12	1.05	3.81
0130P	5.60	0.51	6.11	2.11	0.69	-	1.67	4.47
0131P	4.64	0.88	5.52	1.95	0.63	-	1.53	4.11
0200	5.79	0.60	6.39	1.80	1.40	0.42	-	3.62
0201	5.32	0.67	5.99	1.65	1.29	0.38	-	3.32
0210P	6.00	0.63	6.63	1.75	1.37	0.27	0.59	3.98
0211P	5.61	0.96	6.57	1.61	1.25	0.25	0.54	3.65
0220P	6.09	0.56	6.65	1.71	1.33	0.13	1.14	4.31
0221P	5.70	0.95	6.65	1.56	1.22	0.12	1.05	3.95
0230P	6.07	0.54	6.61	1.66	1.29	-	1.67	4.62
0231P	5.64	0.83	6.47	1.53	1.19	-	1.54	4.26
0300	6.14	0.45	6.59	1.35	2.02	0.42	-	3.79
0301	6.07	0.51	6.58	1.23	1.84	0.38	-	3.45
0310P	6.30	0.46	6.76	1.32	1.97	0.27	0.59	4.15
0311P	6.20	0.49	6.69	1.21	1.80	0.25	0.54	3.80
0320P	6.34	0.47	6.81	1.28	1.91	0.13	1.14	4.46
0321P	5.95	0.53	6.48	1.17	1.76	0.12	1.05	4.10
0330P	6.42	0.46	6.88	1.25	1.86	-	1.67	4.78
0331P	6.18	0.54	6.72	1.15	1.71	-	1.53	4.39

Note:- * = expressed with respect to the mass of the bar

TABLE 8.1 Continued

MIX REF. NO.	ALKALI CONTENT OF SPECIMENS (kg Na_2O_{eq}/m^3)			ALKALI CONTENT FROM MIX CONSTITUENTS * (kg Na_2O_{eq}/m^3)				
	BAR	LIQUID*	BAR + LIQUID	CEMENT	P.F.A.	SAND	PYREX GLASS	Σ CONSTITUENTS
1000	3.04	0.84	3.88	4.42	-	0.41	-	4.83
1001	2.99	0.88	3.87	4.05	-	0.38	-	4.43
1010P	3.70	0.77	4.47	4.31	-	0.27	0.58	5.16
1011P	3.13	0.99	4.12	3.95	-	0.25	0.53	4.73
1020P	5.25	0.57	5.82	4.20	-	0.13	1.14	5.47
1021P	4.75	0.79	5.54	3.85	-	0.12	1.04	5.01
1030P	5.66	0.42	6.08	4.09	-	-	1.66	5.75
1031P	5.42	0.46	5.88	3.76	-	-	1.53	5.29
1100	5.47	0.77	6.24	3.58	0.74	0.42	-	4.74
1101	4.56	0.88	5.44	3.26	0.67	0.38	-	4.31
1110P	6.09	0.74	6.83	3.48	0.72	0.27	0.59	5.06
1111P	4.73	0.88	5.61	3.19	0.66	0.25	0.54	4.64
1120P	6.35	0.64	6.99	3.38	0.70	0.13	1.14	5.35
1121P	6.03	0.77	6.80	3.11	0.65	0.12	1.05	4.93
1130P	7.13	0.54	7.67	3.30	0.68	-	1.66	5.64
1131P	6.37	0.49	6.86	3.04	0.63	-	1.53	5.20
1200	6.55	0.69	7.24	2.82	1.40	0.42	-	4.64
1201	5.93	0.79	6.72	2.57	1.28	0.38	-	4.23
1210P	6.51	0.68	7.19	2.74	1.36	0.27	0.59	4.96
1211P	5.96	0.89	6.85	2.51	1.24	0.25	0.54	4.54
1220P	7.38	0.59	7.97	2.66	1.33	0.13	1.14	5.26
1221P	6.81	0.99	7.80	2.45	1.22	0.12	1.04	4.83
1230P	7.77	0.51	8.28	2.59	1.29	-	1.66	5.54
1231P	6.92	0.74	7.66	2.39	1.19	-	1.53	5.11
1300	6.73	0.48	7.21	2.11	2.02	0.42	-	4.55
1301	6.57	0.74	7.31	1.92	1.84	0.38	-	4.14
1310P	6.75	0.65	7.40	2.05	1.96	0.27	0.59	4.87
1311P	7.17	0.42	7.59	1.88	1.80	0.25	0.54	4.47
1320P	7.35	0.73	8.08	2.00	1.91	0.13	1.14	5.18
1321P	6.92	0.73	7.65	1.84	1.75	0.12	1.05	4.76
1330P	7.51	0.57	8.08	1.94	1.86	-	1.67	5.47
1331P	6.98	0.54	7.52	1.79	1.71	-	1.53	5.03

Note:- * = expressed with respect to the mass of the bar

TABLE 8.1 Continued

MIX REF. NO.	ALKALI CONTENT OF SPECIMENS (kg Na_2O_{eq}/m^3)			ALKALI CONTENT FROM MIX CONSTITUENTS * (kg Na_2O_{eq}/m^3)				
	BAR	LIQUID*	BAR + LIQUID	CEMENT	P.F.A.	SAND	PYREX GLASS	Σ CONSTITUENTS
2000	5.53	1.62	7.15	7.16	-	0.41	-	7.57
2001	4.27	1.60	5.87	6.56	-	0.38	-	6.94
2010P	6.94	0.93	7.87	6.98	-	0.27	0.58	7.83
2011P	6.40	1.02	7.42	6.40	-	0.25	0.53	7.18
2020P	6.49	0.62	7.11	6.81	-	0.13	1.13	8.07
2021P	7.66	0.65	8.31	6.25	-	0.12	1.04	7.41
2030P	6.51	0.51	7.02	6.63	-	-	1.66	8.29
2031P	5.72	0.40	6.12	6.10	-	-	1.52	7.62
2100	7.14	1.03	8.17	5.79	0.74	0.41	-	6.94
2101	6.24	1.20	7.44	5.30	0.67	0.38	-	6.35
2110P	7.34	0.83	8.17	5.64	0.72	0.27	0.58	7.21
2111P	7.39	1.19	8.58	5.17	0.66	0.25	0.53	6.61
2120P	8.85	0.58	9.43	5.49	0.70	0.13	1.13	7.45
2121P	8.13	0.84	8.97	5.04	0.65	0.12	1.04	6.85
2130P	8.74	liquid	lost	5.35	0.68	-	1.66	7.69
2131P	7.98	0.67	8.65	4.94	0.63	-	1.53	7.10
2200	7.93	0.81	8.74	4.56	1.39	0.42	-	6.37
2201	7.05	1.16	8.21	4.17	1.28	0.38	-	5.83
2210P	7.89	0.80	8.69	4.45	1.36	0.27	0.58	6.66
2211P	7.72	0.94	8.66	4.07	1.24	0.25	0.54	6.10
2220P	8.35	0.72	9.07	4.33	1.33	0.13	1.14	6.93
2221P	7.60	0.75	8.35	3.98	1.22	0.12	1.04	6.36
2230P	8.30	0.61	8.91	4.21	1.29	-	1.66	7.16
2231P	7.96	0.64	8.60	3.87	1.18	-	1.53	6.58
2300	8.47	0.68	9.15	3.42	2.01	0.42	-	5.85
2301	7.69	0.68	8.37	3.12	1.84	0.38	-	5.34
2310P	7.60	0.45	8.05	3.32	1.96	0.27	0.58	6.13
2311P	6.83	0.73	7.56	3.04	1.79	0.25	0.53	5.61
2320P	8.71	0.58	9.29	3.24	1.91	0.13	1.14	6.42
2321P	7.74	0.68	8.42	2.98	1.75	0.12	1.05	5.90
2330P	7.98	0.47	8.45	3.16	1.86	-	1.67	6.69
2331P	8.22	0.76	8.98	2.90	1.71	-	1.53	6.14

Note:- * = expressed with respect to the mass of the bar

TABLE 8.2 COMPARISON OF RESULTS FROM THE CHEMICAL ANALYSIS OF THE ALKALI CONTENT OF THE MORTAR BARS AND LIQUIDS WITH THE ALKALIS PROVIDED BY THE MIX CONSTITUENTS

SERIES 2, EXTRA PYREX SET OF MIXES

MIX REF. NO.	ALKALI CONTENT OF SPECIMENS (kg Na_2O_{eq}/m^3)			ALKALI CONTENT FROM MIX CONSTITUENTS * (kg Na_2O_{eq}/m^3)				
	BAR	LIQUID*	BAR + LIQUID	CEMENT	P.F.A.	SAND	PYREX GLASS	Σ CONSTITUENTS
2032P	8.48	0.60	9.08	6.82	-	-	1.71	8.53
2033P	8.51	0.57	9.08	6.45	-	-	1.61	8.06
2034P	8.29	0.46	8.75	6.27	-	-	1.57	7.84
2035P	7.53	0.58	8.11	5.94	-	-	1.49	7.43
2041P	4.37	1.47	5.84	6.53	-	0.36	0.07	6.96
2051P	4.42	1.42	5.84	6.53	-	0.35	0.13	7.01
2061P	5.03	1.58	6.61	6.49	-	0.33	0.20	7.02
2071P	4.71	1.34	6.05	6.48	-	0.31	0.26	7.05
2431P	9.37	0.54	9.91	5.80	0.42	-	1.45	7.67
2531P	9.27	0.72	9.99	5.53	0.80	-	1.38	7.71
2631P	9.29	0.93	10.22	5.28	1.13	-	1.32	7.73
2731P	9.27	0.91	10.18	5.05	1.45	-	1.26	7.76
3031P	3.85	0.55	4.40	2.68	-	-	2.27	4.95
4031P	4.32	0.40	4.72	3.23	-	-	2.15	5.38
5031P	5.21	0.52	5.73	3.74	-	-	2.04	5.78
6031P	6.70	0.61	7.31	4.64	-	-	1.85	6.49

Note:- * = expressed with respect to the mass of the bar

TABLE 8.3 COMPARISON OF RESULTS FROM THE CHEMICAL ANALYSIS OF THE ALKALI CONTENT OF THE MORTAR BARS AND LIQUIDS WITH THE ALKALIS PROVIDED BY THE MIX CONSTITUENTS

SERIES 2, BELTANE OPAL SET OF MIXES

MIX REF. NO.	ALKALI CONTENT OF SPECIMENS (Na_2O_{eq} kg/m^3)			ALKALI CONTENT FROM MIX CONSTITUENTS * (Na_2O_{eq} kg/m^3)				
	BAR	LIQUID*	BAR + LIQUID	CEMENT	P.F.A.	SAND	PYREX GLASS	Σ CONSTITUENTS
2001	4.27	1.601	5.871	6.56	-	0.38	-	6.94
2011B	6.63	0.097	6.727	6.27	-	0.24	0.12	6.63
2021B	6.01	0.085	6.095	6.01	-	0.12	0.23	6.36
2031B	5.47	0.118	5.588	5.77	-	-	0.33	6.10
2041B	5.53	0.563	6.093	6.52	-	0.36	0.02	6.90
2051B	6.15	0.312	6.462	6.48	-	0.34	0.03	6.85
2061B	6.14	0.191	6.331	6.45	-	0.33	0.04	6.82
2071B	6.41	0.194	6.604	6.42	-	0.31	0.06	6.79
2301	7.69	0.676	8.366	3.12	1.84	0.38	-	5.34
2311B	8.29	0.215	8.505	2.98	1.76	0.24	0.12	5.10
2321B	7.59	0.134	7.724	2.87	1.68	0.12	0.23	4.90
2331B	7.72	0.109	7.829	2.75	1.61	-	0.33	4.69
2341B	7.49	0.476	7.966	3.11	1.83	0.36	0.02	5.32
2351B	7.90	0.307	8.207	3.09	1.81	0.35	0.03	5.28
2361B	7.84	0.297	8.137	3.08	1.81	0.33	0.05	5.27
2371B	7.99	0.249	8.239	3.07	1.80	0.31	0.06	5.24

Note:- * = expressed with respect to the mass of the bar

```
PAGE   9     ALKALI CONTENT RESULTS , ANOVAS & F-TESTS  SERIES 2
ANALYSIS OF VARIANCE FOR  1-ST
DEPENDENT VARIABLE - NA2OB

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

       SOURCE       EFFECT            SUM OF     DEGREES OF       MEAN           F       TAIL
                                      SQUARES     FREEDOM        SQUARE                  PROB.

       MEAN                         3300.22240        1        3300.22240    20102.09    0.
       CEMENT        A               107.35109        2          53.67554      326.94    0.
       PFA           B               110.39031        3          36.79694      224.14    0.
       AGGRGATE      C                16.40247        3           5.46749       33.30    0.0000
       FREEWCON      D                 5.26260        1           5.26260       32.06    0.0000
       CP            AB               10.84227        6           1.80705       11.01    0.0000
       CA            AC                2.85821        6           0.47637        2.90    0.0390
       PA            BC                5.51241        9           0.61249        3.73    0.0094
       CF            AD                0.00968        2           0.00484        0.03    0.9710
       PF            BD                0.35211        3           0.11737        0.71    0.5565
       AF            CD                0.06548        3           0.02183        0.13    0.9391
       CPA           ABC               4.30106       18           0.23895        1.46    0.2220
       CPF           ABD               0.38996        6           0.06499        0.40    0.8715
       CAF           ACD               0.80689        6           0.13448        0.82    0.5702
       PAF           BCD               0.27141        9           0.03016        0.18    0.9932
  1    ERROR                           2.79094       17           0.16417
```

TABLE 8.4 ANOVA TABLE FOR CHEMICAL ANALYSIS RESULTS - BARS ONLY

```
PAGE  10     ALKALI CONTENT RESULTS , ANOVAS & F-TESTS  SERIES 2
ANALYSIS OF VARIANCE FOR  2-ND
DEPENDENT VARIABLE - NA2OL

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

       SOURCE        EFFECT             SUM OF     DEGREES OF       MEAN          F       TAIL
                                        SQUARES     FREEDOM        SQUARE                 PROB.

       MEAN                            48.46124         1        48.46124     5463.44    0.
       CEMENT          A                0.20428         2         0.10214       11.52    0.0007
       PFA             B                0.78014         3         0.26005       29.32    0.0000
       AGGRGATE        C                0.69605         3         0.23202       26.16    0.0000
       FREEWCON        D                0.50074         1         0.50074       56.45    0.0000
       CP              AB               0.16177         6         0.02696        3.04    0.0330
       CA              AC               1.06500         6         0.17750       20.01    0.0000
       PA              BC               0.40001         9         0.04445        5.01    0.0021
       CF              AD               0.02612         2         0.01306        1.47    0.2572
       PF              BD               0.07667         3         0.02556        2.88    0.0663
       AF              CD               0.02167         3         0.00722        0.81    0.5036
       CPA             ABC              0.83751        18         0.04653        5.25    0.0006
       CPF             ABD              0.09778         6         0.01630        1.84    0.1513
       CAF             ACD              0.05257         6         0.00876        0.99    0.4639
       PAF             BCD              0.04760         9         0.00529        0.60    0.7831
 1     ERROR                            0.15079        17         0.00887
```

TABLE 8.5   ANOVA TABLE FOR CHEMICAL ANALYSIS RESULTS - LIQUIDS ONLY

```
PAGE  11     ALKALI CONTENT RESULTS , ANOVAS & F-TESTS  SERIES 2
ANALYSIS OF VARIANCE FOR  3-RD
DEPENDENT VARIABLE - NA2OBL

THE HIGHEST ORDER INTERACTION IN EACH TABLE HAS BEEN REMOVED FROM THE MODEL DUE TO ONE SUBJECT PER CELL.

      SOURCE       EFFECT            SUM OF     DEGREES OF       MEAN          F         TAIL
                                    SQUARES      FREEDOM        SQUARE                   PROB.

      MEAN                        4148.51582        1         4148.51582    26130.27    0.
      CEMENT         A             115.54290        2           57.77145      363.89    0.
      PFA            B              98.78341        3           32.92780      207.40    0.
      AGGRGATE       C              11.65966        3            3.88655       24.48    0.0000
      FREEWCON       D               2.51668        1            2.51668       15.85    0.0010
      CP             AB             11.04914        6            1.84152       11.60    0.0000
      CA             AC              2.38920        6            0.39820        2.51    0.0634
      PA             BC              3.71499        9            0.41278        2.60    0.0429
      CF             AD              0.06716        2            0.03358        0.21    0.8115
      PF             BD              0.13658        3            0.04553        0.29    0.8343
      AF             CD              0.10760        3            0.03587        0.23    0.8771
      CPA            ABC             4.28278       18            0.23793        1.50    0.2047
      CPF            ABD             0.35379        6            0.05896        0.37    0.8871
      CAF            ACD             0.72995        6            0.12166        0.77    0.6064
      PAF            BCD             0.38531        9            0.04281        0.27    0.9747
  1   ERROR                          2.69897       17            0.15876

NUMBER OF INTEGER WORDS OF STORAGE USED IN PRECEDING    PROBLEM   12648
CPU TIME USED      53.153 SECONDS
```

TABLE 8.6 ANOVA TABLE FOR CHEMICAL ANALYSIS RESULTS - BARS + LIQUIDS

TABLE 8.7 SUMMARY OF ANOVA TABLES FOR CHEMICAL ANALYSIS RESULTS

	Source of Variation	Significance level of effect		
		Alkali Content of Bar	**Alkali Content of Liquid**	**Alkali Content of Overall System (Bar + Liquid)**
MAIN EFFECTS	Cement -A	*	*	*
	P.f.a. -B	*	*	*
	Aggregate -C	*	*	*
	Free water content -D	*	*	*
1st ORDER INTER-ACTIONS	AB	*	***	*
	AC	***	*	****
	BC	**	**	***
	AD	0	0	0
	BD	0	***	0
	CD	0	0	0
2nd ORDER INTER-ACTIONS	ABC	0	0	0
	ABD	0	*	0
	ACD	0	0	0
	BCD	0	0	0

Notes:- * = Tail probability ⩽ 0.0010 ; Significance level ⩾ 99.9%
** = 0.0010 < Tail probability ⩽ 0.0100 ; 99.9% > Significance level ⩾ 99%
*** = 0.0100 < Tail probability ⩽ 0.0500 ; 99% > Significance level ⩾ 95%
**** = 0.0500 < Tail probability ⩽ 0.1000 ; 95% > Significance level ⩾ 90%
0 = 0.1000 < Tail probability ; 90% > Significance level

TABLE 8.8 ESTIMATION OF PERCENTAGE REACTION OF P.F.A. FOR MIXES CONTAINING NO REACTIVE AGGREGATE, NO ALLOWANCE MADE FOR ORIGINAL ALKALI CONTENT OF SAND OR P.F.A.

Mix Ref No.	Alkali Content (kg Na_2O_{eq}/m^3)						Percentage Reaction = C/D x 100%
	Bar Alkali Content	Liquid Alkali Content *	A Bar + Liquid	B Alkali Content From Cement *	C A - B	D Total Alkali Content From P.f.a. *	
0000	1.83	0.39	2.22	2.84	-0.62	-	-
0100	3.98	0.71	4.69	2.29	2.40	2.53	95%
0200	5.79	0.60	6.39	1.80	4.59	4.79	96%
0300	6.14	0.45	6.59	1.35	5.24	6.89	76%
0001	1.56	0.51	2.07	2.59	-0.52	-	-
0101	3.76	0.81	4.57	2.09	2.48	2.32	107%
0201	5.32	0.67	5.99	1.65	4.34	4.39	99%
0301	6.07	0.51	6.58	1.23	5.35	6.29	85%
1000	3.04	0.84	3.88	4.42	-0.54	-	-
1100	5.47	0.77	6.24	3.58	2.66	2.53	105%
1200	6.55	0.69	7.24	2.82	4.42	4.79	92%
1300	6.73	0.48	7.21	2.11	5.10	6.89	74%
1001	2.99	0.88	3.87	4.05	-0.18	-	-
1101	4.56	0.88	5.44	3.26	2.18	2.30	95%
1201	5.93	0.79	6.72	2.57	4.15	4.36	95%
1301	6.57	0.74	7.31	1.92	5.39	6.29	86%
2000	5.53	1.62	7.15	7.16	-0.01	-	-
2100	7.14	1.03	8.17	5.79	2.38	2.53	94%
2200	7.93	0.81	8.74	4.56	4.18	4.76	88%
2300	8.47	0.68	9.15	3.42	5.73	6.86	84%
2001	4.27	1.60	5.87	6.56	-0.69	-	-
2101	6.24	1.20	7.44	5.30	2.14	2.30	93%
2201	7.05	1.16	8.21	4.17	4.04	4.36	93%
2301	7.69	0.68	8.37	3.12	5.25	6.29	83%

Note: * = expressed with respect to the mass of the bar.

TABLE 8.9 ESTIMATION OF PERCENTAGE REACTION OF P.F.A. FOR MIXES CONTAINING NO REACTIVE AGGREGATE, FULL ALLOWANCE MADE FOR ORIGINAL ACID SOLUBLE ALKALI CONTENT OF SAND AND P.F.A.

MIX REF NO.	ALKALI CONTENT (kg Na_2O_{eq}/m^3)										PERCENTAGE REACTION = C/Ex100%
	BAR ALKALI CONTENT	LIQUID ALKALI CONTENT *	A	b_1	b_2	b_3	B	C	D	E	
			BAR + LIQUID	ALKALI CONTENT FROM CEMENT *	ALKALI CONTENT FROM P.F.A *	ALKALI CONTENT FROM SAND *	b	A - B	TOTAL ALKALI CONTENT FROM PFA *	D - b2	
0000	1.83	0.39	2.22	2.84	-	0.42	3.26	-1.04	-	-	-
0100	3.98	0.71	4.69	2.29	0.74	0.42	3.45	1.24	2.53	1.79	69%
0200	5.79	0.60	6.39	1.80	1.40	0.42	3.62	2.77	4.79	3.39	82%
0300	6.14	0.45	6.59	1.35	2.02	0.42	3.79	2.80	6.89	4.87	57%
0001	1.56	0.51	2.07	2.59	-	0.38	2.97	-0.90	-	-	-
0101	3.76	0.81	4.57	2.09	0.68	0.38	3.15	1.42	2.32	1.64	87%
0201	5.32	0.67	5.99	1.65	1.29	0.38	3.32	2.67	4.39	3.10	86%
0301	6.07	0.51	6.58	1.23	1.84	0.38	3.45	3.13	6.29	4.45	70%
1000	3.04	0.84	3.88	4.42	-	0.41	4.83	-0.95	-	-	-
1100	5.47	0.77	6.24	3.58	0.74	0.42	4.74	1.50	2.53	1.79	84%
1200	6.55	0.69	7.24	2.82	1.40	0.42	4.64	2.60	4.79	3.39	77%
1300	6.73	0.48	7.21	2.11	2.02	0.42	4.55	2.66	6.89	4.87	55%
1001	2.99	0.88	3.87	4.05	-	0.38	4.43	-0.56	-	-	-
1101	4.56	0.88	5.44	3.26	0.67	0.38	4.31	1.13	2.30	1.63	69%
1201	5.93	0.79	6.72	2.57	1.28	0.38	4.23	2.49	4.36	3.08	81%
1301	6.57	0.74	7.31	1.92	1.84	0.38	4.14	3.17	6.29	4.45	71%
2000	5.53	1.62	7.15	7.16	-	0.41	7.57	-0.42	-	-	-
2100	7.14	1.03	8.17	5.79	0.74	0.41	6.94	1.23	2.53	1.79	69%
2200	7.93	0.81	8.74	4.56	1.39	0.42	6.37	2.37	4.76	3.37	70%
2300	8.47	0.68	9.15	3.42	2.01	0.42	5.85	3.30	6.86	4.85	68%
2001	4.27	1.60	5.87	6.56	-	0.38	6.94	-1.07	-	-	-
2101	6.24	1.20	7.44	5.30	0.67	0.38	6.35	1.09	2.30	1.63	67%
2201	7.05	1.16	8.21	4.17	1.28	0.38	5.83	2.38	4.36	3.08	77%
2301	7.69	0.68	8.37	3.12	1.84	0.38	5.34	3.03	6.29	4.45	68%

Note:- * = expressed with respect to the mass of the bar

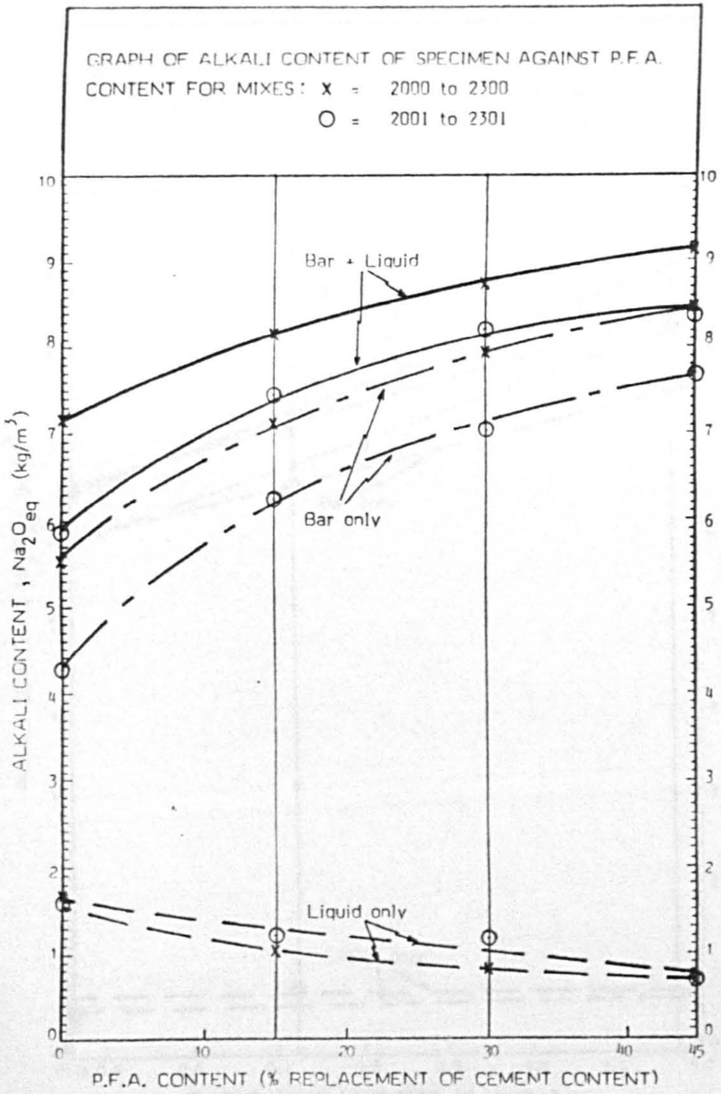


Figure 8.1

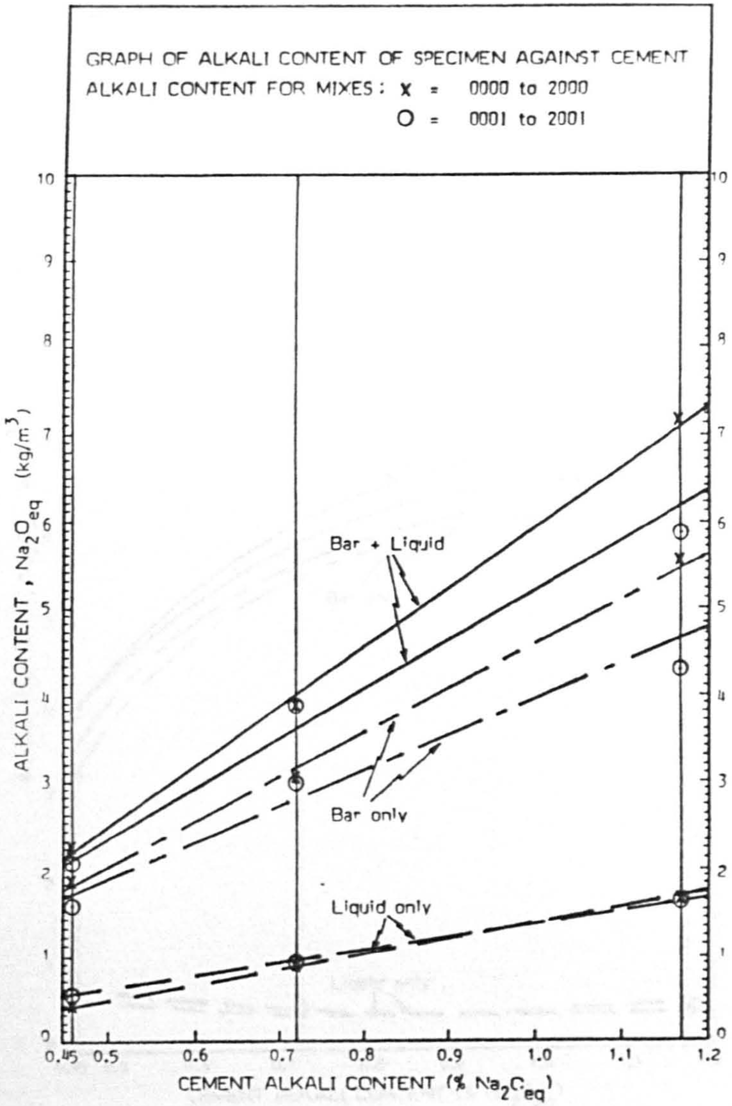


Figure 8.2

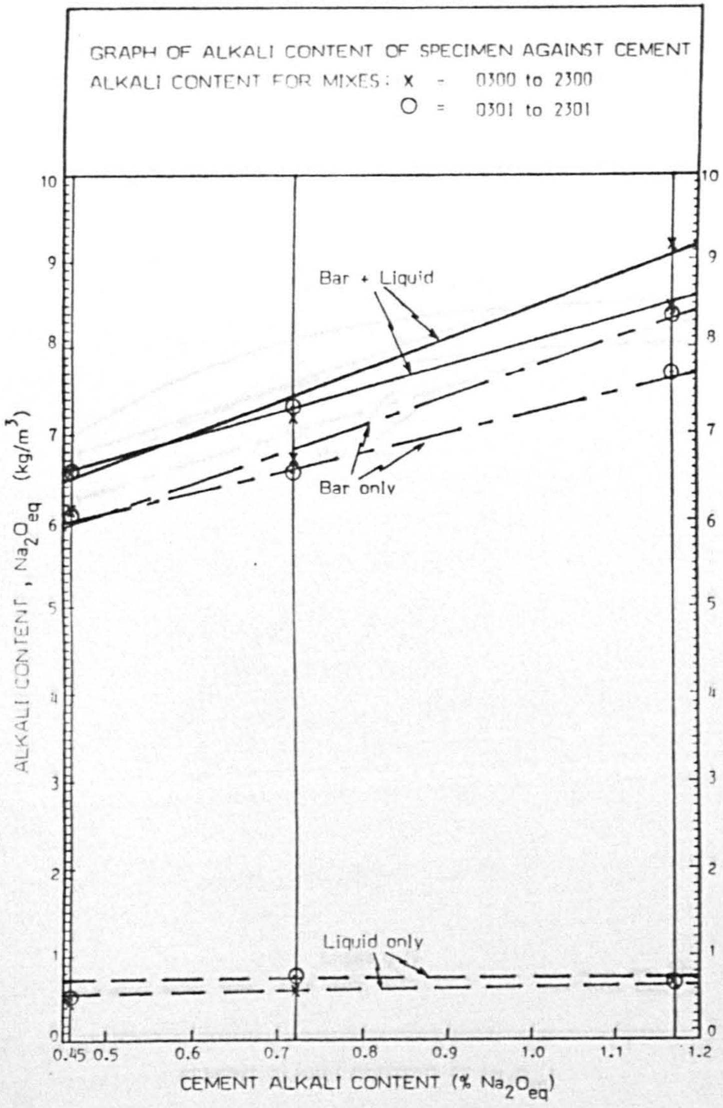


Figure 8.3

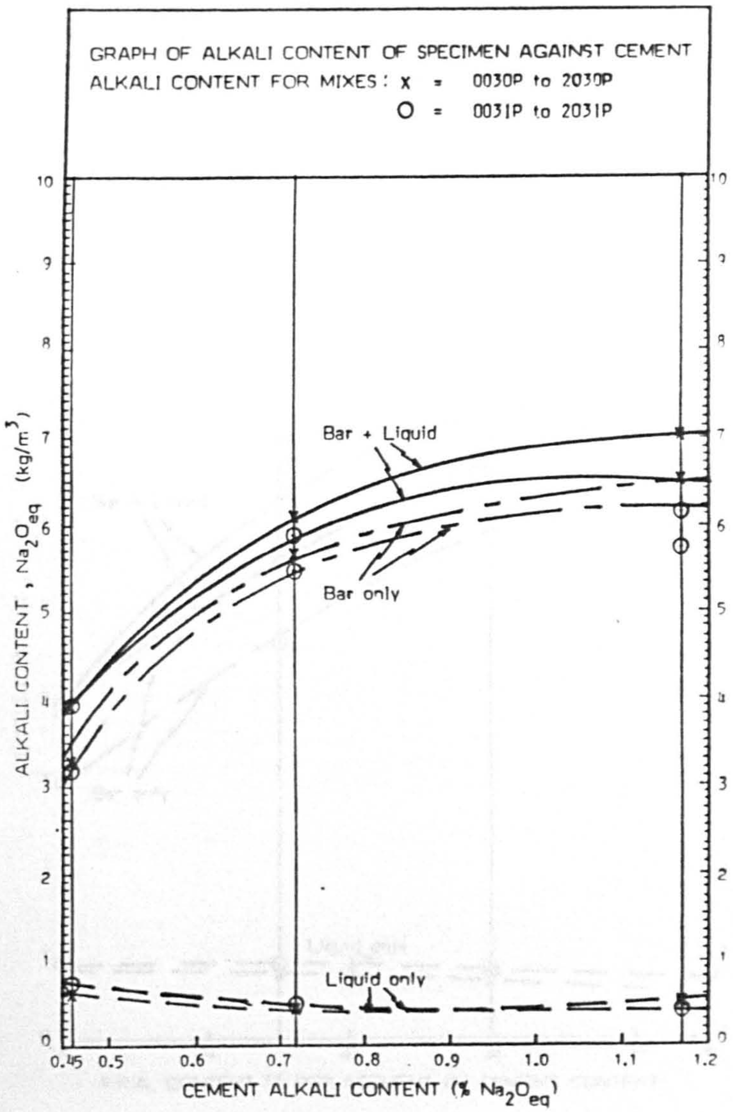


Figure 8.4

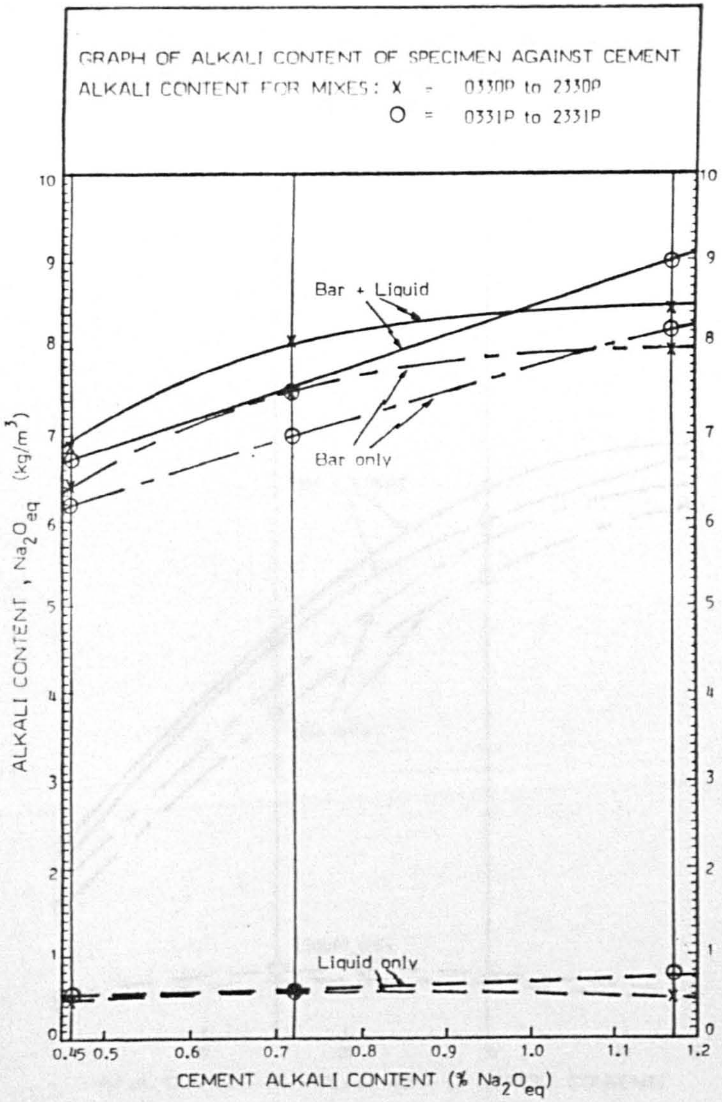


Figure 8.5

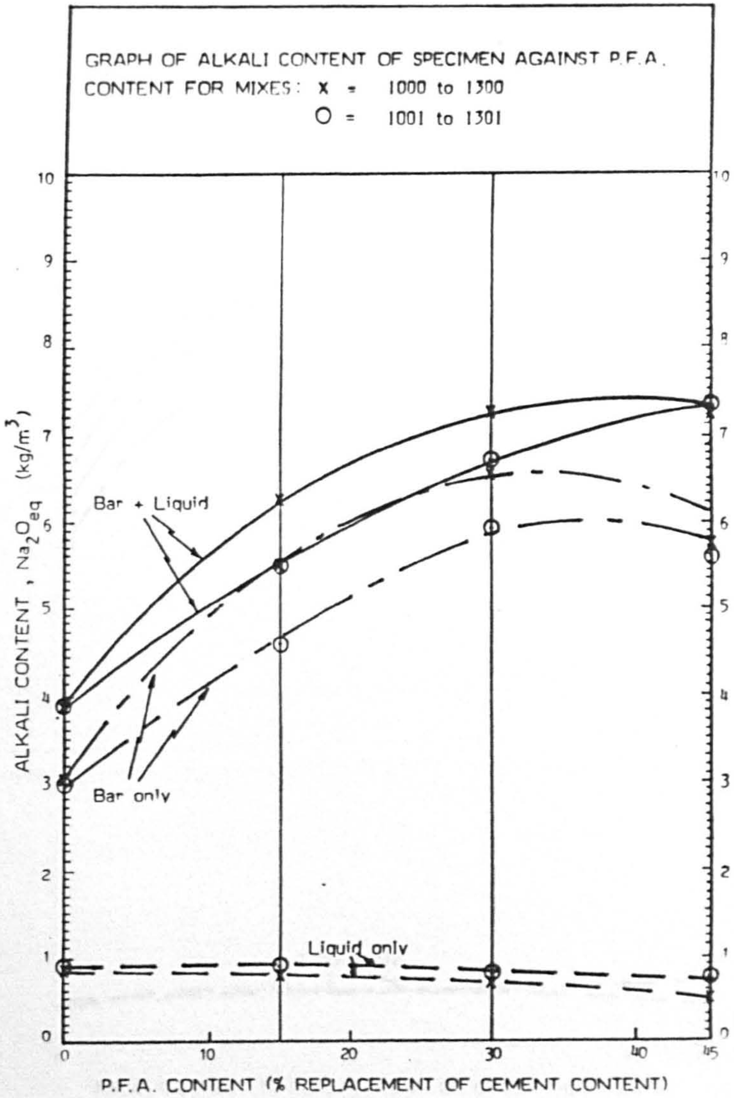
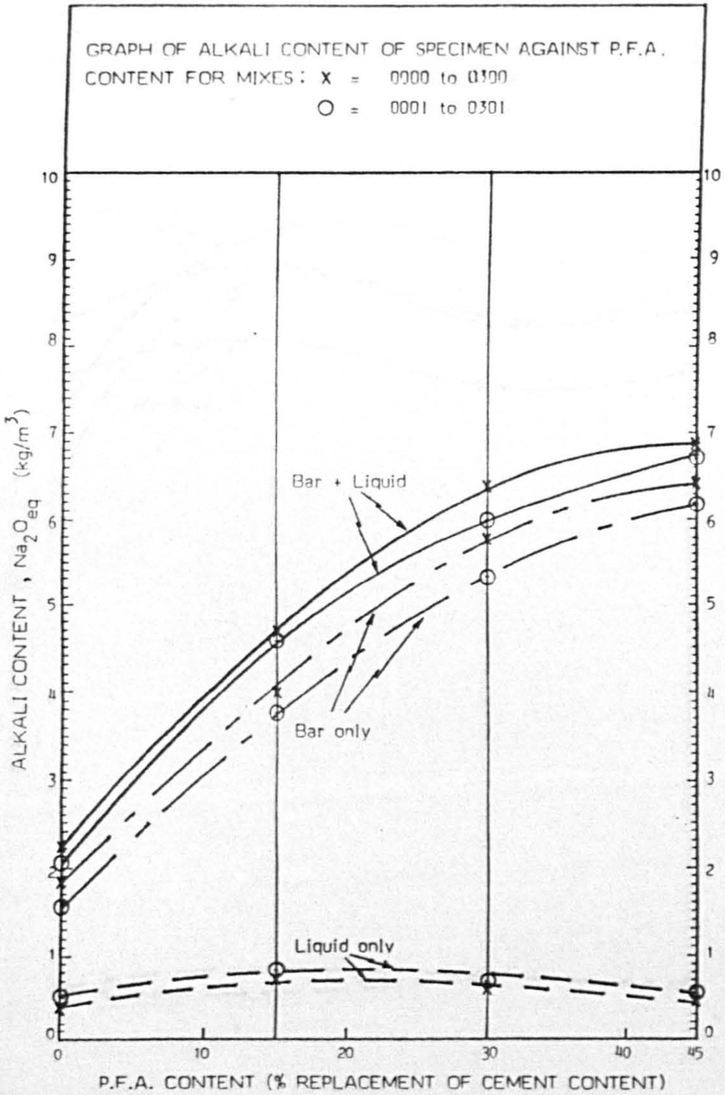


Figure 8.6



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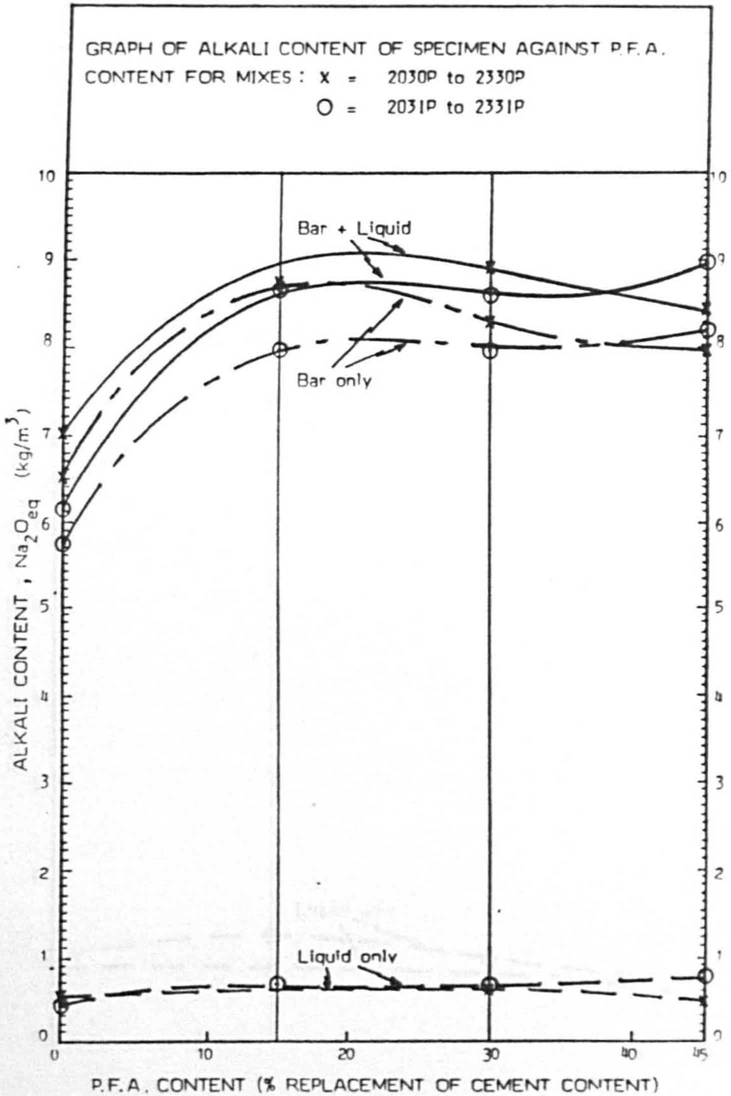

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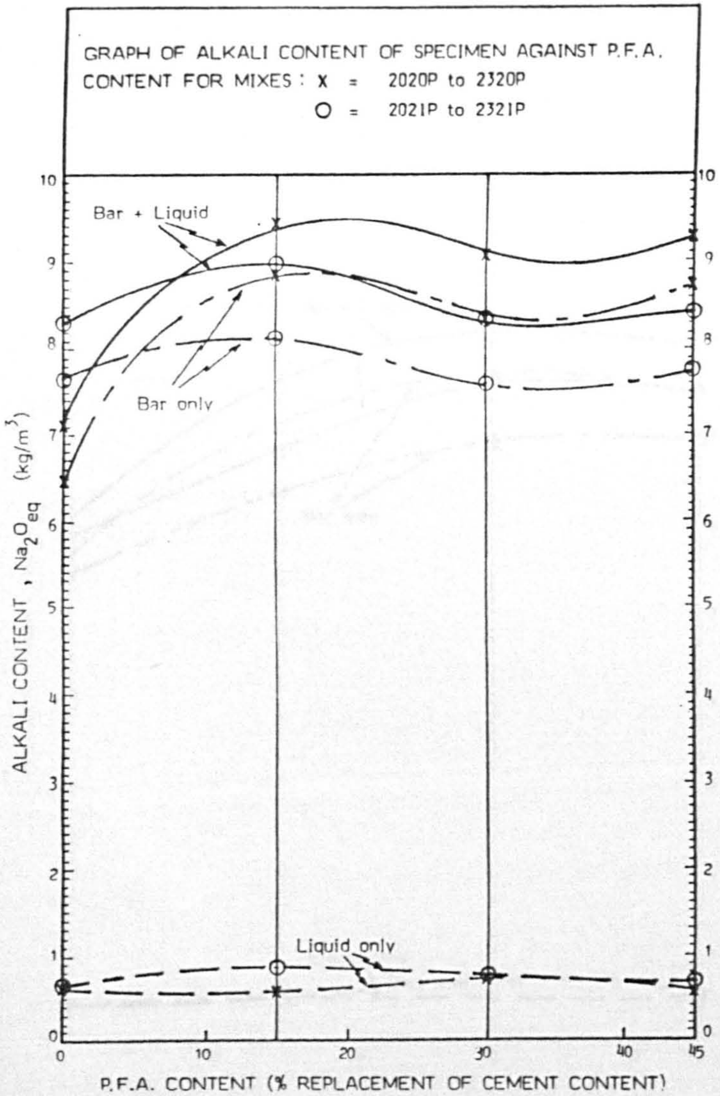

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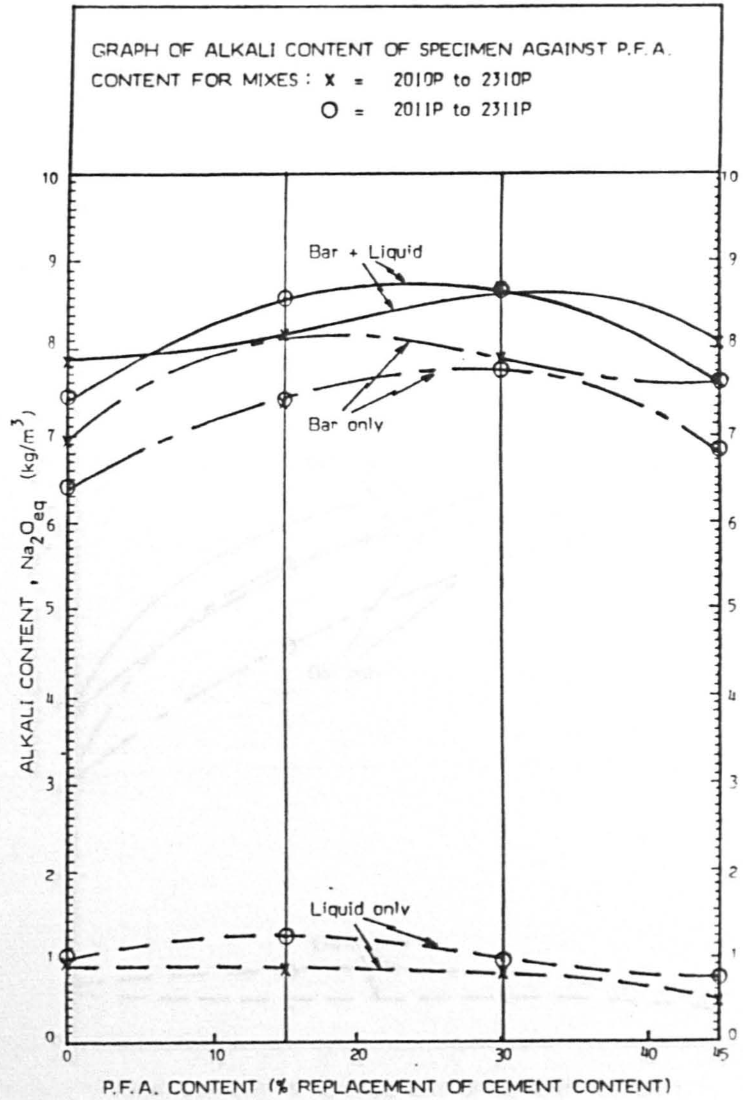

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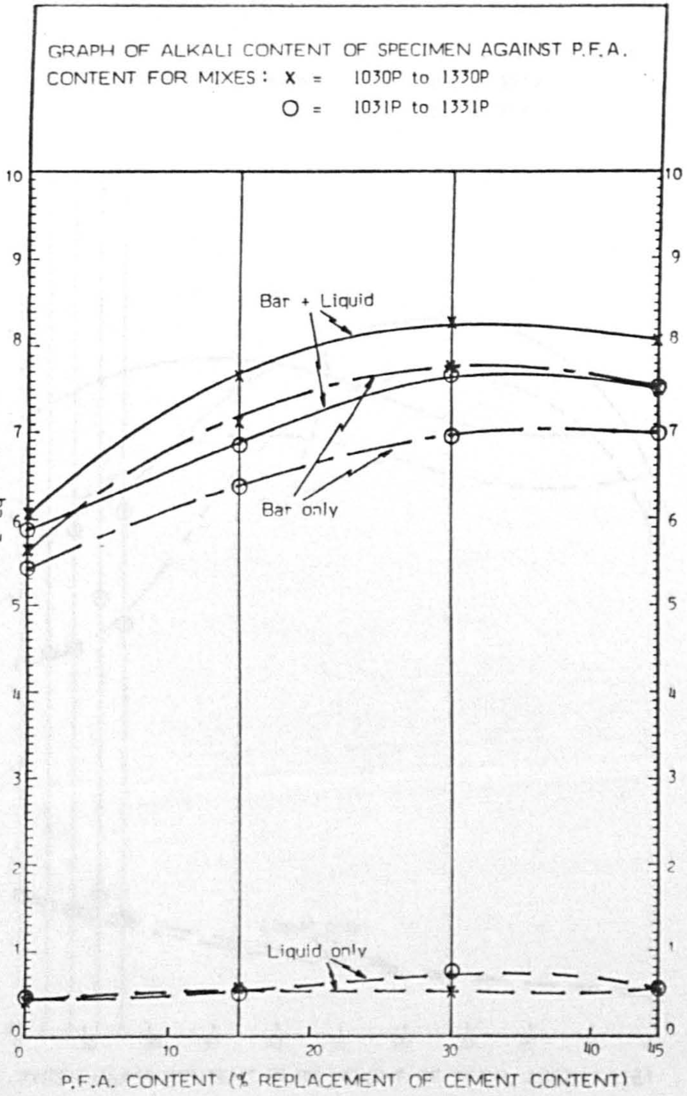


Figure 8.11

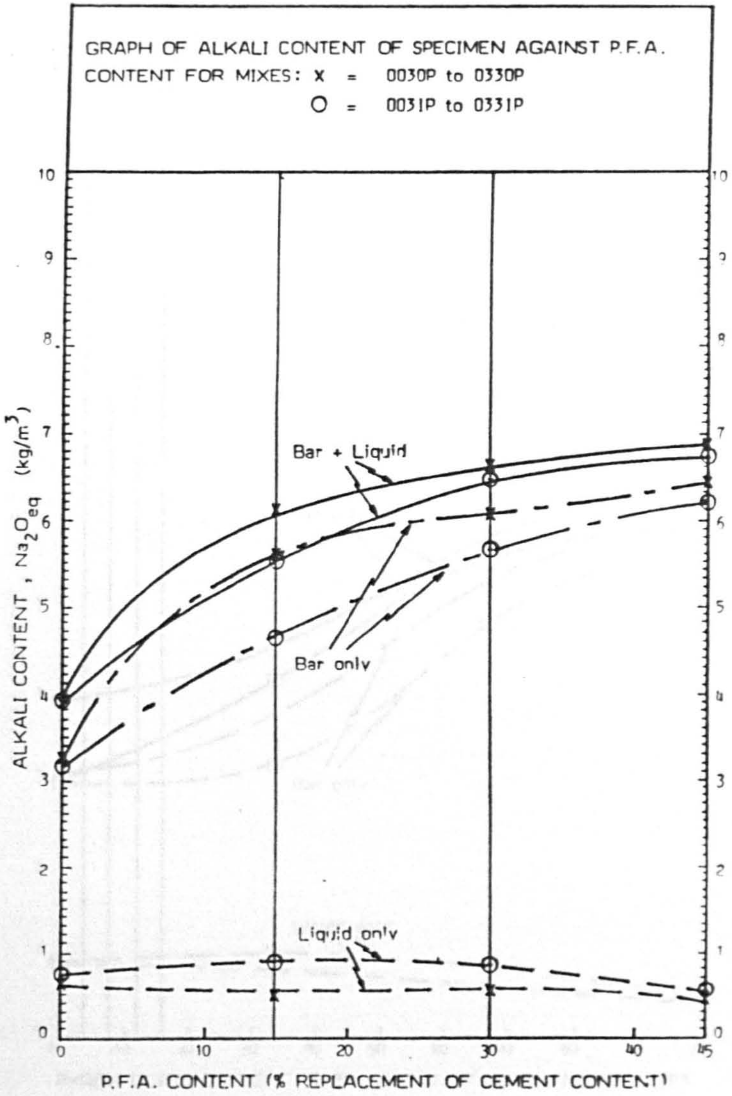


Figure 8.12

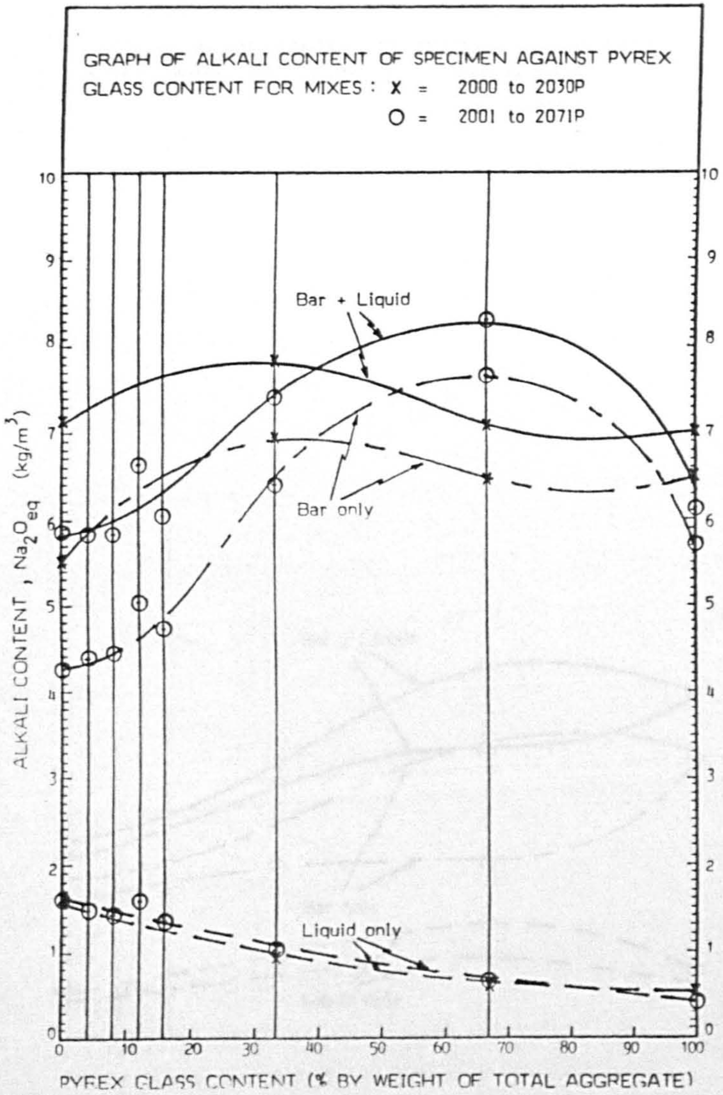


Figure 8.13

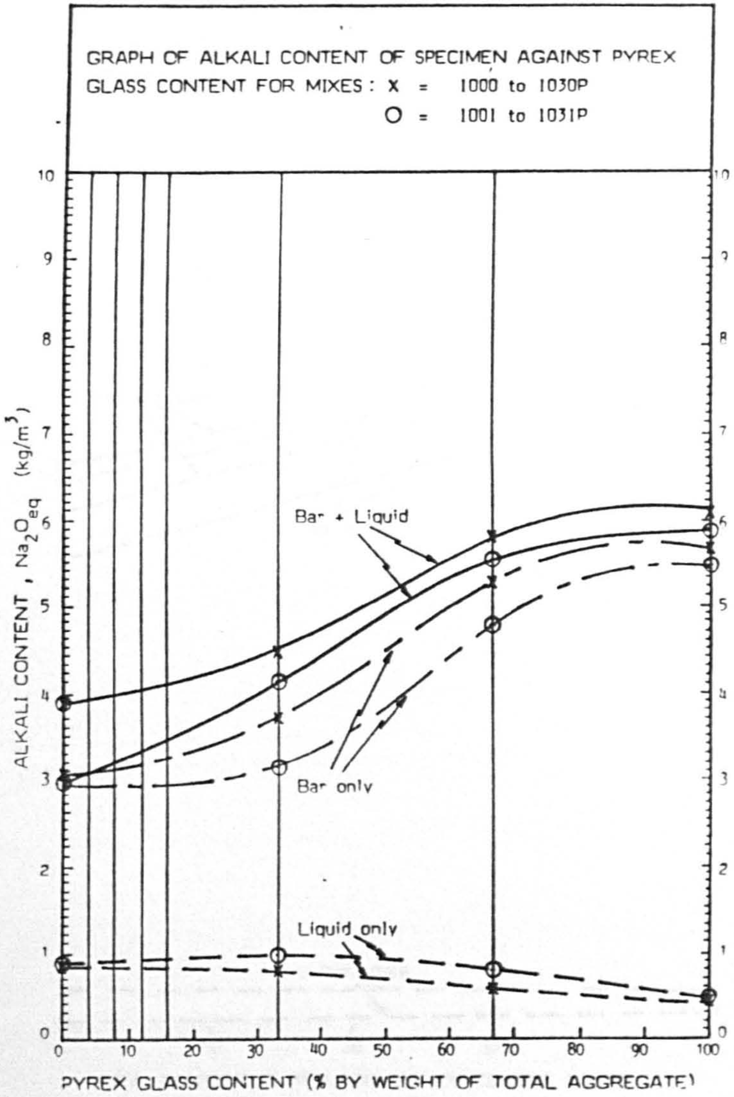
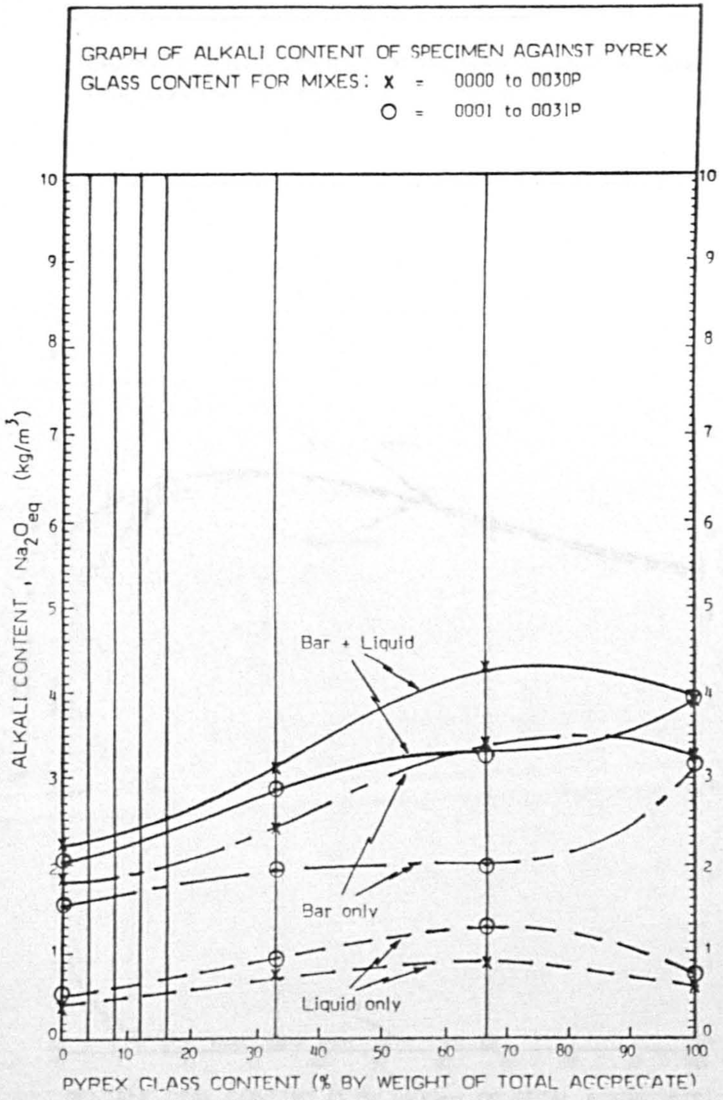


Figure 8.14



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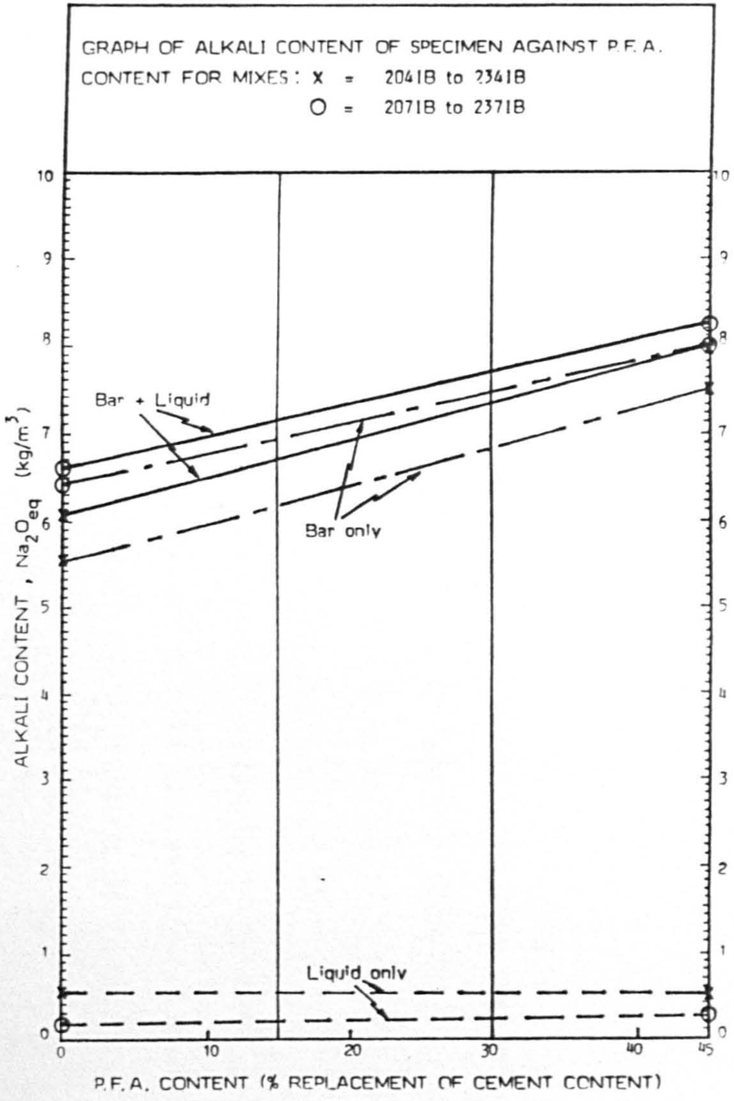


Figure 8.16

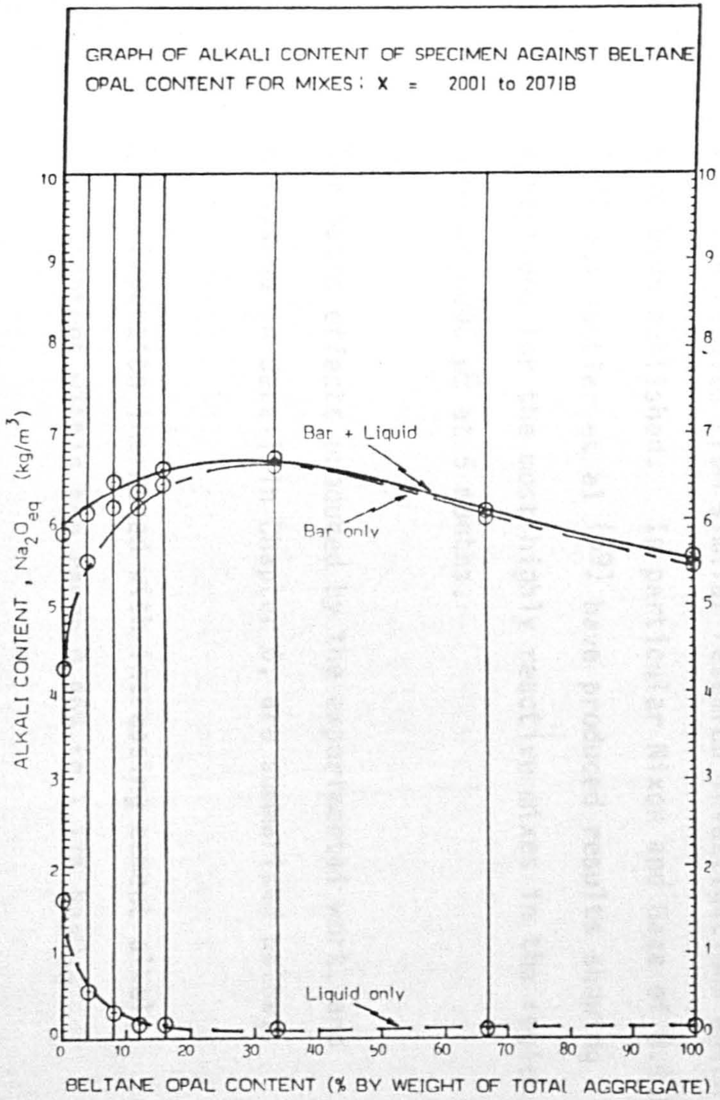

GRAPH OF ALKALI CONTENT OF SPECIMEN AGAINST BELTANE OPAL CONTENT FOR MIXES : X = 2001 to 2071B
ALKALI CONTENT , Na_2O_{eq} (kg/m^3)
BELTANE OPAL CONTENT (% BY WEIGHT OF TOTAL AGGREGATE)
Bar + Liquid
Bar only
Liquid only
0
1
2
3
4
5
6
7
8
9
10
0
10
20
30
40
50
60
70
80
90
100

Figure 8.17

CHAPTER 9 FURTHER DISCUSSION

9.1 COMPARISON OF THE EXPERIMENTAL RESULTS WITH PUBLISHED INFORMATION

In Chapters 6 and 8 the experimental results were presented and their meaning discussed. Following on from this discussion the results will now be compared with other similar information gained from comparable experimental investigations. The literature referred to originates primarily from British based research but extends to overseas work if no suitable British reference could be found.

9.1.1 Mortars containing Pyrex Glass

The order of magnitude of the expansion results obtained from the mortar bars containing Pyrex glass compares favourably with the results from similar research investigations which have been published. In particular Nixon and Gaze of B.R.E. (60) and Buttler et al (69) have produced results showing expansions for the most highly reactive mixes in the region of 5000 to 6000 $\mu\varepsilon$ at 6 months.

The basic effects produced by the experimental work, and discussed in detail in Chapter 6, are summarised below.

(i) Expansion increased with increasing cement alkali content within the range 0.46% to 1.17% Na_2O_{eq}

(ii) Expansion was reduced by the inclusion of p.f.a. in the mix as a partial replacement of the cement content. Greater reduction was achieved by increased cement replacement in the range 15% to 45%.

(iii) Pyrex glass did not exhibit a pessimum content below a concentration of 100% expansion increasing with increasing concentration of Pyrex glass.

(iv) There was a pessimum effect associated with free water/cement ratio, with increased expansion being promoted by increasing free water/cement ratio in the range 0.30 to 0.55 followed by a reduction in expansion with a further increase in free water/cement ratio to 0.60.

Each of these effects is consistent with the findings from work already published (55, 60, 69) notwithstanding the slight variations that have been noted in certain instances.

With respect to cement alkali content, within the range dictated by the experimental materials employed, there seems little doubt that expansions increase with increasing alkali content. This is confirmed by published British research (69) and dates back to Stanton's original report (1).

Results produced using p.f.a. as a cement replacement material have not always produced consistent results for expansion reduction. Buttler et al (69) found that under some circumstances there was an optimum content for p.f.a., above which expansion began to increase again. There was no evidence of such an effect from the observations produced by the present investigation despite the p.f.a. used being from one of the sources used in this earlier research investigation.

The trend for increasing expansion with increasing Pyrex glass concentration throughout the range 0% to 100% is the most common observation. However, Figg (59) has commented that Pyrex glass can produce a pessimum effect. In the present investigation one set of expansion results did indicate the possibility of a pessimum value in the range 33.3% to 100%. This occurred only for the mixes containing the highest alkali cement in conjuction with the lower free water content.

The pessimum effect with free water/cement ratio is in agreement with the work published by Lenzner (55), though the values obtained are slightly different. Lenzner achieved maximum expansion for a water/cement ratio of 0.60 as compared with the value of 0.55 obtained in the present investigation. The close relationship between these two values is worthy of note since Lenzner's work was carried out using a reactive opaline sandstone and not Pyrex glass.

9.1.2 Mortars containing Beltane Opal

The pessimum behaviour observed for Beltane opal, is consistent with the effect found by other research investigations using the same material. This work is well documented in the literature. In particular, Hobbs et al of the C α C A have published information on this subject which was derived from experimental work conducted in the UK (21, 45, 53, 92). In this work, for mortars having mix proportions similar to those specified in the A.S.T.M. mortar bar test, Hobbs generally found the pessimum to be in the region of 2.5% to 6% by weight of total aggregate. This compares favourably with the value of 4% gained from the results of the Series 2 mortar bar tests conducted in the present investigation. The similarity of the results does not, however, extend to the remaining characteristics of the expansion versus Beltane opal content curve.

In Figure 9.1 the expansion versus Beltane opal content curves are compared for the results obtained in the present investigation and the results published by Hobbs (21). Both the form of the overall curve and the scale of the measured expansions are shown to be significantly different.

Considering the difference in the magnitude of the expansion values, those obtained by Hobbs are in the region of a factor of thirty larger than the expansions observed in the present investigation. To find the possible reasons for this vast difference the two experimental regimes require careful comparison.

The experimental methods employed by Hobbs were different with respect to the following details

(i) the particle size of the reactive aggregate
(ii) the storage temperature for the specimens
(iii) the type of storage container used

(iv) the technique used to monitor any length changes.

The latter two differences are not considered to be of major importance except that it does not seem clear whether or not the type of storage container used by Hobbs would allow the mortar bar specimens to be in continuous contact with the reservoir of water. This could possibly have a considerable effect on the expansion level. However, it is the storage temperature and, in particular, the particle size of the reactive aggregate which are considered to be of fundamental importance and merit further discussion.

Some of the early work by Hobbs (53) studied the effect of particle size on the expansion level. At the pessimum concentration, it was shown that Beltane opal in the particle size range of 150µm to 300µm produced the greatest expansion in comparison to the other size ranges used. These other size ranges were 300µm to 600µm, 600µm to 1.2mm and 2.4 to 4.8mm. Consequently, all further work by Hobbs et al included the Beltane opal in the 150µm to 300µm size fraction only, as compared with the present investigation where equal proportions were used in each of five size fractions between 150µm and 4.75mm.

With respect to storage temperature, the ASTM temperature of 37.8°C was not used by Hobbs. The storage temperature selected was 20°C, this being the normal storage temperature for concrete specimens made to British Standard methods.

These two features, in conjunction with the known chemical and physical properties inherent to Beltane opal, may provide a possible reason for the different scales of expansion from the two experimental programmes. It is recognised that Beltane opal is a very highly reactive material in the sense of alkali silica reaction and that as a material it is very porous. In the present investigation Beltane opal was found to have a water absorption coefficient of 3.2%. Further details on the general chemical and physical properties of the material are provided by Gutteridge and Hobbs (57) who analysed Beltane opal and concluded that it contained 82%, by weight, of potentially alkali-reactive siliceous material.

In the present investigation, the use of equal proportions of Beltane opal in the five particle size ranges must produce a considerably different surface area to weight ratio than that produced by using Beltane opal entirely in the smallest particle size range. It seems reasonable to contend that the smaller particles will react more quickly than the larger particles, since the smaller particles offer a greater concentration of available reactive sites per unit mass.

To some extent the porous nature of the aggregate will offset this effect by allowing ready access to reactive sites other than those on the external surface. However, it is considered that this counter effect is unlikely to be sufficiently large as to neutralise the particle size contribution. The main contribution of the porous nature of Beltane opal probably lies in the ability of the larger reactive particles to absorb the initial quantities of the reaction product, namely the gel, and allow it to expand unhindered and without imparting internal stress to the mortar.

The combination of high porosity and lower surface area to weight ratio of the larger particles may result in the reaction starting more slowly in addition to the initial expansion being absorbed. Both of these effects will tend to reduce both the early and the ultimate expansion.

Part of this hypothesis may appear contradictory to the discussion given by Hobbs (53). He concluded that for particle sizes from 150µm to 2.4mm, the factor controlling the rate of gel production is the chemical reaction itself, which takes place between the hydroxyl ions and the reactive silica. The basis for this conclusion was that the graphs of expansion versus reactive aggregate content were found not to broaden with increasing particle size within the defined limits, it being considered that they would broaden if particle surface area were having a significant effect. However, for the largest particle size range of 2.4mm to 4.8mm some broadening of the curve was found to be evident and it was concluded that diffusion of the hydroxyl ion was therefore beginning to be important. This implies a surface area to weight ratio related effect.

If the Hobbs description is correct, it does not necessarily negate the explanation presented earlier, since the largest particle size range represents a 20% proportion of the total reactive material employed in the present investigation. In addition, the work published by Hobbs did not include the second largest size of reactive aggregate, namely 1.18mm to 2.36mm which may also fall into the 'broadening' category.

The small particle size of the Beltane opal used by Hobbs is therefore likely to produce a higher level of expansion. This is due to the probability that the small particles will react more quickly and have a reduced ability to absorb the expanding gel.

The other major difference between the two experimental regimes is the storage temperature, where the lower value is associated with the higher expansion. It is difficult to see how the lower storage temperature could increase the rate of A.S.R. and thus influence expansion. Consequently the most likely contribution made by the temperature is that the high value of 38°C may have a secondary effect by accelerating the curing rate of the mortar. This would enable the mortar to withstand the expansive forces more successfully and accordingly reduce the expansion value obtained.

Given the considerable difference in the particle size ranges utilised for the Beltane opal, and the effect which particle size is known to have on expansion, the similarity of the values obtained for the pessimum reactive aggregate content might be somewhat unexpected. If, however, the critical factor which determines the maximum expansion is the alkali-silica ratio, as has been suggested in the literature (45, 60, 94), then the particle size should not have a serious effect on the position of the pessimum. This ratio is between the amount of alkalis available to produce OH^- and the amount of silica available for reaction. Hence it may be more appropriate to refer to the effective alkali-reactive silica ratio, since materials such as Beltane opal contain both reactive and non-reactive forms of silica.

The second difference noted between the characteristics of the expansion versus Beltane opal content curves shown in Figure 9.1 was the form of the overall curve. This is mainly concerned with the higher Beltane opal concentrations, that is above the pessimum value. The curves reproduced from the work by Hobbs are shown to fall steeply after the pessimum and attain an expansion value of virtually zero. From the results obtained in the present investigation, however, for Beltane opal contents above 16% the curve is shown to increase again. For the 12 month results, the curve plotted in Figure 9.2 shows that the mix containing 100% Beltane opal produced an expansion which was approximately 60% of the value observed at the pessimum. This is a highly significant proportion of the pessimum value, particularly when considering the very localised nature of the pessimum effect itself. Moreover, another feature of the curve in Figure 9.2 is that the slope upwards to the secondary maximum expansion at 100% Beltane opal content is continuous for all mixes above 16% Beltane opal content.

Examination of the same curve, but plotted for the 14 day results, reveals that its form changes progressively with time. At this early age the pessimum remains at 4% Beltane opal content, but the minimum expansion for Beltane opal concentrations above the pessimum is not reached until 66.6%.

Figure 9.3 shows the set of curves for the four ages of measurement, namely 14 days, 2 months, 6 months and 12 months. Comparison of these curves shows that there is a transition with time with respect to the form of the curve beyond the pessimum. Two effects are apparent, firstly the point of minimum expansion moves from 66.6% Beltane opal content at 14 days to 12% Beltane opal content at 12 months and secondly the slope of the curve beyond the minimum shows some evidence of getting progressively steeper with time.

This transitional effect towards relatively greater expansions with the higher Beltane opal concentration mixes at later ages, is considered to be evidence of a slower reaction taking place in the larger Beltane opal particles and the capacity of these particles to absorb a certain initial quantity of gel. In the lower Beltane opal concentration mixes there is not sufficient quantity of the larger particles to significantly add to the early expansions observed around the pessimum. However, for the higher Beltane opal concentrations the amount of material available for the slower expansion is large enough to create the observed effect.

It is interesting to note from Figure 9.3 that, on a much reduced expansion scale, the graph of expansion versus Beltane opal content for the later ages of measurement begins to look more like the equivalent curve for Pyrex glass except for the very localised and extraordinary discontinuity represented by the pessimum.

9.2 BEHAVIOURAL DIFFERENCES BETWEEN PYREX GLASS AND BELTANE OPAL

In the foregoing Section 9.1 the results gained from the present investigation were compared with relevant published information. Following on from this discussion, the results from within the present investigation will now be examined with respect to the important behavioural differences noted between Pyrex glass and Beltane opal.

It is known that the alkali-silica reaction occurs between hydroxyl ions (OH^-) and reactive silica (SiO_2). Both Pyrex glass and Beltane opal are primarily composed of silica, not all of which is in a reactive form. There are, however, significant differences between the two materials with respect to their subsidiary composition, as shown below in the chemical analyses for typical samples:-

Pyrex Glass	SiO_2	80.20%	Beltane opal	SiO_2	91.38%
	Al_2O_3	2.33%		Al_2O_3	2.10%
	Fe_2O_3	0.11%		Fe_2O_3	0.13%
	K_2O	0.10%		K_2O	0.20%
	Na_2O	4.07%		Na_2O	0.10%
	MgO	0.02%		MgO	0.10%
	Others	13.13%	Loss on ignition, as H_2O		5.60%
			as CO_2		0.30%
(Pyrex glass - Corning Ware Glassworks, after Figg (59))			(after Gutteridge and Hobbs (57))		

The Beltane opal composition shows that it is only an 'acid' type system due to its very low proportion of alkaline material represented as K_2O and Na_2O, which combine together to give a total alkali content of 0.23% Na_2O_{eq}. This compares favourably with the value of 0.20% obtained from the analysis conducted on the Beltane opal used for the present work.

Conversely, the Pyrex glass contains a considerable amount of alkalis and therefore constitutes a 'basic' type system in addition to the main 'acid' system. For the Pyrex glass used in the Series 2 set of experiments from the present investigation a value of 3.06% Na_2O_{eq} was obtained by chemical analysis. This again compares favourably with the value of 4.14% Na_2O_{eq} obtained by combining the two individual alkali content figures from the typical analysis listed above.

The high level of alkaline material in the Pyrex glass means that it is not only able to react with the OH^- in the pore fluids because of its reactive silica content but that it is also a source of Na^+ and K^+. Thus the Pyrex glass can be its own source of positive counter ions, which help to maintain electrical neutrality during A.S.R.. In addition, the production of the Na^+ and K^+ ion may assist in enhancing the OH^- concentration in the pore fluids and thereby generate reactive sites as the A.S.R. progresses.

The Beltane opal, on the other hand, has less ability to provide its own positive counter ion and is therefore less likely to generate OH^-. Thus, it is more reliant on those positive ions found in the pore fluids and acts to reduce the OH^- concentration as A.S.R progresses. Therefore, Beltane opal has less ability to generate reactive sites as the reaction proceeds.

Beltane opal, then, appears to be a much better 'sink' for OH^- since, as the A.S.R. proceeds, it is constantly drawing on the OH^- from the pore fluids without generating any in return. The only significant source of OH^- remains the cement, as a result of the calcium hydroxide ($Ca(OH)_2$) produced during the normal hydration process of all Portland cements and the alkalis which differ in concentration for different cements. The $Ca(OH)_2$ is relatively insoluble in water and therefore only maintains a pH level of about 12. This is the pH value which would be expected for a low alkali cement and consequently would not be expected to support A.S.R. if the alkali content of the cement is a critical factor. The sodium and potassium alkalis, however, are more readily soluble and for high alkali cements boost the pH up to a value which can support vigorous A.S.R..

The action of Beltane opal as a 'sink' for OH^- may be sufficient to quickly reduce the pH of the pore fluids in the vicinity of the reactive particles to a level below the threshold value required for A.S.R.. Conversely the Pyrex glass assists in maintaining a pH level above any threshold which may exist. Threshold values have also been discussed by Hobbs (45).

The additional material which can effect the alkali balance of the pore fluids is p.f.a., which in some respects seems to act in a similar manner to Pyrex glass. P.f.a. is composed primarily of silica and reacts strongly with OH^-. Moreover, as it reacts, because of its high alkali content, the p.f.a. releases alkalis into the pore fluids. Initially most of the alkali in the p.f.a. is in solid solution within the glassy matrix of the particles and therefore not available to enter the pore fluids. However, in Chapter 8 it was shown that these alkalis are released as the pozzolanic reaction progresses. Again this release of alkalis could produce an increase in the OH^- concentration.

The p.f.a. is apparently able to react at OH^- concentrations below the apparent threshold for A.S.R. with Pyrex glass or Beltane opal. The level of OH^- concentration produced by the $Ca(OH)_2$ only would appear to be sufficient for the pozzolanic reaction to occur, but higher concentrations are likely to accelerate the process.

In addition to the differences in the chemistry of the two reactive aggregates, there is one very important physical difference, this being the porosity of the materials. The highly porous nature of the Beltane opal was discussed in Section 9.1.2 and results in the material having a high coefficient of water absorption. The Pyrex glass, however does not have this porous characteristic and yields a water absorption coefficient of effectively zero.

The differences in expansion behaviour of mortars containing Pyrex glass and Beltane opal, both with and without p.f.a. in the mix, will now be discussed in light of these physical and chemical differences inherent in the two materials. The discussion will centre on the apparent differences shown by each material with respect to their relative affinities for the hydroxyl ion.

9.2.1 Mixes without Pulverised Fuel Ash

To review the discussion of Chapter 6, in the absence of p.f.a., two fundamental differences were noted from the Series 2 experimental results regarding the expansion data obtained from mortar bars containing either Pyrex glass or Beltane opal as the reactive aggregate. These differences are that the expansions gained from bars containing Pyrex glass were an order of magnitude greater than those gained using Beltane opal and that the Beltane opal aggregate produce a marked pessimum concentration of approximately 4% by weight of total aggregate.

The difference in the scale of the expansion data obtained is further demonstrated in Figures 9.4 and 9.5 where, for all reactive aggregate concentrations except the pessimum for Beltane opal, the Pyrex glass promotes larger expansions. This occurs despite the probability that Pyrex glass contains less reactive silica than the extremely reactive Beltane opal.

The explanation for the difference in expansion levels is considered to involve the particular experimental regime used for the laboratory specimens in addition to physical and chemical differences inherent in the materials. It is these physical and chemical differences which will be discussed initially.

Firstly, there is the ability of the Beltane opal to act as a much better 'sink' for OH^- than the Pyrex glass, thus tending to reduce the OH^- concentration. Secondly, the very porous nature of the Beltane opal has been discussed as a possible mechanism for reaction to proceed without any expansion in the initial stages. This feature has been linked, in particular, to the larger Beltane opal particles. Pyrex glass does not have this porous characteristic and, therefore, does not offer any facility to allow the gel to expand in an unrestrained manner. For mortars containing reactive aggregate of fairly large particle size, if similar amounts of gel are produced then more expansion may be anticipated with the Pyrex glass mixes. Both these features of Beltane opal would, then, tend to reduce the amount of expansion caused by Beltane opal in comparison to that produced by Pyrex glass.

In addition, Beltane opal has been shown by Hobbs (21, 45, 53) to be capable, under different laboratory and experimental conditions, of producing expansions which exceed by a factor of thirty those measured in the present investigation. Such an expansion level is considerably greater than that produced by the most highly expansive mortar containing Pyrex glass from the Series 2 set of experiments. This could be an indication that the A.S.T.M. mortar bar method represents ideal conditions for expansion with Pyrex glass but poor conditions for expansion with Beltane opal.

At the pessimum concentration the expansion produced by Beltane opal is seen in Figure 9.4 to exceed the corresponding expansion due to Pyrex glass. The expansion of the Pyrex glass mix is in line with the low concentration of reactive aggregate and follows the general trend of the expansion versus reactive aggregate curve. Thus, the reversal of the normal relationship between the expansions produced by the two reactive materials is entirely associated with the apparently disproportionate amount of expansion produced by the Beltane opal at its pessimum.

At the pessimum the Beltane opal effectively offers few reactive sites and is fully saturated with OH^-, that is the optimum reactive combination exists (94). This situation has been described by Hobbs (45) as one where complete reaction of the Beltane opal will just reduce the OH^- concentration in the pore fluids to the threshold level. Both these explanations relate to the pessimum being the occurrence of the most critical value of the alkali/silica ratio, resulting in an extraordinary level of expansion.

9.2.2 Mixes including P.F.A.

When the 45% level of p.f.a. was included in the mix containing the pessimum Beltane opal content a dramatic reduction of approximately 50% was achieved in the measured expansion. At all other Beltane opal concentrations an opposite effect was observed with the p.f.a. resulting in an increased expansion value. The increase, however, was less marked than the 50% reduction at the pessimum. These two different effects are illustrated in Figure 9.6.

For 45% p.f.a. used in conjunction with Pyrex glass, a reduction in the measured value of expansion of approximately 90% was observed for all the Pyrex glass concentrations that were examined.

It is therefore apparent that there is a considerable difference between the responses of the two reactive aggregates to the use of p.f.a.. Despite this difference, however, for all the reactive aggregate concentrations used in conjunction with p.f.a. and both types of reactive aggregate, namely 33.3%, 66.6% and 100%, the expansion for the mixes including Beltane opal remained below its Pyrex glass equivalents. This is demonstrated in Figure 9.7. The two curves, however, become very much closer together when p.f.a. is introduced into the mix, as can be seen by comparing Figure 9.7 and Figure 9.5.

At the lower reactive aggregate concentrations of 4%, 8%, 12% and 16% the mixes containing p.f.a. were not included in the experimental work with Pyrex glass. However, on Figure 9.8 the curves for the equivalent Pyrex glass mixes without p.f.a. have been superimposed over the curves for the Beltane opal mixes which did include p.f.a.. The comparison of the pairs of curves obtained indicates that, except for the 4% and 8% concentrations, the curves for Pyrex glass remain above the corresponding curves for Beltane opal. This presumes that the p.f.a. would not have caused any increase in expansion for any of the Pyrex glass mixes which would be in keeping with all the results which have been obtained in the current investigation.

Now, let us firstly consider the case of Pyrex glass and p.f.a.. The experimental results show that, for all the Pyrex glass concentrations investigated, the introduction of p.f.a. causes a reduction in expansion. This observation extends over all the results obtained for the three levels of cement replacement that were employed, that is 15%, 30% and 45% p.f.a.. The inference is, then, that the p.f.a. has a much higher affinity for OH^- than the Pyrex glass. This is further substantiated by Table 9.1 where the mass ratio of Pyrex glass to p.f.a. is calculated for each of the nine mixes containing both materials. The data show that even when the mass ratio of Pyrex glass : p.f.a. is 15.7 : 1, as in Mix Ref 2131P, a reduction in expansion was still achieved. Thus, the p.f.a. has a far more preferential reaction with OH^- and may be considered to starve the Pyrex glass of the OH^- concentration it requires for A.S.R. to proceed.

The explanation must now be extended to the expansion gained from mixes containing Beltane opal. It has been stated earlier that Beltane opal contains a very high proportion of potentially reactive material (over 80%). In addition, a typical Pyrex glass contains approximately 80% of siliceous material, not all of which will be in reactive forms. Consequently, it is most likely that the Beltane opal contains considerably more reactive material and will therefore have a much higher affinity for OH^-. This being true, two possibilities remain with respect to OH^- affinity, and they are:-

Beltane opal > p.f.a. > Pyrex glass CASE A
p.f.a. > Beltane opal > Pyrex glass CASE B

If CASE B were correct, then, based on the argument put forward for the Pyrex glass mixes, it would be anticipated that p.f.a. would reduce expansions for Beltane opal mixes. This is clearly shown by the expansion results to not be the general observation, which indicates that CASE A may provide the answer.

For all Beltane opal concentrations above the pessimum the p.f.a. fails to reduce the measured expansion and actually has the opposite effect (see for example Figure 9.9). The only feasible situation with respect to OH^- affinity is, therefore, that the Beltane opal has a greater affinity than the p.f.a.. However, despite this situation under normal circumstances, at the pessimum concentration where the non-p.f.a. mix produces such an extraordinarily large expansion, the introduction of p.f.a. into the mix results in a reduction in expansion (see Figure 9.10). Thus, it appears that at the pessimum the p.f.a. does maintain a preferential reaction with the OH^- and thereby starve the Beltane opal from reaction.

This may arise from the occurrence of the pessimum at such a low Beltane opal concentration and does not necessarily imply that CASE B, above, applies at the pessimum. It is possible that the large amount of p.f.a. in comparison to the small amount of Beltane opal is sufficient to make up for the difference in their relative affinities for OH^-.

At the next higher Beltane opal concentration of 8% the situation is already back to normal, the p.f.a. causing a slight increase in expansion (Figure 9.9). Thus, the difference in the amount of p.f.a. relative to the amount of Beltane opal is no longer sufficient to compensate for their different affinities for OH^-.

In Table 9.2 the mass ratio of Beltane opal to p.f.a. is calculated for each of the seven mixes containing both materials. From this data it can be seen that at the pessimum there is 4.3 times the quantity of p.f.a. than Beltane opal, whereas for the 8% Beltane opal concentration this ratio is reduced to 2.2. It may be deduced, therefore, that the relative affinity for OH^- between Beltane opal and p.f.a. is somewhere between 2.2 and 4.3 and that CASE A, above, satisfies all the expansions observed in the present investigation.

The discussion so far indicates that the use of p.f.a. in conjunction with the pessimum Beltane opal content produces an effect very similar to that observed with Pyrex glass. However, at concentrations above the pessimum zone the Beltane opal produces a totally opposite effect. Let us now take the discussion a stage further and consider the effects of time, again taking Pyrex glass first.

Table 9.3 shows the change in expansion for the relevant Pyrex glass mixes over each increment between the specimen ages selected for expansion measurement. This change in expansion is also converted into an expansion rate, in microstrain per day, for each age increment. A graph can now be plotted of expansion rate against age of specimen, as in Figures 9.11 to 9.13 for the mortars containing 33.3%, 66.6% and 100% Pyrex glass respectively. For convenience these curves are plotted to a log scale on the y-axis, all values extending beyond the minimum value available on the scale being plotted on the x-axis at the base of the graph.

Figures 9.11 to 9.13 show that at early ages the curve for the mix containing p.f.a. stays below the curve for the corresponding non-p.f.a. mix. At later ages, however, there is a tendency for the curves to be much closer together and in the case of the mortar containing 100% Pyrex glass the curve for the mix containing p.f.a. actually intersects its non-p.f.a. equivalent.

This effect was first mentioned in Chapter 6 and can be further demonstrated by taking the difference between the expansions from the non-p.f.a. mix and its equivalent mixes containing p.f.a.. This is done for the high free water content mixes in Table 9.4 and the data are plotted in Figures 9.14 to 9.16 for the mixes containing 45% p.f.a., 30% p.f.a. and 15% p.f.a. respectively. The trend is for these curves to rise rapidly to the earliest measurement age followed by a transition towards a falling curve at later ages. This effect is more pronounced for higher Pyrex glass concentrations and lower p.f.a. contents.

The evidence is, then, that mixes containing Pyrex glass and p.f.a. tend to expand more slowly at early ages, but continue their expansive activity beyond the time when expansion has virtually ceased in the non-p.f.a. mixes. This is particularly true for the higher free water content mortars.

In the earlier discussion it was deduced that the p.f.a., when used in conjunction with Pyrex glass, promotes a preferential reaction with the OH^- (the pozzolanic reaction) due to its greater affinity for this negative ion. This effectively starves the Pyrex glass of the OH^- it requires for A.S.R. to occur. We therefore have

$$\frac{\text{Rate of expansion of Pyrex/p.f.a. mixes}}{\text{Rate of expansion of Pyrex/non-p.f.a. mixes}} < 1 \qquad 9.1$$

As time progresses there are three factors which could contribute towards increasing the value of this quotient, the experimental results having shown this to be the trend.

Firstly, it has been shown in Chapter 8 that as the pozzolanic reaction proceeds a significant amount of alkalis are released into the pore fluids from their previous state of being in solid solution within the glassy particles of the p.f.a.. Secondly, also in Chapter 8, the p.f.a. was shown to reduce the amount of alkalis which leach out of the mortar bars and enter the water at the bottom of the storage containers. Finally, the power of the pozzolanic reaction itself will show some tendency to diminish with time and thereby reduce the ability of the p.f.a. to starve the Pyrex glass of OH^-.

Thus, all three of these time dependent factors act to increase the amount of OH^- which is available for reaction with the Pyrex glass which itself remains in an unchanged reactive state. The pozzolanic reaction has not fundamentally altered the potential for A.S.R. which Pyrex glass provides and therefore the reaction process is delayed rather than completely avoided.

At the stage where the reaction between the p.f.a. and the OH^- begins to diminish, the rate of expansion due to A.S.R. does not, however, increase dramatically and remains at only a fraction of the level observed for the early expansions of the non-p.f.a. mortars. The reasons for this are two-fold. At the later ages involved, the mortar has matured in respect to its strength and therefore its ability to withstand the internal pressures created by the expanding gel. In addition, and perhaps more importantly, the pozzolanic reaction will have reduced the permeability of the cement paste by the production of C-S-H gel as discussed in Chapter 3. This reduced permeability will both reduce the mobility of the OH^- ions, thereby restricting their ability to get to the reactive sites, and also reduce the speed at which water can be absorbed into the reacted particles to initiate swelling.

In some instances the power of this transition with time is sufficient to produce the situation where:-

$$\frac{\text{Rate of expansion of Pyrex/p.f.a. mixes}}{\text{Rate of expansion of Pyrex/non-p.f.a. mixes}} > 1 \qquad 9.2$$

Now let us study the comparable time related effects for p.f.a. used in conjunction with Beltane opal. Table 9.5 shows the changes in expansion over each increment in specimen age and also the expansion rate for each age increment. The two graphs of expansion rate against age of specimen for each of the seven Beltane opal concentrations are given in Figures 9.17 to 9.23, again with the expansion rate plotted to a log scale.

At the pessimum Beltane opal content, the form of the two curves is very similar to the ones obtained for the 100% Pyrex glass mix (cf Figure 9.17 with Figure 9.13). The curve for the mix containing p.f.a. starting beneath the corresponding curve for the mix without p.f.a. but this situation being reversed at later ages.

Above the pessimum Beltane opal concentration (Figures 9.18 to 9.23), at early ages the p.f.a. curve is generally above the corresponding non-p.f.a. curve. At later ages, however, there is a tendency for the curves to become closer together. This tendency increases with increasing Beltane opal concentration. For all Beltane opal concentrations of 16% and above, at later ages the two curves actually intersect indicating that the non-p.f.a. mix was then expanding more rapidly than the corresponding mix containing p.f.a.. These trends are totally opposite to the situation observed with Pyrex glass.

Data derived from subtracting the expansion measured for a mix containing p.f.a. from that measured for its non-p.f.a. counterpart are presented in Table 9.6. Here, most of the figures are negative due to the common effect of increased expansion when p.f.a. is used with Beltane opal. When the difference in expansion is plotted against age of specimen the curves in Figure 9.24 are obtained. These curves correspond to a p.f.a. content of 45%, this being the only p.f.a. content used in conjunction with Beltane opal. Comparing Figure 9.24 with Figure 9.14, the corresponding figure for Pyrex glass, emphasises the opposite nature of the effect produced by Beltane opal at all concentrations above the pessimum.

At the pessimum concentration the arguments presented earlier in respect of expansion with Pyrex glass and p.f.a. seem to hold true for the Beltane opal observations. This is because the p.f.a. is present in sufficient quantity, relative to the amount of Beltane opal, for it to react preferentially with the OH^-.

For the other Beltane opal concentrations there is evidence of a slight tendency for p.f.a. to delay the onset of the very early expansion, an effect noted in the discussion of Chapter 6. The general form of the curves in Figure 9.24, however, is for a rapid early fall followed by a transition towards a rising curve. This effect shows evidence of being more pronounced with increasing Beltane opal concentration and is further confirmation of the opposite trends associated with p.f.a. when used in conjunction with Beltane opal.

The evidence obtained from this investigation indicates that mixes containing p.f.a. and Beltane opal, the latter being above the pessimum concentration, expand more rapidly at early ages than the corresponding non-p.f.a. mixes. At later ages, the non-p.f.a. mixes continue to expand beyond the time at which expansion has virtually ceased for the p.f.a. mixes. We now need to consider how this situation fits into the hypothesised concept of the relative affinity for OH^- exhibited by the materials involved.

It has been deduced earlier that Beltane opal has a higher affinity for OH^- than p.f.a., and also that Beltane opal is a good sink for OH^- since it is not itself a source of OH^-. The Beltane opal therefore has a preferential reaction with OH^- and to a certain extent starves the p.f.a. of the material it needs for the pozzolanic reaction. The ability of the p.f.a. to slow down the expansion promoted by Beltane opal is therefore drastically reduced. Under these circumstances, the situation with respect to expansion rates is:-

$$\frac{\text{Rate of expansion of Beltane opal/p.f.a. mixes}}{\text{Rate of expansion of Beltane opal/non-p.f.a. mixes}} > 1 \qquad 9.3$$

(i.e. opposite to effect with Pyrex glass, cf 9.3 with 9.1)

The preferential reaction between the Beltane opal and the OH^- does not fully explain the negative effect of increased expansion when p.f.a. is used. It would explain either a reduction in the ability of p.f.a. to inhibit expansion or a completely null effect. For the increased expansion to occur, other factors must be acting.

It seems unlikely that the Beltane opal will be able to completely stop the pozzolanic reaction but, more probably, allow it to commence at a reduced rate. Thus, a certain amount of alkalis will be released from solid solution within the p.f.a. thus helping to replenish the OH^- used by the Beltane opal. Further, the p.f.a. was shown in Chapter 8 to restrict the amount of alkalis leached out of the bar, thus slightly enhancing the OH^- concentration in the bar. It is also possible that the p.f.a. has a physical effect in reducing the ability of the larger Beltane opal particles to absorb some of the early gel production. These three secondary effects may be responsible for the increased expansion with p.f.a..

As time progresses the capacity of the Beltane opal to act as an OH^- sink will reduce as the most highly reactive sites are consumed and only the less reactive sites remain. The ability of the Beltane opal to maintain its preferential reaction with OH^- will likewise diminish allowing the pozzolanic reaction to flourish. This in turn will help to curtail the A.S.R. and the expansion process by reducing the permeability of the cement paste thus restricting the supply of OH^- to the reactive sites and the water supply to the expansive gel.

In the non-p.f.a. mixes, provided the OH^- concentration remains above any threshold level, the less accessible and less reactive sites within the Beltane opal will continue to react, though more slowly, and promote continuing expansion. Thus, with time, the relationship between the rates of expansion of the two types of mixes becomes:-

$$\frac{\text{Rate of expansion of Beltane opal/p.f.a. mixes}}{\text{Rate of expansion of Beltane opal/non-p.f.a. mixes}} < 1 \qquad 9.4$$

(again opposite to the effect with Pyrex glass, cf 9.4 with

9.2)

9.3 IMPLICATIONS FOR THE USE OF P.F.A. IN CONCRETE

The discussion so far has further highlighted the considerable difference between the two reactive materials most commonly used in experimental research into A.S.R.. Indeed, with respect to p.f.a., the two materials have been shown to behave in a virtually opposite manner. In extrapolating the information gained from the small scale mortar specimens into the domain of concrete and full size structural components, one fundamental question still remains. That is whether concrete aggregates which are susceptible to A.S.R. will produce characteristics similar to Beltane opal or Pyrex glass. The observed differences in their behaviour can be summarised as below:-

	Pyrex Glass	Beltane Opal
1.	Maximum expansion at 100% concentration	Maximum expansion at 4% concentration.
2.	No pessimum behaviour, increased expansion with increasing reactive aggregate concentration.	Pessimum type behaviour at low concentrations.
3.	Maximum recorded expansion in mortar bar at age 6 months = 6948$\mu\varepsilon$	Maximum recorded expansion in mortar bar at age 6 months = 529$\mu\varepsilon$

4.	P.f.a. causes a reduction in expansion for all mixes containing reactive aggregate	P.f.a. causes an increase in expansion for all mixes containing reactive aggregate above the pessimum concentration
5.	The reduction in expansion caused by p.f.a. tends to diminish with time	The increase in expansion caused by p.f.a. tends to diminish with time

Should any aggregate behave in a similar way to Beltane opal, then, above its pessimum content, there would be no case for using the material with p.f.a. since this would only exacerbate the situation. In the case of aggregates which behave like Pyrex glass or Beltane opal at its pessimum then p.f.a. offers one possible means of protection against A.S.R..

It seems more likely that the Pyrex glass type behaviour will be the norm since the pessimum effect with the Beltane opal is very localised and occurs at such low reactive aggregate concentrations. In addition, much of the behavioural characteristics of Beltane opal depend on its highly porous nature which is not a feature common to normal aggregates. Nevertheless it is important to remember that neither of the experimental materials can be considered as 'natural' in terms of a concrete aggregate material.

Following research using British aggregates, it has been stated by Nixon and Gaze (60) that none of the aggregates that were found to be reactive showed any pessimum effect. More recent research and field investigation work (95), however, has indicated that some U.K. reactive aggregates may have a pessimum characteristic. This is normally associated with higher reactive aggregate concentrations than the 4% observed for Beltane opal.

Under normal circumstances the use of p.f.a. represents a means by which an extra safeguard can be provided against the often disastrous effects of A.S.R., provided that care is taken to examine aggregates for materials which exhibit Beltane opal type behaviour. However, the practical implications of the time dependent trends must be considered. The tendency for expansions at later ages to fall into the category of

$$\frac{\text{Rate of expansion with p.f.a.}}{\text{Rate of expansion without p.f.a.}} > 1 \qquad 9.5$$

provides some uncertainty against the long term stability of the p.f.a. effect in reducing expansion and damage due to A.S.R.. This is despite the association of this time dependent effect with primarily the higher free water content mortars.

In any Portland cement/aggregate system, be it concrete or mortar, the reactive silica in the aggregate will always out live the active life of the p.f.a.. Further, since it has been shown that p.f.a. tends to increase the concentration of the alkali metal ions Na^+ and K^+, there may be a corresponding increase in the OH^- concentration. If this is so, the potential for A.S.R. in the long term will at least be maintained. Rather than a permanent inhibitor the p.f.a. can, therefore, only be seen to delay the onset of reaction.

In the experimental situation, the amount of expansion beyond the point at which the situation expressed in Equation 9.5 is reached, is relatively small even for the most highly active mortars. This observation must be considered in relation to the tremendous pressure which is being placed on the p.f.a. in the experimental test. Under no reasonable circumstances would the p.f.a. be expected to combat a material as reactive as Pyrex glass in a structural concrete, since such a material would not be a viable aggregate.

In practice the p.f.a. would be used with aggregates which are only mildly reactive in comparison to Pyrex glass, but which could cause serious problems if used with a high alkali cement. The inference is, therefore, that when the stage is reached where the reactive silica in the aggregate is able to gain a sufficient supply of OH^- for A.S.R. to proceed, the reaction will be so slow that the forces generated within the concrete will not be sufficient to cause significant damage.

Further, for the full benefits of the p.f.a. to be realised, it should be ensured that a reasonably high replacement level is used. The experimental results suggest that 30% - 45% replacement is suitable since at this level a very high proportion of the p.f.a. is still able to react.

The use of p.f.a. as one possible means of minimising the risk of A.S.R. has been advocated by two comparatively recent UK publications (48, 72) which proffer advice on all aspects of A.S.R. in structural concrete. In particular, the report of the working party (48), which was set up to make specific recommendations to aid practising engineers in the field of concrete construction, presents guidelines on how to assess the quantity of p.f.a. required to avert a potentially reactive aggregate/cement combination.

The basis of the reasoning behind these guidelines is that p.f.a. does not contribute any 'reactive' alkali to the concrete. It is also stated that the true 'inhibiting mechanism is not understood'. Hence, dilution theory is assumed. When used as a cement replacement material, the p.f.a. is considered to dilute the alkali level of the cementitious material and consequently the 'reactive' alkali concentration in the concrete.

The criterion used to assess sufficient alkali dilution is 3.0kg Na_2O_{eq}/m^3 (acid soluble), which has previously been discussed and found to be an unsuitable parameter, at least under certain circumstances. Provided that the alkali content of the concrete, contributed by the cement only, falls below this limit the mix design is considered safe, subject to an additional requirement that a minimum of 25% p.f.a. is used.

The cement/p.f.a. cementitious mixture arrived at in this way will no doubt achieve a beneficial effect in reducing expansion. It may also be possible to equate expansion reduction to an 'equivalent alkali dilution'. However, based on the experimental results obtained, the actual mechanism of the beneficial effect has been shown in the earlier discussion to be not simply related to alkali dilution.

Recommendations for the use of p.f.a. are still not universally supported. Recent information provided by Hobbs (96) contains very pessimistic conclusions as to whether or not p.f.a. actually reduces expansion in practical situations.

In this document Hobbs refers to much published research involving the use of p.f.a., some of which produced results showing little or no success with respect to expansion reduction. From the limited detail of the information presented, most of the experimental work which yielded a negative response to p.f.a. appears to have been based on the use of reactive aggregates only in their most reactive size fraction. Often these reactive aggregates appear also to be Beltane opal or similar material. In such cases, the findings of the current work could not disagree with the inability of p.f.a. to reduce expansion, particularly if the proportion of the reactive material is remote from the pessimum concentration.

Hobbs also concludes that the effectiveness of p.f.a. in preventing deleterious expansion is dependent upon the total alkali content of the p.f.a. and that the p.f.a. contributes significant quantities of alkali to the reaction. Both of these statements may be seen to have some link with the explanations presented earlier in the present discussion, but do not necessarily have a serious effect on the benefits attributable to the use of p.f.a..

It may be that the total alkali content of the p.f.a. has some bearing on the overall effectiveness of p.f.a. since it has been argued that this alkali is released during the pozzolanic reaction. Further, it has also been shown that there is a tendency for mixes containing p.f.a. to begin expanding more rapidly at later ages. That is, after the pozzolanic reaction has started to diminish. Such expansion may well be adversely affected by high levels of alkali in the p.f.a. and consequently released into solution.

The statement that p.f.a. contributes significant quantities of alkali to the reaction is completely the opposite from previous explanations that the expansion reduction capabilities of p.f.a. were due purely to alkali dilution. It does, however, agree with the findings of the current investigation which has showed that p.f.a. does substantially increase the alkali content of mature mortar bars, and yet normally produces a massive reduction in expansion.

The obstacle which remains for the successful use of p.f.a. to minimise the risk of A.S.R., is the possibility of Beltane opal type behaviour being commonplace in concrete aggregates. In the event that this is the true situation, then p.f.a. should still produce a beneficial effect where the concentration of the reactive material is at or very near to its pessimum value. This is when such an aggregate would produce the maximum expansion and greatest destructive effect. At concentrations above the pessimum it seems likely, however, that there will be some risk of p.f.a. exacerbating expansion.

Table 9.1 MASS RATIO OF REACTIVE AGGREGATE TO P.F.A. FOR MIXES CONTAINING PYREX GLASS

Mix Ref No.	Dry Pyrex Glass Content (kg/m^3)	P.F.A. Content (kg/m^3)	Mass Ratio Pyrex glass : P.f.a
2111P	411	78	5.3 : 1
2121P	802	77	10.4 : 1
2131P	1176	75	15.7 : 1
2211P	412	148	2.8 : 1
2221P	803	145	5.5 : 1
2231P	1175	141	8.3 : 1
2311P	411	213	1.9 : 1
2321P	804	208	3.9 : 1
2331P	1176	203	5.8 : 1

Table 9.2 MASS RATIO OF REACTIVE AGGREGATE TO P.F.A. FOR MIXES CONTAINING BELTANE OPAL

Mix Ref No.	Dry Beltane Opal Content (kg/m^3)	P.F.A. Content (kg/m^3)	Mass Ratio Beltane Opal : P.f.a
2311B	403	209	1.9 : 1
2321B	774	200	3.9 : 1
2331B	1114	192	5.8 : 1
2341B	51	218	1 : 4.3
2351B	100	216	1 : 2.2
2361B	150	215	1 : 1.4
2371B	198	214	1 : 1.1

TABLE 9.3 CALCULATED CHANGES IN EXPANSION AND EXPANSION RATES FOR CERTAIN MIXES CONTAINING PYREX GLASS

Mix Ref No.	0 - 14 Days		14 Days to 2 Months		2 Months to 6 Months		6 Months to 12 Months	
	Change in Expansion (μE)	Expansion Rate (μE/day)	Change in Expansion (μE)	Expansion Rate (μE/day)	Change in Expansion (μE)	Expansion Rate (μE/day)	Change in Expansion (μE)	Expansion Rate (μE/day)
2001	92	6.5714	3	0.0714	10	0.0893	-17	-0.1012
2011P	1309	93.5000	1622	38.6190	117	1.0446	-20	-0.1190
2021P	3432	245.1429	2785	66.3095	250	2.2321	145	0.8631
2031P	4680	334.2857	2097	44.9286	171	1.5268	-100	-0.5952
2041P	117	8.3571	43	1.0238	21	0.1875	11	0.0655
2051P	153	10.9286	75	1.7857	17	0.1518	-13	-0.0774
2061P	232	16.5714	187	4.4524	70	0.6250	-17	-0.1012
2071P	372	26.5714	333	7.9286	66	0.5893	1	0.0060
2301	96	6.8571	35	0.8333	32	0.2857	-11	-0.0655
2311P	187	13.3571	50	1.1905	72	0.6429	-9	-0.0536
2321P	375	26.7857	90	2.1429	78	0.6964	57	0.3393
2331P	593	42.3571	90	2.1429	117	1.0446	64	0.3810
2031P	4680	334.2857	2097	49.9286	171	1.5268	-100	-0.5952
2131P	2163	154.5000	756	18.0000	282	2.5179	87	0.5179
2231P	1125	80.3571	336	8.0000	411	3.6696	232	1.3810
2331P	593	42.3571	90	2.1429	117	1.0446	64	0.3810

TABLE 9.4 CALCULATED DIFFERENCE BETWEEN EXPANSIONS OF NON-P.F.A. MIXES AND THEIR EQUIVALENT P.F.A. MIXES FOR CERTAIN MORTARS CONTAINING PYREX GLASS

Mix Ref No. For Non-p.f.a. Mix	Mix Ref No. For P.f.a. Mix	Reactive Aggregate Content (%)	P.f.a. Content (%)	Expansion Difference (μƐ)			
				14 days	2 months	6 months	12 months
2101	2001	0	15	-33	-29	-51	-68
2111P	2011P	33.3	15	801	1962	1316	1164
2121P	2021P	66.6	15	1664	3306	2910	2936
2131P	2031P	100	15	2517	3858	3747	3560
2201	2001	0	30	-4	-34	-72	-88
2211P	2011P	33.3	30	1008	2524	2557	2508
2221P	2021P	66.6	30	2725	5313	5303	5164
2231P	2031P	100	30	3555	5316	5076	4744
2301	2001	0	45	-4	-36	-58	-64
2311P	2011P	33.3	45	1122	2694	2739	2728
2321P	2021P	66.6	45	3057	5752	5924	6012
2331P	2031P	100	45	4087	6094	6148	5984

TABLE 9.5 CALCULATED CHANGES IN EXPANSION AND EXPANSION RATES FOR MIXES CONTAINING BELTANE OPAL

Mix Ref No.	0 - 14 Days		14 Days to 2 Months		2 Months to 6 Months		6 Months to 12 Months	
	Change in Expansion (μƐ)	Expansion Rate (μƐ/day)	Change in Expansion (μƐ)	Expansion Rate (μƐ/day)	Change in Expansion (μƐ)	Expansion Rate (μƐ/day)	Change in Expansion (μƐ)	Expansion Rate (μƐ/day)
2011B	88	6.2857	51	1.2143	25	0.2232	8	0.0476
2021B	84	6.0000	88	2.0952	44	0.3929	20	0.1190
2031B	120	8.5714	115	2.7381	60	0.5357	9	0.0536
2041B	276	19.7143	217	5.1667	36	0.3214	-1	-0.0060
2051B	144	10.2857	45	1.0714	11	0.0982	0	0.0000
2061B	112	8.0000	40	0.9524	7	0.0625	-7	-0.0417
2071B	109	7.7857	31	0.7381	13	0.1161	3	0.0179
2311B	112	8.0000	126	3.0000	24	0.2143	2	0.0119
2321B	127	9.0714	166	3.9524	44	0.3929	-13	-0.0774
2331B	160	11.4286	208	4.9524	48	0.4286	-20	-0.1190
2341B	129	9.2143	90	2.1429	32	0.2857	17	0.1012
2351B	121	8.6429	102	2.4286	24	0.2143	1	0.0060
2361B	109	7.7857	115	2.7381	15	0.1339	5	0.0298
2371B	92	6.5714	131	3.1190	29	0.2589	-28	-0.1667
2001	92	6.5714	3	0.0714	10	0.0893	-17	-0.1012
2301	96	6.8571	35	0.8333	32	0.2857	-11	-0.0655

TABLE 9.6 CALCULATED DIFFERENCE BETWEEN EXPANSIONS OF NON-P.F.A. MIXES AND THEIR EQUIVALENT P.F.A. MIXES FOR MORTARS CONTAINING BELTANE OPAL

Mix Ref No. For Non-p.f.a. Mix	Mix Ref No. For P.f.a. Mix	Reactive Aggregate Content (%)	P.f.a. Content (%)	Expansion Difference (με)			
				14 days	2 months	6 months	12 months
2001B	2301B	0	45	-4	-36	-58	-64
2011B	2311B	33.3	45	-24	-99	-98	-92
2021B	2321B	66.6	45	-43	-121	-121	-88
2031B	2331B	100	45	-40	-133	-121	-92
2041B	2341B	4	45	147	274	278	260
2051B	2351B	8	45	23	-34	-47	-48
2061B	2361B	12	45	3	-72	-80	-92
2071B	2371B	16	45	17	-83	-99	-68

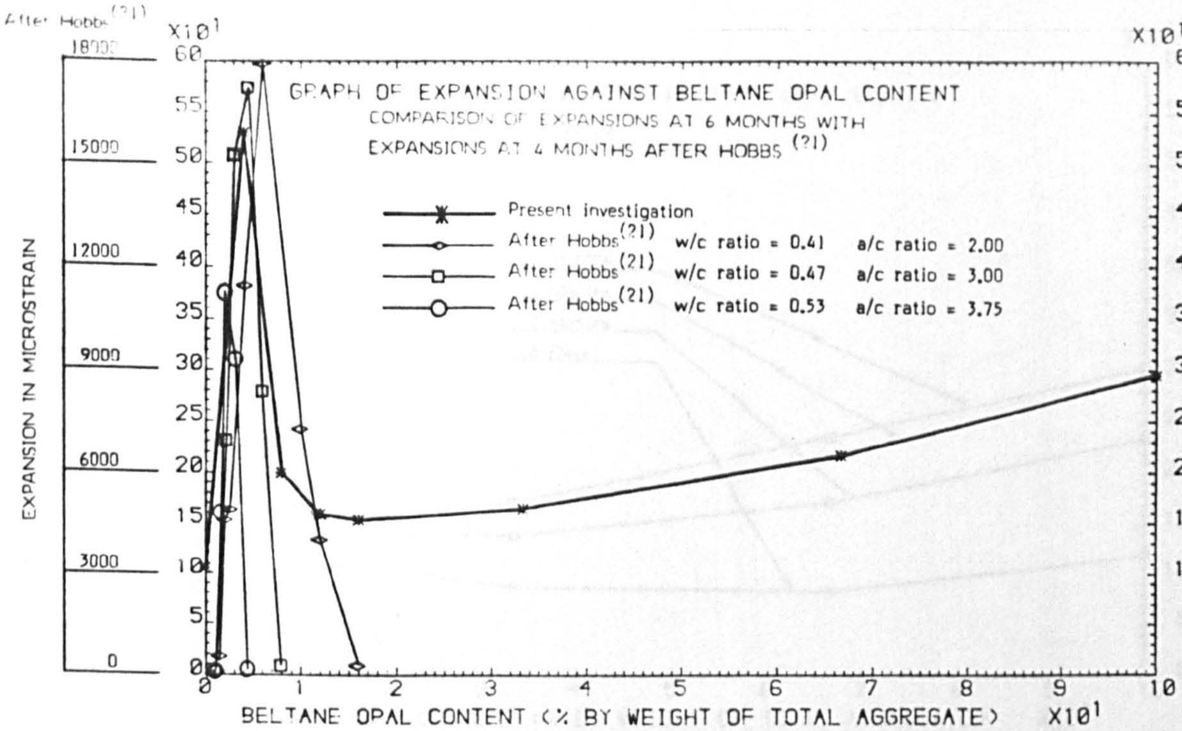


Figure 9.1

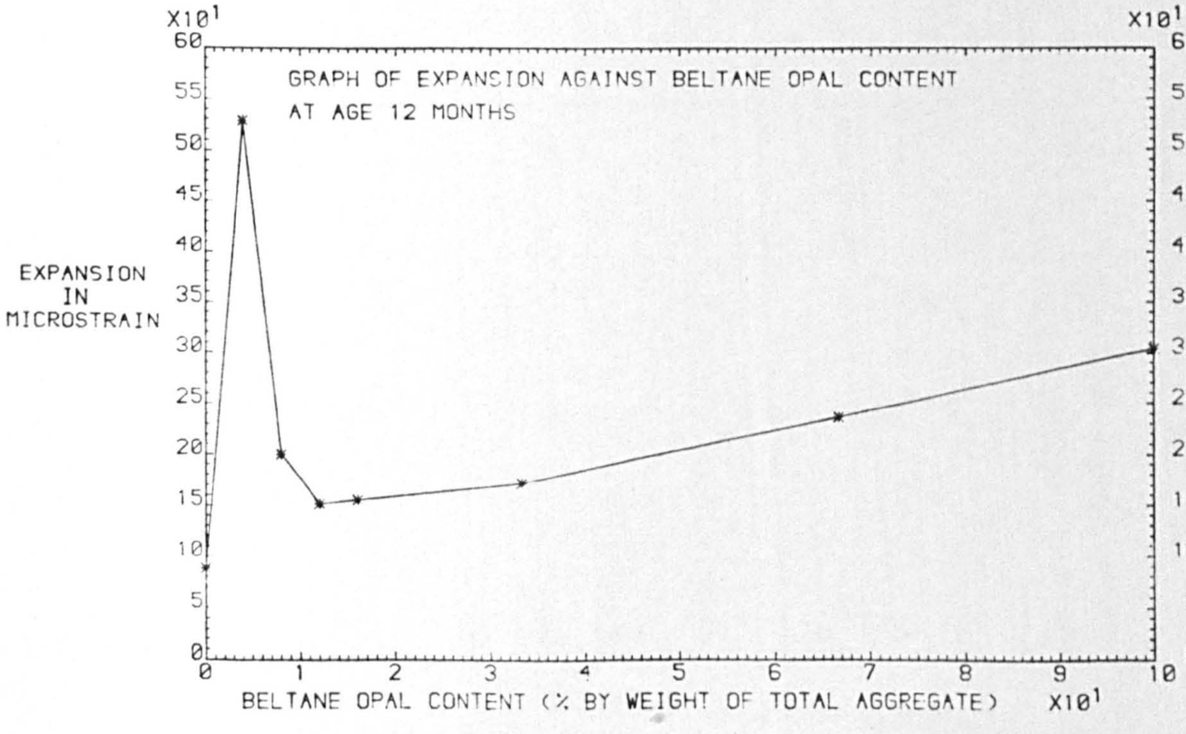


Figure 9.2

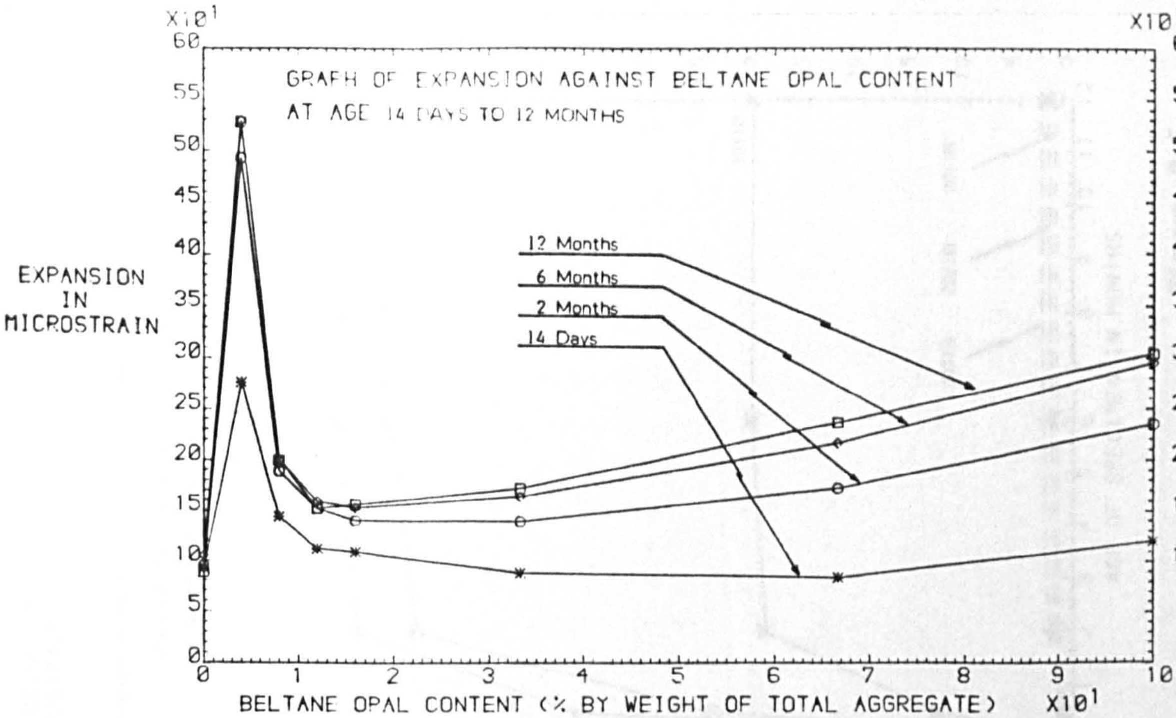
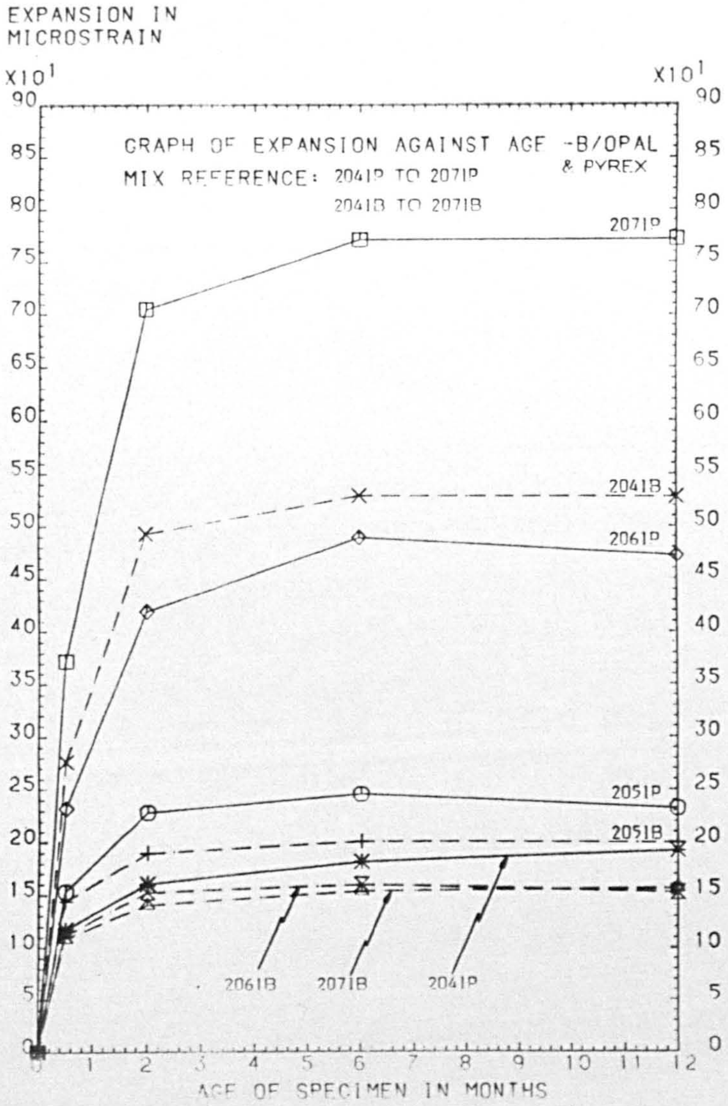

X10¹
60
55
50
45
40
35
30
25
20
15
10
5
0
X10¹
GRAPH OF EXPANSION AGAINST BELTANE OPAL CONTENT
AT AGE 14 DAYS TO 12 MONTHS
EXPANSION IN MICROSTRAIN
12 Months
6 Months
2 Months
14 Days
0
1
2
3
4
5
6
7
8
9
10
BELTANE OPAL CONTENT (% BY WEIGHT OF TOTAL AGGREGATE)
X10¹

Figure 9.3



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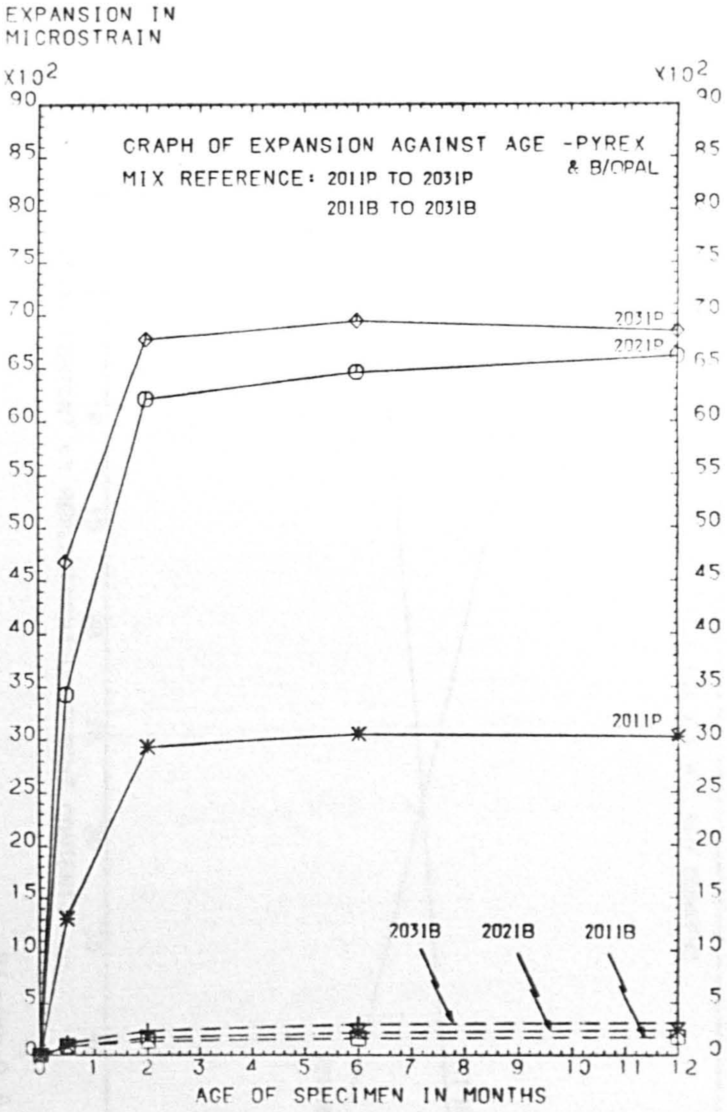
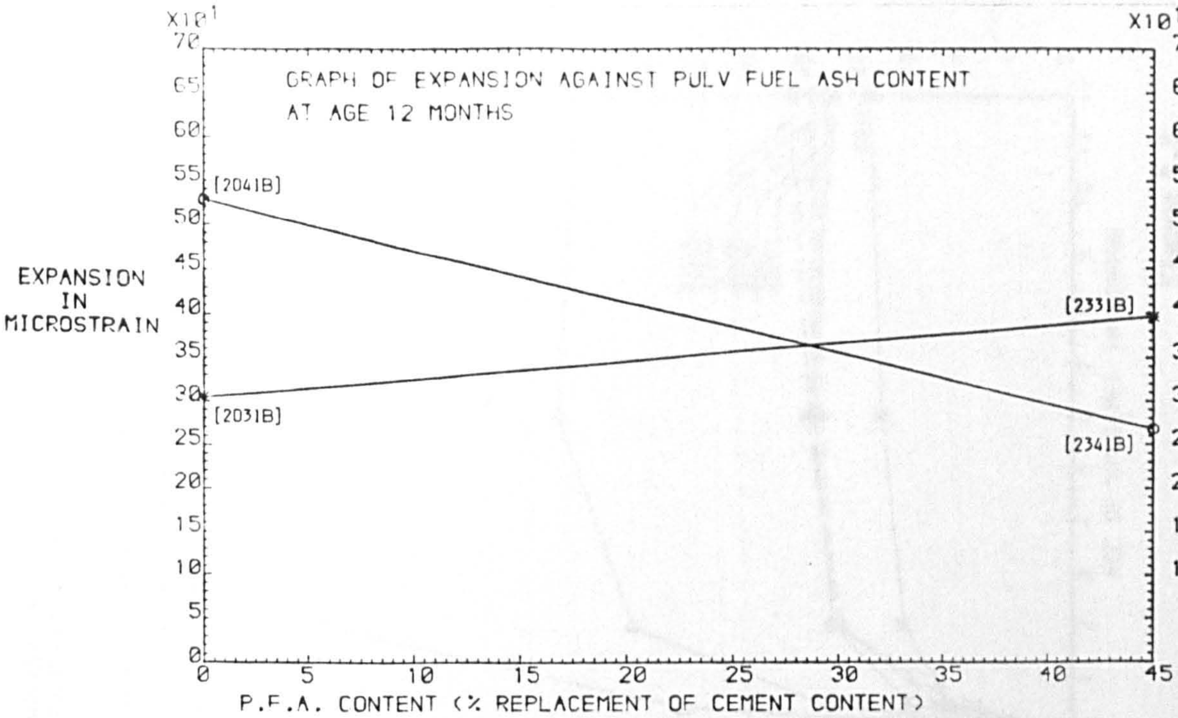
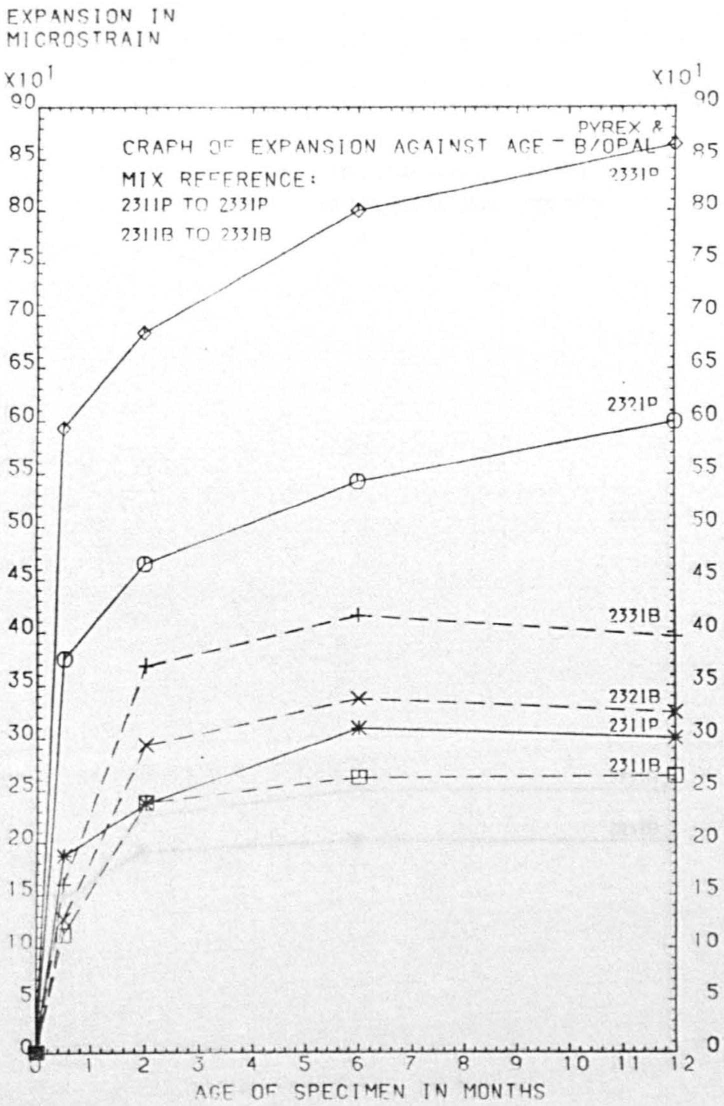


Figure 9.5



X10^1
X10^1
GRAPH OF EXPANSION AGAINST PULV FUEL ASH CONTENT
AT AGE 12 MONTHS
EXPANSION IN MICROSTRAIN
[2041B]
[2331B]
[2031B]
[2341B]
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
P.F.A. CONTENT (% REPLACEMENT OF CEMENT CONTENT)

Figure 9.6



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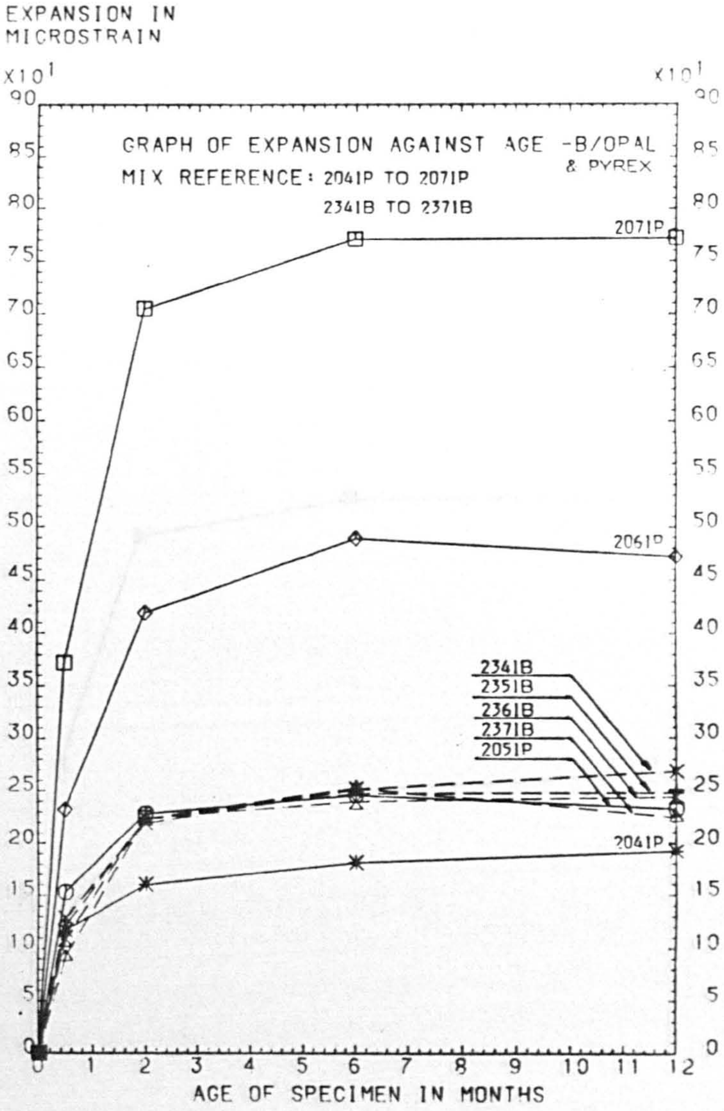

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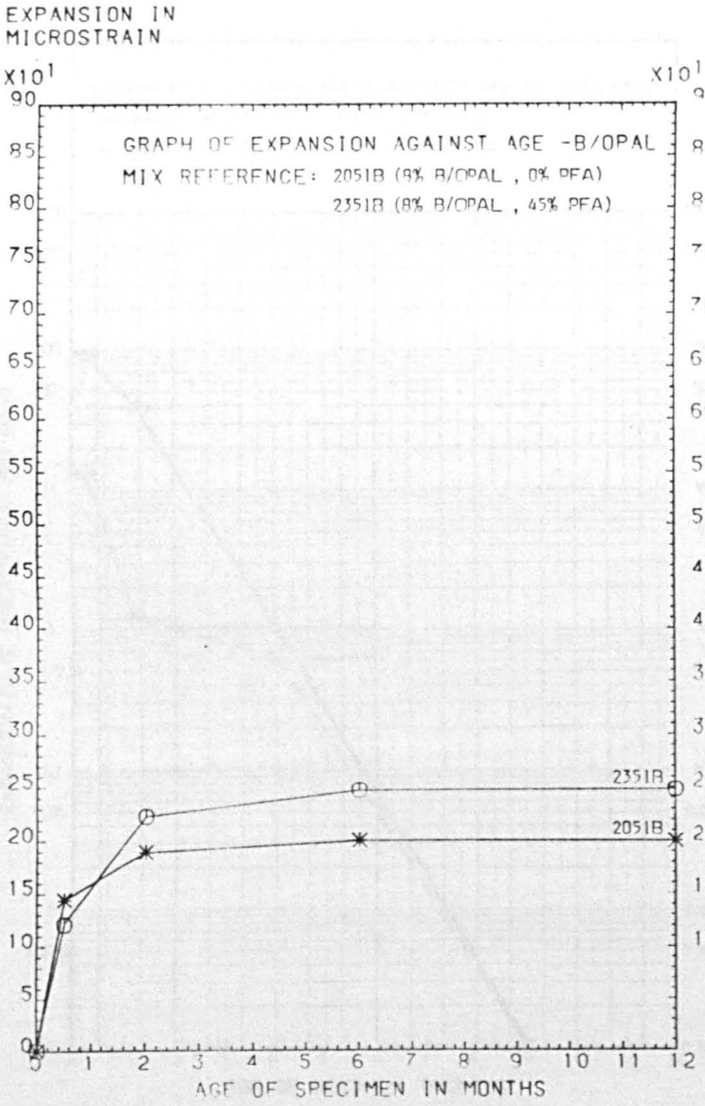

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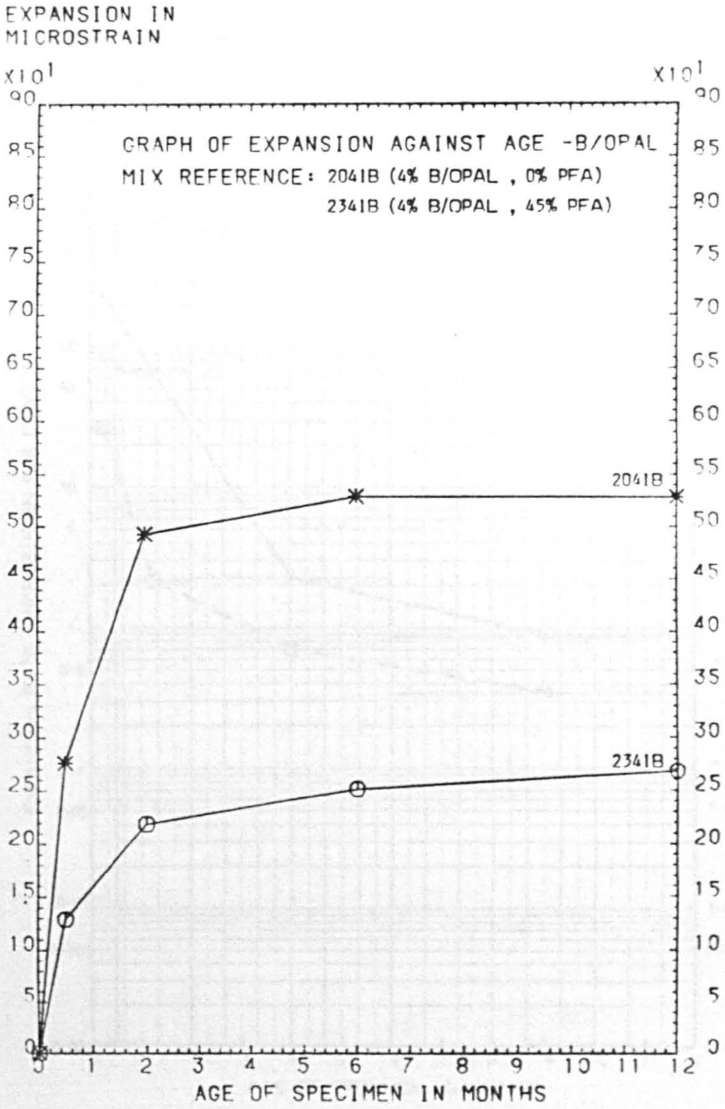

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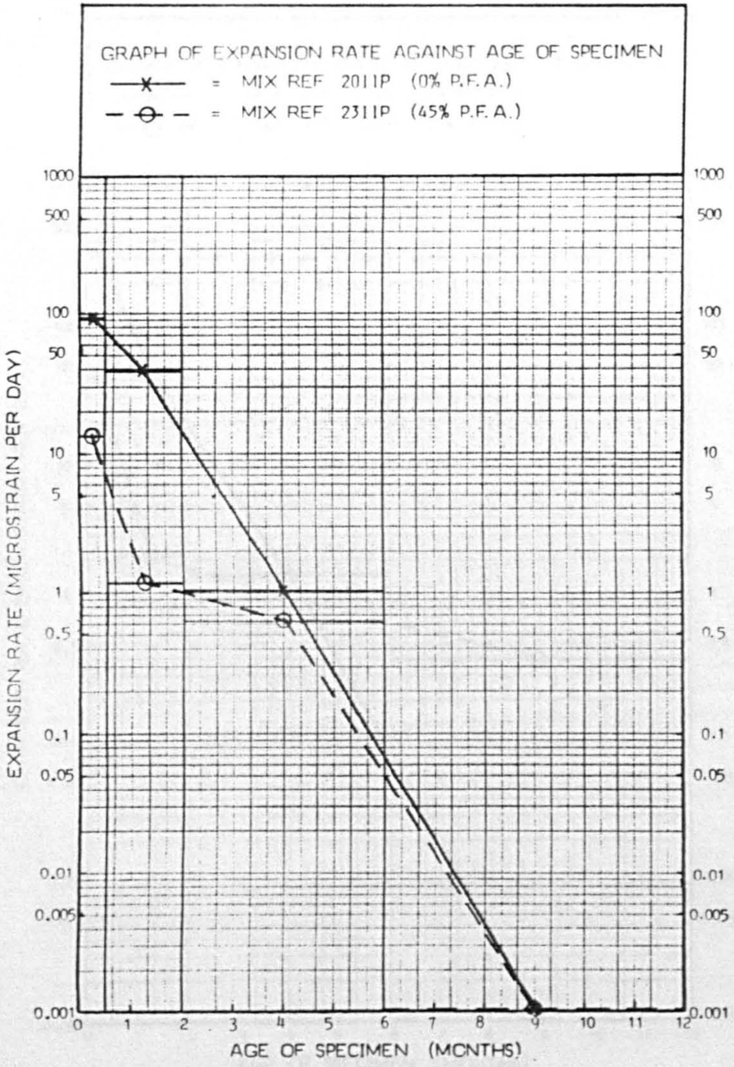

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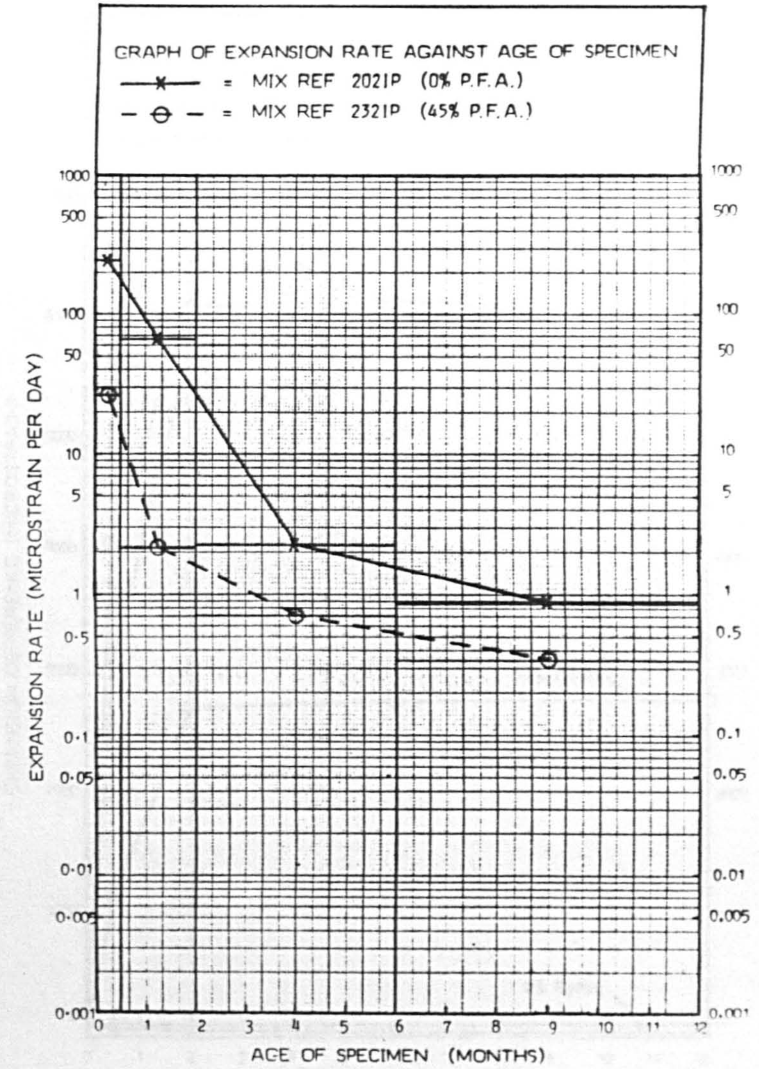

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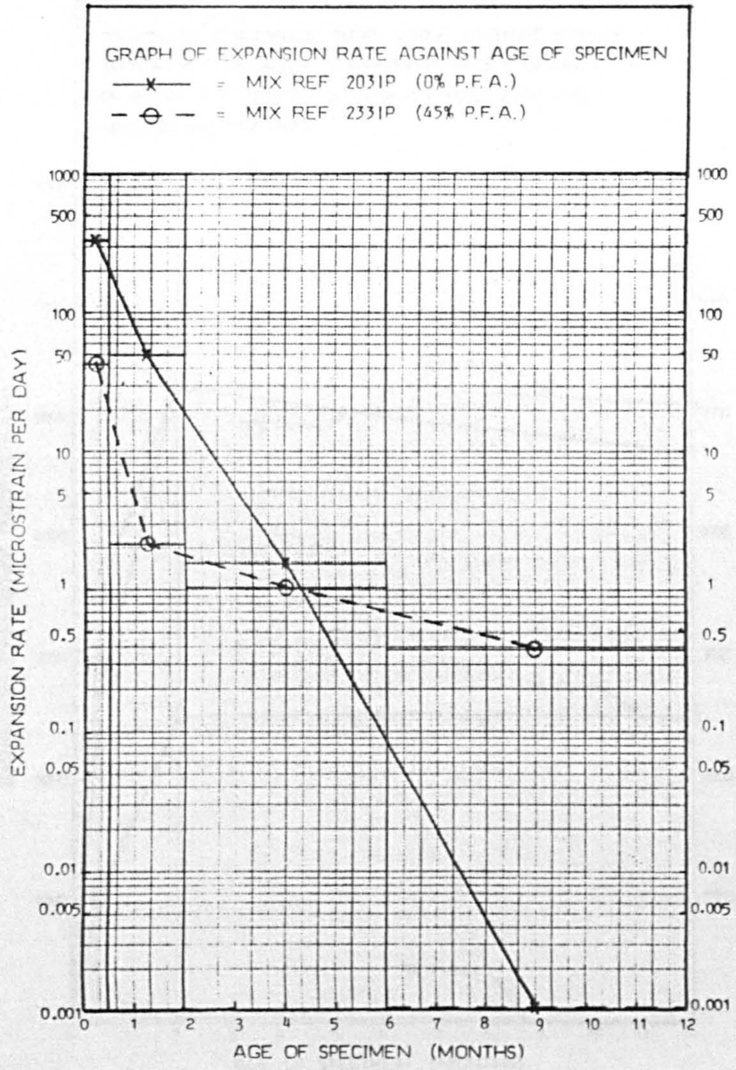

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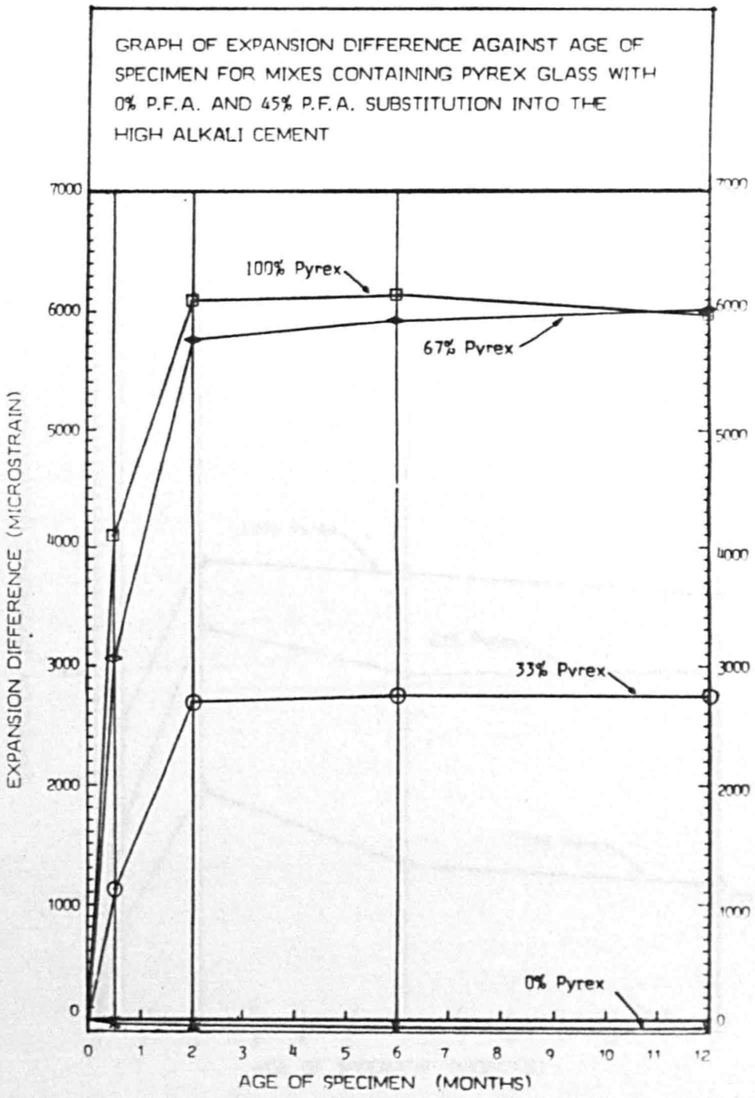

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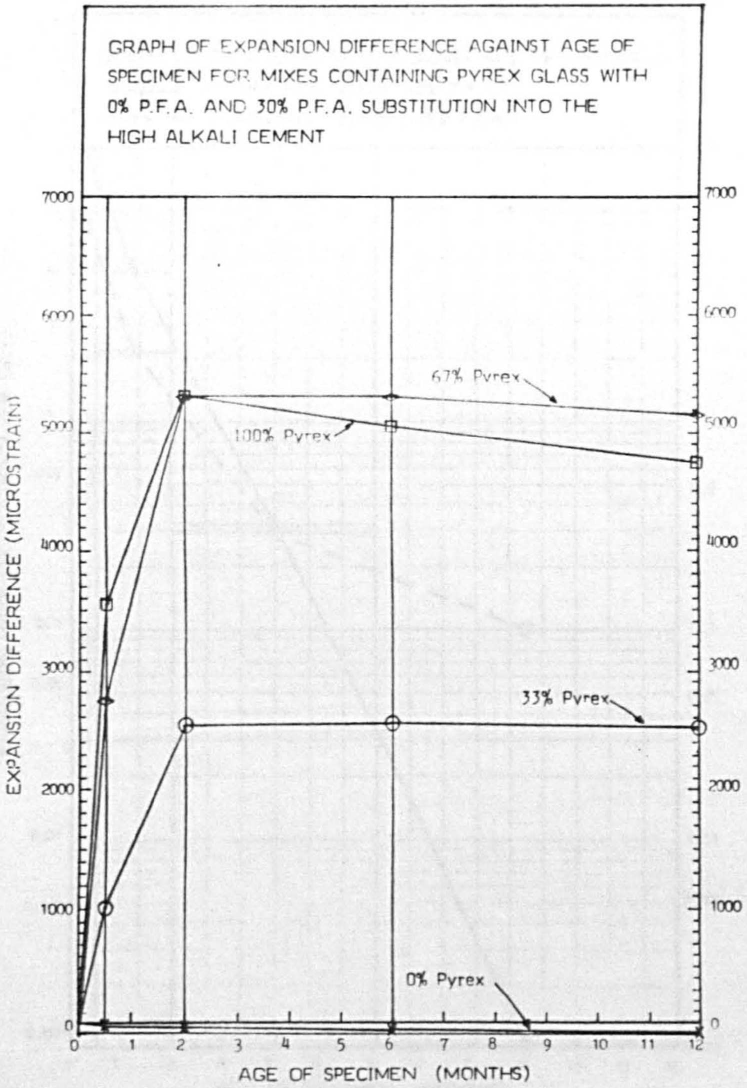

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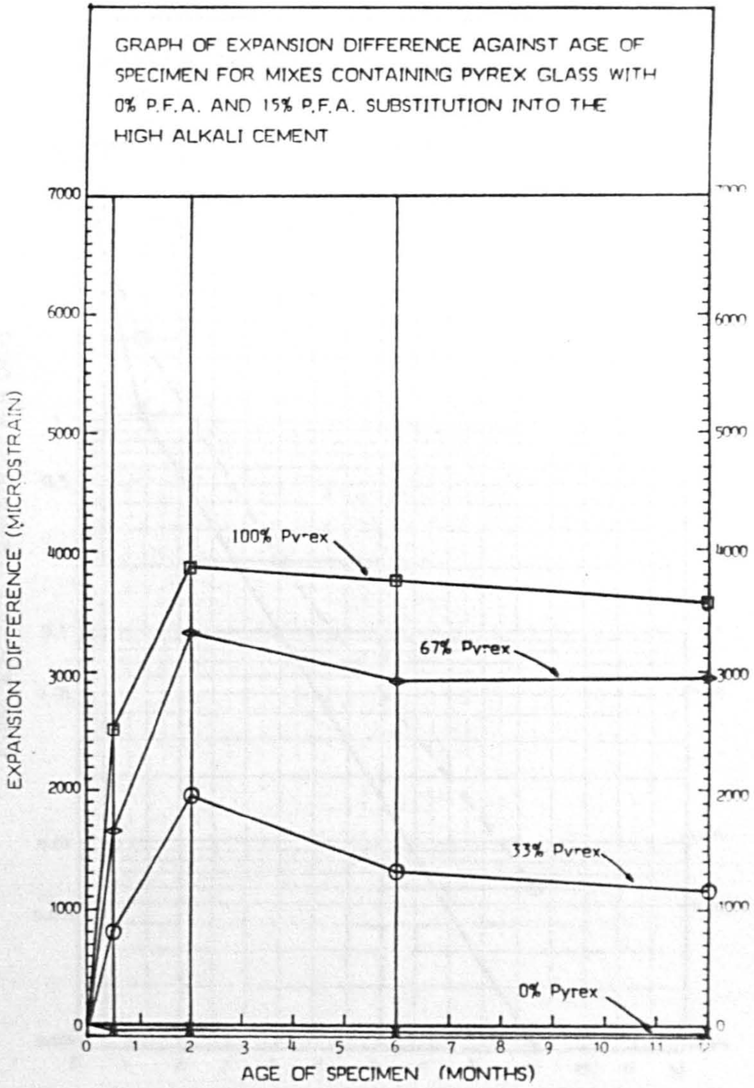

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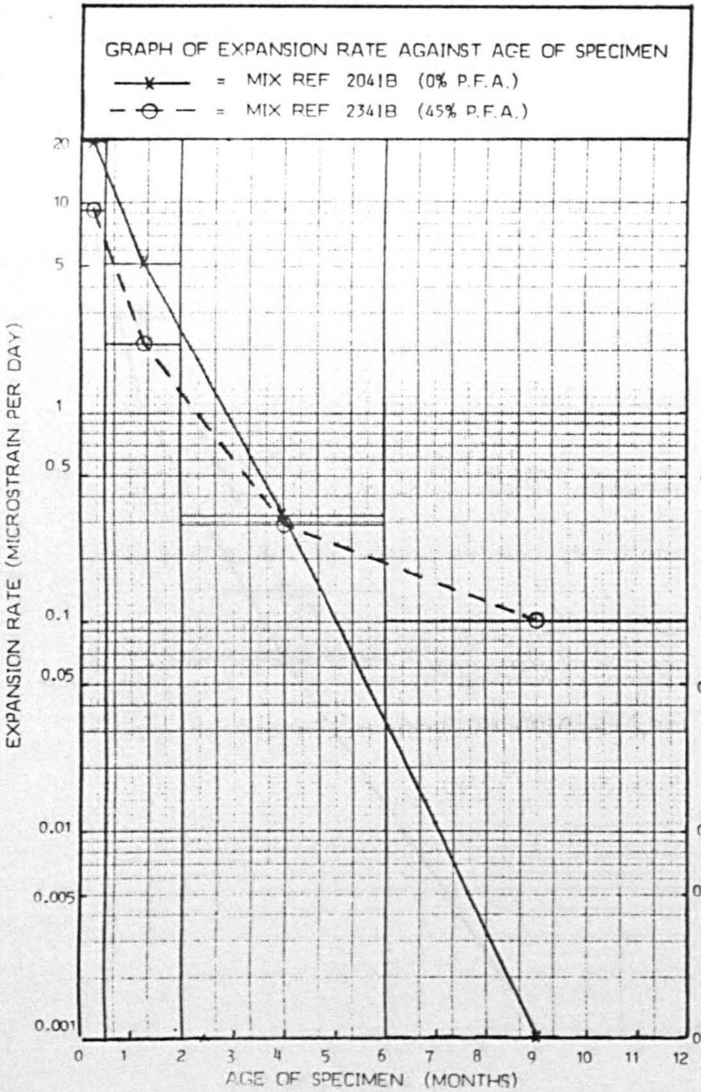

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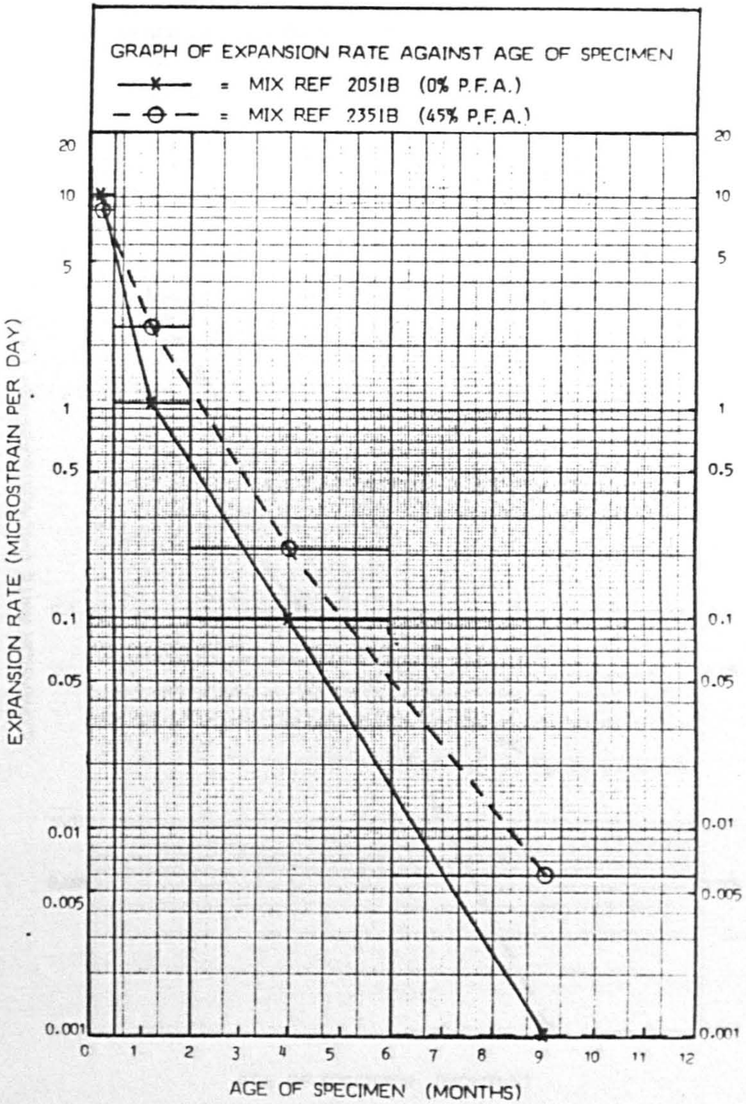
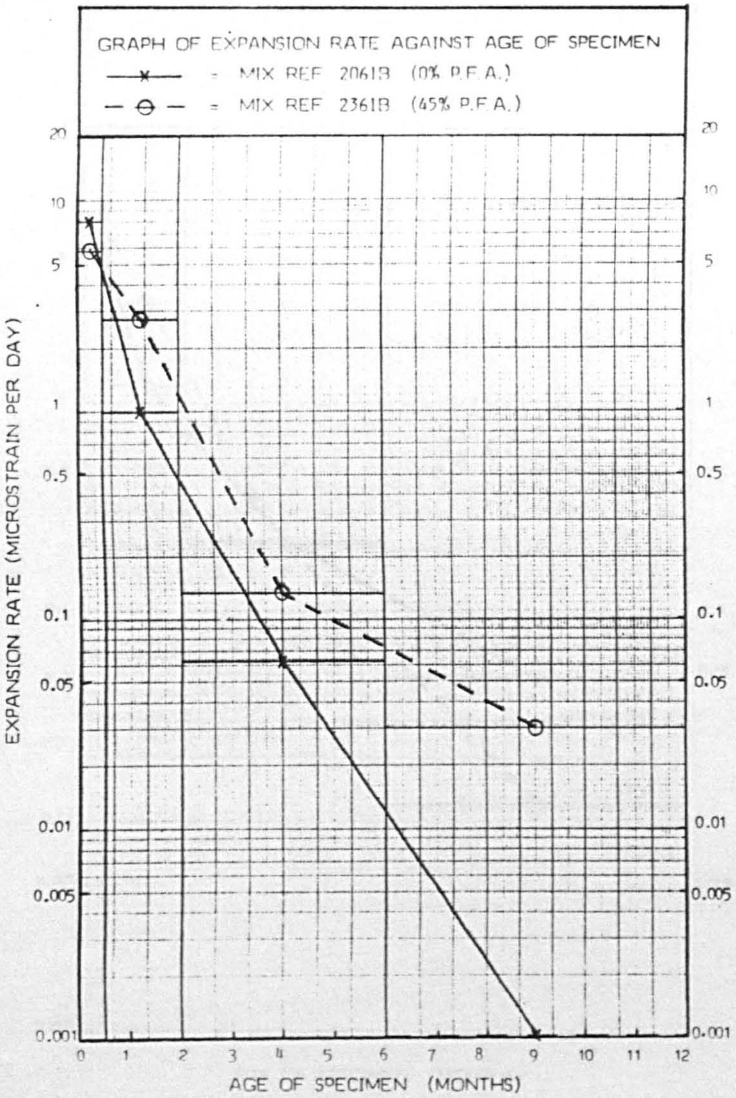


Figure 9.18



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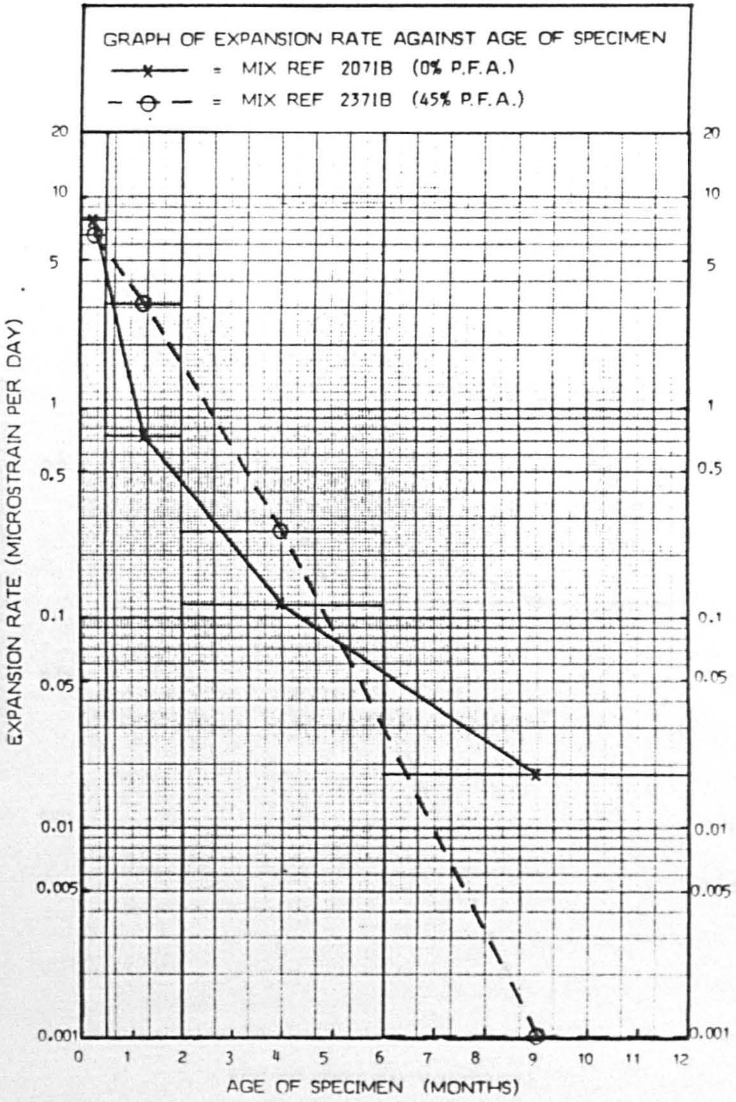

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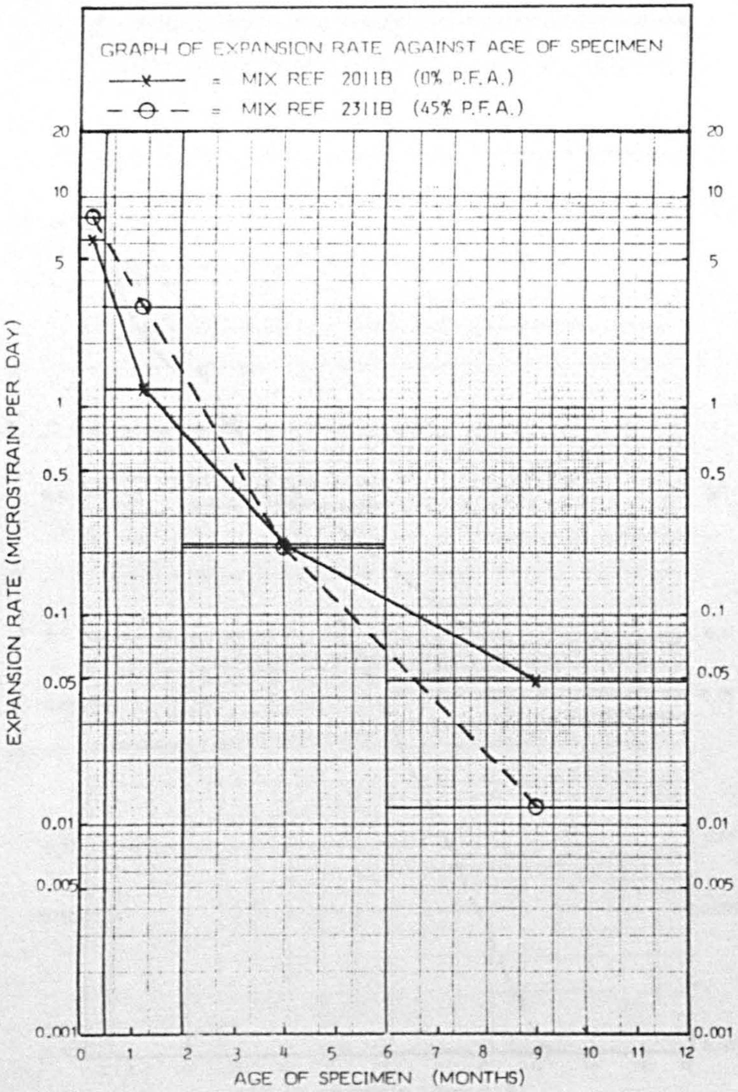

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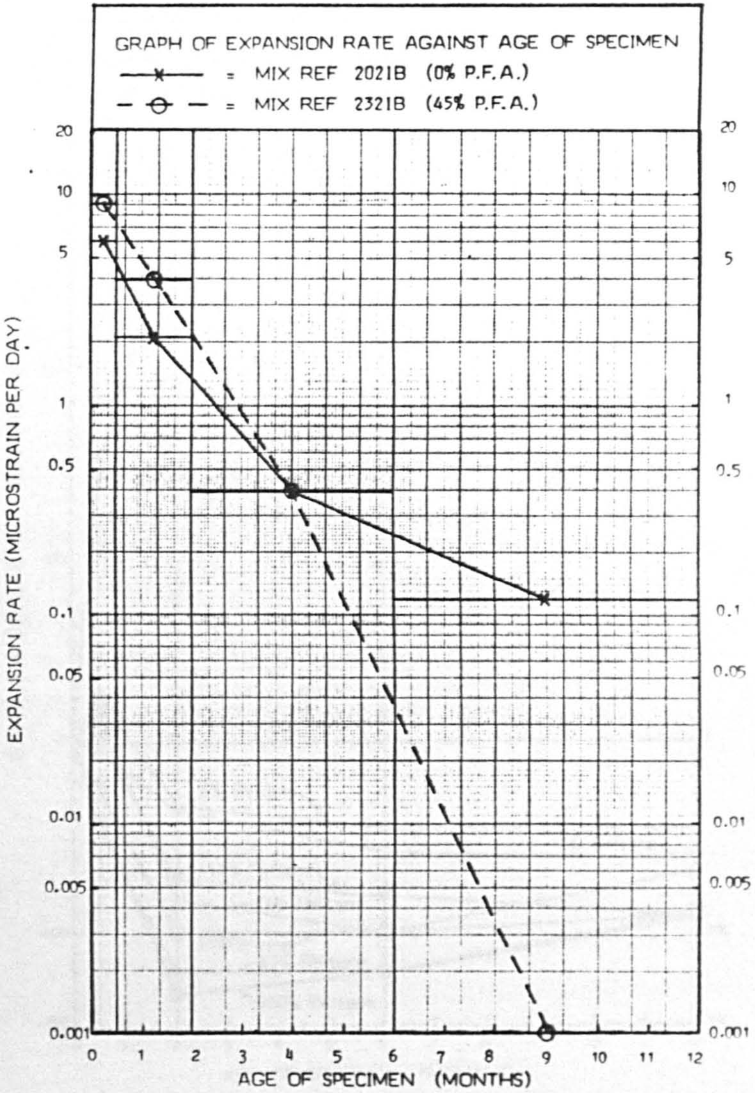

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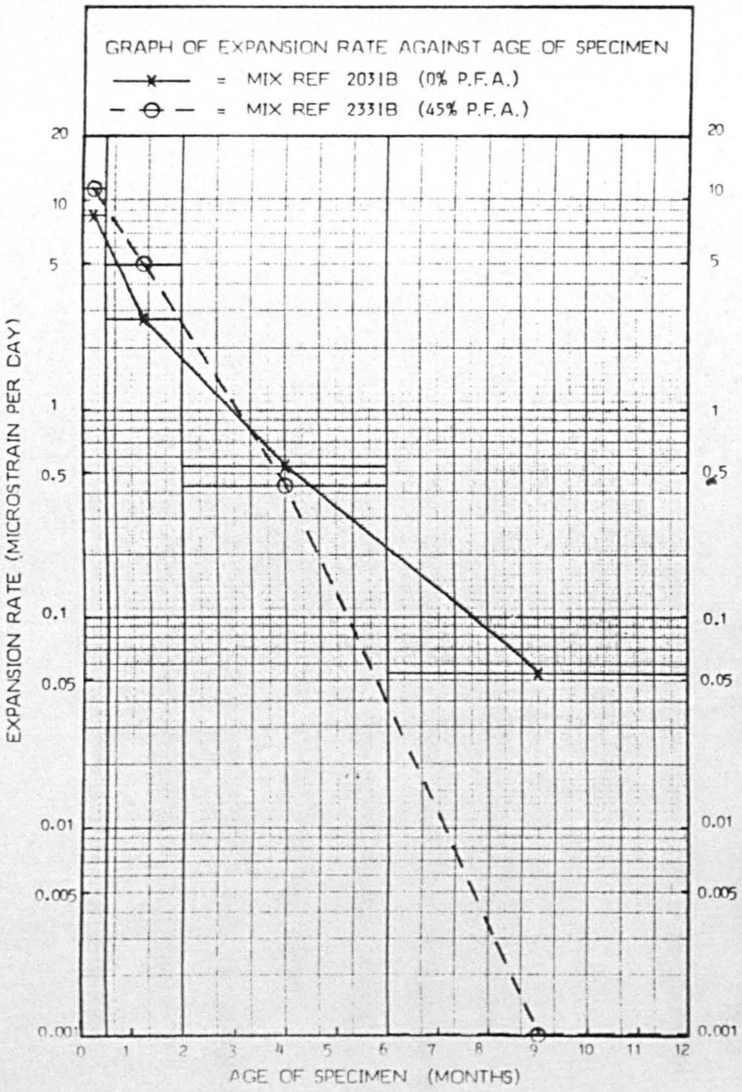

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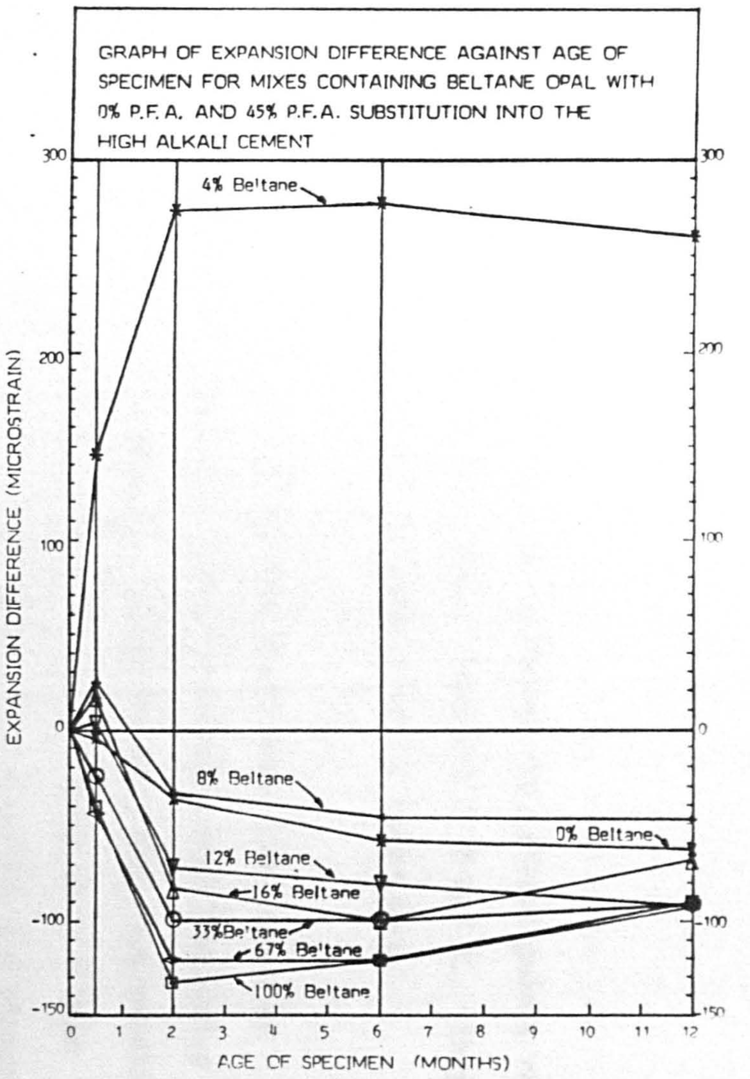


Figure 9.24

CHAPTER 10 CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

10.1 CONCLUSIONS

The following conclusions represent the salient points raised in the discussion sections of Chapters 6, 8 and 9. They are not presented in any specific order of importance.

10.1.1 The expansion of mortar bars containing Pyrex glass as reactive aggregate was found to increase with increasing cement alkali content. For mature specimens, approaching ultimate expansion, there was some evidence of a linear relationship between the cement alkali content and expansion.

10.1.2 The expansion of mortar bars was found to increase with increasing Pyrex glass concentration throughout the range 0% to 100% by weight of total aggregate. There was little evidence to suggest that Pyrex glass exhibits a pessimum concentration of anything less than 100%.

10.1.3 The expansion of mortar bars containing Pyrex glass as reactive aggregate was found to increase with increasing free water content. For mixes which did not contain p.f.a., expansion was found to increase with increasing free water/cement ratio, up to a maximum expansion for a value of 0.55. Above this value a further increase in free water/cement ratio was found to decrease the expansion observed.

10.1.4 The expansion of mortar bars containing Pyrex glass as reactive aggregate was found to be reduced by the use of p.f.a.. This effect was greater when the p.f.a. was used as a cement replacement material but also occurred when the p.f.a. was used as an admixture. Only one exception to this generalisation was noted, for a 20% p.f.a. substitution into the low alkali cement used with 100% Pyrex glass in the preliminary experiments of Series 1.

10.1.5 The effects produced by the cement alkali content, p.f.a., Pyrex glass concentration and free water content on the expansion of mortar bars were found to show considerable interaction. Thus, the effect of each individual factor is partially dependent on the remaining factors.

10.1.6 The reduction in expansion resulting from the use of p.f.a. as a cement replacement material in mixes containing Pyrex glass, was found to be greater than could be explained by alkali dilution theory. That is the p.f.a. does not act as a simple alkali diluent but undergoes a reaction process which causes expansion due to A.S.R. to be inhibited.

10.1.7 P.f.a. was found to be more efficient in reducing the expansion of mixes which contained Pyrex glass when the higher free water content was employed.

10.1.8 The reduction in expansion attributable to the use of p.f.a. in mixes containing Pyrex glass was found to be more stable with time for mixes employing the lower free water content. In addition, this expansion reduction effect was found to be more stable with time for all mixes where the p.f.a. was able to reduce the observed expansion to a value below the A.S.T.M. 'harmful' limits of $500\mu\varepsilon$ at 6 months and $1000\mu\varepsilon$ at 12 months.

10.1.9 The expansion results showed evidence that p.f.a. acts to delay the expansive reaction of mixes containing Pyrex glass, rather than averting it completely. This delay enables the mortar to have gained greater strength before the expansive forces begin to develop more rapidly and, therefore, be more capable of withstanding the internal pressures created by A.S.R.. Further, the lower permeability of the more mature mortar restricts the movement of both the hydroxyl ion to the reactive sites and also the water required for the reaction product to expand.

10.1.10 The rate of expansion of mortar bars containing Pyrex glass was found to decline more slowly with time when p.f.a. was included in the mix. This effect was more prominent for mixes containing the lower p.f.a. substitutions of 15% and 30%.

10.1.11 Reducing the amount of alkali provided by the cement by using a smaller quantity of high alkali cement was found to have a relatively greater effect in reducing expansion than was obtained by using a cement of lower alkali content.

10.1.12 Beltane opal was found to exhibit pronounced pessimum behaviour, the expansion of mortar bars containing Beltane opal as reactive aggregate being at a maximum for a concentration of 4% by weight of total aggregate.

10.1.13 The expansion of mortar bars containing the pessimum Beltane opal concentration was found to be considerably reduced by the use of p.f.a. in partial replacement of the cement content.

10.1.14 The expansion of mortar bars containing Beltane opal in concentrations above the pessimum was found to be increased by the use of p.f.a. in partial replacement of the cement content. This increase was generally less than the reduction in expansion achieved at the pessimum concentration.

10.1.15 The use of p.f.a. in mixes containing Beltane opal was found to cause a complete transformation in the form of the expansion versus Beltane opal content curve.

10.1.16 With both Pyrex glass and Beltane opal as the reactive aggregate the most rapid expansion of the mortar bars was found to occur during the first two months after casting.

10.1.17 There was considerably less expansion observed for the mortar bars containing Beltane opal when compared with their Pyrex glass equivalents.

10.1.18 The overall level of expansion observed with mortar bars containing Beltane opal is very sensitive to changes in experimental conditions. Large differences of up to a factor of thirty can be expected.

10.1.19 In comparing the expansion of mixes containing Pyrex glass with the expansion of mixes containing Beltane opal at its pessimum concentration, the effects produced by p.f.a. were found to be very similar.

10.1.20 In comparing the expansion of mixes containing Pyrex glass with the expansion of mixes containing Beltane opal at any concentration above its pessimum, the effects produced by p.f.a were found to be totally opposite (contrast with 10.1.19).

10.1.21 The differences in the response to p.f.a. which were observed for mixes containing Pyrex glass and Beltane opal can be explained in terms of the relative affinity for the hydroxyl ion (OH^-) shown by the three materials, and the preferential reactions which result. The order of relative affinity for OH^- is considered to be

Beltane opal > p.f.a. > Pyrex glass.

10.1.22 In mortar bars the relative affinity for OH^- shown by p.f.a. is more than 15 times greater than the relative affinity for OH^- shown by Pyrex glass.

10.1.23 In mortar bars the relative affinity for OH^- shown by Beltane opal is in the range 2 to 4 times greater than the relative affinity for OH^- shown by p.f.a.

10.1.24 The substitution of p.f.a for part of the cement content was found to produce an increase in the combined amount of alkali found in the mortar bars and their storage environment. This combined increase was made up from an increase in the acid soluble alkali content of the mortar bars and a decrease in the amount of alkali leached out of the bars.

10.1.25 P.f.a. was found to release sodium and potassium ions, often referred to as 'alkali', as the pozzolanic reaction progressed.

10.1.26 The amount of alkali in the mortar bars and their storage environment was found to increase linearly with increasing cement alkali content. This overall increase comprised increases in both the alkali content of the mortar bars and the amount of alkali leached from the bars and into the liquid at the bottom of the storage containers.

10.1.27 The amount of alkali in the mortar bars and their storage environment was found to increase with increasing Pyrex glass concentration. This overall increase comprised an increase in the alkali content of the mortar bars but a decrease in the amount of alkali leached into the liquid at the bottom of the storage containers.

10.1.28 The effects produced by the cement alkali content, p.f.a., and Pyrex glass concentration on the alkali content of the mortar bars and their storage environment were found to show considerable interaction. Thus, the effect of each individual factor is partially dependent on the remaining factors.

10.1.29 The amount of alkali leached from the bars into the liquid at the bottom of the storage container was found to reduce as the concentration of Beltane opal was increased.

10.1.30 It was found that mortar bars containing considerably more alkali than 3.0kg Na_2O_{eq}/m^3 can be relatively non-expansive, particularly when p.f.a. is used. Conversely, it was also found that mortar bars containing less than 3.0kg Na_2O_{eq}/m^3 can be expansive.

10.1.31 The experimental results contained evidence that in the mortar bars the p.f.a. underwent considerable reaction for the 15%, 30% and 45% cement replacement levels employed. Using the group of mixes 2000 to 2300 and the methods of calculation proposed, the percentage reaction of the p.f.a. was found to lie between a minimum of 68% and a maximum of 94%, based on the amount of alkali released from the p.f.a. during its reaction.

10.1.32 It was found that a sufficient level of p.f.a. substitution should be used to achieve reliable expansion reduction. From the experimental results, 30% to 45% substitution was shown to be satisfactory.

10.1.33 The beneficial effect of p.f.a. in reducing expansion due to A.S.R. is primarily associated with OH^- starvation.

10.1.34 The use of a statistically based method of experimentation, in particular Factorial Analysis, allows deeper insight into the mechanism of the effects produced by the experimental factors than would be gained by more traditional experimentation with concrete. This advantage is primarily associated with the interactions which occur between the experimental factors.

These conclusions are based on observations made with mortar bars produced and conditioned in accordance with the A.S.T.M. mortar bar test method. They are, therefore, directly related to the particular materials used and test method employed. Whilst no particular reasons have been found to suggest that the information presented does not extend beyond the scope of the experimental work, such inference would require verification as suggested in the following section.

10.2 SUGGESTIONS FOR FUTURE WORK

The experimental work carried out in this investigation offers some scope for continuation using the materials and methods already employed. Additional work could also be devised expanding the frame of reference set by the present experimental regime. This latter approach could, in particular, enable the ideas which have been hypothesised to be explored under differing conditions and thus examine whether or not they are universally applicable.

Using similar materials and methods there are certain aspects of the investigation which, with the benefit of hindsight, require examination.

Considering the work carried out using Pyrex glass as the reactive aggregate, the effect produced by p.f.a. has been studied in some considerable depth. However, the effect of p.f.a. in conjunction with the very low Pyrex glass concentrations between 4% and 16% inclusive has not been investigated. It is important that this work should be carried out to ensure that there is no tendency for p.f.a. to increase expansions at such low Pyrex glass concentrations.

With respect to the section of the investigation using Beltane opal as the reactive aggregate, only a limited number of mixes were included. This allowed the use of only the highest alkali cement, the higher free water content and a single level of p.f.a. substitution, namely 45%. A study of the remaining mixes required to bring the experimental coverage with Beltane opal up to the level set by the experiments using Pyrex glass would provide useful extra information. The examination of the effect of lower p.f.a. substitutions would be of particular significance since they might offer further information on why the response is so different to that observed with Pyrex glass.

There is also scope for additional work with Beltane opal at reactive aggregate concentrations other than those already employed. The pessimum effect has been shown to be both of prime importance and, apparently, very localised. It would therefore be pertinent to investigate the response of mixes with Beltane opal concentrations very close to the pessimum value in order to examine precisely how localised the pessimum effect is and over what range of Beltane opal concentration p.f.a. produces a beneficial effect on expansion. Further, from the experimental work conducted, no information is available concerning Beltane opal concentrations below the pessimum. Hence it has not been proven experimentally whether p.f.a. produces an increase or decrease in expansion in this region.

In the discussion of Chapter 9, one aspect of A.S.R. which was considered to have an important effect on expansion was the particle size of the reactive aggregate. This discussion was primarily in relation to two different experimental regimes, both using Beltane opal as the reactive aggregate. One investigation used Beltane opal only in its most reactive size fraction whereas the other, the present investigation, used Beltane opal throughout the whole particle size distribution of the aggregate material. The latter approach was found to be associated with much lower expansions, notwithstanding the other differences in the experimental procedures. Useful information might well be gained from experimental work using Beltane opal where the particle size distribution of the reactive material is the only differing factor. Such work could also be extended to examine the effect on particle size on expansions observed with Pyrex glass. One of the fundamental objectives of any work concerning particle size would be to examine whether or not experimental investigations using only the most highly reactive particle size fraction are a fair representation of the practical situation likely to occur outside the laboratory.

The section of the present investigation involving the chemical analysis of the mortar bars and the liquid from the storage containers was, of necessity, carried out in a step-by-step fashion. Moreover, this section of the work was not part of the originally planned experimental programme. Now that this work has been shown to produce important information it would be appropriate to repeat the exercise using a more formal and controlled experimental procedure. This would enable more definitive comparisons to be made between the results obtained and, if repeated at various intervals, allow examination of how the alkali contents of the mortar bars and their storage environment alter with time.

A natural extension to the overall coverage of the experimental work would be to examine the effects of similar experimental factors on concrete specimens. This could incorporate the use of naturally occurring aggregates, in particular those from the UK, which have been found to cause deleterious reaction in actual concrete structures. The effect produced by the use of p.f.a. would be one of the most important aspects of this type of experimental programme. Presuming that p.f.a. was found to produce significant reductions in the expansion of concrete specimens, equivalent to those predicted by the mortar bar tests, then the performance of p.f.a. in actual structures would remain as the final stage of the argument. Some light could possibly be thrown on this by a field study of structures constructed with concrete containing p.f.a. with an aggregate found elsewhere to be associated with deleterious reaction.

The above suggestions for future work do not attempt to cover the whole topic of A.S.R.. They are, however, intended to draw attention to a few of the possible examples of work which could be undertaken. These would further investigate the hypotheses which have been developed in explanation of the experimental results obtained in this research project.

APPENDIX A

Series 1 - full details of mortar mixes

Tables A.1 to A.2

TABLE A.1 BATCH QUANTITIES, SERIES 1 MORTAR MIXES

Mix Ref No.	Batch Quantities (kg)						Flow Reading
	Cement Content and Type	P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
000	0.440	-	0.990	-	0.240	0.220	108%
001P	0.440	-	0.495	0.495	0.240	0.230	107%
002P	0.440	-	-	0.990	0.257	0.257	115%
010	0.328	0.082	0.990	-	0.225	0.205	102%
011P	0.328 Southam	0.082	0.495	0.495	0.235	0.225	101%
012P	0.328 S.R.P.C.	0.082	-	0.990	0.250	0.250	114%
020	0.230	0.154	0.990	-	0.210	0.190	115%
021P	0.230	0.154	0.495	0.495	0.202	0.192	117%
022P	0.230	0.154	-	0.990	0.230	0.230	115%
100	0.440	-	0.990	-	0.235	0.215	116%
101P	0.440	-	0.495	0.495	0.245	0.235	116%
102P	0.440	-	-	0.990	0.265	0.265	115%
110	0.328	0.082	0.990	-	0.225	0.205	111%
111P	0.328 Masons	0.082	0.495	0.495	0.235	0.225	107%
112P	0.328 O.P.C.	0.082	-	0.990	0.230	0.230	112%
120	0.230	0.154	0.990	-	0.200	0.180	117%
121P	0.230	0.154	0.495	0.495	0.213	0.203	113%
122P	0.230	0.154	-	0.990	0.218	0.218	109%
200	0.440	-	0.990	-	0.240	0.220	116%
201P	0.440	-	0.495	0.495	0.233	0.223	110%
202P	0.440	-	-	0.990	0.245	0.245	111%
210	0.328	0.082	0.990	-	0.205	0.185	108%
211P	0.328 Plymstock	0.082	0.495	0.495	0.235	0.225	107%
212P	0.328 O.P.C.	0.082	-	0.990	0.240	0.240	116%
220	0.230	0.154	0.990	-	0.210	0.190	109%
221P	0.230	0.154	0.495	0.495	0.215	0.205	106%
222P	0.230	0.154	-	0.990	0.207	0.207	100%

TABLE A.2 MIX PROPORTIONS, SERIES 1 MORTAR BARS

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Pyrex	P.f.a.	Free Water	
000	574	1318	0	0	287	2180
001P	551	632	619	0	288	2090
002P	516	0	1162	0	302	1980
010	437	1346	0	109	273	2165
011P	413	636	624	103	283	2060
012P	389	0	1173	97	296	1955
020	313	1374	0	210	258	2155
021P	302	663	650	202	252	2070
022P	282	0	1216	189	268	1945
100	576	1322	0	0	282	2180
101P	544	624	612	0	290	2070
102P	510	0	1148	0	307	1965
110	435	1339	0	109	272	2155
111P	411	633	621	103	282	2050
112P	396	0	1197	99	278	1970
120	316	1389	0	212	248	2165
121P	297	652	639	199	262	2050
122P	282	0	1216	189	268	1955
200	574	1318	0	0	287	2180
201P	554	636	624	0	281	2095
202P	524	0	1179	0	292	1995
210	450	1384	0	112	254	2200
211P	412	635	622	103	283	2055
212P	393	0	1186	98	288	1965
220	313	1374	0	210	258	2155
221P	297	652	639	199	264	2050
222P	287	0	1237	192	259	1975

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%.

APPENDIX B

Series 2 - full details of mortar mixes

Tables B.1 to B.6

TABLE B.1 BATCH QUANTITIES, SERIES 2 FACTORIAL SET OF MORTAR MIXES

Mix Ref No.	Batch Quantities (kg)							Flow Reading
	Cement Content and Type		P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
0000	0.440	Southam S.R.P.C.	-	0.990	-	0.190	0.176	51%
0001	0.440		-	0.990	-	0.256	0.242	25 Blows*
0010P	0.440		-	0.660	0.330	0.185	0.176	35%
0011P	0.440		-	0.660	0.330	0.251	0.242	22 Blows*
0020P	0.440		-	0.330	0.660	0.181	0.176	52%
0021P	0.440		-	0.330	0.660	0.247	0.242	20 Blows*
0030P	0.440		-	-	0.990	0.176	0.176	60%
0031P	0.440		-	-	0.990	0.242	0.242	20 Blows*
0100	0.355		0.063	0.990	-	0.190	0.176	69%
0101	0.355		0.063	0.990	-	0.256	0.242	22 Blows*
0110P	0.355		0.063	0.660	0.330	0.185	0.176	84%
0111P	0.355		0.063	0.660	0.330	0.251	0.242	20 Blows*
0120P	0.355		0.063	0.330	0.660	0.181	0.176	86%
0121P	0.355		0.063	0.330	0.660	0.247	0.242	22 Blows*
0130P	0.355		0.063	-	0.990	0.176	0.176	71%
0131P	0.355		0.063	-	0.990	0.242	0.242	23 Blows*
0200	0.279		0.119	0.990	-	0.190	0.176	99%
0201	0.279		0.119	0.990	-	0.256	0.242	21 Blows*
0210P	0.279		0.119	0.660	0.330	0.185	0.176	107%
0211P	0.279		0.119	0.660	0.330	0.251	0.242	19 Blows*
0220P	0.279		0.119	0.330	0.660	0.181	0.176	90%
0221P	0.279		0.119	0.330	0.660	0.247	0.242	21 Blows*
0230P	0.279		0.119	-	0.990	0.176	0.176	105%
0231P	0.279		0.119	-	0.990	0.242	0.242	18 Blows*
0300	0.209		0.171	0.990	-	0.190	0.176	103%
0301	0.209		0.171	0.990	-	0.256	0.242	18 Blows*
0310P	0.209		0.171	0.660	0.330	0.185	0.176	105%
0311P	0.209		0.171	0.660	0.330	0.251	0.242	16 Blows*
0320P	0.209		0.171	0.330	0.660	0.181	0.176	113%
0321P	0.209		0.171	0.330	0.660	0.247	0.242	17 Blows*
0330P	0.209		0.171	-	0.990	0.176	0.176	115%
0331P	0.209		0.171	-	0.990	0.242	0.242	17 Blows*

Notes:- * denotes FLOW > 150% (number of blows causing mortar to flow off edge of flow table recorded).

TABLE B.1 continued

Mix Ref No.	Batch Quantities (kg)						Flow Reading
	Cement Content and Type	P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
1000	0.440	-	0.990	-	0.190	0.176	50%
1001	0.440	-	0.990	-	0.256	0.242	25 Blows*
1010P	0.440	-	0.660	0.330	0.185	0.176	51%
1011P	0.440	-	0.660	0.330	0.251	0.242	22 Blows*
1020P	0.440	-	0.330	0.660	0.181	0.176	54%
1021P	0.440	-	0.330	0.660	0.247	0.242	22 Blows*
1030P	0.440	-	-	0.990	0.176	0.176	55%
1031P	0.440	-	-	0.990	0.242	0.242	22 Blows*
1100	0.355	0.063	0.990	-	0.190	0.176	79%
1101	0.355	0.063	0.990	-	0.256	0.242	25 Blows*
1110P	0.355	0.063	0.660	0.330	0.185	0.176	74%
1111P	0.355	0.063	0.660	0.330	0.251	0.242	21 Blows*
1120P	0.355	0.063	0.330	0.660	0.181	0.176	85%
1121P	0.355	0.063	0.330	0.660	0.247	0.242	20 Blows*
1130P	0.355	0.063	-	0.990	0.176	0.176	79%
1131P	0.355 Masons	0.063	-	0.990	0.242	0.242	21 Blows*
1200	0.279 O.P.C.	0.119	0.990	-	0.190	0.176	94%
1201	0.279	0.119	0.990	-	0.256	0.242	18 Blows*
1210P	0.279	0.119	0.660	0.330	0.185	0.176	91%
1211P	0.279	0.119	0.660	0.330	0.251	0.242	19 Blows*
1220P	0.279	0.119	0.330	0.660	0.181	0.176	98%
1221P	0.279	0.119	0.330	0.660	0.247	0.242	18 Blows*
1230P	0.279	0.119	-	0.990	0.176	0.176	102%
1231P	0.279	0.119	-	0.990	0.242	0.242	19 Blows*
1300	0.209	0.171	0.990	-	0.190	0.176	105%
1301	0.209	0.171	0.990	-	0.256	0.242	19 Blows*
1310P	0.209	0.171	0.660	0.330	0.185	0.176	108%
1311P	0.209	0.171	0.660	0.330	0.251	0.242	17 Blows*
1320P	0.209	0.171	0.330	0.660	0.181	0.176	108%
1321P	0.209	0.171	0.330	0.660	0.247	0.242	16 Blows*
1330P	0.209	0.171	-	0.990	0.176	0.176	114%
1331P	0.209	0.171	-	0.990	0.242	0.242	14 Blows*

Notes:- * denotes FLOW > 150% (number of blows causing mortar to flow off edge of flow table recorded).

TABLE B.1 continued

Mix Ref No.	Batch Quantities (kg)						Flow Reading
	Cement Content and Type	P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
2000	0.440	-	0.990	-	0.190	0.176	67%
2001	0.440	-	0.990	-	0.256	0.242	24 Blows*
2010P	0.440	-	0.660	0.330	0.185	0.176	64%
2011P	0.440	-	0.660	0.330	0.251	0.242	25 Blows*
2020P	0.440	-	0.330	0.660	0.181	0.176	64%
2021P	0.440	-	0.330	0.660	0.247	0.242	23 Blows*
2030P	0.440	-	-	0.990	0.176	0.176	66%
2031P	0.440	-	-	0.990	0.242	0.242	22 Blows*
2100	0.355	0.063	0.990	-	0.190	0.176	81%
2101	0.355	0.063	0.990	-	0.256	0.242	20 Blows*
2110P	0.355	0.063	0.660	0.330	0.185	0.176	82%
2111P	0.355	0.063	0.660	0.330	0.251	0.242	21 Blows*
2120P	0.355	0.063	0.330	0.660	0.181	0.176	94%
2121P	0.355	0.063	0.330	0.660	0.247	0.242	19 Blows*
2130P	0.355	0.063	-	0.990	0.176	0.176	92%
2131P	0.355 Oxford	0.063	-	0.990	0.242	0.242	17 Blows*
2200	0.279 O.P.C.	0.119	0.990	-	0.190	0.176	105%
2201	0.279	0.119	0.990	-	0.256	0.242	17 Blows*
2210P	0.279	0.119	0.660	0.330	0.185	0.176	108%
2211P	0.279	0.119	0.660	0.330	0.251	0.242	17 Blows*
2220P	0.279	0.119	0.330	0.660	0.181	0.176	111%
2221P	0.279	0.119	0.330	0.660	0.247	0.242	18 Blows*
2230P	0.279	0.119	-	0.990	0.176	0.176	113%
2231P	0.279	0.119	-	0.990	0.242	0.242	17 Blows*
2300	0.209	0.171	0.990	-	0.190	0.176	113%
2301	0.209	0.171	0.990	-	0.256	0.242	17 Blows*
2310P	0.209	0.171	0.660	0.330	0.185	0.176	123%
2311P	0.209	0.171	0.660	0.330	0.251	0.242	15 Blows*
2320P	0.209	0.171	0.330	0.660	0.181	0.176	119%
2321P	0.209	0.171	0.330	0.660	0.247	0.242	14 Blows*
2330P	0.209	0.171	-	0.990	0.176	0.176	124%
2331P	0.209	0.171	-	0.990	0.242	0.242	-

Notes:- * denotes FLOW > **150%** (number of blows causing mortar to flow off edge of flow table recorded).

TABLE B.2 MIX PROPORTIONS, SERIES 2 FACTORIAL SET OF MORTAR BARS

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Pyrex	P.f.a.	Free Water	
0000	617	1407	0	0	247	2270
0001	564	1286	0	0	310	2160
0010P	601	913	451	0	240	2205
0011P	550	836	412	0	302	2100
0020P	584	445	877	0	234	2140
0021P	537	409	805	0	295	2045
0030P	570	0	1282	0	228	2080
0031P	525	0	1181	0	289	1995
0100	498	1407	0	88	247	2240
0101	455	1288	0	81	310	2135
0110P	485	913	451	86	240	2175
0111P	444	837	413	79	303	2075
0120P	471	445	876	84	234	2110
0121P	433	409	806	77	295	2020
0130P	459	0	1281	82	228	2050
0131P	423	0	1179	75	288	1965
0200	392	1409	0	167	247	2215
0201	358	1289	0	153	311	2110
0210P	381	914	451	163	241	2150
0211P	349	837	413	149	303	2050
0220P	371	445	877	158	234	2085
0221P	340	409	805	145	295	1995
0230P	361	0	1282	154	228	2025
0231P	333	0	1181	142	289	1945
0300	293	1409	0	240	247	2190
0301	268	1287	0	219	310	2085
0310P	286	914	451	234	241	2125
0311P	262	838	413	214	303	2030
0320P	278	445	877	227	234	2060
0321P	255	409	806	209	296	1975
0330P	271	0	1284	222	228	2005
0331P	249	0	1179	204	288	1920

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%

TABLE B.2 continued

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Pyrex	P.f.a.	Free Water	
1000	614	1401	0	0	246	2260
1001	562	1283	0	0	309	2155
1010P	598	909	449	0	239	2195
1011P	548	834	411	0	302	2095
1020P	583	444	875	0	233	2135
1021P	535	408	803	0	294	2040
1030P	568	0	1279	0	227	2075
1031P	522	0	1175	0	287	1985
1100	497	1404	0	88	246	2235
1101	453	1282	0	80	309	2125
1110P	484	911	450	86	240	2170
1111P	443	835	412	79	302	2070
1120P	470	444	874	83	233	2105
1121P	432	408	804	77	295	2015
1130P	458	0	1278	81	227	2045
1131P	422	0	1176	75	287	1960
1200	391	1406	0	167	246	2210
1201	357	1286	0	152	310	2105
1210P	380	912	450	162	240	2145
1211P	348	835	412	148	302	2045
1220P	370	444	875	158	233	2085
1221P	340	408	803	145	295	1990
1230P	360	0	1279	154	227	2020
1231P	332	0	1178	142	288	1940
1300	293	1406	0	240	247	2185
1301	267	1284	0	219	310	2080
1310P	285	912	450	233	240	2120
1311P	261	836	412	214	302	2025
1320P	278	445	877	227	234	2060
1321P	255	408	804	208	295	1970
1330P	270	0	1281	221	228	2000
1331P	249	0	1179	204	288	1920

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%

TABLE B.2 continued

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Pyrex	P.f.a.	Free Water	
2000	612	1398	0	0	245	2255
2001	561	1280	0	0	309	2150
2010P	597	907	447	0	239	2190
2011P	547	832	410	0	301	2090
2020P	582	443	873	0	233	2130
2021P	534	407	801	0	294	2035
2030P	567	0	1276	0	227	2070
2031P	521	0	1172	0	287	1980
2100	495	1401	0	88	246	2230
2101	453	1282	0	80	309	2125
2110P	482	909	448	86	239	2165
2111P	442	833	411	78	301	2065
2120P	469	443	872	83	233	2100
2121P	431	407	802	77	294	2010
2130P	457	0	1275	81	227	2040
2131P	422	0	1176	75	287	1960
2200	390	1403	0	166	246	2205
2201	356	1282	0	152	309	2100
2210P	380	910	449	162	239	2140
2211P	348	835	412	148	302	2045
2220P	370	444	875	158	233	2080
2221P	340	408	803	145	295	1990
2230P	360	0	1279	154	227	2020
2231P	331	0	1175	141	287	1935
2300	292	1403	0	239	246	2180
2301	267	1284	0	219	310	2080
2310P	284	910	449	233	239	2115
2311P	260	834	411	213	302	2020
2320P	277	444	874	227	233	2055
2321P	255	408	804	208	295	1970
2330P	270	0	1281	221	228	2000
2331P	248	0	1176	203	288	1915

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%

TABLE B.3 BATCH QUANTITIES, SERIES 2 'EXTRA PYREX' MORTAR MIXES

Mix Ref No.	Batch Quantities (kg)						Flow Reading
	Cement Content and Type	P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
2032P	0.440	-	-	0.990	0.154	0.154	Friable **
2033P	0.440	-	-	0.990	0.198	0.198	111%
2034P	0.440	-	-	0.990	0.220	0.220	136%
2035P	0.440	-	-	0.990	0.264	0.264	17 Blows *
2041P	0.440	-	0.950	0.040	0.255	0.242	22 Blows *
2051P	0.440	-	0.911	0.079	0.255	0.242	22 Blows *
2061P	0.440	-	0.871	0.119	0.254	0.242	23 Blows *
2071P	0.440	-	0.832	0.158	0.254	0.242	24 Blows *
2431P	0.440 Oxford	0.044	-	0.990	0.266	0.266	19 Blows *
2531P	0.440 O.P.C.	0.088	-	0.990	0.290	0.290	16 Blows *
2631P	0.440	0.132	-	0.990	0.315	0.315	13 Blows *
2731P	0.440	0.176	-	0.990	0.339	0.339	11 Blows *
3031P	0.130	-	-	0.990	0.072	0.072	Friable **
4031P	0.165	-	-	0.990	0.098	0.098	Friable **
5031P	0.202	-	-	0.990	0.111	0.111	Friable **
6031P	0.277	-	-	0.990	0.152	0.152	38%

Note:- * = Flow > 150% (number of blows causing mortar to flow off edge of flow table recorded).

** = Mix too dry for any measurable flow.

TABLE B.4 MIX PROPORTIONS, SERIES 2 'EXTRA PYREX' SET OF MORTAR BARS

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Pyrex	P.f.a.	Free Water	
2032P	583	0	1313	0	204	2100
2033P	551	0	1241	0	248	2040
2034P	536	0	1206	0	268	2010
2035P	508	0	1143	0	305	1955
2041P	558	1224	51	0	307	2140
2051P	558	1171	100	0	307	2135
2061P	555	1114	150	0	305	2125
2071P	554	1062	199	0	305	2120
2431P	496	0	1115	50	300	1960
2531P	473	0	1065	95	312	1945
2631P	451	0	1015	135	323	1925
2731P	432	0	972	173	333	1910
3031P	229	0	1744	0	127	2100
4031P	276	0	1657	0	152	2085
5031P	320	0	1569	0	176	2065
6031P	397	0	1420	0	218	2035

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%.

TABLE B.5 BATCH QUANTITIES, SERIES 2 'BELTANE OPAL' MORTAR MIXES

Mix Ref No.	Batch Quantities (kg)						Flow Reading
	Cement Content and Type	P.f.a. Content	Dry Sand Content	Pyrex Glass Content	Total Water Content	Free Water Content	
2011B	0.440	-	0.660	0.330	0.262	0.242	142%
2021B	0.440	-	0.330	0.660	0.268	0.242	117%
2031B	0.440	-	-	0.990	0.274	0.242	97%
2041B	0.440	-	0.950	0.040	0.257	0.242	154%
2051B	0.440	-	0.911	0.079	0.257	0.242	25 Blows *
2061B	0.440	-	0.871	0.119	0.258	0.242	25 Blows *
2071B	0.440 Oxford	-	0.832	0.158	0.259	0.242	24 Blows *
2311B	0.209 O.P.C.	0.171	0.660	0.330	0.262	0.242	21 Blows *
2321B	0.209	0.171	0.330	0.660	0.268	0.242	25 Blows *
2331B	0.209	0.171	-	0.990	0.274	0.242	132%
2341B	0.209	0.171	0.950	0.040	0.257	0.242	17 Blows *
2351B	0.209	0.171	0.911	0.079	0.257	0.242	16 Blows *
2361B	0.209	0.171	0.871	0.119	0.258	0.242	16 Blows *
2371B	0.209	0.171	0.832	0.158	0.259	0.242	17 Blows *

Note:- * = Flow > 150% (number of blows causing mortar to flow off edge of flow table recorded).

TABLE B.6 MIX PROPORTIONS, SERIES 2 'BELTANE OPAL' SET OF MORTAR BARS

Mix Ref No.	Mix Proportions (kg/m^3)					* Calculated Wet Density (kg/m^3)
	Cement	S.S.D. Sand	Beltane Opal	P.f.a.	Free Water	
2011B	536	815	415	0	295	2060
2021B	514	392	796	0	283	1985
2031B	493	0	1146	0	271	1910
2041B	557	1220	52	0	306	2135
2051B	554	1163	103	0	305	2125
2061B	551	1106	154	0	303	2115
2071B	549	1051	203	0	302	2105
2311B	255	818	417	209	296	1995
2321B	245	393	798	200	284	1920
2331B	235	0	1150	192	272	1850
2341B	266	1226	52	218	308	2070
2351B	264	1169	104	216	306	2060
2361B	263	1112	155	215	305	2050
2371B	262	1056	204	214	303	2040

Note:- * = Air content of fully compacted wet mortar assumed to be 1.5%.

APPENDIX C

Series 1 - full details of expansion results from 3^3 full factorial set of experiments

Tables C.1 to C.3

Figures C.1 to C.27

TABLE C.1 SERIES 1 MORTAR BAR EXPANSION RESULTS

MIX REF NO.	EXPANSION IN MICROSTRAIN ($\mu\varepsilon$) AT AGE:-						'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	28 DAYS	2 MONTHS	4 MONTHS	14 MONTHS	18 MONTHS	
000	84	100	85	113	121	-	-
001 P	75	95	79	105	127 *	-	-
002 P	165	305 *	495 +	571 +	661 +	-	-
010	43	55	15	41	83	-	-
011 P	59	60	52	73	129	-	-
012 P	132	179	243	413	892	-	-
020	57	44	25	49	124	-	-
021 P	68	48	31	35	116	-	-
022 P	136	135	163	191	289	-	-
100	61	67	93	107	105 *	-	-
101 P	249	341	405	445	472	-	-
102 P	1151	1672	1835	1987	2075	-	Harmful
110	45	31	21	27	95	-	-
111 P	121	120	135	157	240	-	-
112 P	523	577	636	712	927	-	Harmful
120	51	55	47	53	141	-	-
121 P	77	95	99	119	188	-	-
122 P	229	257	277	301	376	-	-
200	129	153	172	169	204	188	-
201 P	1759	2909	3448	3639	3676	3700	Harmful
202 P	3355	5408	5964	6269	6337	6337	Harmful
210	99	103	95	120	224	213	-
211 P	424	495	563	709	1079	1071	Harmful
212 P	985	1167	1325	1552	2016	1997	Harmful
220	104	120	141	181	293	303	-
221 P	247	271	304	339	444	409	-
222 P	629	676	713	732	848	884	Harmful

Note :- * denotes result from 1 bar failed the ASTM repeatability test

\+ denotes results from 2 bars failed the ASTM repeatability test

** denotes based on limit referred to in ASTM C227

TABLE C.2(a) REPEATABILITY COMPUTATIONS FOR SERIES 1, 14 DAY RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
 No.      EXPANSION     STRAIN
 000       0.021          84.
 001P      0.019          75.
 002P      0.041         165.
 010       0.011          43.
 011P      0.015          59.
 012P      0.033         132.
 020       0.014          57.
 021P      0.017          68.
 022P      0.034         136.
 100       0.015          61.
 101P      0.062         249.
 102P      0.288        1151.
 110       0.011          45.
 111P      0.030         121.
 112P      0.131         523.
 120       0.013          51.
 121P      0.019          77.
 122P      0.057         229.
 200       0.032         129.
 201P      0.440        1759.
 202P      0.839        3355.
 210       0.025          99.
 211P      0.106         424.
 212P      0.246         985.
 220       0.026         104.
 221P      0.062         247.
 222P      0.157         629.
```

```
Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
 No.                                                  DIFFERENCE
 000          0.000        -0.000         0.000         0.003
 001P        -0.001        -0.003         0.003         0.003
 002P         0.002         0.001        -0.002         0.003
 010          0.000         0.001        -0.002         0.003
 011P         0.000        -0.001         0.000         0.003
 012P        -0.001        -0.002         0.003         0.003
 020          0.002         0.001        -0.002         0.003
 021P         0.001         0.000        -0.001         0.003
 022P        -0.001         0.001         0.            0.003
 100          0.001         0.001        -0.001         0.003
 101P         0.005        -0.004        -0.000         0.009
 102P        -0.004         0.003         0.000         0.043
 110         -0.000        -0.000         0.001         0.003
 111P        -0.001         0.003        -0.001         0.003
 112P        -0.008        -0.005         0.012         0.020
 120         -0.001        -0.001         0.001         0.003
 121P         0.001         0.001        -0.001         0.003
 122P        -0.001        -0.000         0.002         0.009
 200         -0.000         0.002        -0.001         0.003
 201P        -0.007        -0.002         0.008         0.066
 202P         0.044        -0.051         0.006         0.126
 210          0.001         0.001        -0.003         0.003
 211P         0.006        -0.001        -0.005         0.016
 212P         0.007        -0.004        -0.002         0.037
 220         -0.001         0.002        -0.001         0.003
 221P         0.001         0.000        -0.002         0.009
 222P        -0.000        -0.003         0.004         0.024
```

```
Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 000            0.003          0.003          0.003
 001P           0.002          0.000         -0.000
 002P           0.001          0.002          0.001
 010            0.003          0.002          0.001
 011P           0.003          0.002          0.003
 012P           0.002          0.001          0.000
 020            0.001          0.002          0.001
 021P           0.002          0.003          0.002
 022P           0.002          0.002          0.003
 100            0.002          0.002          0.002
 101P           0.005          0.005          0.009
 102P           0.039          0.040          0.043
 110            0.003          0.003          0.002
 111P           0.002          0.000          0.002
 112P           0.012          0.015          0.007
 120            0.002          0.002          0.002
 121P           0.002          0.002          0.002
 122P           0.007          0.008          0.007
 200            0.003          0.001          0.002
 201P           0.059          0.064          0.058
 202P           0.081          0.075          0.119
 210            0.002          0.002          0.000
 211P           0.010          0.015          0.011
 212P           0.030          0.033          0.035
 220            0.002          0.001          0.002
 221P           0.008          0.009          0.008
 222P           0.023          0.020          0.020
```

TABLE C.2(b) REPEATABILITY COMPUTATIONS FOR SERIES 1, 28 DAY RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION     STRAIN
  000       0.025        100.
  001P      0.024         95.
  002P      0.076        305.
  010       0.014         55.
  011P      0.015         60.
  012P      0.045        179.
  020       0.011         44.
  021P      0.012         48.
  022P      0.034        135.
  100       0.017         67.
  101P      0.085        341.
  102P      0.418       1672.
  110       0.008         31.
  111P      0.030        120.
  112P      0.144        577.
  120       0.014         55.
  121P      0.024         95.
  122P      0.064        257.
  200       0.038        153.
  201P      0.727       2909.
  202P      1.352       5408.
  210       0.026        103.
  211P      0.124        495.
  212P      0.292       1167.
  220       0.030        120.
  221P      0.068        271.
  222P      0.169        676.
```

```
Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
  No.                                                DIFFERENCE
  000         0.            0.            0.          0.003
  001P       -0.002        -0.002         0.003       0.003
  002P        0.014        -0.011        -0.002       0.011
  010         0.002         0.000        -0.003       0.003
  011P       -0.001         0.001         0.000       0.003
  012P       -0.001        -0.002         0.002       0.003
  020         0.001         0.000        -0.001       0.003
  021P        0.002         0.           -0.002       0.003
  022P       -0.002         0.001         0.000       0.003
  100         0.001        -0.001        -0.001       0.003
  101P        0.005        -0.004        -0.000       0.013
  102P       -0.016         0.016         0.000       0.063
  110        -0.003         0.001         0.001       0.003
  111P        0.            0.002        -0.002       0.003
  112P       -0.008        -0.006         0.015       0.022
  120         0.000        -0.001         0.000       0.003
  121P        0.000         0.000        -0.001       0.003
  122P       -0.001        -0.001         0.003       0.010
  200         0.001        -0.000        -0.000       0.003
  201P       -0.011        -0.008         0.020       0.109
  202P        0.058        -0.071         0.013       0.203
  210         0.000         0.001        -0.002       0.003
  211P        0.006        -0.001        -0.006       0.019
  212P        0.007        -0.005        -0.003       0.044
  220        -0.001         0.002        -0.001       0.003
  221P        0.002         0.000        -0.003       0.010
  222P       -0.001        -0.003         0.004       0.025
```

```
Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 000             0.003         0.003         0.003
 001P            0.001         0.001        -0.000
 002P           -0.002         0.000         0.009
 010             0.001         0.003         0.000
 011P            0.002         0.002         0.003
 012P            0.002         0.001         0.001
 020             0.002         0.003         0.002
 021P            0.001         0.003         0.001
 022P            0.001         0.002         0.003
 100             0.002         0.002         0.002
 101P            0.008         0.008         0.012
 102P            0.047         0.047         0.063
 110             0.000         0.002         0.002
 111P            0.003         0.001         0.001
 112P            0.013         0.015         0.007
 120             0.003         0.002         0.003
 121P            0.003         0.003         0.002
 122P            0.008         0.008         0.007
 200             0.002         0.003         0.003
 201P            0.098         0.101         0.089
 202P            0.145         0.132         0.190
 210             0.003         0.002         0.001
 211P            0.012         0.018         0.013
 212P            0.036         0.039         0.041
 220             0.002         0.001         0.002
 221P            0.008         0.010         0.007
 222P            0.024         0.022         0.021
```

TABLE C.2(c) REPEATABILITY COMPUTATIONS FOR SERIES 1, 2 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.    AVERAGE     MICRO-
 No.       EXPANSION     STRAIN
 000         0.021         85.
 001P        0.020         79.
 002P        0.124        495.
 010         0.004         15.
 011P        0.013         52.
 012P        0.061        243.
 020         0.006         25.
 021P        0.008         31.
 022P        0.041        163.
 100         0.023         93.
 101P        0.101        405.
 102P        0.459       1835.
 110         0.005         21.
 111P        0.034        135.
 112P        0.159        636.
 120         0.012         47.
 121P        0.025         99.
 122P        0.069        277.
 200         0.043        172.
 201P        0.862       3448.
 202P        1.491       5964.
 210         0.024         95.
 211P        0.141        563.
 212P        0.331       1325.
 220         0.035        141.
 221P        0.076        304.
 222P        0.178        713.
```

```
Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
 No.                                                DIFFERENCE
 000          0.001        -0.001         0.001        0.003
 001P        -0.001        -0.003         0.003        0.003
 002P        -0.059        -0.044        -0.016        0.019
 010          0.001         0.000        -0.002        0.003
 011P        -0.000        -0.000         0.000        0.003
 012P         0.001        -0.004         0.002        0.009
 020          0.002        -0.001        -0.000        0.003
 021P         0.001         0.000        -0.002        0.003
 022P        -0.001         0.002        -0.002        0.003
 100          0.003        -0.002        -0.000        0.003
 101P         0.006        -0.003        -0.002        0.015
 102P        -0.021         0.020         0.000        0.069
 110         -0.003         0.001         0.003        0.003
 111P        -0.001         0.002        -0.002        0.003
 112P        -0.010        -0.007         0.017        0.024
 120         -0.002         0.000         0.001        0.003
 121P         0.000         0.001        -0.002        0.003
 122P        -0.001        -0.002         0.004        0.010
 200          0.            0.001        -0.001        0.003
 201P        -0.033         0.004         0.029        0.129
 202P         0.084        -0.095         0.011        0.224
 210          0.000         0.001        -0.002        0.003
 211P         0.007        -0.002        -0.006        0.021
 212P         0.011        -0.006        -0.004        0.050
 220         -0.000         0.003        -0.002        0.003
 221P         0.003         0.           -0.003        0.011
 222P        -0.000        -0.004         0.005        0.027
```

```
Mix Ref.
 No.    CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 000            0.002          0.002          0.002
 001P           0.002          0.000         -0.000
 002P          -0.041         -0.025          0.003
 010            0.002          0.003          0.001
 011P           0.003          0.003          0.003
 012P           0.008          0.005          0.007
 020            0.001          0.002          0.003
 021P           0.002          0.003          0.001
 022P           0.002          0.001          0.001
 100            0.000          0.001          0.003
 101P           0.010          0.012          0.013
 102P           0.048          0.048          0.068
 110           -0.000          0.002          0.000
 111P           0.002          0.001          0.001
 112P           0.014          0.017          0.007
 120            0.001          0.003          0.002
 121P           0.003          0.002          0.001
 122P           0.009          0.008          0.007
 200            0.003          0.002          0.002
 201P           0.096          0.125          0.100
 202P           0.140          0.129          0.213
 210            0.003          0.002          0.001
 211P           0.014          0.019          0.015
 212P           0.039          0.043          0.045
 220            0.003          0.000          0.001
 221P           0.008          0.011          0.008
 222P           0.026          0.022          0.022
```

TABLE C.2(d) REPEATABILITY COMPUTATIONS FOR SERIES 1, 4 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION     STRAIN
  000       0.028        113.
  001P      0.026        105.
  002P      0.143        571.
  010       0.010         41.
  011P      0.018         73.
  012P      0.103        413.
  020       0.012         49.
  021P      0.009         35.
  022P      0.048        191.
  100       0.027        107.
  101P      0.111        445.
  102P      0.497       1987.
  110       0.007         27.
  111P      0.039        157.
  112P      0.178        712.
  120       0.013         53.
  121P      0.030        119.
  122P      0.075        301.
  200       0.042        169.
  201P      0.910       3639.
  202P      1.567       6269.
  210       0.030        120.
  211P      0.177        709.
  212P      0.388       1552.
  220       0.045        181.
  221P      0.085        339.
  222P      0.183        732.
```

```
Mix Ref.  EXPN(1)-AVGE   EXPN(2)-AVGE   EXPN(3)-AVGE   ALLOWABLE
  No.                                                  DIFFERENCE
  000         0.001         -0.001          0.001         0.003
  001P       -0.001         -0.001          0.003         0.003
  002P        0.071         -0.053         -0.019         0.021
  010         0.001         -0.000         -0.000         0.003
  011P        0.001          0.001         -0.001         0.003
  012P        0.009         -0.006         -0.002         0.015
  020         0.002         -0.001         -0.000         0.003
  021P        0.001          0.000         -0.002         0.003
  022P       -0.002          0.003         -0.002         0.003
  100         0.002         -0.002         -0.001         0.003
  101P        0.008         -0.005         -0.002         0.017
  102P       -0.025          0.024          0.000         0.075
  110        -0.003          0.001          0.001         0.003
  111P       -0.000          0.003         -0.002         0.003
  112P       -0.011         -0.010          0.021         0.027
  120        -0.001         -0.000          0.002         0.003
  121P       -0.001          0.002         -0.002         0.003
  122P       -0.001         -0.004          0.006         0.011
  200         0.001          0.001         -0.001         0.003
  201P       -0.034          0.000          0.033         0.136
  202P        0.105         -0.108          0.004         0.235
  210         0.001          0.001         -0.002         0.003
  211P        0.009         -0.000         -0.008         0.027
  212P        0.013         -0.009         -0.004         0.058
  220        -0.000          0.003         -0.002         0.003
  221P        0.002         -0.001         -0.002         0.013
  222P       -0.001         -0.004          0.005         0.027
```

```
Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 000            0.002          0.002          0.002
 001P           0.002          0.002          0.000
 002P          -0.050         -0.031          0.003
 010            0.002          0.003          0.003
 011P           0.002          0.002          0.002
 012P           0.007          0.009          0.013
 020            0.001          0.002          0.003
 021P           0.002          0.003          0.001
 022P           0.001         -0.000          0.001
 100            0.001          0.001          0.002
 101P           0.009          0.011          0.014
 102P           0.050          0.050          0.074
 110            0.000          0.002          0.002
 111P           0.003          0.000          0.001
 112P           0.016          0.017          0.006
 120            0.002          0.003          0.001
 121P           0.002          0.001          0.001
 122P           0.010          0.007          0.006
 200            0.002          0.002          0.002
 201P           0.103          0.136          0.103
 202P           0.130          0.127          0.231
 210            0.002          0.002          0.001
 211P           0.018          0.026          0.018
 212P           0.045          0.049          0.054
 220            0.003          0.000          0.001
 221P           0.010          0.012          0.011
 222P           0.026          0.023          0.022
```

TABLE C.2(e) REPEATABILITY COMPUTATIONS FOR SERIES 1, 14 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
MIX REF.   AVERAGE     MICRO-
 NO.      EXPANSION     STRAIN
000         0.030       121.
001P        0.032       127.
002P        0.165       661.
010         0.021        83.
011P        0.032       129.
012P        0.223       892.
020         0.031       124.
021P        0.029       116.
022P        0.072       289.
100         0.026       105.
101P        0.118       472.
102P        0.519      2075.
110         0.024        95.
111P        0.060       240.
112P        0.232       927.
120         0.035       141.
121P        0.047       188.
122P        0.094       376.
200         0.051       204.
201P        0.919      3676.
202P        1.584      6337.
210         0.056       224.
211P        0.270      1079.
212P        0.504      2016.
220         0.073       293.
221P        0.111       444.
222P        0.212       848.
```

```
Mix Ref.  EXPN(1)-AVGE   EXPN(2)-AVGE   EXPN(3)-AVGE   ALLOWABLE
 No.                                                    DIFFERENCE
 000        -0.000          -0.001          0.002          0.003
 001P       -0.003          -0.002          0.004          0.003
 002P        0.065          -0.057         -0.007          0.025
 010        -0.002           0.000          0.001          0.003
 011P        0.001           0.001         -0.001          0.003
 012P        0.020          -0.014         -0.006          0.033
 020         0.003          -0.002         -0.001          0.003
 021P        0.002          -0.001         -0.001          0.003
 022P        0.002           0.003         -0.004          0.011
 100         0.004          -0.000         -0.003          0.003
 101P        0.007          -0.004         -0.003          0.018
 102P       -0.025           0.024          0.000          0.078
 110        -0.002           0.000          0.001          0.003
 111P        0.002           0.001         -0.003          0.009
 112P       -0.016          -0.012          0.027          0.035
 120        -0.001          -0.000          0.002          0.003
 121P        0.              0.003         -0.003          0.003
 122P       -0.002          -0.005          0.007          0.014
 200         0.002           0.000         -0.002          0.008
 201P       -0.033          -0.001          0.034          0.138
 202P        0.104          -0.108          0.005          0.238
 210         0.002           0.001         -0.003          0.008
 211P        0.009           0.001         -0.011          0.040
 212P        0.018          -0.015         -0.003          0.076
 220        -0.000           0.003         -0.002          0.011
 221P        0.004          -0.002         -0.002          0.017
 222P       -0.001          -0.005          0.006          0.032
```

```
Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 000            0.003           0.002           0.001
 001P           0.000           0.001          -0.001
 002P          -0.040          -0.033           0.017
 010            0.001           0.003           0.002
 011P           0.002           0.002           0.002
 012P           0.013           0.019           0.027
 020            0.000           0.001           0.002
 021P           0.001           0.002           0.002
 022P           0.009           0.008           0.007
 100           -0.001           0.003          -0.000
 101P           0.011           0.014           0.015
 102P           0.053           0.053           0.077
 110            0.001           0.003           0.002
 111P           0.007           0.008           0.006
 112P           0.019           0.023           0.007
 120            0.002           0.003           0.001
 121P           0.003           0.000           0.000
 122P           0.012           0.009           0.007
 200            0.006           0.008           0.006
 201P           0.105           0.137           0.104
 202P           0.134           0.129           0.233
 210            0.006           0.007           0.005
 211P           0.031           0.039           0.030
 212P           0.058           0.061           0.073
 220            0.011           0.008           0.009
 221P           0.013           0.015           0.015
 222P           0.031           0.027           0.026
```

TABLE C.2(f) REPEATABILITY COMPUTATIONS FOR SERIES 1, 18 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
          Mix Ref.   AVERAGE    MICRO-
            No.     EXPANSION     STRAIN
            200       0.047        188.
            201P      0.925       3700.
            202P      1.534       6337.
            210       0.053        213.
            211P      0.268       1071.
            212P      0.499       1997.
            220       0.076        303.
            221P      0.102        409.
            222P      0.221        884.

     Mix Ref. EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
       No.                                                DIFFERENCE
       200          0.002        -0.001        -0.001        0.003
       201P        -0.033        -0.001         0.034        0.139
       202P         0.100        -0.104         0.005        0.238
       210          0.003         0.004        -0.006        0.008
       211P         0.007         0.003        -0.011        0.040
       212P         0.013        -0.011        -0.001        0.075
       220          0.000         0.004        -0.005        0.011
       221P         0.004        -0.000        -0.003        0.015
       222P        -0.001        -0.005         0.006        0.033

Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
  200               0.001         0.002         0.002
  201P              0.106         0.138         0.105
  202P              0.138         0.133         0.233
  210               0.005         0.004         0.002
  211P              0.033         0.037         0.029
  212P              0.062         0.064         0.074
  220               0.011         0.007         0.007
  221P              0.012         0.015         0.012
  222P              0.032         0.028         0.027
```

TABLE C.3 SERIES 1 VISUAL EXAMINATION OF MORTAR BARS, AFTER EXPANSION MEASUREMENTS AT 4 MONTHS

Mix Ref No.	Date Cast	Date of Examination	Specimen Ref.	Warping	Twisting	Compaction	Visible Aggregate	Surface Mottling	Surficial Exudation	Other Comments
000	16.7.80	5.11.80	A	Zero	Zero	Average	None	None	None	
			B	Zero	Zero					
			C	Zero	Zero					
001 P	22.7.80	11.11.80	A	Zero	Zero	Average	None	Slight	Slight	
			B	Zero	Zero					
			C	Zero	Zero					
002 P	2.10.80	29.1.81	A	Negligible	Zero	Below average	Slight - At ends and on surfaces	Slight	Slight	Some large white deposits
			B	Zero	Zero					
			C	Negligible	Zero					
010	24.7.80	13.11.80	A	Zero	Zero	Average	Slight - At ends	Slight	Slight	Specimen quite wet on surface. Very few white deposits.
			B	Zero	Zero					
			C	Negligible	Negligible					
011 P	24.7.80	13.11.80	A	Zero	Zero	Average	None	Slight	Slight	Specimen quite wet on surface.
			B	Zero	Negligible					
			C	Zero	Negligible					
012 P	16.9.80	13.1.81	A	Zero	Zero	Good	None	Yes	Slight	Specimens quite wet
			B	Zero	Zero					
			C	Zero	Zero					
020	30.9.80	27.1.81	A	Zero	Zero	Below average	None	Yes	None	
			B	Zero	Zero					
			C	Zero	Zero					
021 P	15.10.80	11.2.81	A	Zero	Zero	Poor	Slight - At surface	Yes	None	
			B	Zero	Zero					
			C	Zero	Zero					
022 P	17.9.80	14.1.81	A	Negligible	Zero	Average	None	Slight	None	
			B	Zero	Zero					
			C	Negligible	Zero					

TABLE C.3 continued

Mix Ref No.	Date Cast	Date of Examination	Specimen Ref.	Warping	Twisting	Compaction	Visible Aggregate	Surface Mottling	Surficial Exudation	Other Comments
100	30.9.80	27.1.81	A	Slight	Zero	Average	None	None	None	
			B	Zero	Zero					
			C	Zero	Zero					
101 P	24.9.80	21.1.81	A	Zero	Zero	Below average	None	Yes	Yes	White deposits and 'runs' on surface.
			B	Zero	Zero					
			C	Zero	Zero					
102 P	23.7.80	12.11.80	A	Negligible	Zero	Average	None	Yes	Extensive	Signs of condensation on specimens. Appearance dark grey/green with white deposits and light patches
			B	Negligible	Slight					
			C	-	-					
110	23.9.80	20.1.81	A	Zero	Zero	Average	None	Yes	Slight	Minor white staining on surface.
			B	Zero	Zero					
			C	Slight	Zero					
111 P	18.9.80	15.1.81	A	Zero	Zero	Average	None	Yes	None	
			B	Zero	Zero					
			C	Slight	Zero					
112 P	9.10.80	5.2.81	A	Zero	Zero	Below average	None	Yes	Slight	
			B	Zero	Zero					
			C	Zero	Zero					
120	14.10.80	10.2.81	A	Zero	Zero	Below average	Slight - At ends and at surfaces	Yes	None	
			B	Zero	Zero					
			C	Zero	Zero					
121 P	25.9.80	22.1.81	A	Negligible	Zero	Poor	None	Slight	None	
			B	Zero	Zero					
			C	Negligible	Zero					
122 P	25.9.80	22.1.81	A	Zero	Zero	Poor	None	Slight	None	
			B	Zero	Zero					
			C	Negligible	Zero					

TABLE C.3 continued

Mix Ref No.	Date Cast	Date of Examination	Specimen Ref.	Warping	Twisting	Compaction	Visible Aggregate	Surface Mottling	Surficial Exudation	Other Comments
200	16.9.80	13.1.81	A	Zero	Slight	Good	None	Slight	Slight	
			B	Zero	Zero					
			C	Zero	Negligible					
201 P	1.10.80	28.1.81	A	≃1mm	Zero	Below average	None	Yes	Extensive	No visible cracking. Possible start of surface spalling. Extensive white deposits.
			B	Zero	Zero					
			C	≃1mm	Zero					
202 P	7.10.80	3.2.81	A	Negligible	Zero	Average	None	None	Extensive	No visible cracking. Extensive white deposits.
			B	≃1mm	Zero					
			C	Zero	Zero					
210	14.10.80	10.2.81	A	Negligible	Zero	Good	None	Yes	None	
			B	Zero	Zero					
			C	Zero	Zero					
211 P	23.9.80	20.1.81	A	Zero	Zero	Average	None	Slight	Yes	Extensive white staining/deposits on surface.
			B	Zero	Zero					
			C	Zero	Zero					
212 P	7.10.80	3.2.81	A	Negligible	Zero	Average	None	Yes	Slight	
			B	Zero	Zero					
			C	Zero	Zero					
220	17.9.80	14.1.81	A	Zero	Zero	Average	None	Extensive	None	
			B	Zero	Negligible					
			C	Zero	Zero					
221 P	18.9.80	15.1.81	A	Zero	Zero	Average	None	Yes	None	
			B	Zero	Zero					
			C	Zero	Zero					
222 P	9.10.80	5.2.81	A	Zero	Zero	Below average	None	Slight	None	
			B	Zero	Zero					
			C	Slight	Zero					

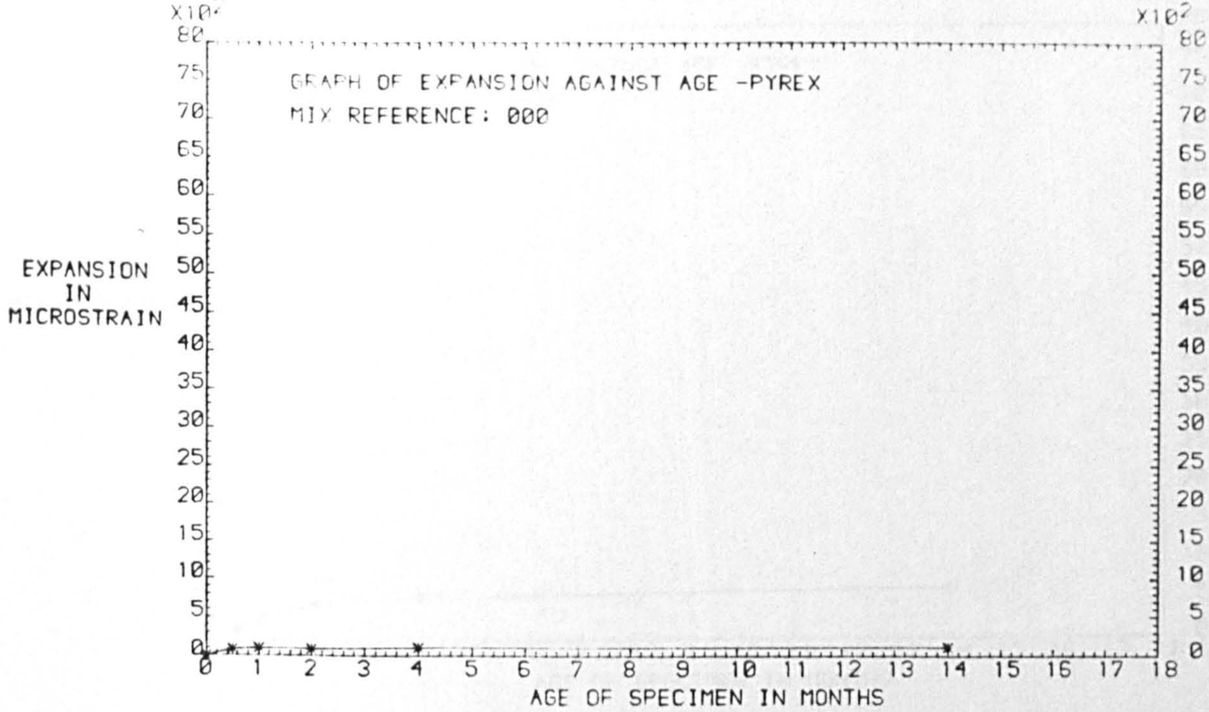


Figure C.1

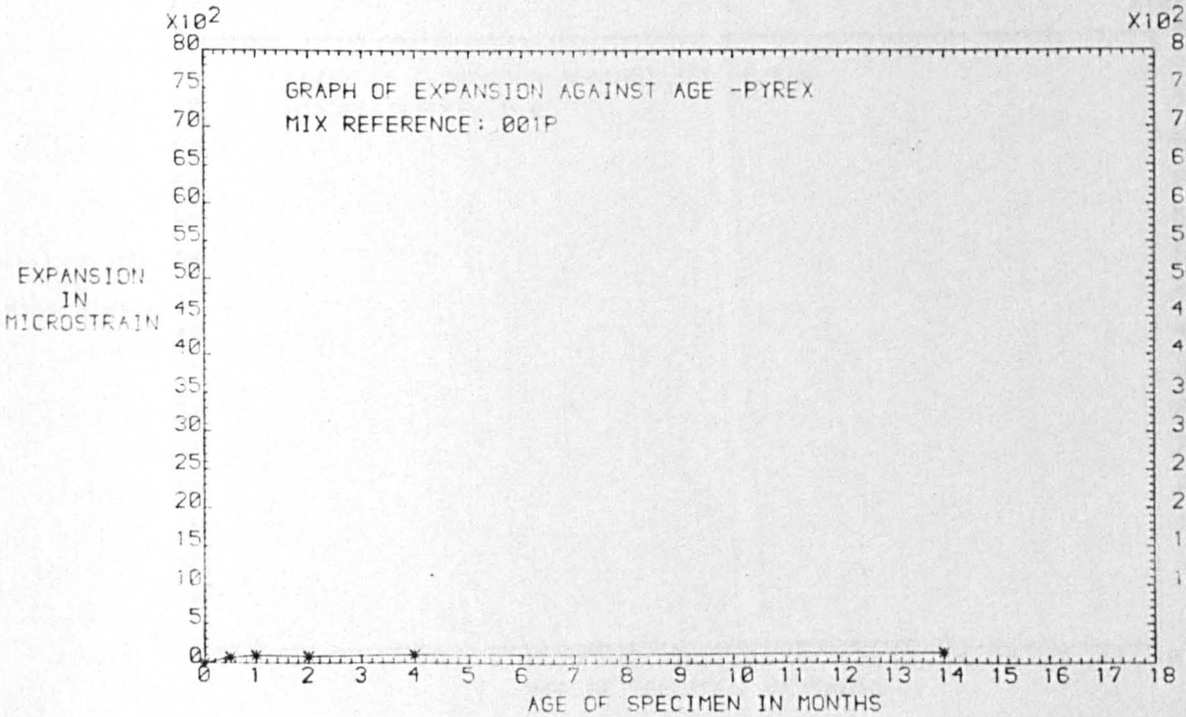


Figure C.2

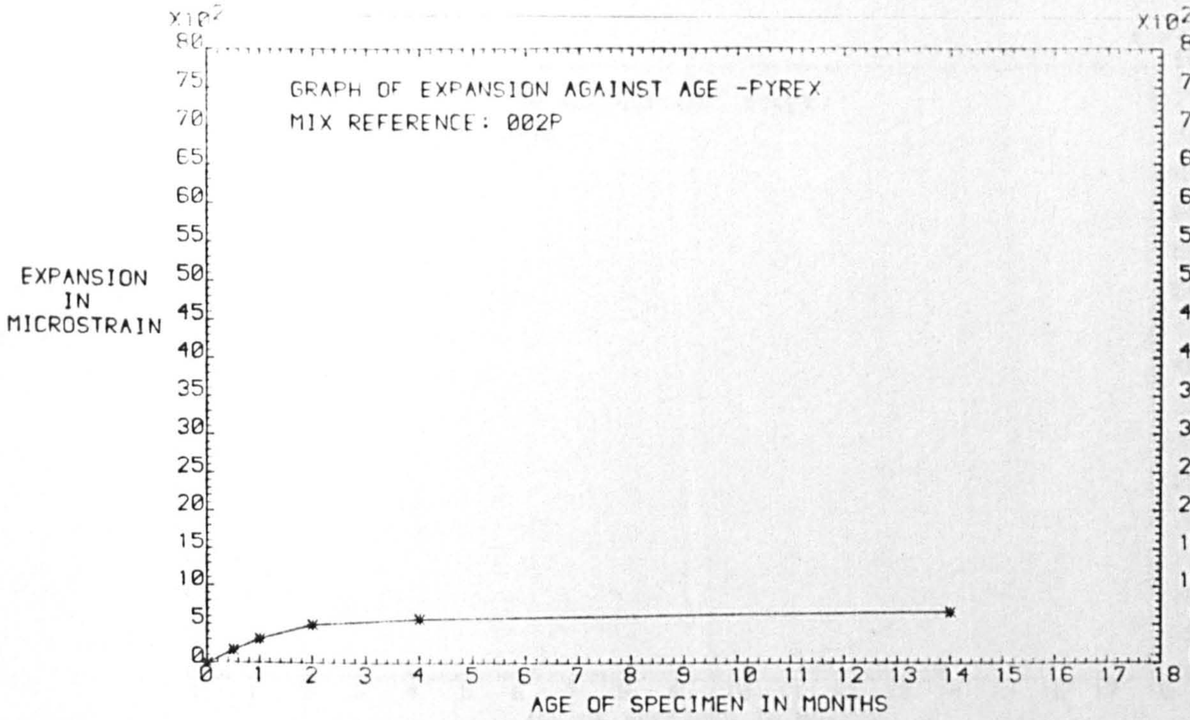


Figure C.3

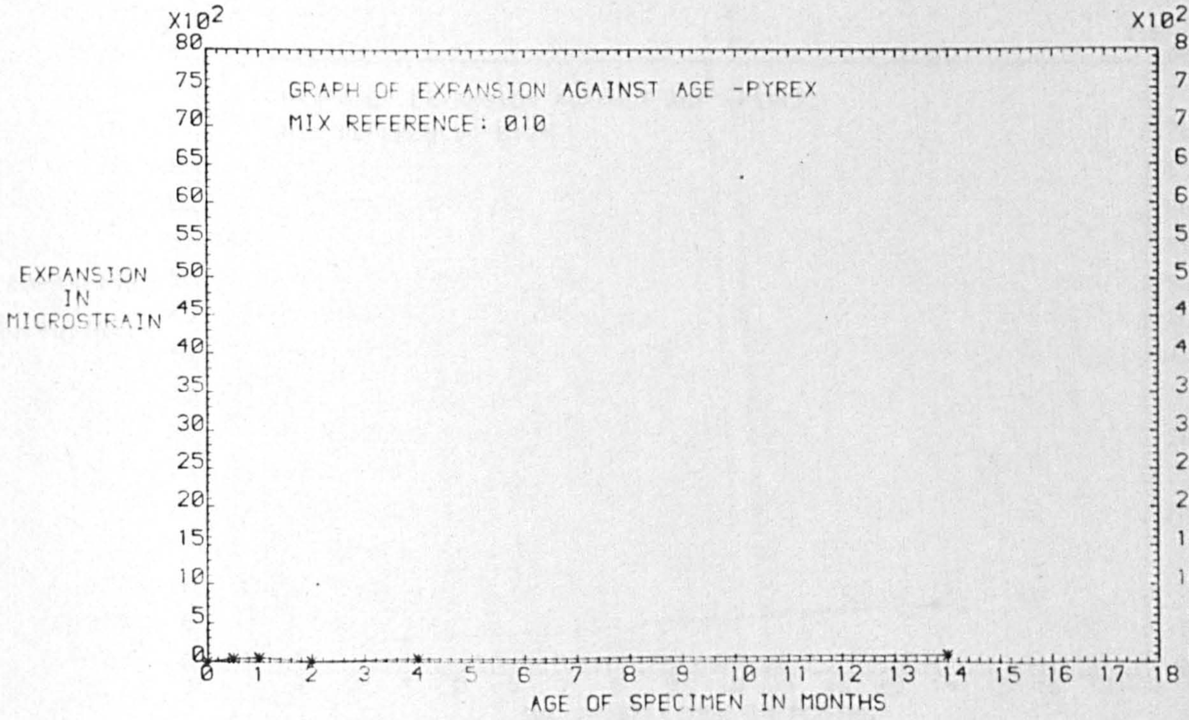


Figure C.4

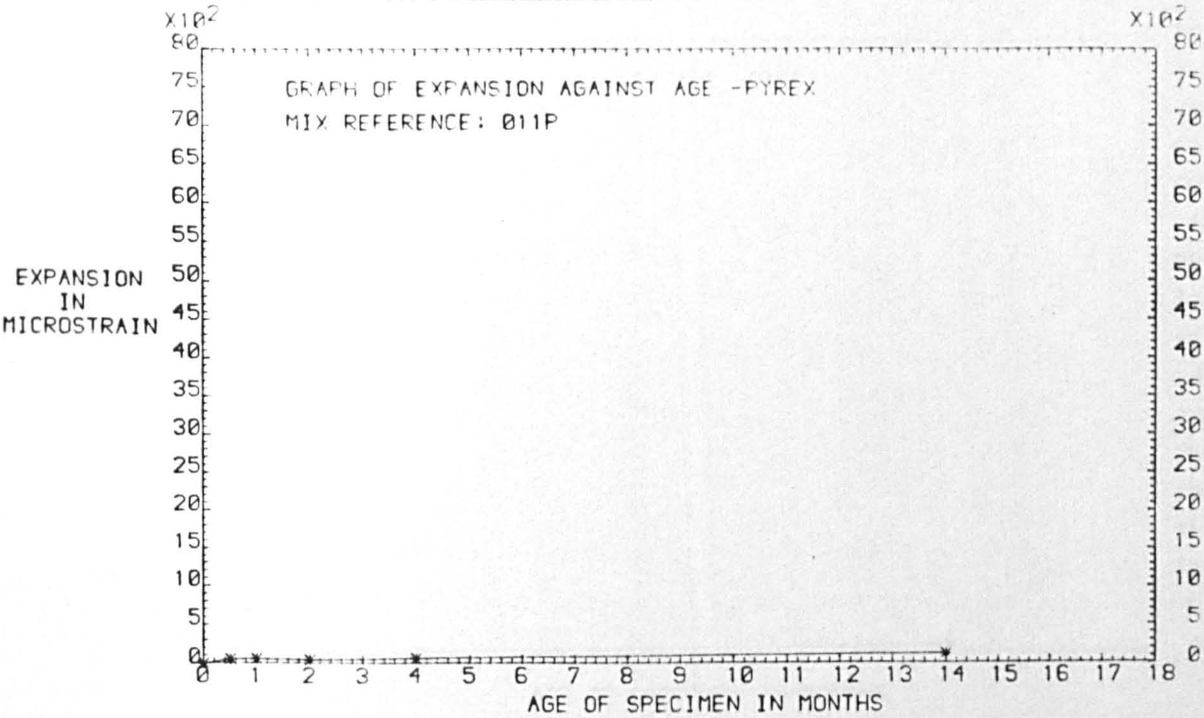


Figure C.5

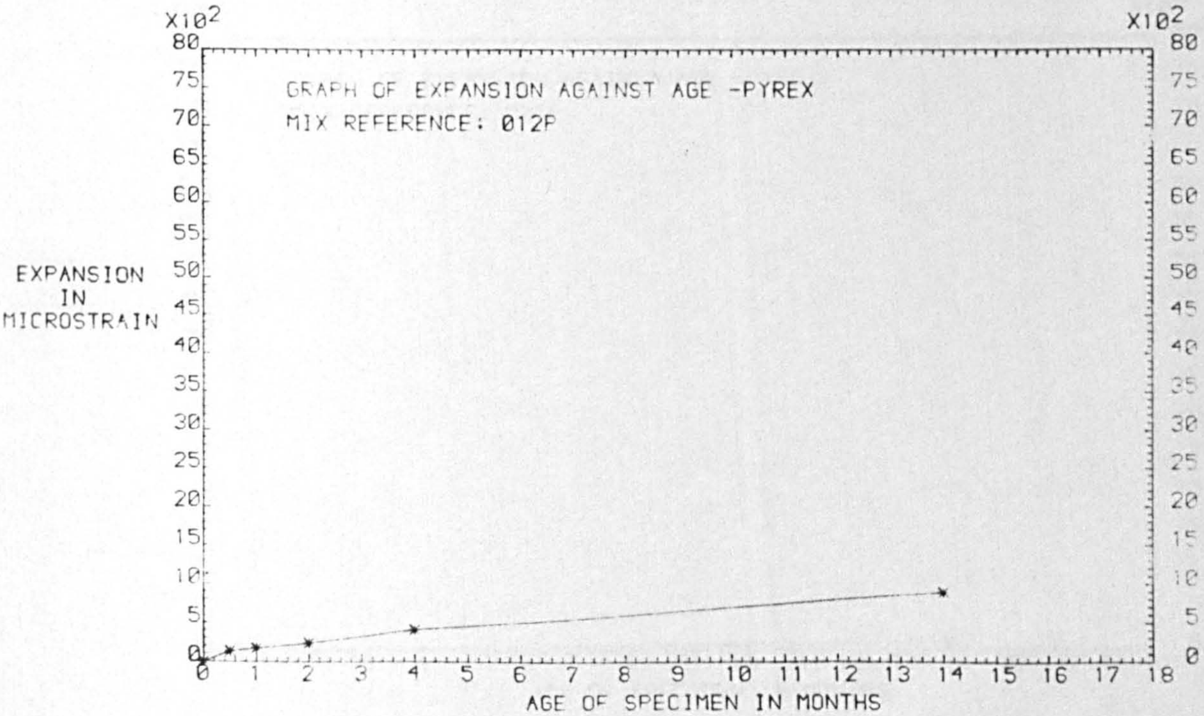


Figure C.6

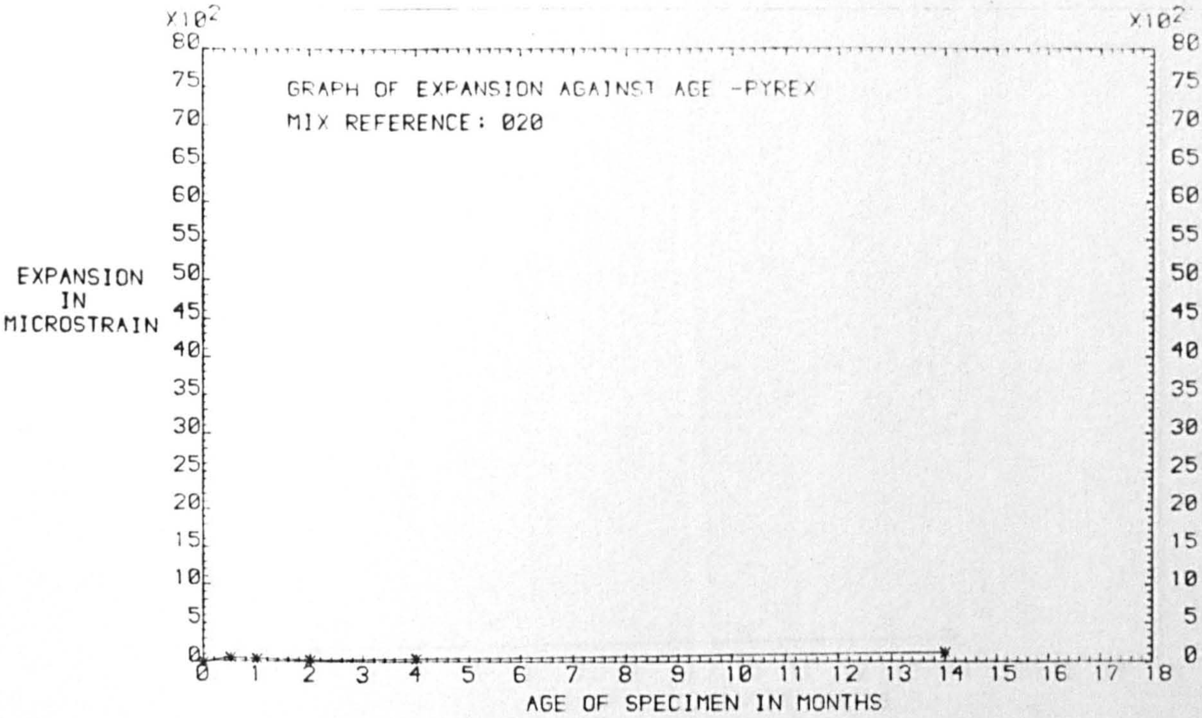


Figure C.7

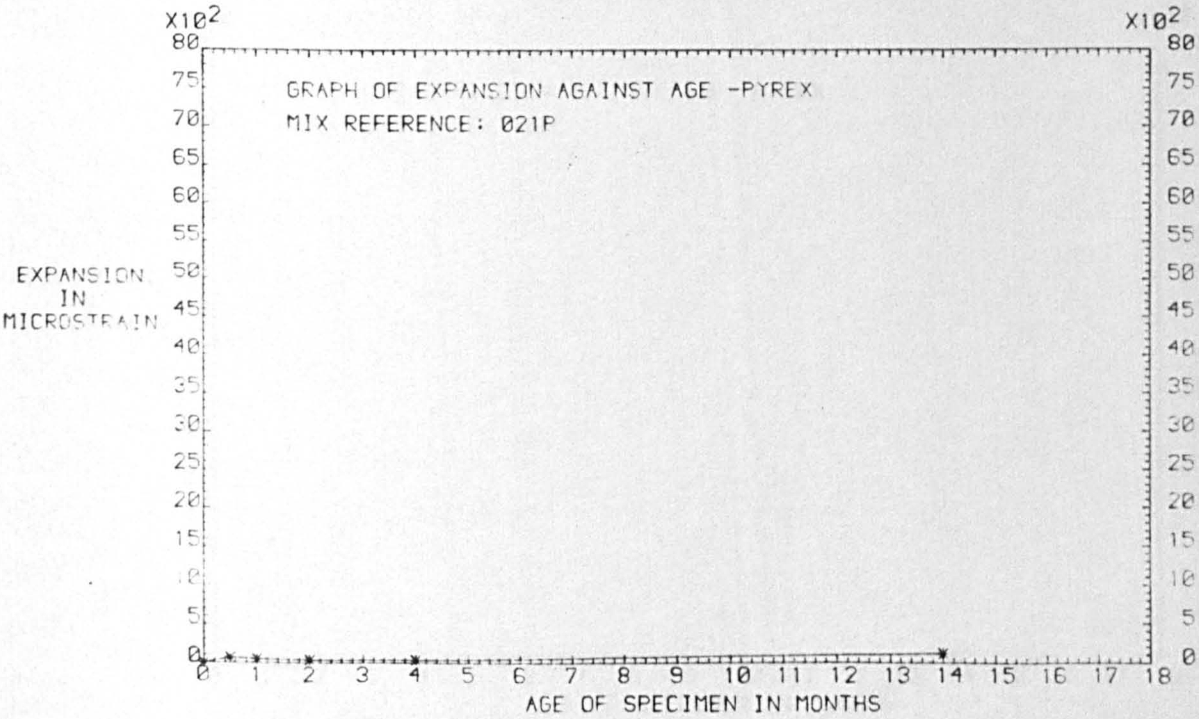


Figure C.8

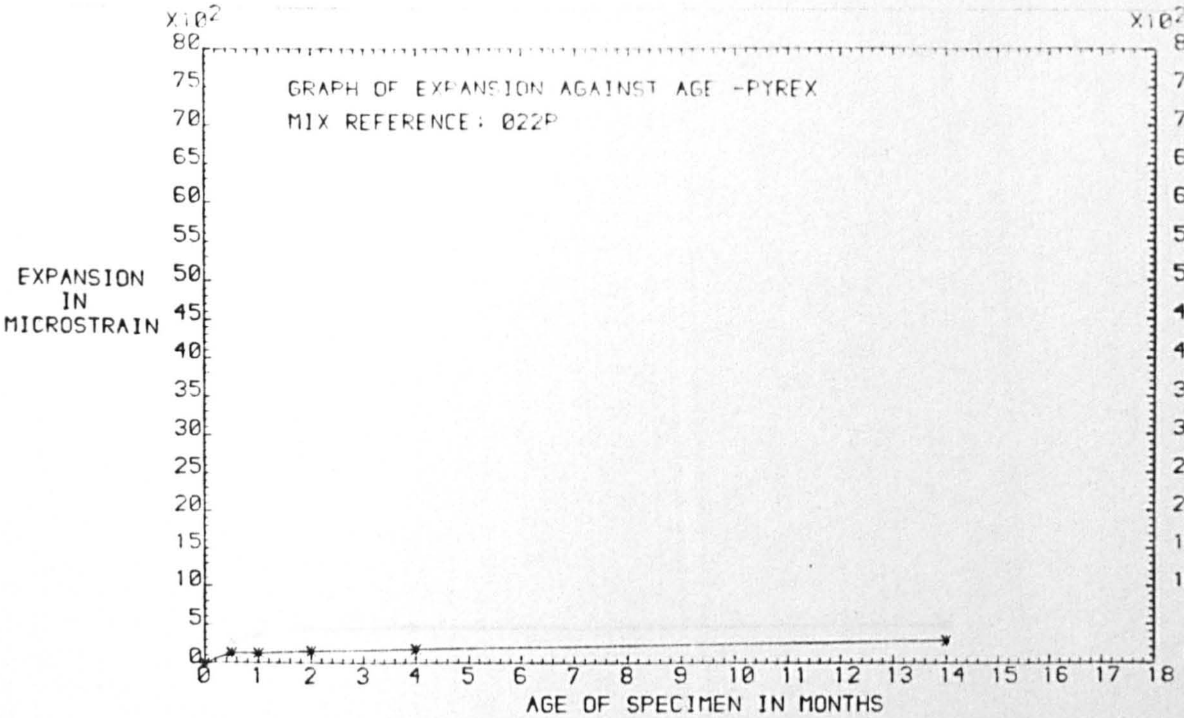


Figure C.9

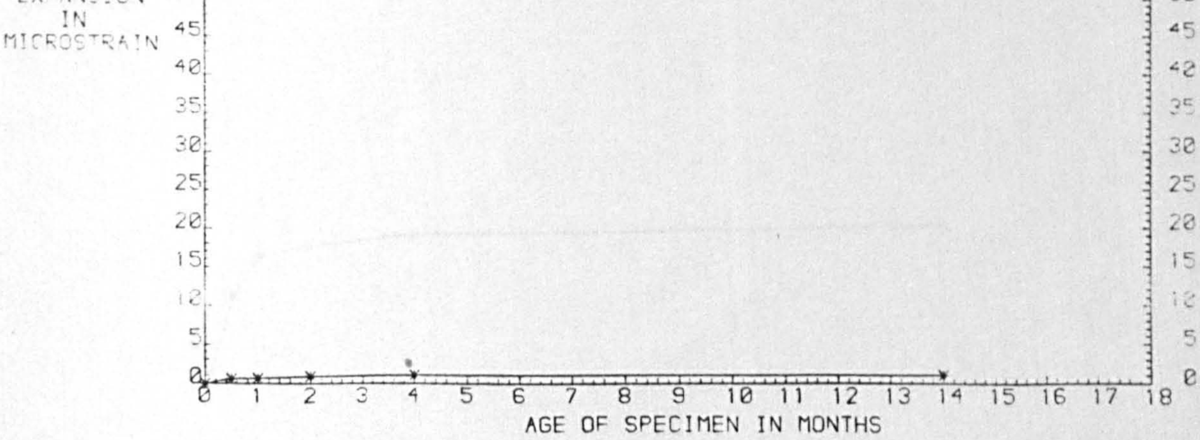


Figure C.10

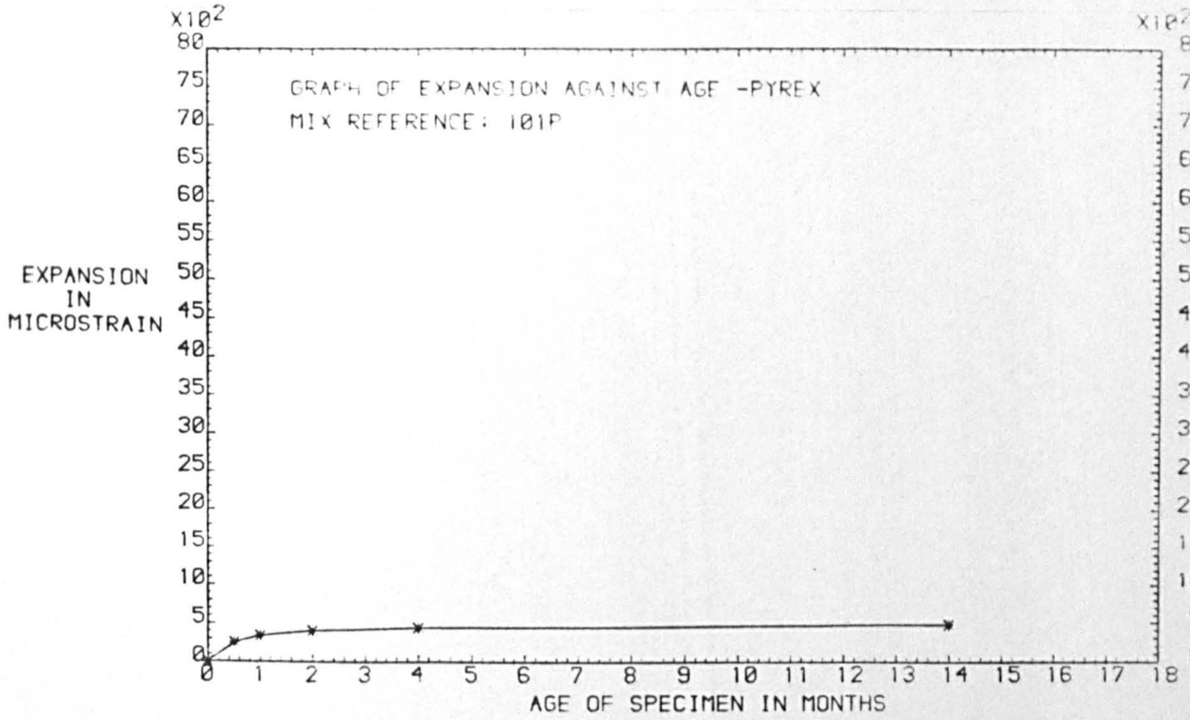


Figure C.11

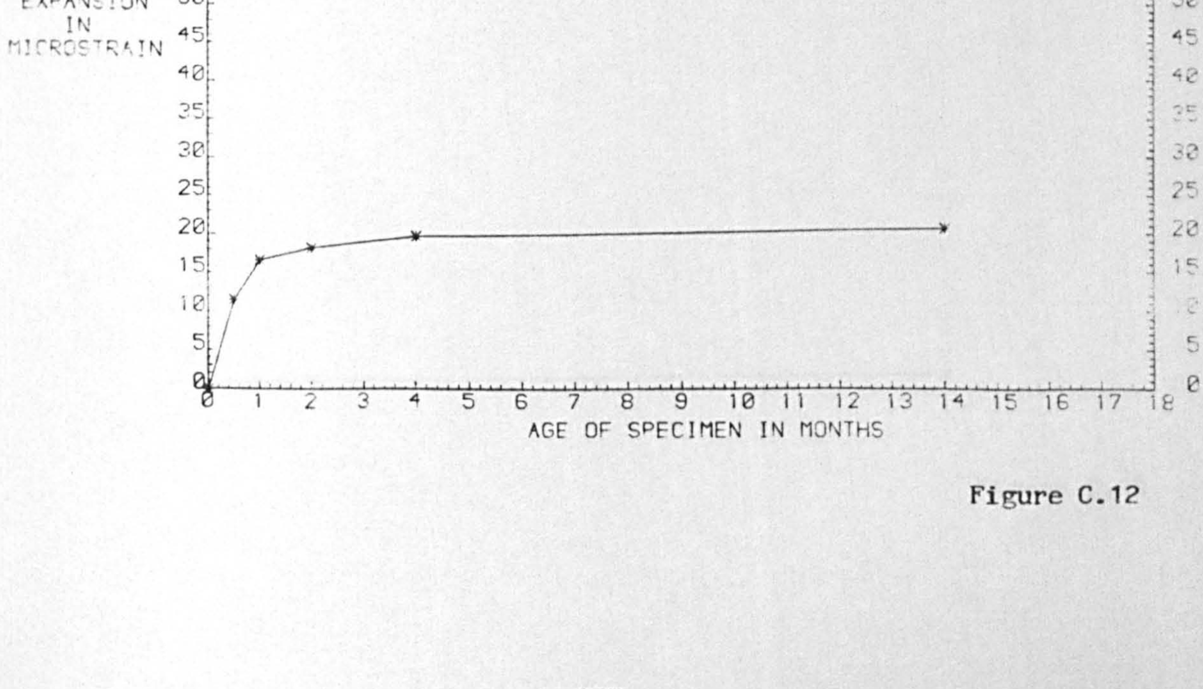


Figure C.12

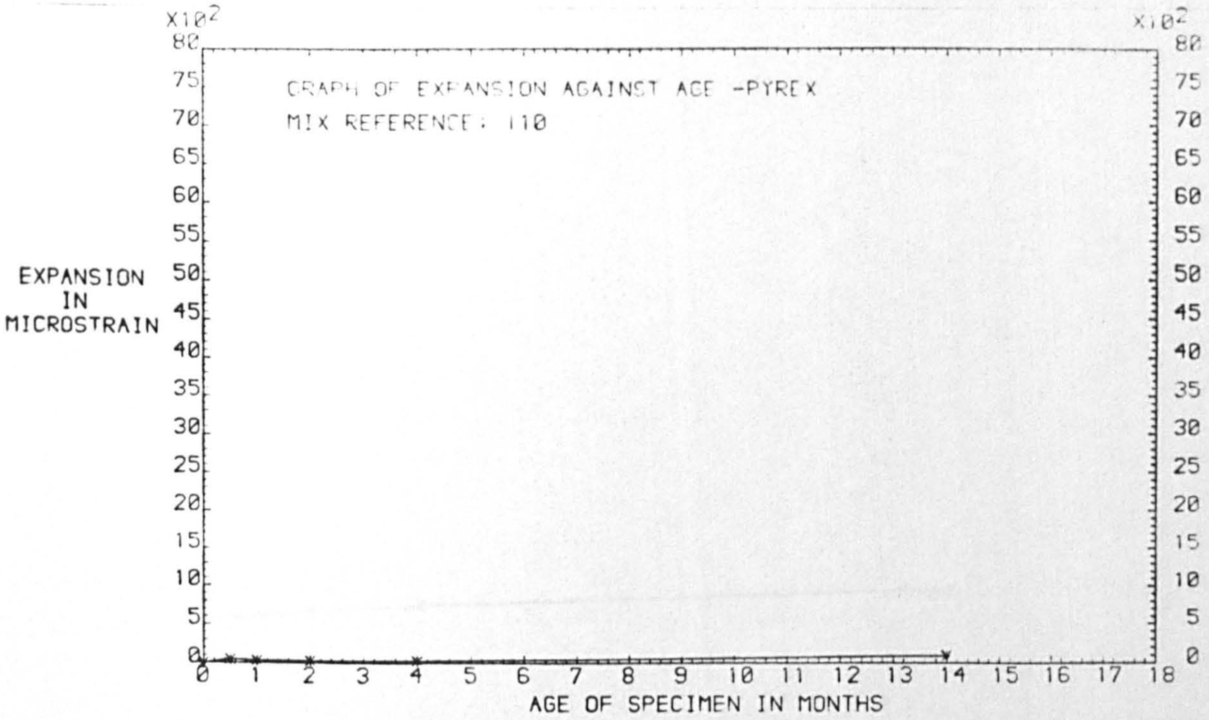


Figure C.13

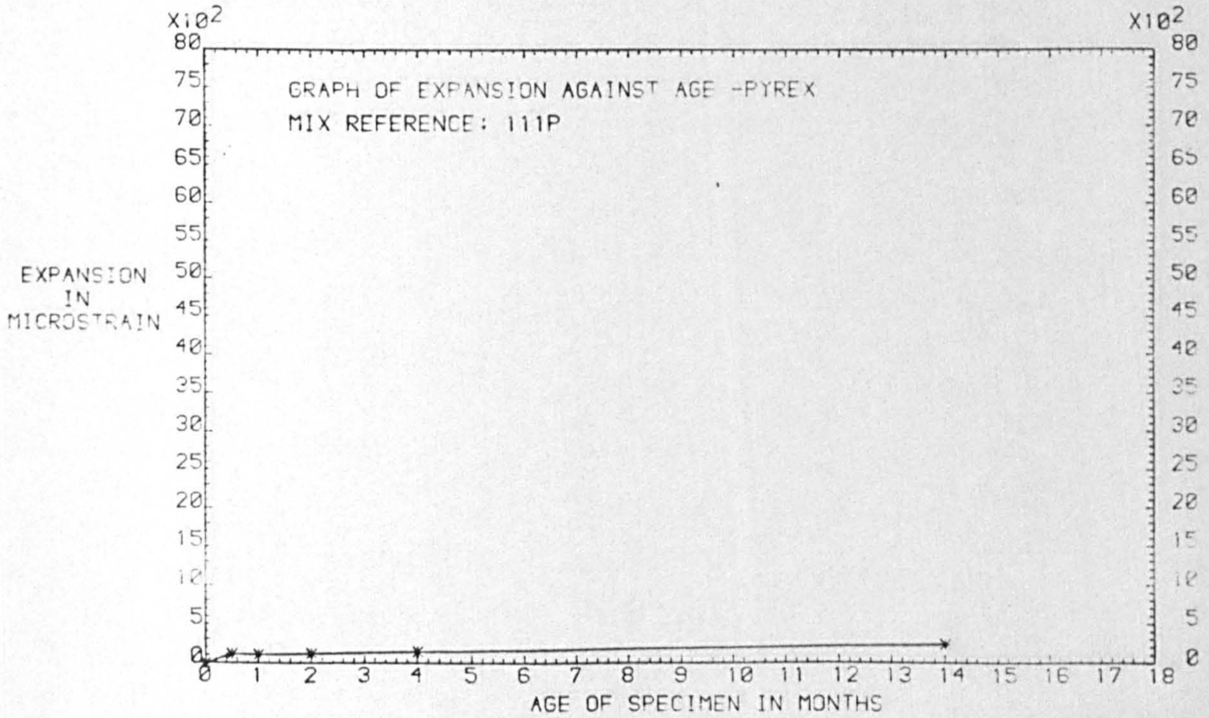


Figure C.14

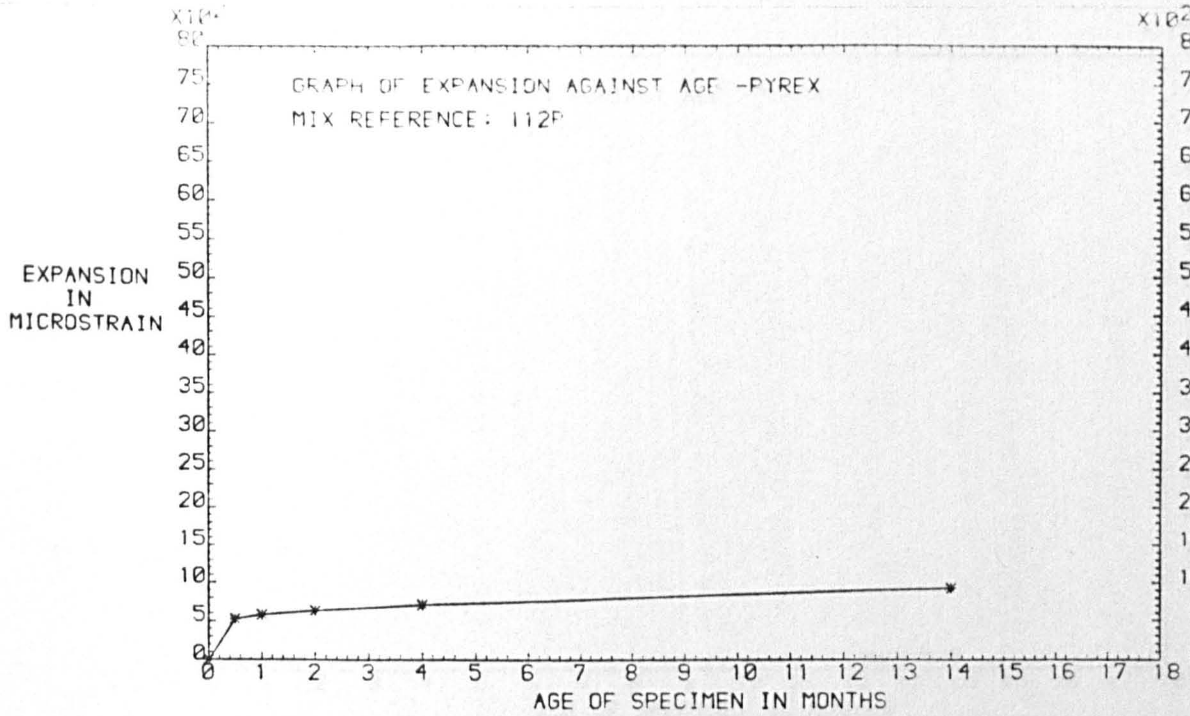


Figure C.15

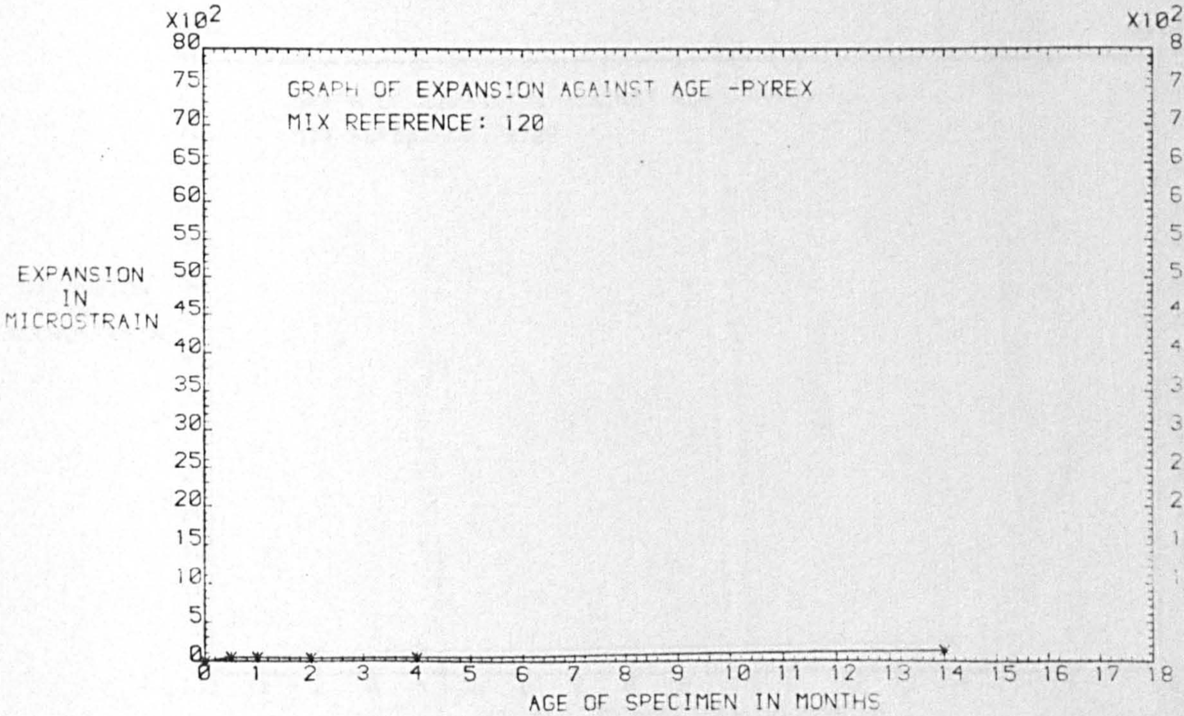


Figure C.16

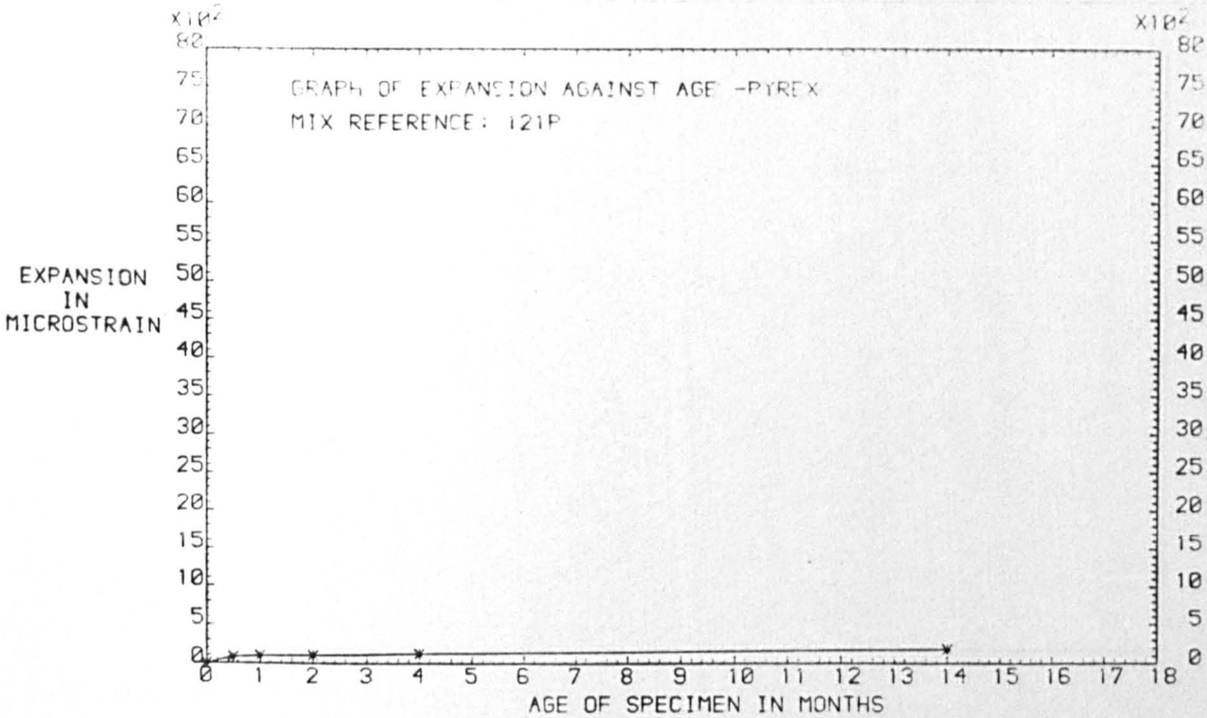


Figure C.17

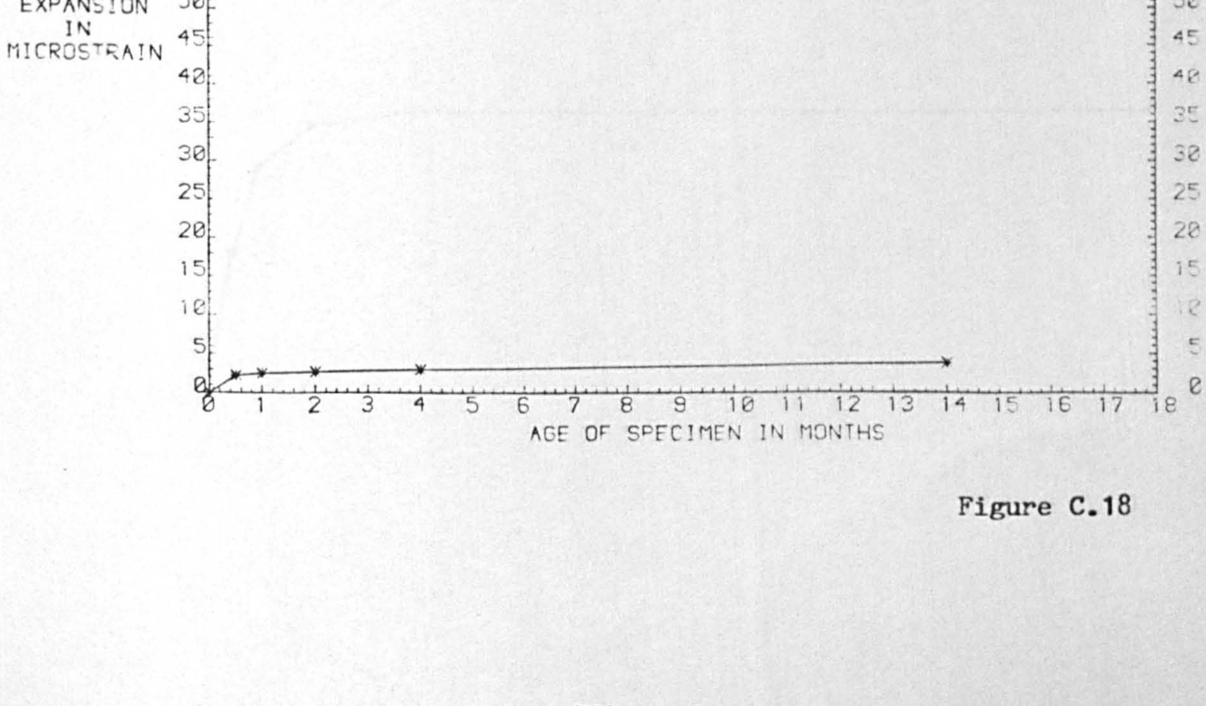


Figure C.18

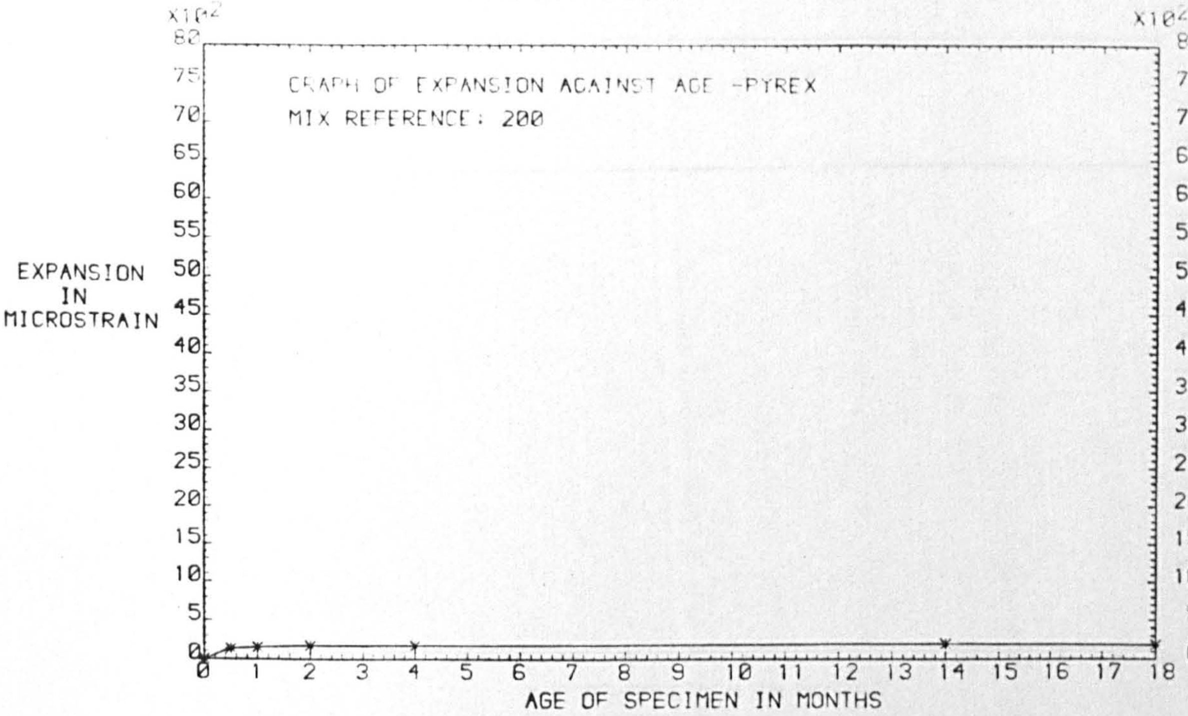


Figure C.19

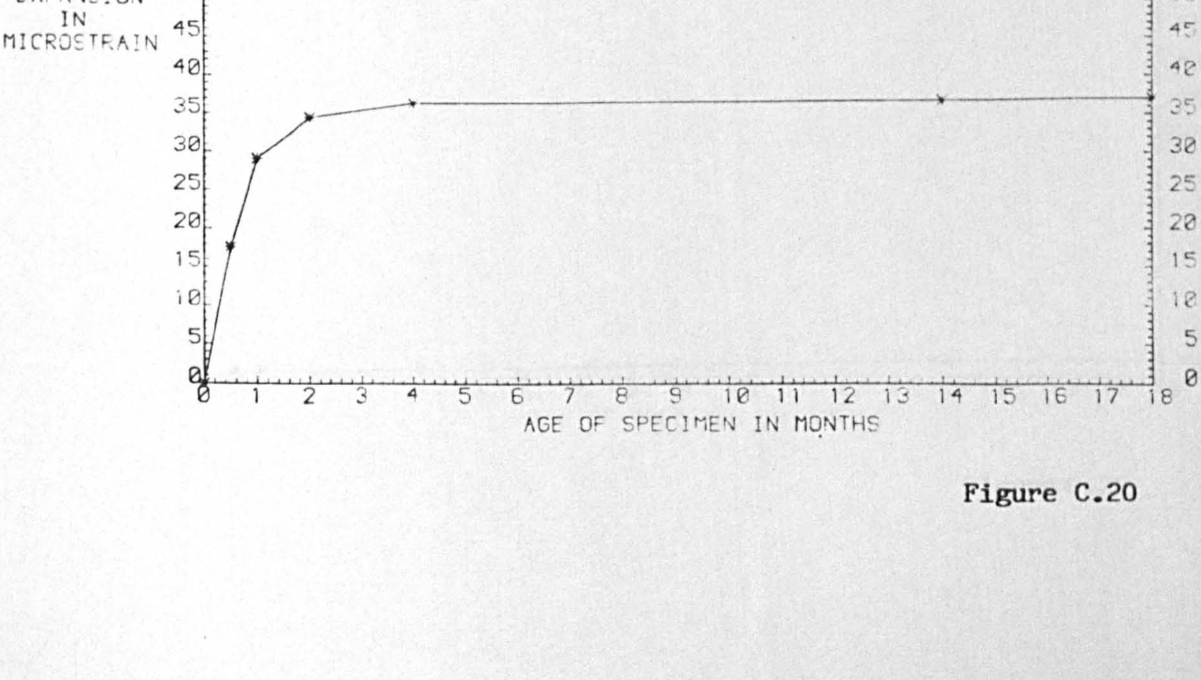


Figure C.20

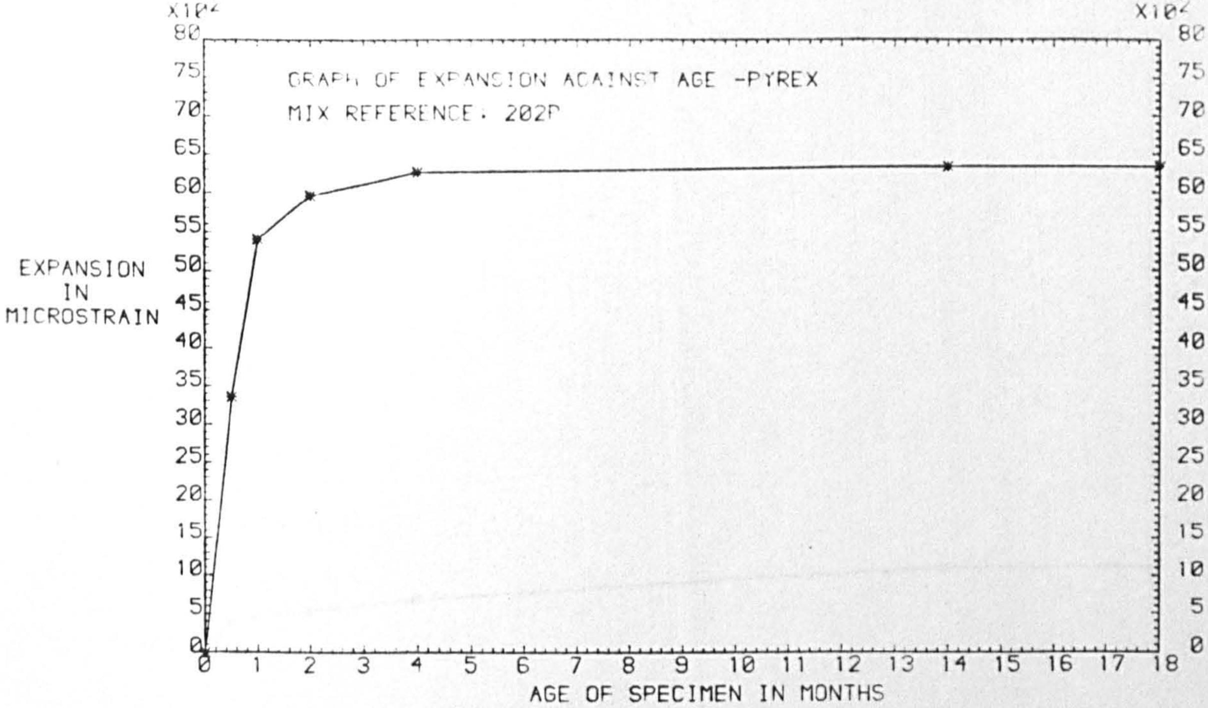


Figure C.21

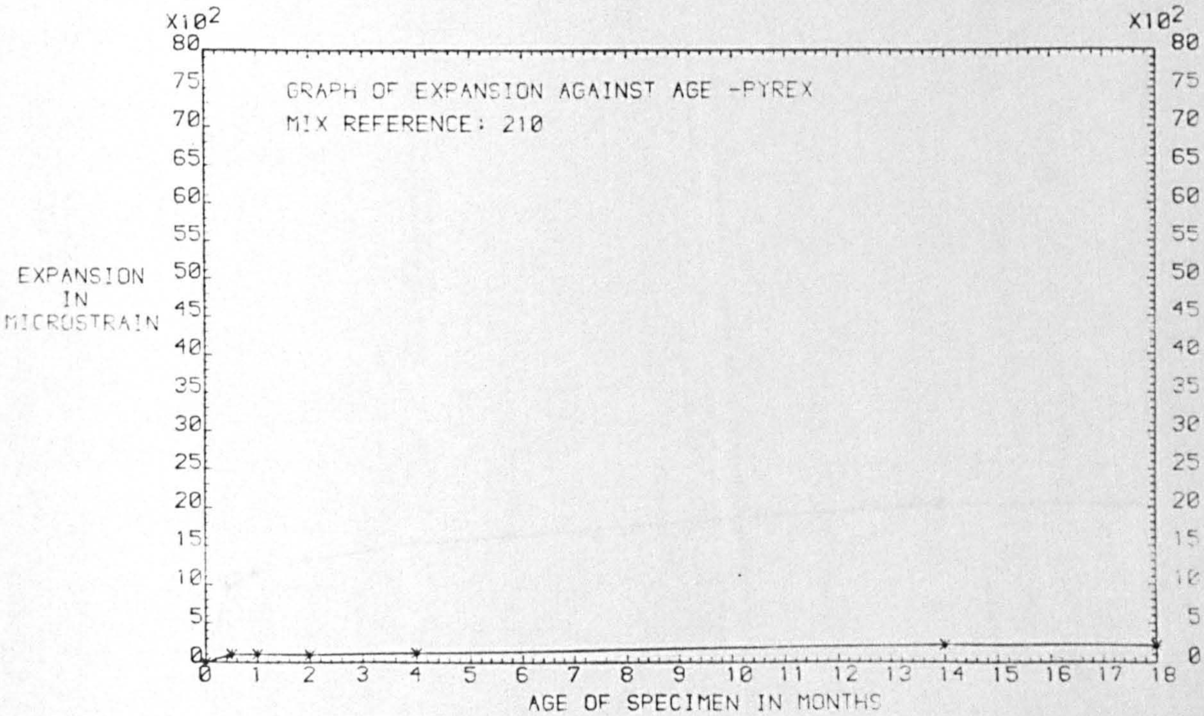


Figure C.22

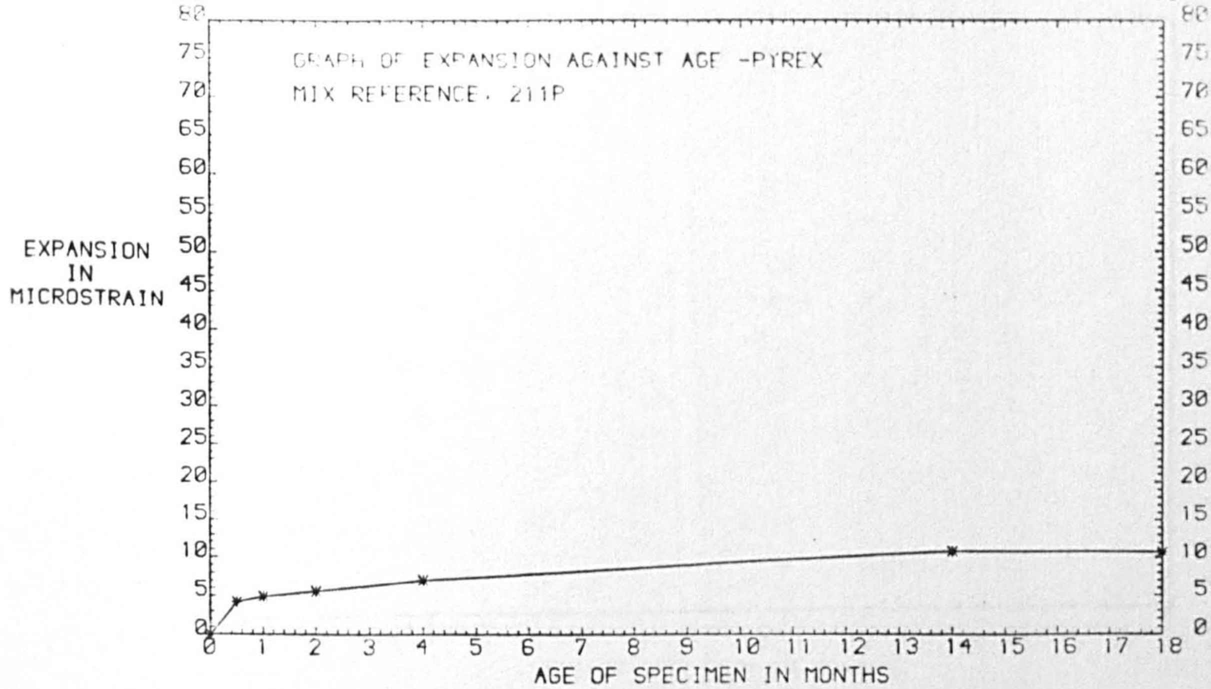


Figure C.23

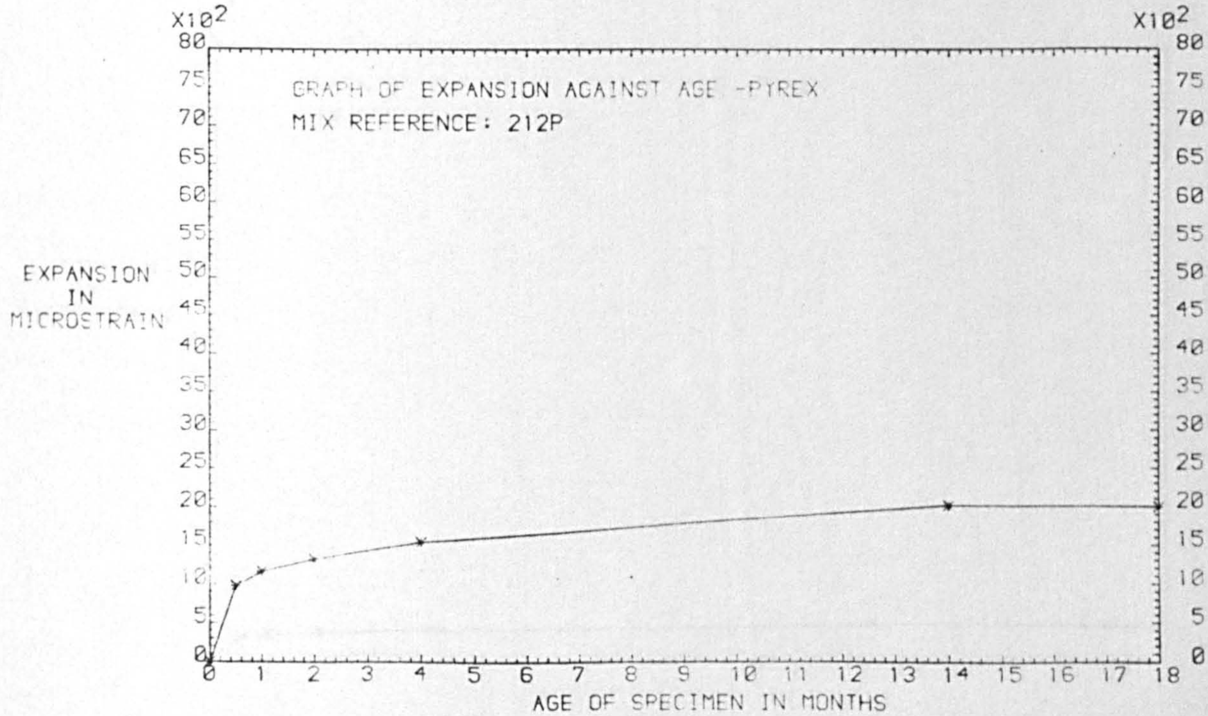


Figure C.24

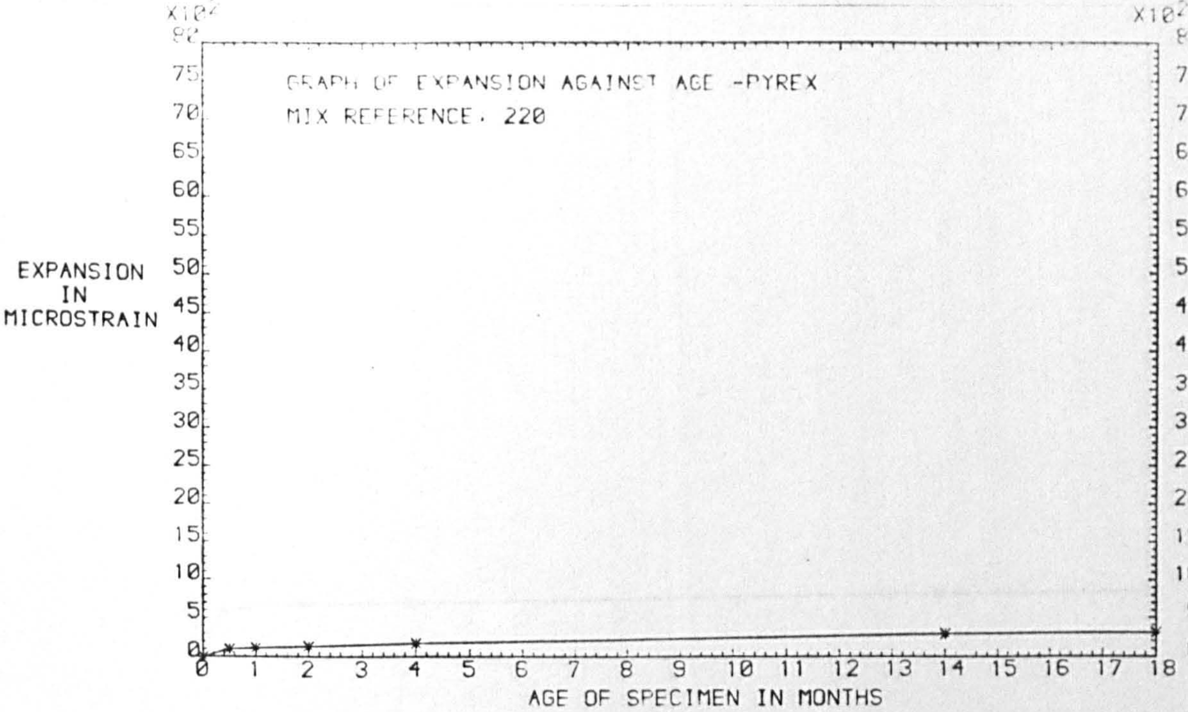


Figure C.25

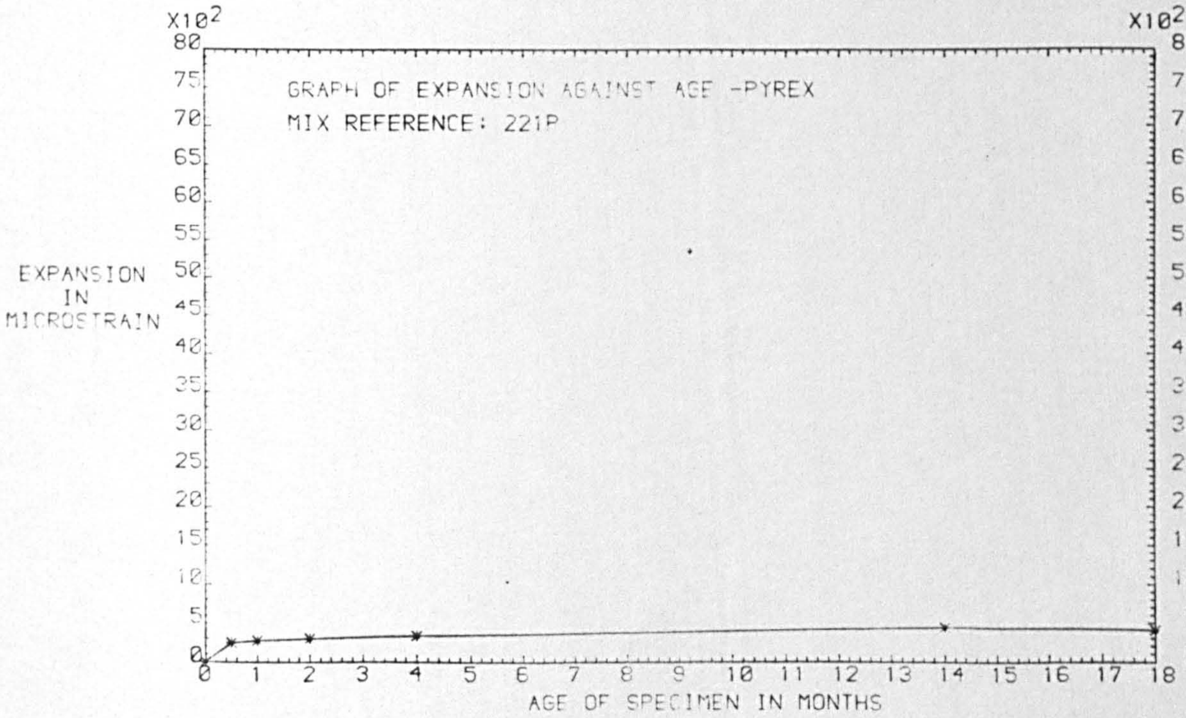


Figure C.26

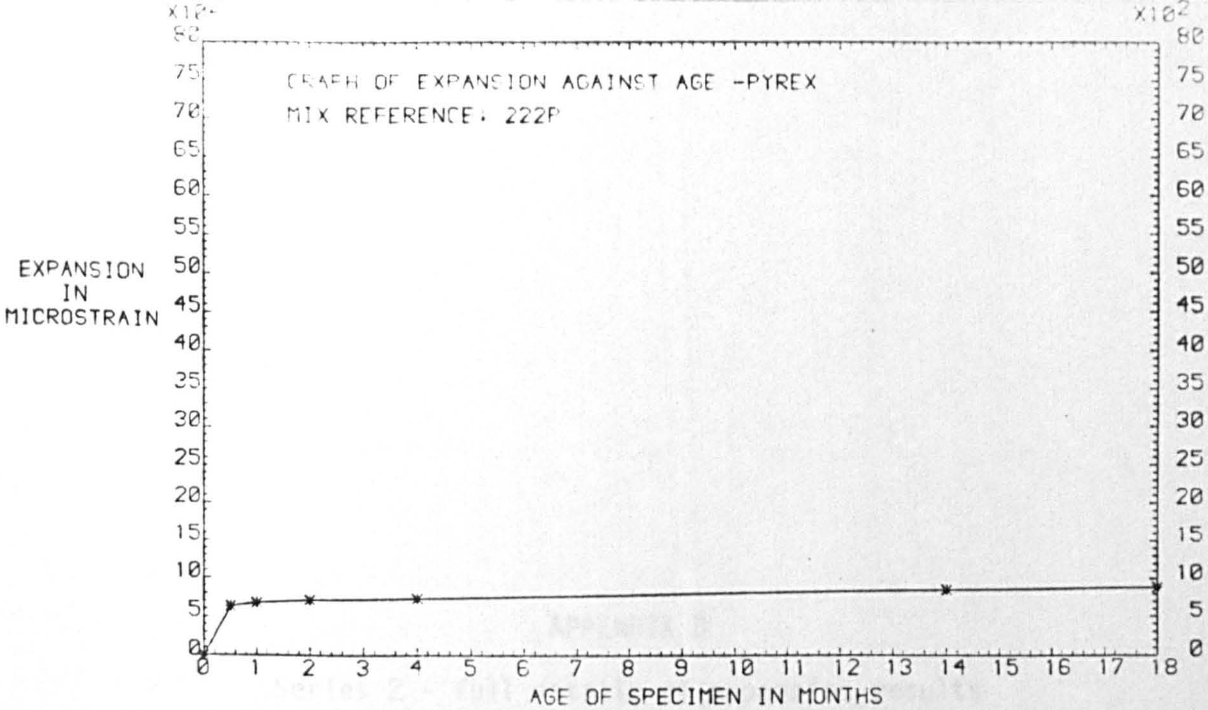

X10²
GRAPH OF EXPANSION AGAINST AGE -PYREX
MIX REFERENCE: 222P
EXPANSION IN MICROSTRAIN
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
AGE OF SPECIMEN IN MONTHS

Figure C.27

APPENDIX D

Series 2 - full details of expansion results from 3 X 4 X 4 X 2 factorial set of experiments

Tables D.1 to D.2

Figures D.1 to D.96

TABLE D.1 SERIES 2 MORTAR BAR EXPANSION RESULTS - 3x4x4x2 FACTORIAL MIXES

MIX REF NO.	EXPANSION IN MICROSTRAIN ($\mu\varepsilon$) AT AGE:-				'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	2 MONTHS	6 MONTHS	12 MONTHS	
0000	64	88	91	67	-
0001	77	80	81	21*	-
0010 P	56	68	80	37	-
0011 P	71	85*	91*	83	-
0020 P	99	149	172	139	-
0021 P	64	104	99	80	-
0030 P	912	1421	1425	1379	Harmful
0031 P	193*	1889	2819	2827	Harmful
0100	53	37	52*	24	-
0101	65	81	95	88+	-
0110 P	67	59	64	59+	-
0111 P	84	76*	88*	69	-
0120 P	60	65	93*	88+	-
0121 P	69	61	99	107	-
0130 P	283	353	399	376	-
0131 P	153+	771	2023	2243	Harmful
0200	40	0	5	-21	-
0201	93	53	48	43	-
0210 P	60	40	63*	21+	-
0211 P	80	60	78	40	-
0220 P	75	68	60	48	-
0221 P	48	27	29	29	-
0230 P	149	161	180	176	-
0231 P	117	157	297	587	-
0300	83	59	73	67	-
0301	96	91	99	91	-
0310 P	43	27	31	16	-
0311 P	77	57	79*	59	-
0320 P	77	68*	79	61	-
0321 P	87	101*	115+	104+	-
0330 P	135	133	147	131*	-
0331 P	96	115	148	168+	-

Note:- * denotes result from 1 bar failed the ASTM repeatability test
+ denotes results from 2 bars failed the ASTM repeatability test
** denotes based on limit referred to in ASTM C227

TABLE D.1 continued

MIX REF NO.	EXPANSION IN MICROSTRAIN (μE) AT AGE:-				'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	2 MONTHS	6 MONTHS	12 MONTHS	
1000	64	88	103	91	-
1001	48	85	87	51*	-
1010 P	208	291	311	283	-
1011 P	141	252*	271	261	-
1020 P	1252	1516	1533	1533	Harmful
1021 P	1467	3077	3164	3128	Harmful
1030 P	2633	2831	2864	2859	Harmful
1031 P	3025	4367	4385	4381	Harmful
1100	63	40	51	29^{+}	-
1101	67	83	84	64^{+}	-
1110 P	109	136	153*	115*	-
1111 P	139	167^{+}	196	200*	-
1120 P	408	453	452	440	-
1121 P	552	1301	2092	2328	Harmful
1130 P	1099	1197	1208	1187	Harmful
1131 P	1215	2245	2649	2739	Harmful
1200	57	73	91	77	-
1201	80	73	99	96^{+}	-
1210 P	67	108	128	117*	-
1211 P	75	84	124	115	-
1220 P	184	221	251	237	-
1221 P	144^{+}	209	340	448	-
1230 P	725	775	807	808	-
1231 P	576	804	1236	1517	Harmful
1300	77	117	148	163*	-
1301	77	97	125	99*	-
1310 P	92	107	137	141^{+}	-
1311 P	69	95	123	120	-
1320 P	149	187	208	211	-
1321 P	119	157	209	216	-
1330 P	388	407	433	443	-
1331 P	221	261	351	381	-

Note:- * denotes result from 1 bar failed the ASTM repeatability test
+ denotes results from 2 bars failed the ASTM repeatability test
** denotes based on limit referred to in ASTM C227

TABLE D.1 continued

MIX REF NO.	EXPANSION IN MICROSTRAIN ($\mu\varepsilon$) AT AGE:-				'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	2 MONTHS	6 MONTHS	12 MONTHS	
2000	96	101	125	92	-
2001	92	95	105	88	-
2010 P	1317	1953	1993	1952	Harmful
2011 P	1309	2931	3048	3028	Harmful
2020 P	3447	4065	4099	4112	Harmful
2021 P	3432	6217	6467	6612	Harmful
2030 P	3643	3843	3887	3920	Harmful
2031 P	4680	6777	6948	6848	Harmful
2100	71	71	104	96	-
2101	125	124	156*	156	-
2110 P	539	597	597	584	-
2111 P	508	969	1732	1864	Harmful
2120 P	1307	1441	1479	1464	Harmful
2121 P	1768	2911	3557	3676	Harmful
2130 P	1743	1873	1924	1936	Harmful
2131 P	2163	2919	3201	3288	Harmful
2200	61	87*	137	156+	-
2201	96+	129+	177+	176+	-
2210 P	301	335	371	372	-
2211 P	301	407	491	520	-
2220 P	855	916	964	964	-
2221 P	707	904	1164	1448	Harmful
2230 P	1412	1505	1556	1552	Harmful
2231 P	1125	1461	1872	2104	Harmful
2300	91	113	133	140	-
2301	96*	131	163	152	-
2310 P	253	299	331	328	-
2311 P	187	237	309	300	-
2320 P	501	532	568	572	-
2321 P	375	465	543	600	-
2330 P	985	1047	1077	1084	Harmful
2331 P	593	683	800	864	-

Note:- * denotes result from 1 bar failed the ASTM repeatability test
+ denotes results from 2 bars failed the ASTM repeatability test
** denotes based on limit referred to in ASTM C227

TABLE D.2(a) REPEATABILITY COMPUTATIONS FOR SERIES 2 FACTORIAL SET, 14 DAY RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.  AVERAGE    MICRO-
  No.    EXPANSION   STRAIN
 0000      0.016       64.
 0001      0.019       77.
 0010P     0.014       56.
 0011P     0.018       71.
 0020P     0.025       99.
 0021P     0.016       64.
 0030P     0.228      912.
 0031P     0.048      193.
 0100      0.013       53.
 0101      0.016       65.
 0110P     0.017       67.
 0111P     0.021       84.
 0120P     0.015       60.
 0121P     0.017       69.
 0130P     0.071      283.
 0131P     0.038      153.
 0200      0.010       40.
 0201      0.023       93.
 0210P     0.015       60.
 0211P     0.020       80.
 0220P     0.019       75.
 0221P     0.012       48.
 0230P     0.037      149.
 0231P     0.029      117.
 0300      0.021       83.
 0301      0.024       96.
 0310P     0.011       43.
 0311P     0.019       77.
 0320P     0.019       77.
 0321P     0.022       87.
 0330P     0.034      135.
 0331P     0.024       96.
 1000      0.016       64.
 1001      0.012       48.
 1010P     0.052      208.
 1011P     0.035      141.
 1020P     0.313     1252.
 1021P     0.367     1467.
 1030P     0.658     2633.
 1031P     0.756     3025.
 1100      0.016       63.
 1101      0.017       67.
 1110P     0.027      109.
 1111P     0.035      139.
 1120P     0.102      408.
 1121P     0.138      552.
 1130P     0.275     1099.
 1131P     0.304     1215.
 1200      0.014       57.
 1201      0.020       80.
 1210P     0.017       67.
 1211P     0.019       75.
 1220P     0.046      184.
 1221P     0.036      144.
 1230P     0.181      725.
 1231P     0.144      576.
 1300      0.019       77.
 1301      0.019       77.
 1310P     0.023       92.
 1311P     0.017       69.
 1320P     0.037      149.
 1321P     0.030      119.
 1330P     0.097      388.
 1331P     0.055      221.
 2000      0.024       96.
 2001      0.023       92.
 2010P     0.329     1317.
 2011P     0.327     1309.
 2020P     0.862     3447.
 2021P     0.858     3432.
 2030P     0.911     3643.
 2031P     1.170     4680.
 2100      0.018       71.
 2101      0.031      125.
 2110P     0.135      539.
 2111P     0.127      508.
 2120P     0.327     1307.
 2121P     0.442     1768.
 2130P     0.436     1743.
 2131P     0.541     2163.
 2200      0.015       61.
 2201      0.024       96.
 2210P     0.075      301.
 2211P     0.075      301.
 2220P     0.214      855.
 2221P     0.177      707.
 2230P     0.353     1413.
 2231P     0.281     1125.
 2300      0.023       91.
 2301      0.024       96.
 2310P     0.063      253.
 2311P     0.047      187.
 2320P     0.125      501.
 2321P     0.094      375.
 2330P     0.246      985.
 2331P     0.148      593.
```

TABLE D.2(a) continued

[illegible] REF. NO.	EXPN(1)-AVGE	EXPN(2)-AVGE	EXPN(3)-AVGE	ALLOWABLE DIFFERENCE
0000	0.	0.	0.	0.003
0001	0.001	0.001	-0.001	0.003
0010P	-0.002	0.000	0.002	0.003
0011P	-0.002	0.002	-0.001	0.003
0020P	0.001	0.001	-0.003	0.003
0021P	-0.000	0.000	0.000	0.003
0030P	0.008	0.010	-0.018	0.034
0031P	-0.000	-0.003	0.004	0.003
0100	0.001	-0.001	0.001	0.003
0101	-0.002	0.001	0.002	0.003
0110P	0.001	-0.002	0.000	0.003
0111P	-0.001	-0.001	0.002	0.003
0120P	-0.000	-0.001	0.001	0.003
0121P	-0.000	0.001	-0.000	0.003
0130P	0.003	-0.003	-0.001	0.011
0131P	-0.005	0.002	0.004	0.003
0200	-0.000	-0.000	0.000	0.003
0201	-0.001	0.001	0.001	0.003
0210P	0.001	-0.001	-0.000	0.003
0211P	-0.002	-0.000	0.002	0.003
0220P	-0.001	0.001	-0.001	0.003
0221P	0.000	0.000	-0.000	0.003
0230P	-0.001	0.001	0.001	0.003
0231P	0.001	0.001	-0.001	0.003
0300	-0.001	0.001	-0.001	0.003
0301	0.	0.	0.	0.003
0310P	0.001	-0.001	-0.001	0.003
0311P	-0.001	0.001	0.001	0.003
0320P	-0.001	0.001	0.001	0.003
0321P	0.000	-0.001	0.000	0.003
0330P	0.002	-0.001	-0.002	0.003
0331P	0.001	-0.000	-0.001	0.003
1000	0.	0.	0.	0.003
1001	0.	0.	0.	0.003
1010P	0.	0.	0.	0.008
1011P	0.003	-0.003	0.001	0.003
1020P	0.014	-0.017	0.003	0.047
1021P	0.001	-0.019	0.017	0.055
1030P	-0.021	0.012	0.010	0.099
1031P	-0.005	0.002	0.004	0.113
1100	-0.001	0.000	0.000	0.003
1101	0.001	-0.001	-0.001	0.003
1110P	0.001	0.001	-0.001	0.003
1111P	0.001	0.001	-0.003	0.003
1120P	-0.002	0.000	0.002	0.015
1121P	0.006	-0.008	0.002	0.021
1130P	-0.001	-0.011	0.011	0.041
1131P	0.022	-0.027	0.004	0.046
1200	0.002	-0.001	-0.000	0.003
1201	0.000	0.000	-0.000	0.003
1210P	0.001	-0.001	-0.001	0.003
1211P	-0.001	0.000	0.000	0.003
1220P	0.	-0.002	0.002	0.003
1221P	0.000	-0.004	0.004	0.003
1230P	0.013	-0.011	-0.001	0.027
1231P	-0.004	-0.002	0.006	0.022
1300	0.001	0.001	-0.001	0.003
1301	-0.001	0.001	0.001	0.003
1310P	0.000	0.001	-0.001	0.003
1311P	0.001	0.001	-0.001	0.003
1320P	-0.001	0.003	-0.001	0.003
1321P	0.000	-0.002	0.001	0.003
1330P	0.003	-0.004	0.001	0.015
1331P	0.001	-0.001	0.001	0.008
2000	0.	0.	0.	0.003
2001	-0.001	0.000	0.001	0.003
2010P	0.009	-0.009	0.001	0.049
2011P	-0.001	-0.000	0.002	0.049
2020P	0.010	-0.013	0.002	0.129
2021P	-0.032	-0.010	0.042	0.129
2030P	-0.005	0.007	-0.003	0.137
2031P	0.026	-0.014	-0.012	0.175
2100	0.000	0.000	-0.001	0.003
2101	0.001	-0.001	0.001	0.003
2110P	-0.001	-0.001	0.001	0.020
2111P	0.009	0.000	-0.009	0.019
2120P	-0.007	0.005	0.001	0.049
2121P	-0.012	0.008	0.004	0.066
2130P	0.010	-0.006	-0.005	0.065
2131P	0.003	0.007	-0.011	0.081
2200	-0.002	0.001	0.002	0.003
2201	-0.002	-0.004	0.006	0.003
2210P	0.001	-0.001	0.001	0.011
2211P	0.001	-0.003	0.003	0.011
2220P	-0.010	-0.001	0.010	0.032
2221P	0.011	-0.012	0.000	0.027
2230P	0.005	-0.013	0.009	0.053
2231P	0.007	-0.011	0.005	0.042
2300	-0.001	-0.001	0.001	0.003
2301	0.004	-0.002	-0.002	0.003
2310P	-0.001	0.003	-0.001	0.010
2311P	-0.001	-0.003	0.003	0.003
2320P	-0.001	-0.001	0.003	0.019
2321P	0.004	-0.003	-0.002	0.014
2330P	-0.003	-0.006	0.010	0.037
2331P	-0.008	-0.005	0.014	0.022

TABLE D.2(a) continued

```
[illegible]
  NO.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 0000         0.003           0.003           0.003
 0001         0.002           0.002           0.002
 0010P        0.001           0.003           0.001
 0011P        0.001           0.001           0.002
 0020P        0.002           0.002           0.000
 0021P        0.003           0.003           0.003
 0030P        0.026           0.024         . 0.016
 0031P        0.003          -0.000          -0.001
 0100         0.002           0.002           0.002
 0101         0.001           0.002           0.001
 0110P        0.002           0.001           0.003
 0111P        0.002           0.002           0.001
 0120P        0.003           0.002           0.002
 0121P        0.003           0.002           0.003
 0130P        0.007           0.008           0.010
 0131P       -0.002           0.001          -0.001
 0200         0.003           0.003           0.003
 0201         0.002           0.002           0.002
 0210P        0.002           0.002           0.003
 0211P        0.001           0.003           0.001
 0220P        0.002           0.002           0.002
 0221P        0.003           0.003           0.003
 0230P        0.002           0.002           0.002
 0231P        0.002           0.002           0.002
 0300         0.002           0.002           0.002
 0301         0.003           0.003           0.003
 0310P        0.002           0.002           0.002
 0311P        0.002           0.002           0.002
 0320P        0.002           0.002           0.002
 0321P        0.003           0.002           0.003
 0330P        0.001           0.002           0.001
 0331P        0.002           0.003           0.002
 1000         0.003           0.003           0.003
 1001         0.003           0.003           0.003
 1010P        0.008           0.008           0.008
 1011P        0.000          -0.000           0.002
 1020P        0.033           0.030           0.044
 1021P        0.054           0.036           0.038
 1030P        0.077           0.027           0.089
 1031P        0.108           0.112           0.110
 1100         0.002           0.003           0.003
 1101         0.002           0.002           0.002
 1110P        0.002           0.002           0.002
 1111P        0.002           0.002           0.000
 1120P        0.013           0.015           0.013
 1121P        0.015           0.013           0.019
 1130P        0.041           0.031           0.030
 1131P        0.023           0.019           0.041
 1200         0.001           0.002           0.003
 1201         0.003           0.003           0.003
 1210P        0.002           0.002           0.002
 1211P        0.002           0.003           0.003
 1220P        0.003           0.001           0.001
 1221P        0.003          -0.001          -0.001
 1230P        0.015           0.016           0.026
 1231P        0.018           0.020           0.016
 1300         0.002           0.002           0.002
 1301         0.002           0.002           0.002
 1310P        0.003           0.002           0.002
 1311P        0.002           0.002           0.002
 1320P        0.002           0.000           0.002
 1321P        0.003           0.001           0.002
 1330P        0.012           0.011           0.014
 1331P        0.008           0.007           0.008
 2000         0.003           0.003           0.003
 2001         0.002           0.003           0.002
 2010P        0.041           0.040           0.049
 2011P        0.043           0.049           0.047
 2020P        0.119           0.117           0.127
 2021P        0.097           0.119           0.087
 2030P        0.132           0.129           0.134
 2031P        0.150           0.161           0.164
 2100         0.003           0.003           0.002
 2101         0.002           0.002           0.002
 2110P        0.020           0.020           0.019
 2111P        0.010           0.019           0.010
 2120P        0.042           0.044           0.048
 2121P        0.054           0.058           0.062
 2130P        0.055           0.060           0.061
 2131P        0.078           0.074           0.070
 2200         0.001           0.002           0.001
 2201         0.001          -0.001          -0.003
 2210P        0.011           0.010           0.011
 2211P        0.011           0.008           0.009
 2220P        0.022           0.031           0.022
 2221P        0.015           0.015           0.026
 2230P        0.048           0.040           0.044
 2231P        0.036           0.031           0.038
 2300         0.002           0.002           0.002
 2301        -0.001           0.001           0.001
 2310P        0.008           0.007           0.008
 2311P        0.002           0.000          -0.000
 2320P        0.017           0.017           0.016
 2321P        0.010           0.011           0.012
 2330P        0.034           0.031           0.027
 2331P        0.014           0.017           0.009
```

TABLE D.2(b) REPEATABILITY COMPUTATIONS FOR SERIES 2 FACTORIAL SET, 2 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE    MICRO-
  No.     EXPANSION     STRAIN
 0000       0.022        88.
 0001       0.020        80.
 0010P      0.017        68.
 0011P      0.021        85.
 0020P      0.037       149.
 0021P      0.026       104.
 0030P      0.355      1421.
 0031P      0.472      1889.
 0100       0.009        37.
 0101       0.020        81.
 0110P      0.015        59.
 0111P      0.019        76.
 0120P      0.016        65.
 0121P      0.015        61.
 0130P      0.088       353.
 0131P      0.193       771.
 0200      -0.000        -0.
 0201       0.013        53.
 0210P      0.010        40.
 0211P      0.015        60.
 0220P      0.017        68.
 0221P      0.007        27.
 0230P      0.040       161.
 0231P      0.039       157.
 0300       0.015        59.
 0301       0.023        91.
 0310P      0.007        27.
 0311P      0.014        57.
 0320P      0.017        68.
 0321P      0.025       101.
 0330P      0.033       133.
 0331P      0.029       115.
 1000       0.022        88.
 1001       0.021        85.
 1010P      0.073       291.
 1011P      0.063       252.
 1020P      0.379      1516.
 1021P      0.769      3077.
 1030P      0.708      2831.
 1031P      1.092      4367.
 1100       0.010        40.
 1101       0.021        83.
 1110P      0.034       136.
 1111P      0.042       167.
 1120P      0.113       453.
 1121P      0.325      1301.
 1130P      0.299      1197.
 1131P      0.561      2245.
 1200       0.018        73.
 1201       0.018        73.
 1210P      0.027       108.
 1211P      0.021        84.
 1220P      0.055       221.
 1221P      0.052       209.
 1230P      0.194       775.
 1231P      0.201       804.
 1300       0.029       117.
 1301       0.024        97.
 1310P      0.027       107.
 1311P      0.024        95.
 1320P      0.047       187.
 1321P      0.039       157.
 1330P      0.102       407.
 1331P      0.065       261.
 2000       0.025       101.
 2001       0.024        95.
 2010P      0.488      1953.
 2011P      0.733      2931.
 2020P      1.016      4065.
 2021P      1.554      6217.
 2030P      0.961      3843.
 2031P      1.694      6777.
 2100       0.018        71.
 2101       0.031       124.
 2110P      0.149       597.
 2111P      0.242       969.
 2120P      0.360      1441.
 2121P      0.728      2911.
 2130P      0.468      1873.
 2131P      0.730      2919.
 2200       0.022        87.
 2201       0.032       129.
 2210P      0.084       335.
 2211P      0.102       407.
 2220P      0.229       916.
 2221P      0.226       904.
 2230P      0.376      1505.
 2231P      0.365      1461.
 2300       0.028       113.
 2301       0.033       131.
 2310P      0.075       299.
 2311P      0.059       237.
 2320P      0.133       532.
 2321P      0.116       465.
 2330P      0.262      1047.
 2331P      0.171       683.
```

TABLE D.2(b) continued

Mix Ref. No.	EXPN(1)-AVGE	EXPN(2)-AVGE	EXPN(3)-AVGE	ALLOWABLE DIFFERENCE
0000	0.	0.	0.	0.003
0001	0.002	-0.000	-0.002	0.003
0010P	-0.001	0.	0.001	0.003
0011P	-0.002	0.004	-0.001	0.003
0020P	0.001	0.001	-0.001	0.003
0021P	-0.000	0.000	0.000	0.003
0030P	-0.008	0.021	-0.012	0.053
0031P	-0.008	-0.021	0.030	0.071
0100	0.001	-0.001	0.001	0.003
0101	-0.000	0.003	-0.002	0.003
0110P	0.001	-0.003	0.001	0.003
0111P	-0.001	-0.003	0.004	0.003
0120P	0.001	-0.000	-0.000	0.003
0121P	-0.000	0.001	-0.000	0.003
0130P	0.004	-0.002	-0.001	0.013
0131P	-0.017	0.006	0.010	0.029
0200	0.	-0.000	0.000	0.003
0201	-0.001	0.003	-0.001	0.003
0210P	-0.002	-0.001	0.003	0.003
0211P	-0.002	-0.000	0.002	0.003
0220P	0.001	0.000	-0.001	0.003
0221P	-0.001	0.001	-0.001	0.003
0230P	-0.001	-0.000	0.002	0.003
0231P	0.002	-0.001	-0.000	0.003
0300	-0.003	0.001	0.001	0.003
0301	0.001	-0.001	-0.001	0.003
0310P	-0.001	0.001	-0.001	0.003
0311P	-0.002	0.001	0.002	0.003
0320P	-0.003	-0.001	0.004	0.003
0321P	0.003	-0.005	0.003	0.003
0330P	0.003	-0.000	-0.002	0.003
0331P	0.002	-0.001	-0.002	0.003
1000	0.	0.	0.	0.003
1001	-0.001	-0.001	0.003	0.003
1010P	-0.003	0.001	0.001	0.011
1011P	0.007	-0.010	0.003	0.009
1020P	0.008	-0.011	0.003	0.057
1021P	-0.023	-0.019	0.043	0.115
1030P	-0.023	0.012	0.010	0.106
1031P	0.003	-0.025	0.021	0.164
1100	0.000	-0.000	-0.000	0.003
1101	0.000	0.000	-0.001	0.003
1110P	0.003	-0.001	-0.002	0.003
1111P	-0.002	0.006	-0.005	0.003
1120P	-0.001	-0.001	0.003	0.017
1121P	0.017	-0.025	0.009	0.049
1130P	-0.000	-0.011	0.012	0.045
1131P	0.039	-0.033	-0.005	0.084
1200	-0.000	-0.001	0.002	0.003
1201	-0.000	0.001	-0.000	0.003
1210P	0.002	0.000	-0.002	0.003
1211P	-0.001	0.000	0.001	0.003
1220P	0.002	-0.003	0.002	0.008
1221P	-0.000	-0.006	0.007	0.008
1230P	0.012	-0.012	-0.001	0.029
1231P	-0.003	-0.002	0.005	0.030
1300	0.001	0.001	-0.001	0.003
1301	-0.000	0.001	-0.000	0.003
1310P	0.000	0.001	-0.002	0.003
1311P	-0.001	0.000	0.000	0.003
1320P	-0.003	0.003	-0.001	0.003
1321P	0.001	-0.002	0.002	0.003
1330P	0.001	-0.004	0.002	0.015
1331P	0.001	-0.001	0.001	0.010
2000	0.001	0.001	-0.001	0.003
2001	-0.002	0.001	0.000	0.003
2010P	0.018	-0.007	-0.010	0.073
2011P	-0.023	0.014	0.008	0.110
2020P	0.026	-0.007	-0.018	0.152
2021P	-0.019	0.008	0.012	0.233
2030P	-0.005	0.008	-0.004	0.144
2031P	0.054	-0.041	-0.012	0.254
2100	0.000	-0.002	0.001	0.003
2101	-0.001	-0.002	0.003	0.003
2110P	-0.001	0.001	0.001	0.022
2111P	0.024	0.007	-0.030	0.036
2120P	-0.005	0.004	0.002	0.054
2121P	0.000	0.000	-0.001	0.109
2130P	0.014	-0.006	-0.007	0.070
2131P	0.014	-0.003	-0.012	0.109
2200	-0.005	0.001	0.003	0.003
2201	-0.002	-0.005	0.008	0.003
2210P	0.003	-0.002	-0.002	0.013
2211P	0.000	-0.004	0.003	0.015
2220P	-0.007	0.000	0.007	0.034
2221P	0.016	-0.016	0.	0.034
2230P	0.006	-0.014	0.009	0.056
2231P	0.011	-0.011	0.001	0.055
2300	-0.001	-0.000	0.002	0.003
2301	0.001	0.001	-0.003	0.003
2310P	-0.001	0.001	-0.001	0.011
2311P	-0.003	-0.003	0.007	0.009
2320P	-0.002	-0.001	0.003	0.020
2321P	0.004	-0.003	-0.000	0.017
2330P	-0.003	-0.008	0.010	0.039
2331P	-0.009	-0.006	0.014	0.026

TABLE D.2(b)   continued

```
Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD UE POSITIVE
0000            0.003          0.003          0.003
0001            0.001          0.003          0.001
0010P           0.002          0.003          0.002
0011P           0.001         -0.001          0.002
0020P           0.002          0.002          0.002
0021P           0.003          0.003          0.003
0030P           0.045          0.033          0.041
0031P           0.063          0.050          0.041
0100            0.002          0.002          0.002
0101            0.003          0.000          0.001
0110P           0.002          0.000          0.002
0111P           0.002         -0.000         -0.001
0120P           0.002          0.003          0.003
0121P           0.003          0.002          0.003
0130P           0.010          0.011          0.012
0131P           0.012          0.023          0.019
0200            0.003          0.003          0.003
0201            0.002          0.000          0.002
0210P           0.001          0.002         -0.000
0211P           0.001          0.003          0.001
0220P           0.002          0.003          0.002
0221P           0.002          0.002          0.002
0230P           0.002          0.003          0.001
0231P           0.001          0.002          0.003
0300            0.000          0.002          0.002
0301            0.002          0.002          0.002
0310P           0.002          0.002          0.002
0311P           0.001          0.002          0.001
0320P          -0.000          0.002         -0.001
0321P           0.000         -0.002          0.000
0330P           0.000          0.003          0.001
0331P           0.001          0.002          0.001
1000            0.003          0.003          0.003
1001            0.002          0.002          0.000
1010P           0.008          0.010          0.010
1011P           0.002         -0.001          0.006
1020P           0.049          0.046          0.054
1021P           0.092          0.096          0.073
1030P           0.083          0.094          0.096
1031P           0.160          0.139          0.142
1100            0.003          0.003          0.003
1101            0.003          0.003          0.002
1110P          -0.000          0.002          0.001
1111P           0.001         -0.003         -0.002
1120P           0.016          0.016          0.014
1121P           0.032          0.023          0.040
1130P           0.045          0.034          0.033
1131P           0.046          0.051          0.079
1200            0.003          0.002          0.001
1201            0.003          0.002          0.003
1210P           0.001          0.003          0.001
1211P           0.002          0.003          0.002
1220P           0.007          0.005          0.007
1221P           0.008          0.002          0.001
1230P           0.017          0.017          0.028
1231P           0.027          0.028          0.025
1300            0.002          0.002          0.002
1301            0.003          0.002          0.003
1310P           0.003          0.002          0.001
1311P           0.002          0.003          0.003
1320P           0.000         -0.000          0.002
1321P           0.002          0.001          0.001
1330P           0.014          0.012          0.013
1331P           0.009          0.008          0.009
2000            0.002          0.002          0.002
2001            0.001          0.002          0.003
2010P           0.056          0.066          0.063
2011P           0.087          0.096          0.102
2020P           0.127          0.145          0.134
2021P           0.214          0.225          0.221
2030P           0.139          0.136          0.140
2031P           0.200          0.213          0.242
2100            0.003          0.001          0.002
2101            0.002          0.001          0.000
2110P           0.021          0.022          0.022
2111P           0.013          0.030          0.006
2120P           0.049          0.050          0.052
2121P           0.109          0.109          0.108
2130P           0.057          0.064          0.063
2131P           0.095          0.107          0.098
2200           -0.002          0.002         -0.000
2201            0.001         -0.002         -0.005
2210P           0.009          0.011          0.011
2211P           0.015          0.012          0.012
2220P           0.027          0.034          0.027
2221P           0.018          0.018          0.034
2230P           0.051          0.042          0.048
2231P           0.044          0.043          0.054
2300            0.002          0.003          0.001
2301            0.002          0.002          0.000
2310P           0.011          0.010          0.011
2311P           0.006          0.006          0.002
2320P           0.018          0.019          0.017
2321P           0.014          0.014          0.017
2330P           0.037          0.032          0.029
2331P           0.017          0.020          0.011
```

TABLE D.2(c) REPEATABILITY COMPUTATIONS FOR SERIES 2 FACTORIAL SET, 6 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref. AVERAGE    MICRO-
 No.    EXPANSION    STRAIN
0000     0.023        91.
0001     0.020        81.
0010P    0.020        80.
0011P    0.023        91.
0020P    0.043       172.
0021P    0.025        99.
0030P    0.356      1425.
0031P    0.705      2819.
0100     0.013        52.
0101     0.024        95.
0110P    0.016        64.
0111P    0.022        88.
0120P    0.023        93.
0121P    0.025        99.
0130P    0.100       399.
0131P    0.506      2023.
0200     0.001         5.
0201     0.012        48.
0210P    0.016        63.
0211P    0.020        78.
0220P    0.015        60.
0221P    0.007        29.
0230P    0.045       180.
0231P    0.074       297.
0300     0.018        73.
0301     0.025        99.
0310P    0.008        31.
0311P    0.020        79.
0320P    0.020        79.
0321P    0.029       115.
0330P    0.037       147.
0331P    0.037       148.
1000     0.026       103.
1001     0.022        87.
1010P    0.078       311.
1011P    0.068       271.
1020P    0.383      1533.
1021P    0.791      3164.
1030P    0.716      2864.
1031P    1.096      4385.
1100     0.013        51.
1101     0.021        84.
1110P    0.038       153.
1111P    0.049       196.
1120P    0.113       452.
1121P    0.523      2092.
1130P    0.302      1208.
1131P    0.662      2649.
1200     0.023        91.
1201     0.025        99.
1210P    0.032       128.
1211P    0.031       124.
1220P    0.063       251.
1221P    0.085       340.
1230P    0.202       807.
1231P    0.309      1236.
1300     0.037       148.
1301     0.031       125.
1310P    0.034       137.
1311P    0.031       123.
1320P    0.052       208.
1321P    0.052       209.
1330P    0.108       433.
1331P    0.088       351.
2000     0.031       125.
2001     0.026       105.
2010P    0.498      1993.
2011P    0.762      3048.
2020P    1.025      4099.
2021P    1.617      6467.
2030P    0.972      3887.
2031P    1.737      6948.
2100     0.026       104.
2101     0.039       156.
2110P    0.149       597.
2111P    0.433      1732.
2120P    0.370      1479.
2121P    0.889      3557.
2130P    0.481      1924.
2131P    0.800      3201.
2200     0.034       137.
2201     0.044       177.
2210P    0.093       371.
2211P    0.123       491.
2220P    0.241       964.
2221P    0.291      1164.
2230P    0.389      1556.
2231P    0.468      1872.
2300     0.033       133.
2301     0.041       163.
2310P    0.083       331.
2311P    0.077       309.
2320P    0.142       568.
2321P    0.136       543.
2330P    0.269      1077.
2331P    0.200       800.
```

TABLE D.2(c) continued

Mix Ref. No.	EXPN(1)-AVGE	EXPN(2)-AVGE	EXPN(3)-AVGE	ALLOWABLE DIFFERENCE
0000	-0.001	-0.001	0.001	0.003
0001	-0.000	0.003	-0.002	0.003
0010P	-0.001	0.001	-0.000	0.003
0011P	-0.004	0.003	0.000	0.003
0020P	0.001	0.000	-0.001	0.003
0021P	-0.001	0.000	0.000	0.003
0030P	-0.010	0.022	-0.011	0.053
0031P	-0.023	-0.007	0.029	0.106
0100	0.002	-0.004	0.002	0.003
0101	0.000	0.001	-0.002	0.003
0110P	-0.000	-0.003	0.003	0.003
0111P	-0.002	-0.002	0.004	0.003
0120P	0.004	-0.002	-0.001	0.003
0121P	0.001	-0.003	0.001	0.003
0130P	0.004	-0.003	-0.002	0.015
0131P	-0.023	-0.017	0.039	0.076
0200	0.001	0.002	-0.002	0.003
0201	-0.001	0.001	-0.000	0.003
0210P	-0.004	0.000	0.003	0.003
0211P	-0.003	-0.000	0.003	0.003
0220P	0.001	0.000	-0.001	0.003
0221P	0.002	0.001	-0.002	0.003
0230P	-0.000	0.001	-0.001	0.003
0231P	-0.000	-0.000	0.001	0.011
0300	-0.002	0.002	0.001	0.003
0301	-0.001	0.000	0.000	0.003
0310P	0.000	0.000	-0.001	0.003
0311P	-0.004	0.003	0.000	0.003
0320P	-0.002	-0.002	0.003	0.003
0321P	0.002	-0.008	0.005	0.003
0330P	0.001	0.000	-0.002	0.003
0331P	0.003	-0.001	-0.002	0.003
1000	-0.001	-0.002	0.002	0.003
1001	-0.001	-0.002	0.002	0.003
1010P	-0.004	0.000	0.003	0.012
1011P	0.006	-0.010	0.003	0.010
1020P	0.007	-0.011	0.005	0.058
1021P	-0.024	-0.020	0.044	0.119
1030P	-0.021	0.011	0.010	0.107
1031P	0.004	-0.023	0.020	0.164
1100	0.000	-0.001	0.000	0.003
1101	0.003	-0.001	-0.002	0.003
1110P	0.003	0.002	-0.004	0.003
1111P	-0.000	0.003	-0.003	0.003
1120P	-0.001	-0.002	0.003	0.017
1121P	0.017	-0.027	0.010	0.078
1130P	0.000	-0.012	0.012	0.045
1131P	0.036	-0.027	-0.008	0.099
1200	0.001	-0.001	-0.001	0.003
1201	0.000	0.001	-0.002	0.003
1210P	0.001	0.000	-0.001	0.003
1211P	0.001	0.000	-0.001	0.003
1220P	0.001	-0.004	0.002	0.009
1221P	-0.001	-0.006	0.007	0.013
1230P	0.011	-0.011	-0.001	0.030
1231P	-0.001	-0.006	0.007	0.046
1300	0.001	0.001	-0.002	0.003
1301	0.002	-0.001	-0.000	0.003
1310P	-0.000	0.002	-0.001	0.003
1311P	0.001	-0.001	-0.001	0.003
1320P	-0.004	0.004	0.000	0.008
1321P	0.001	-0.003	0.003	0.008
1330P	0.001	-0.004	0.004	0.016
1331P	0.000	-0.003	0.002	0.013
2000	-0.001	0.003	-0.001	0.003
2001	-0.001	0.001	0.001	0.003
2010P	0.018	-0.005	-0.012	0.075
2011P	-0.026	0.022	0.004	0.114
2020P	0.027	-0.008	-0.020	0.154
2021P	-0.023	0.005	0.017	0.243
2030P	-0.005	0.008	-0.004	0.146
2031P	0.031	-0.031	0.000	0.261
2100	-0.000	0.001	-0.001	0.003
2101	-0.002	-0.003	0.005	0.003
2110P	-0.000	-0.000	0.001	0.022
2111P	0.041	0.004	-0.045	0.065
2120P	-0.007	0.004	0.002	0.055
2121P	-0.007	0.001	0.007	0.133
2130P	0.019	-0.008	-0.011	0.072
2131P	0.021	-0.009	-0.011	0.120
2200	-0.003	0.001	0.003	0.003
2201	-0.004	-0.003	0.008	0.003
2210P	0.002	-0.001	-0.002	0.014
2211P	-0.002	-0.003	0.004	0.018
2220P	-0.009	0.000	0.009	0.036
2221P	0.019	-0.019	0.	0.044
2230P	0.006	-0.015	0.009	0.058
2231P	0.016	-0.015	-0.001	0.070
2300	-0.001	-0.001	0.003	0.003
2301	0.001	-0.001	-0.001	0.003
2310P	-0.002	0.001	0.000	0.012
2311P	-0.002	-0.004	0.007	0.012
2320P	-0.001	-0.002	0.003	0.021
2321P	0.007	-0.004	-0.004	0.020
2330P	-0.002	-0.008	0.011	0.040
2331P	-0.008	-0.006	0.014	0.030

TABLE D.2(c)   continued

```
Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
0000          0.002        0.002        0.002
0001          0.003        0.000        0.001
0010P         0.002        0.002        0.003
0011P        -0.001       -0.000        0.003
0020P         0.002        0.003        0.002
0021P         0.002        0.003        0.003
0030P         0.043        0.032      . 0.042
0031P         0.083        0.099        0.076
0100          0.001       -0.001        0.001
0101          0.003        0.002        0.001
0110P         0.003       -0.000       -0.000
0111P         0.001        0.001       -0.001
0120P        -0.001        0.001        0.002
0121P         0.002        0.000        0.002
0130P         0.011        0.012        0.013
0131P         0.053        0.059        0.037
0200          0.002        0.001        0.001
0201          0.002        0.002        0.003
0210P        -0.001        0.003       -0.000
0211P         0.000        0.003        0.000
0220P         0.002        0.003        0.002
0221P         0.001        0.002        0.001
0230P         0.003        0.002        0.002
0231P         0.011        0.011        0.010
0300          0.001        0.001        0.002
0301          0.002        0.003        0.003
0310P         0.003        0.003        0.002
0311P        -0.001       -0.000        0.003
0320P         0.001        0.001       -0.000
0321P         0.001       -0.005       -0.002
0330P         0.002        0.003        0.001
0331P        -0.000        0.002        0.001
1000          0.002        0.001        0.001
1001          0.002        0.001        0.001
1010P         0.008        0.011        0.008
1011P         0.004        0.000        0.007
1020P         0.051        0.046        0.053
1021P         0.095        0.099        0.075
1030P         0.086        0.096        0.097
1031P         0.161        0.141        0.145
1100          0.003        0.002        0.003
1101          0.000        0.002        0.001
1110P         0.000        0.001       -0.001
1111P         0.003       -0.000       -0.000
1120P         0.016        0.015        0.014
1121P         0.061        0.051        0.068
1130P         0.045        0.033        0.033
1131P         0.064        0.072        0.091
1200          0.002        0.002        0.002
1201          0.003        0.002        0.001
1210P         0.002        0.003        0.002
1211P         0.002        0.003        0.002
1220P         0.008        0.006        0.007
1221P         0.012        0.007        0.006
1230P         0.019        0.020        0.030
1231P         0.045        0.040        0.039
1300          0.002        0.002        0.001
1301          0.001        0.002        0.003
1310P         0.003        0.001        0.002
1311P         0.002        0.002        0.002
1320P         0.004        0.004        0.008
1321P         0.007        0.005        0.005
1330P         0.016        0.012        0.013
1331P         0.013        0.010        0.011
2000          0.002        0.000        0.002
2001          0.002        0.002        0.002
2010P         0.057        0.069        0.062
2011P         0.088        0.092        0.110
2020P         0.126        0.146        0.134
2021P         0.220        0.237        0.225
2030P         0.141        0.137        0.142
2031P         0.230        0.230        0.261
2100          0.003        0.002        0.002
2101          0.001       -0.000       -0.002
2110P         0.022        0.022        0.022
2111P         0.024        0.061        0.020
2120P         0.049        0.051        0.053
2121P         0.126        0.133        0.127
2130P         0.053        0.064        0.061
2131P         0.099        0.111        0.109
2200         -0.000        0.002        0.000
2201         -0.001       -0.000       -0.005
2210P         0.012        0.013        0.012
2211P         0.017        0.016        0.014
2220P         0.027        0.036        0.027
2221P         0.025        0.025        0.044
2230P         0.052        0.043        0.049
2231P         0.054        0.055        0.069
2300          0.002        0.002        0.000
2301          0.002        0.002        0.002
2310P         0.011        0.011        0.012
2311P         0.009        0.007        0.005
2320P         0.020        0.019        0.018
2321P         0.013        0.017        0.017
2330P         0.038        0.032        0.030
2331P         0.022        0.024        0.016
```

TABLE D.2(d) REPEATABILITY COMPUTATIONS FOR SERIES 2 FACTORIAL SET, 12 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION     STRAIN
 0000      0.017          67.
 0001      0.005          21.
 0010P     0.009          37.
 0011P     0.021          83.
 0020P     0.035         139.
 0021P     0.020          80.
 0030P     0.345        1379.
 0031P     0.707        2827.
 0100      0.006          24.
 0101      0.022          88.
 0110P     0.015          59.
 0111P     0.017          69.
 0120P     0.022          88.
 0121P     0.027         107.
 0130P     0.094         376.
 0131P     0.561        2243.
 0200     -0.005         -21.
 0201      0.011          43.
 0210P     0.005          21.
 0211P     0.010          40.
 0220P     0.012          48.
 0221P     0.007          29.
 0230P     0.044         176.
 0231P     0.147         587.
 0300      0.017          67.
 0301      0.023          91.
 0310P     0.004          16.
 0311P     0.015          59.
 0320P     0.015          61.
 0321P     0.026         104.
 0330P     0.033         131.
 0331P     0.042         168.
 1000      0.023          91.
 1001      0.013          51.
 1010P     0.071         283.
 1011P     0.065         261.
 1020P     0.383        1533.
 1021P     0.782        3128.
 1030P     0.715        2859.
 1031P     1.095        4381.
 1100      0.007          29.
 1101      0.016          64.
 1110P     0.029         115.
 1111P     0.050         200.
 1120P     0.110         440.
 1121P     0.582        2328.
 1130P     0.297        1187.
 1131P     0.685        2739.
 1200      0.019          77.
 1201      0.024          96.
 1210P     0.029         117.
 1211P     0.029         115.
 1220P     0.059         237.
 1221P     0.112         448.
 1230P     0.202         808.
 1231P     0.379        1517.
 1300      0.041         163.
 1301      0.025          99.
 1310P     0.035         141.
 1311P     0.030         120.
 1320P     0.053         211.
 1321P     0.054         216.
 1330P     0.111         443.
 1331P     0.095         381.
 2000      0.023          92.
 2001      0.022          88.
 2010P     0.488        1952.
 2011P     0.757        3028.
 2020P     1.028        4112.
 2021P     1.653        6612.
 2030P     0.980        3920.
 2031P     1.712        6848.
 2100      0.024          96.
 2101      0.039         156.
 2110P     0.146         584.
 2111P     0.466        1864.
 2120P     0.366        1464.
 2121P     0.919        3676.
 2130P     0.484        1936.
 2131P     0.822        3288.
 2200      0.039         156.
 2201      0.044         176.
 2210P     0.093         372.
 2211P     0.130         520.
 2220P     0.241         964.
 2221P     0.362        1448.
 2230P     0.388        1552.
 2231P     0.526        2104.
 2300      0.035         140.
 2301      0.038         152.
 2310P     0.082         328.
 2311P     0.075         300.
 2320P     0.143         572.
 2321P     0.150         600.
 2330P     0.271        1084.
 2331P     0.216         864.
```

TABLE D.2(d) continued

Mix Ref. No.	EXPN(1)-AVGE	EXPN(2)-AVGE	EXPN(3)-AVGE	ALLOWABLE DIFERENCE
0000	-0.001	-0.003	0.003	0.003
0001	0.005	-0.001	-0.003	0.003
0010P	0.001	0.001	-0.001	0.003
0011P	-0.003	0.003	-0.001	0.003
0020P	0.001	0.001	-0.003	0.003
0021P	0.	0.	0.	0.003
0030P	-0.009	0.019	-0.011	0.052
0031P	-0.027	-0.003	0.029	0.106
0100	0.000	-0.002	0.002	0.003
0101	0.000	-0.004	0.004	0.003
0110P	-0.001	-0.005	0.005	0.003
0111P	-0.003	0.001	0.003	0.003
0120P	0.008	-0.006	-0.002	0.003
0121P	0.001	-0.003	0.001	0.003
0130P	0.004	-0.002	-0.002	0.014
0131P	-0.031	-0.007	0.037	0.084
0200	0.001	0.001	-0.003	0.003
0201	0.001	0.001	-0.003	0.003
0210P	-0.005	0.005	0.001	0.003
0211P	-0.002	-0.000	0.002	0.003
0220P	-0.002	0.000	0.002	0.003
0221P	0.003	0.001	-0.003	0.003
0230P	0.	0.002	-0.002	0.003
0231P	-0.005	0.003	0.001	0.022
0300	-0.001	0.001	-0.001	0.003
0301	-0.003	0.001	0.001	0.003
0310P	0.	0.	0.	0.003
0311P	-0.001	0.001	-0.001	0.003
0320P	0.001	-0.001	0.001	0.003
0321P	0.008	-0.012	0.004	0.003
0330P	0.001	0.003	-0.005	0.003
0331P	0.004	-0.000	-0.004	0.003
1000	-0.001	-0.001	0.001	0.003
1001	0.001	0.003	-0.005	0.003
1010P	-0.001	-0.005	0.005	0.011
1011P	0.005	-0.005	0.001	0.010
1020P	0.009	-0.013	0.005	0.058
1021P	-0.026	-0.020	0.046	0.117
1030P	-0.017	0.005	0.011	0.107
1031P	0.007	-0.025	0.019	0.164
1100	0.007	-0.005	-0.001	0.003
1101	0.006	-0.002	-0.004	0.003
1110P	0.003	0.003	-0.007	0.003
1111P	0.002	0.002	-0.004	0.003
1120P	-0.002	-0.002	0.004	0.017
1121P	0.066	-0.052	-0.014	0.097
1130P	-0.001	-0.011	0.011	0.045
1131P	0.033	-0.027	-0.007	0.103
1200	0.003	0.001	-0.003	0.003
1201	0.	0.004	-0.004	0.003
1210P	0.005	-0.003	-0.001	0.003
1211P	0.003	-0.001	-0.003	0.003
1220P	-0.001	-0.005	0.007	0.009
1221P	-0.002	-0.006	0.008	0.017
1230P	0.012	-0.008	-0.004	0.030
1231P	0.003	-0.013	0.011	0.057
1300	0.001	0.003	-0.005	0.003
1301	0.005	-0.003	-0.003	0.003
1310P	-0.007	0.005	0.003	0.003
1311P	0.000	0.000	-0.000	0.003
1320P	-0.003	0.005	-0.003	0.008
1321P	0.002	-0.006	0.004	0.008
1330P	0.005	-0.009	0.003	0.017
1331P	0.003	-0.005	0.003	0.014
2000	-0.001	0.001	-0.000	0.003
2001	0.000	-0.002	0.002	0.003
2010P	0.016	-0.000	-0.016	0.073
2011P	-0.027	0.027	0.	0.114
2020P	0.024	0.	-0.024	0.154
2021P	-0.019	0.	0.019	0.248
2030P	-0.004	0.004	-0.000	0.147
2031P	0.010	-0.010	0.	0.257
2100	-0.000	0.000	0.000	0.003
2101	-0.003	0.000	0.003	0.003
2110P	0.000	-0.000	-0.000	0.022
2111P	0.046	-0.000	-0.046	0.070
2120P	-0.006	0.006	0.000	0.055
2121P	-0.007	0.	0.007	0.138
2130P	0.012	0.000	-0.012	0.073
2131P	0.018	0.000	-0.018	0.123
2200	-0.007	0.000	0.007	0.003
2201	-0.006	0.000	0.006	0.003
2210P	0.001	-0.000	-0.001	0.014
2211P	0.	-0.002	0.002	0.020
2220P	-0.007	0.000	0.007	0.036
2221P	0.	0.	0.000	0.054
2230P	-0.000	-0.016	0.016	0.058
2231P	0.018	-0.018	0.	0.079
2300	0.	-0.001	0.001	0.003
2301	0.002	-0.002	-0.000	0.003
2310P	-0.004	0.004	0.000	0.012
2311P	0.	-0.005	0.005	0.011
2320P	0.	-0.003	0.003	0.021
2321P	-0.000	0.000	0.000	0.022
2330P	0.	-0.009	0.009	0.041
2331P	-0.014	0.000	0.014	0.032

TABLE D.2(d) continued

Mix Ref. No.	CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE		
0000	0.002	0.000	-0.000
0001	-0.002	0.002	-0.000
0010P	0.002	0.002	0.002
0011P	0.000	-0.000	0.002
0020P	0.002	0.002	0.000
0021P	0.003	0.003	0.003
0030P	0.043	0.032	0.041
0031P	0.079	0.103	0.077
0100	0.003	0.001	0.001
0101	0.003	-0.001	-0.001
0110P	0.002	-0.002	-0.002
0111P	-0.000	0.002	0.000
0120P	-0.005	-0.003	0.001
0121P	0.002	0.000	0.002
0130P	0.010	0.012	0.012
0131P	0.053	0.077	0.047
0200	0.002	0.002	0.000
0201	0.002	0.002	0.000
0210P	-0.002	-0.002	0.002
0211P	0.001	0.003	0.001
0220P	0.001	0.003	0.001
0221P	0.000	0.002	-0.000
0230P	0.003	0.001	0.001
0231P	0.017	0.019	0.021
0300	0.002	0.002	0.002
0301	0.000	0.002	0.002
0310P	0.003	0.003	0.003
0311P	0.002	0.002	0.002
0320P	0.002	0.002	0.002
0321P	-0.005	-0.009	-0.001
0330P	0.002	-0.000	-0.002
0331P	-0.001	0.003	-0.001
1000	0.002	0.002	0.002
1001	0.002	-0.000	-0.002
1010P	0.010	0.006	0.005
1011P	0.005	0.004	0.009
1020P	0.049	0.044	0.053
1021P	0.091	0.097	0.071
1030P	0.091	0.102	0.096
1031P	0.158	0.139	0.146
1100	-0.004	-0.002	0.002
1101	-0.003	0.001	-0.001
1110P	-0.000	-0.000	-0.004
1111P	0.001	0.001	-0.001
1120P	0.014	0.014	0.012
1121P	0.021	0.035	0.073
1130P	0.044	0.034	0.033
1131P	0.069	0.076	0.096
1200	0.000	0.002	-0.000
1201	0.003	-0.001	-0.001
1210P	-0.002	-0.000	0.002
1211P	-0.000	0.002	0.000
1220P	0.008	0.004	0.002
1221P	0.015	0.011	0.009
1230P	0.018	0.022	0.026
1231P	0.054	0.044	0.046
1300	0.002	-0.000	-0.002
1301	-0.002	0.000	0.000
1310P	-0.004	-0.002	0.000
1311P	0.003	0.003	0.003
1320P	0.005	0.003	0.005
1321P	0.006	0.002	0.004
1330P	0.011	0.008	0.013
1331P	0.012	0.009	0.012
2000	0.002	0.002	0.003
2001	0.003	0.001	0.001
2010P	0.057	0.073	0.057
2011P	0.087	0.087	0.114
2020P	0.130	0.154	0.130
2021P	0.229	0.248	0.229
2030P	0.143	0.143	0.147
2031P	0.247	0.247	0.257
2100	0.003	0.003	0.003
2101	0.000	0.003	0.000
2110P	0.022	0.022	0.022
2111P	0.024	0.070	0.024
2120P	0.049	0.049	0.055
2121P	0.131	0.138	0.131
2130P	0.061	0.073	0.061
2131P	0.105	0.123	0.105
2200	-0.004	0.003	-0.004
2201	-0.003	0.003	-0.003
2210P	0.013	0.014	0.013
2211P	0.020	0.018	0.018
2220P	0.029	0.036	0.029
2221P	0.054	0.054	0.054
2230P	0.058	0.042	0.042
2231P	0.061	0.061	0.079
2300	0.003	0.002	0.002
2301	0.001	0.001	0.003
2310P	0.008	0.008	0.012
2311P	0.011	0.006	0.006
2320P	0.021	0.018	0.018
2321P	0.022	0.022	0.022
2330P	0.041	0.032	0.032
2331P	0.018	0.032	0.018

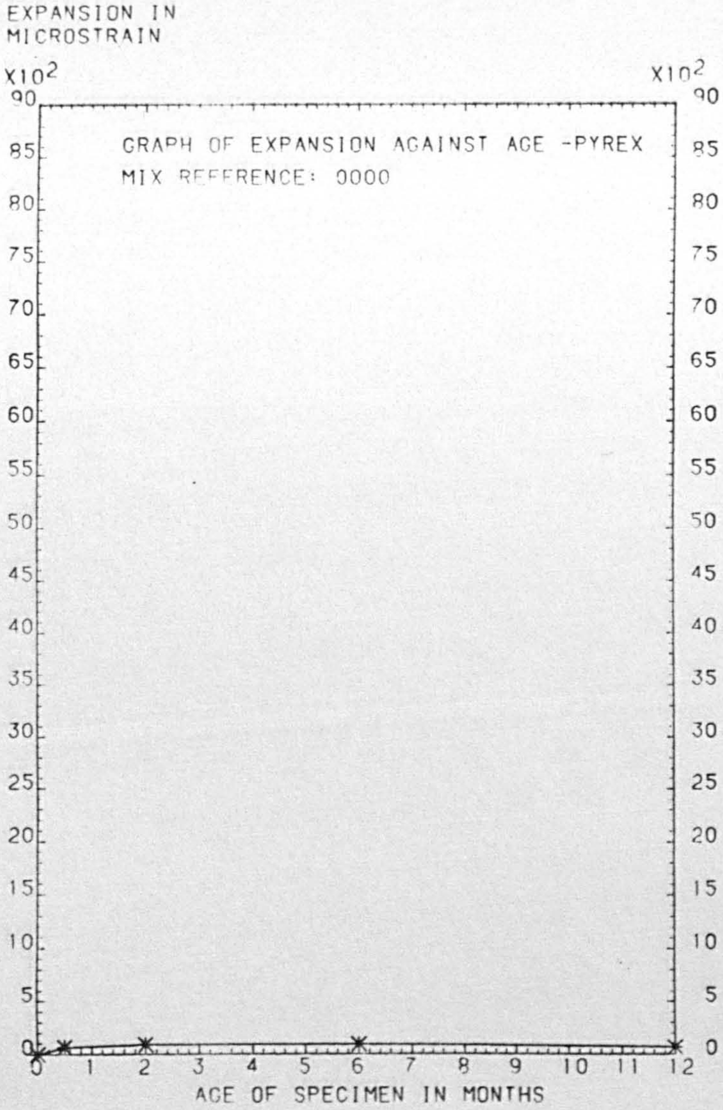


Figure D.1

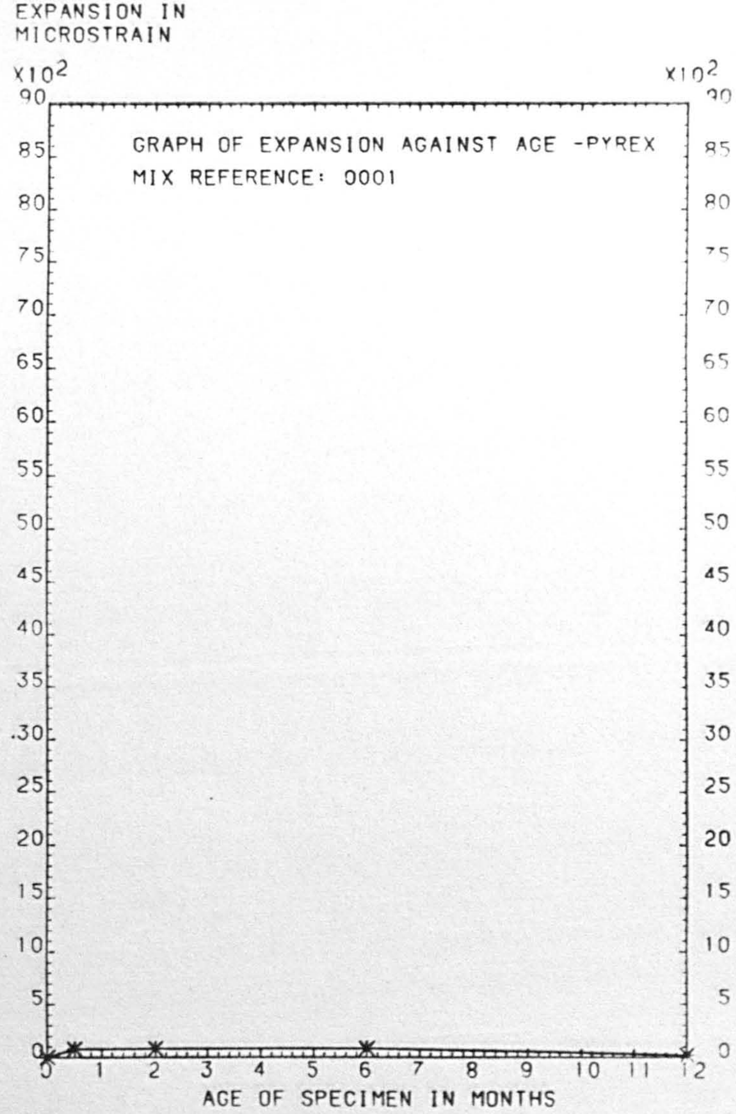


Figure D.2

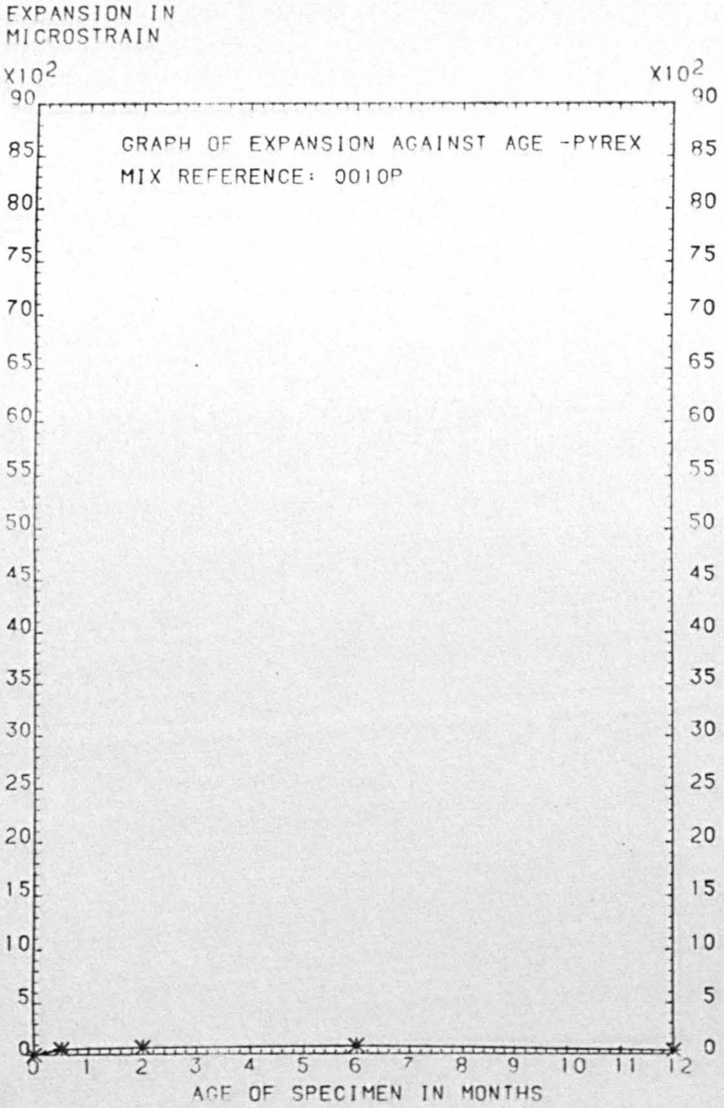


Figure D.3

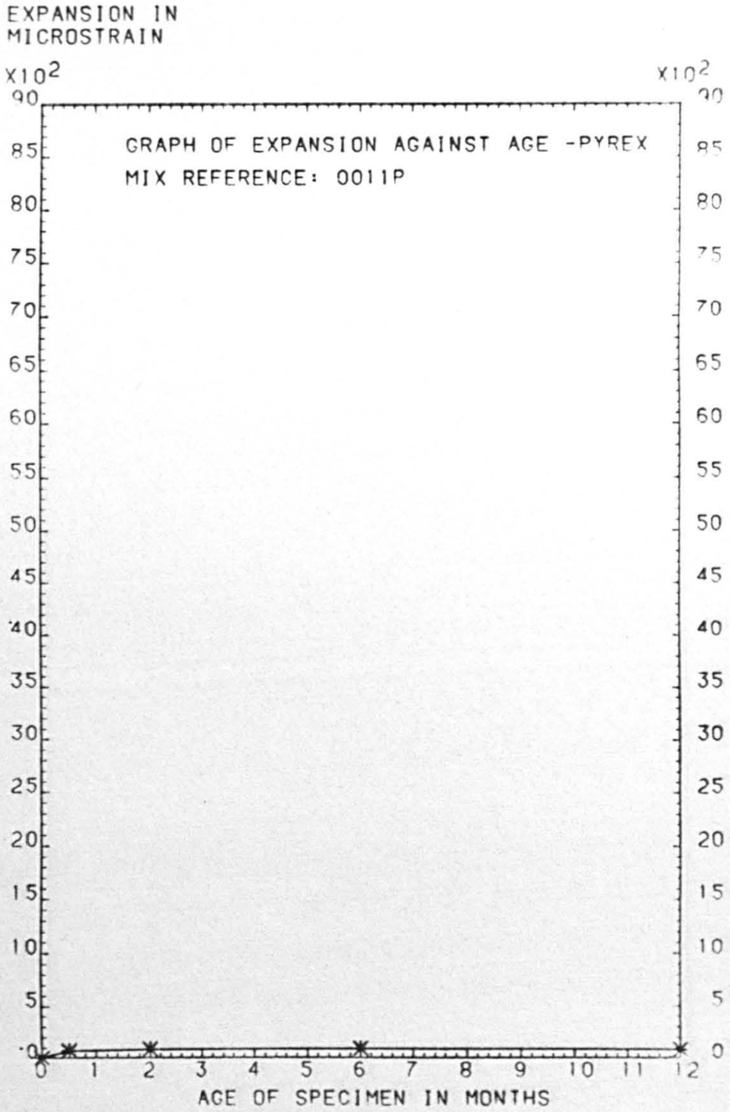


Figure D.4

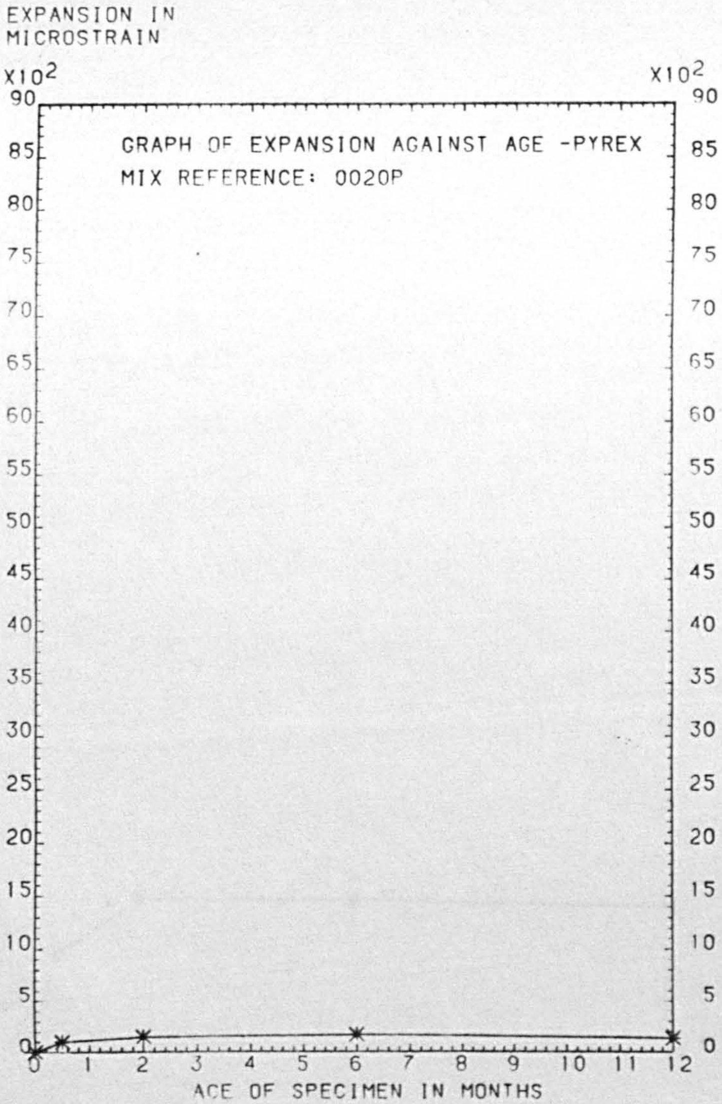


Figure D.5

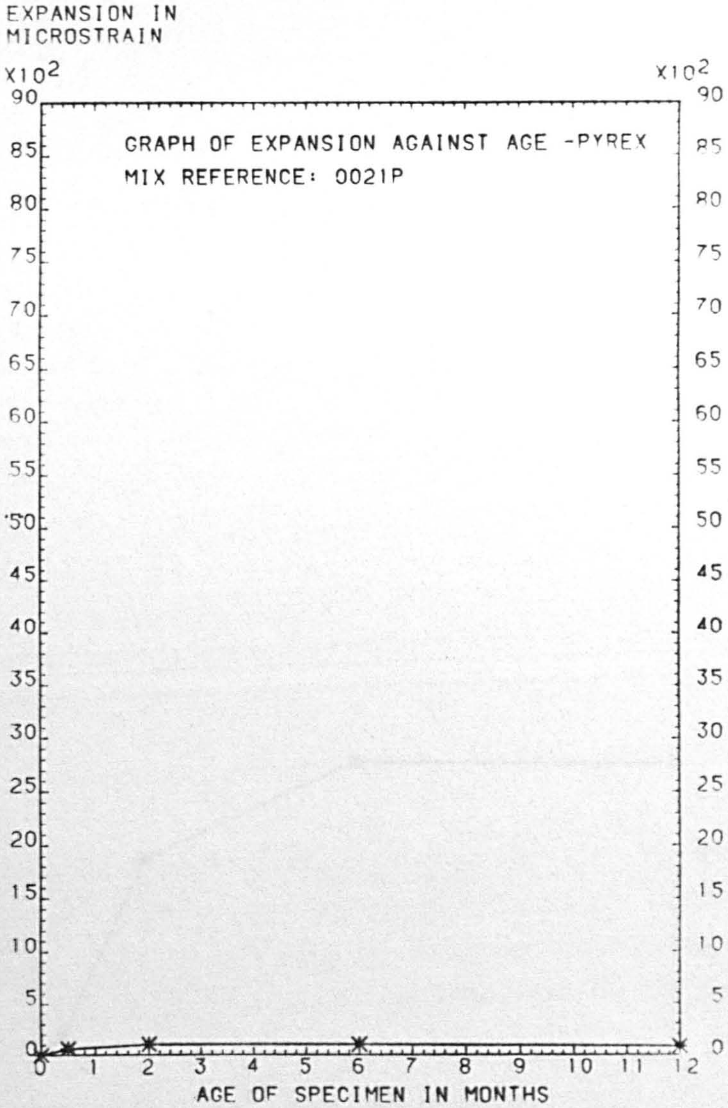


Figure D.6

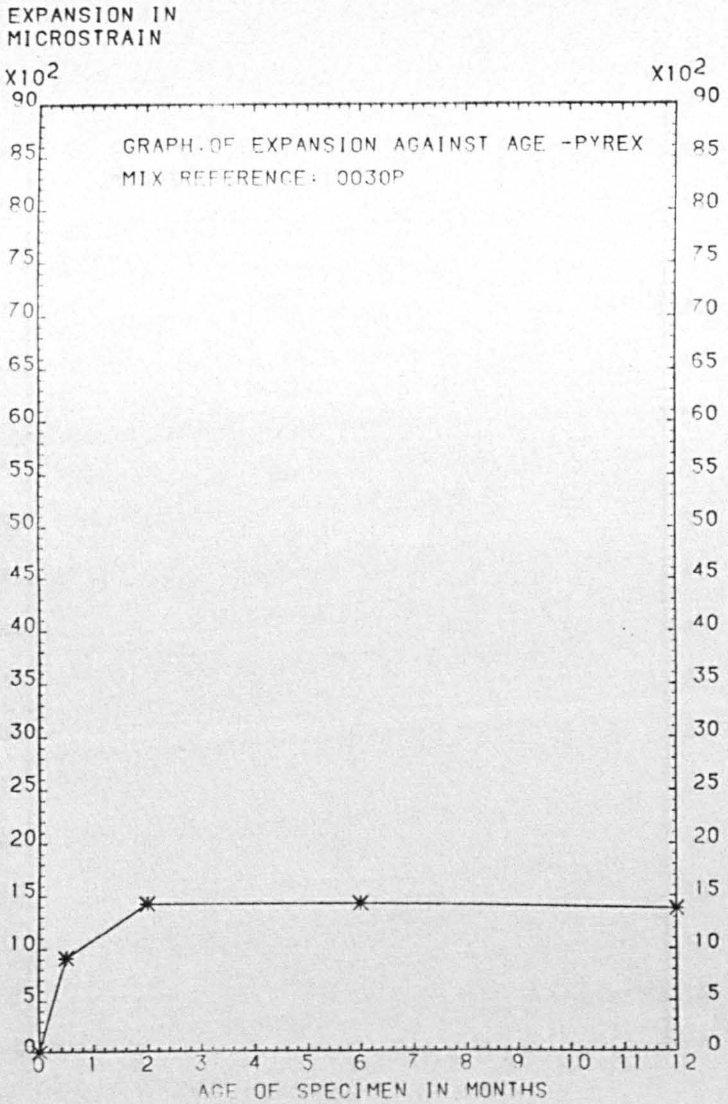


Figure D.7

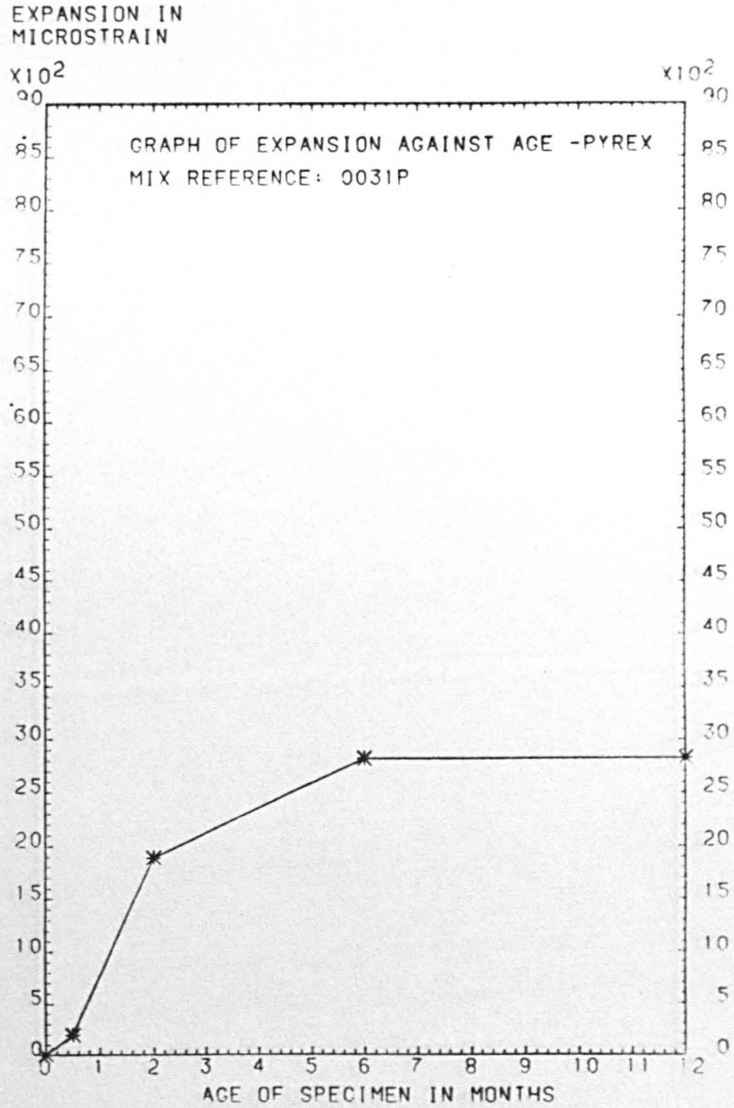


Figure D.8

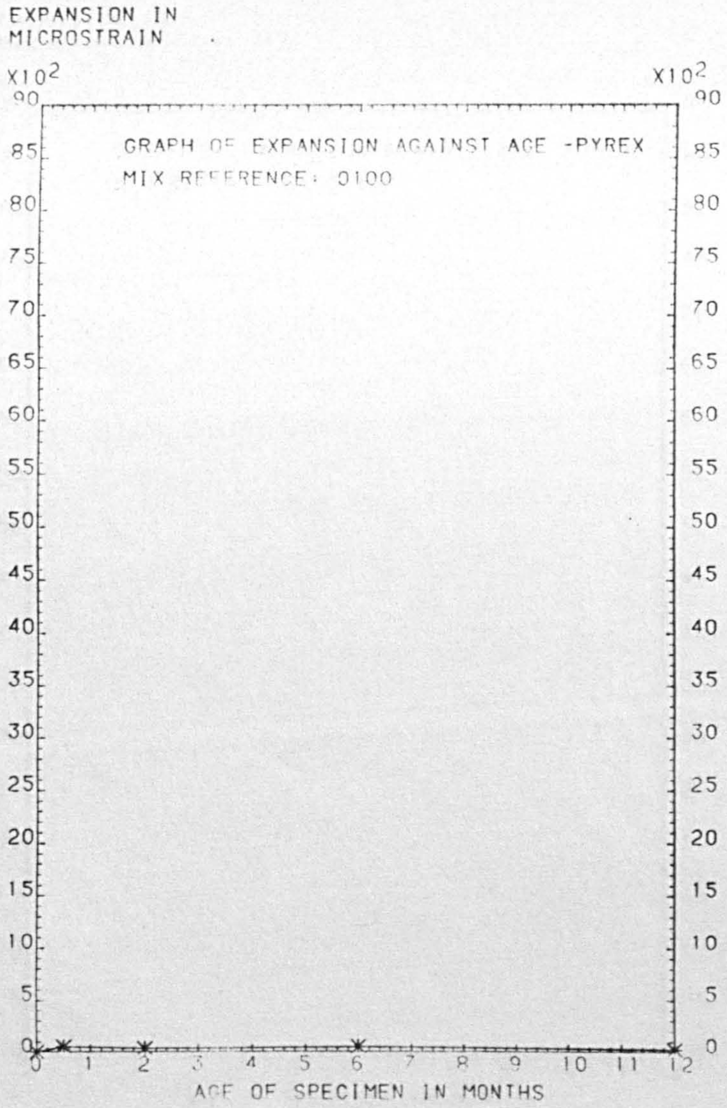


Figure D.9

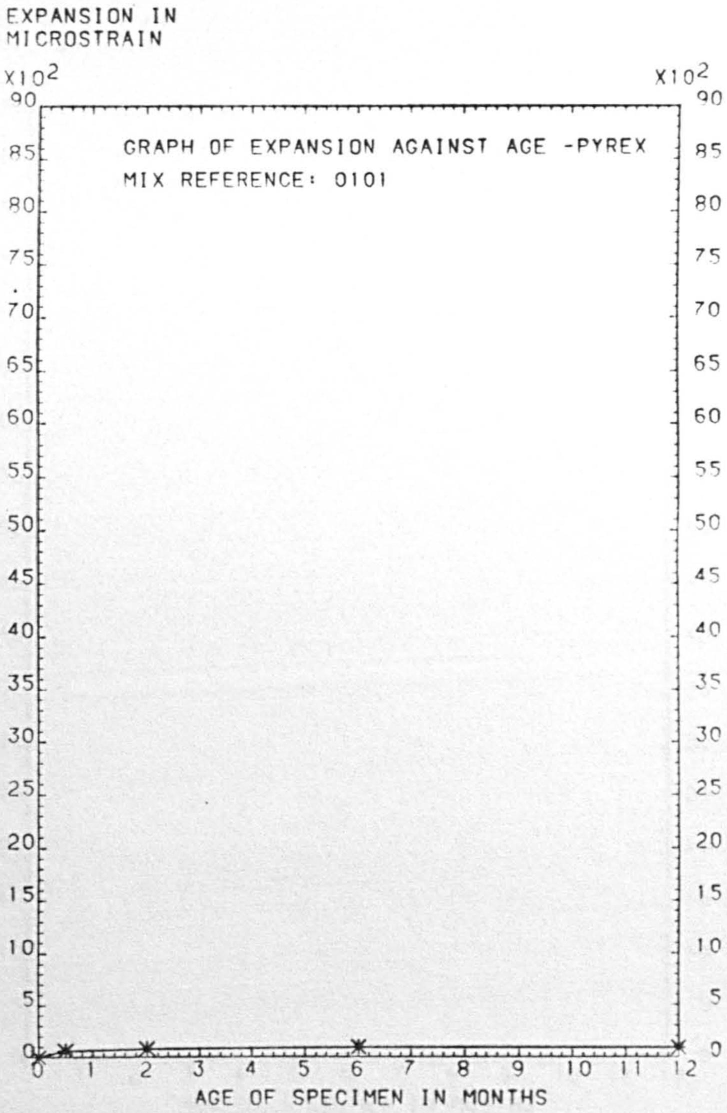
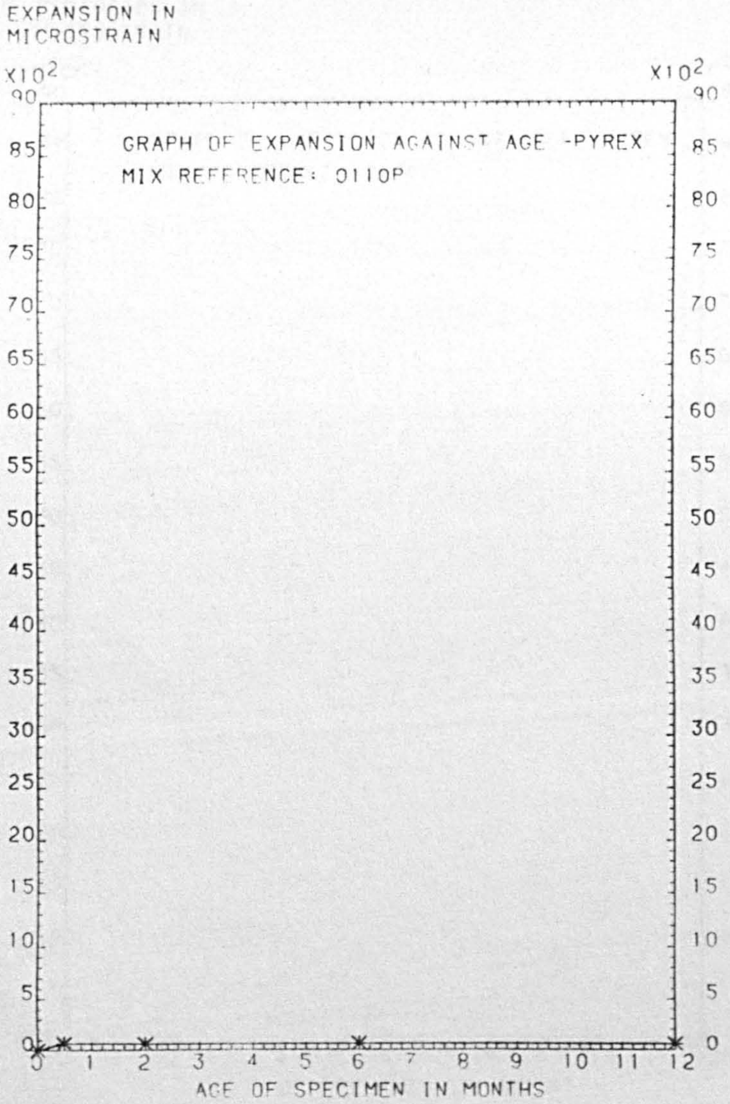


Figure D.10



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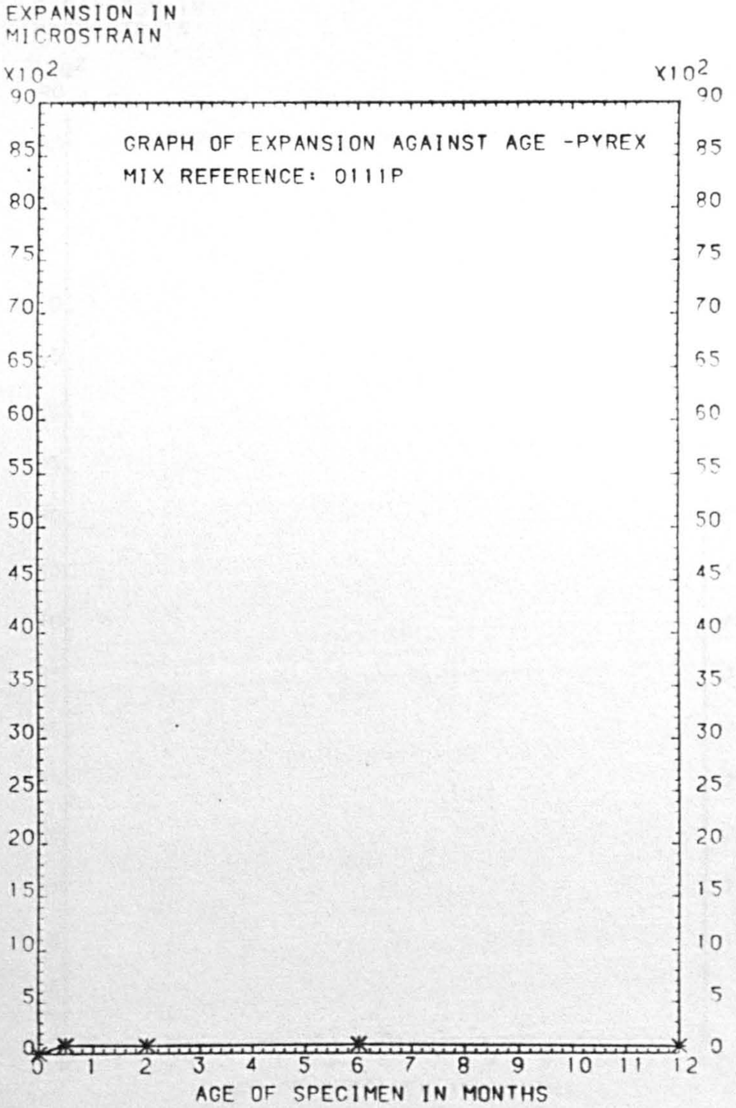

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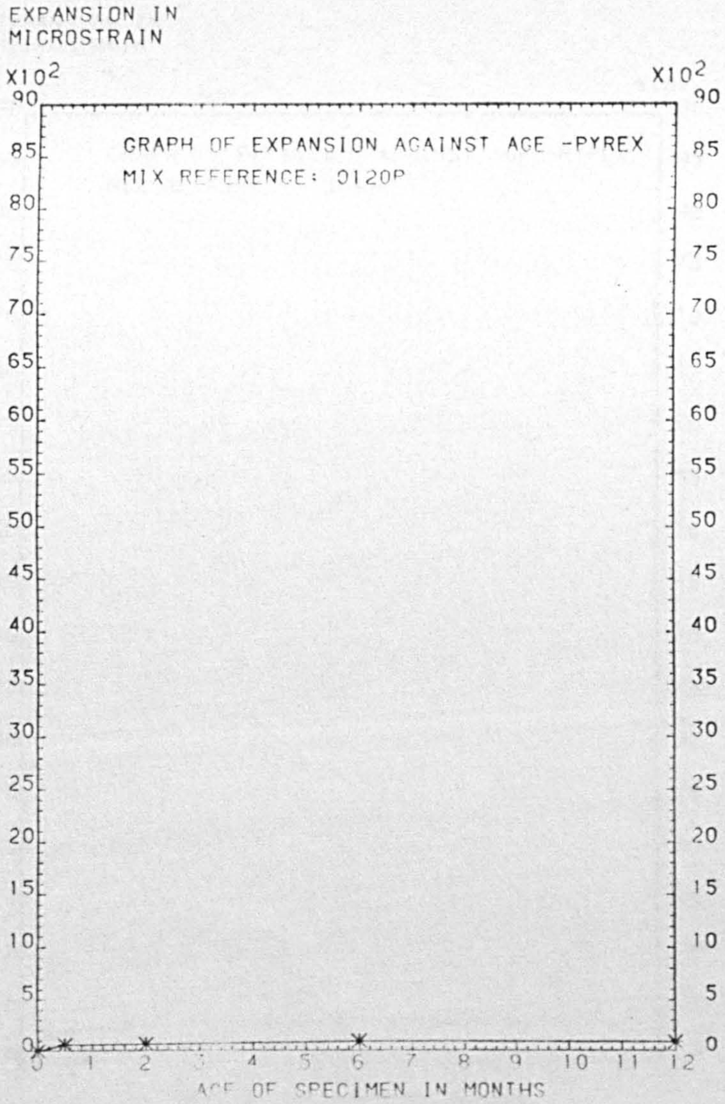

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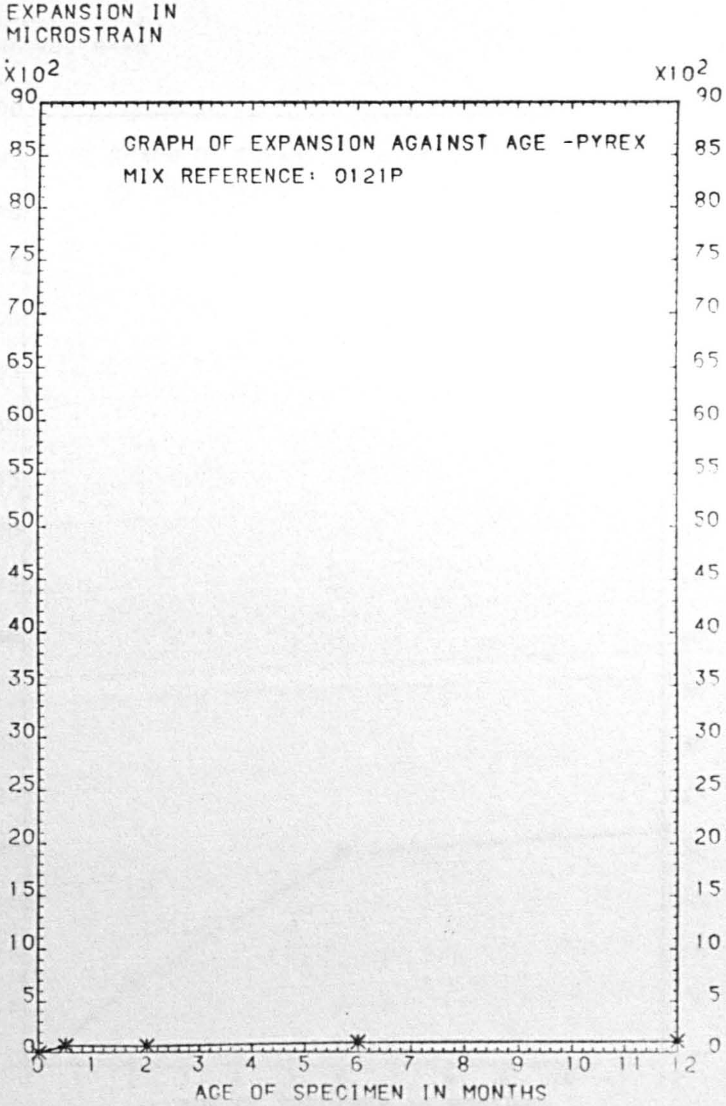

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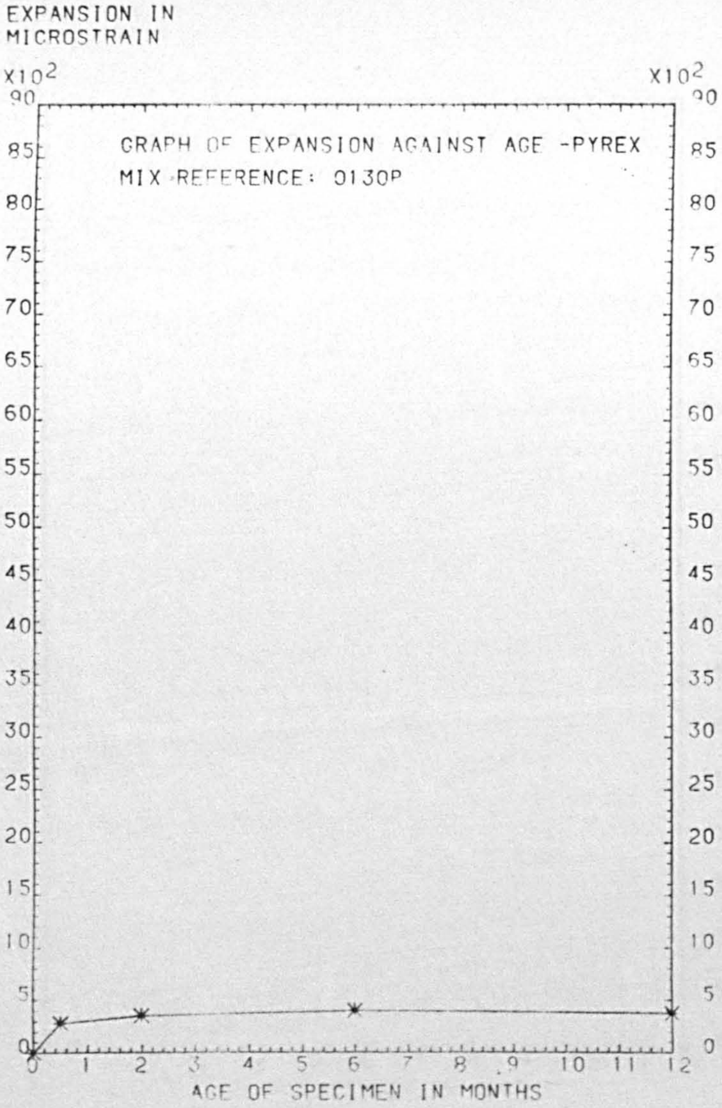

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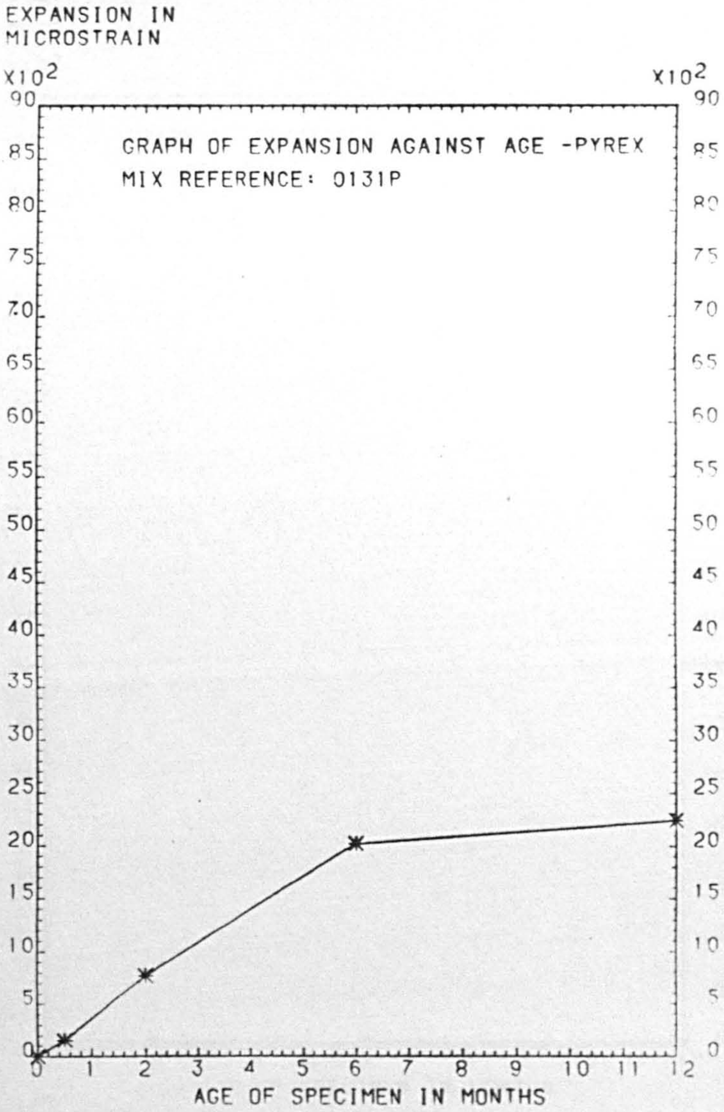

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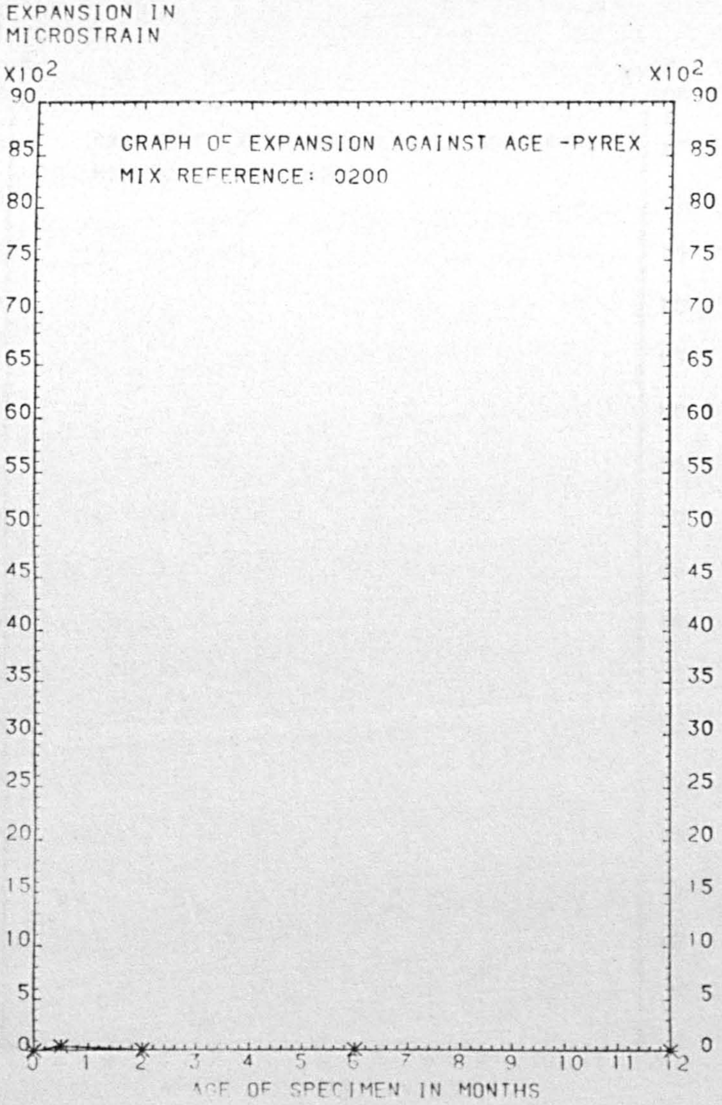

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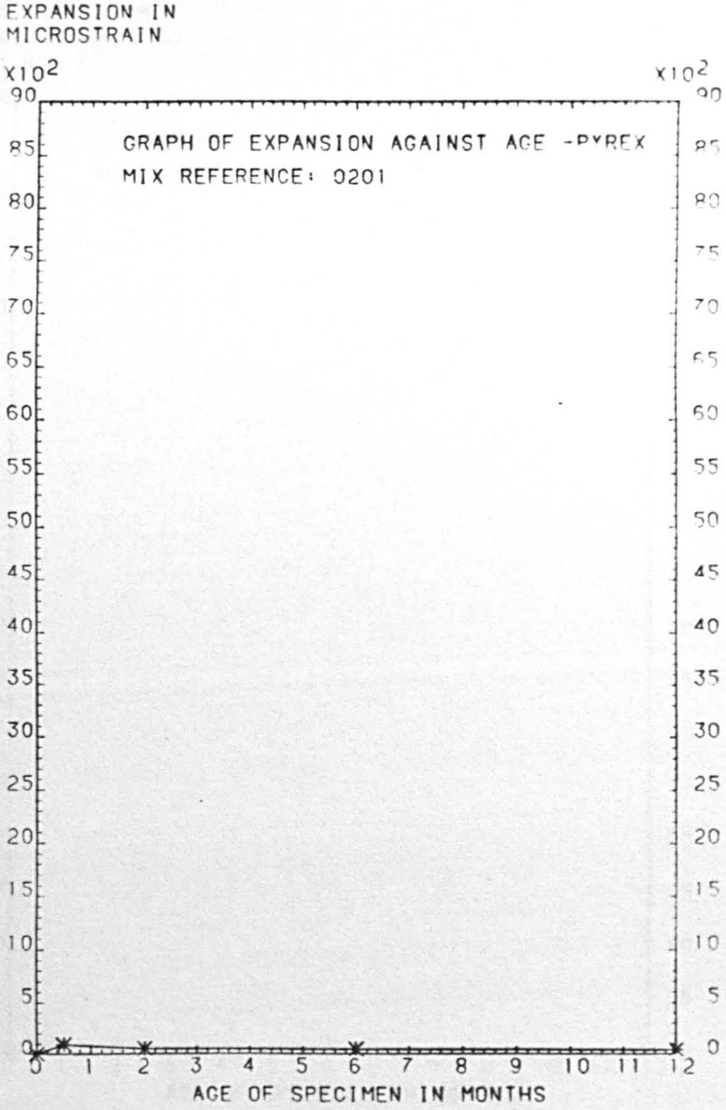

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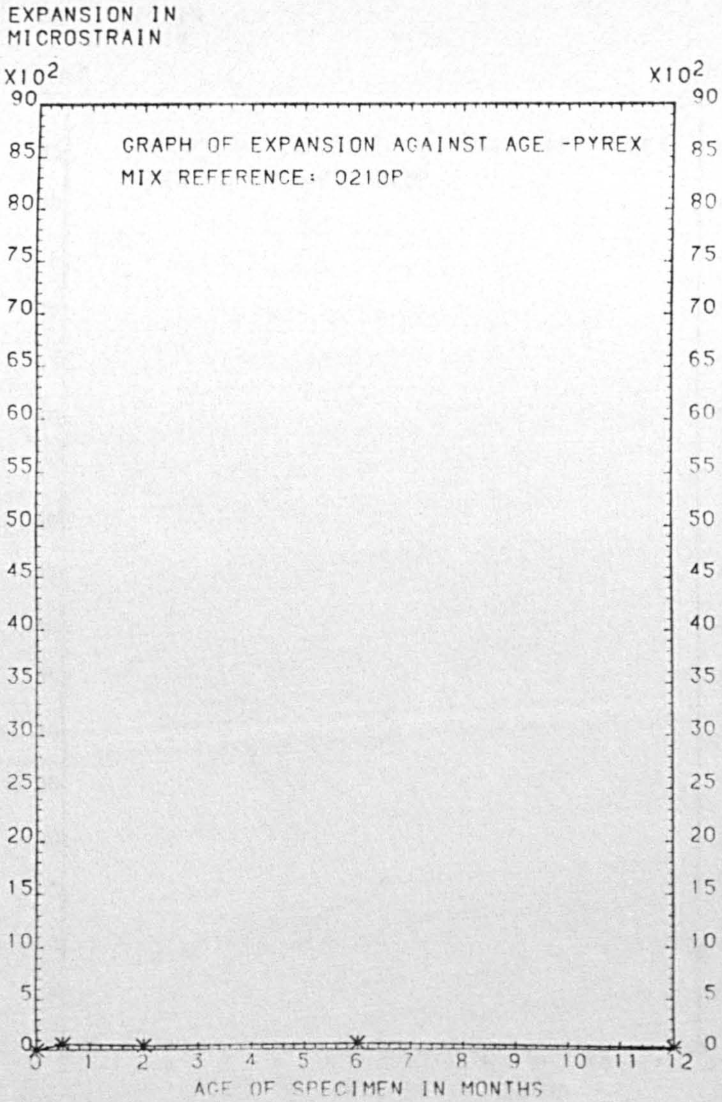

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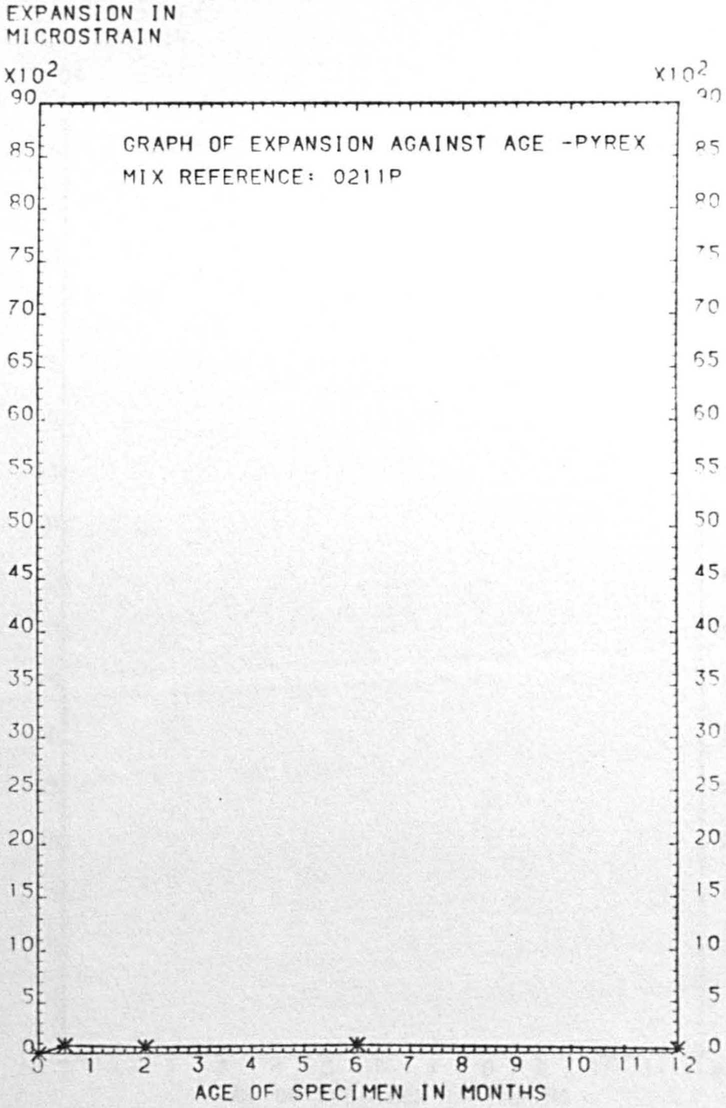

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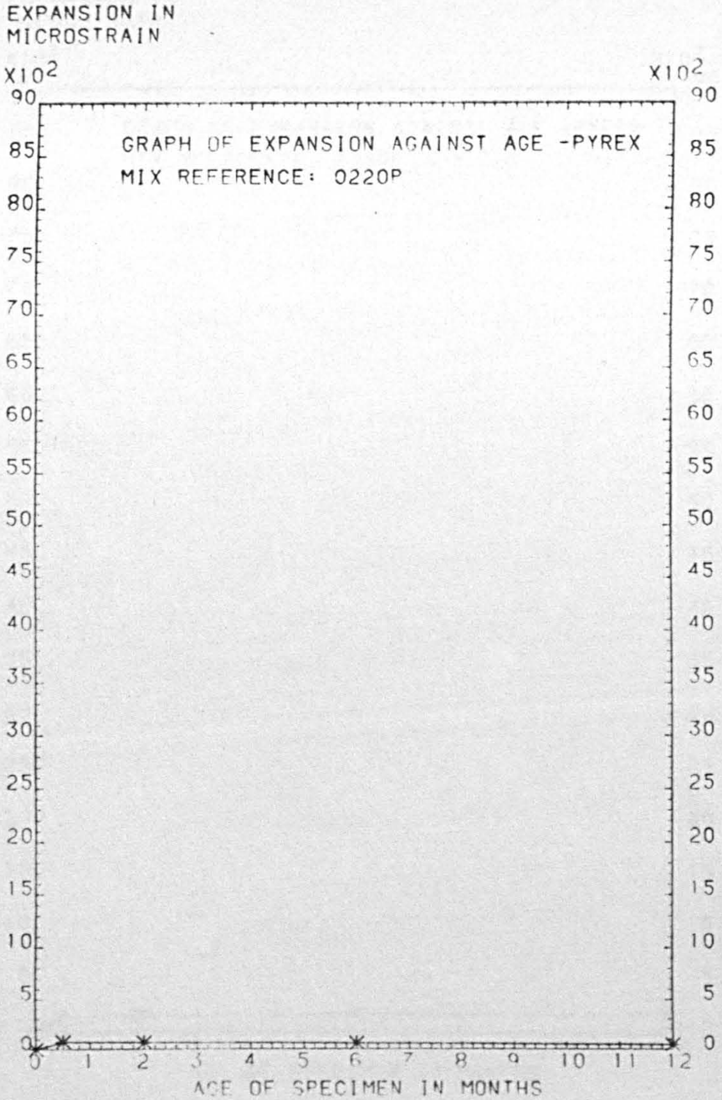

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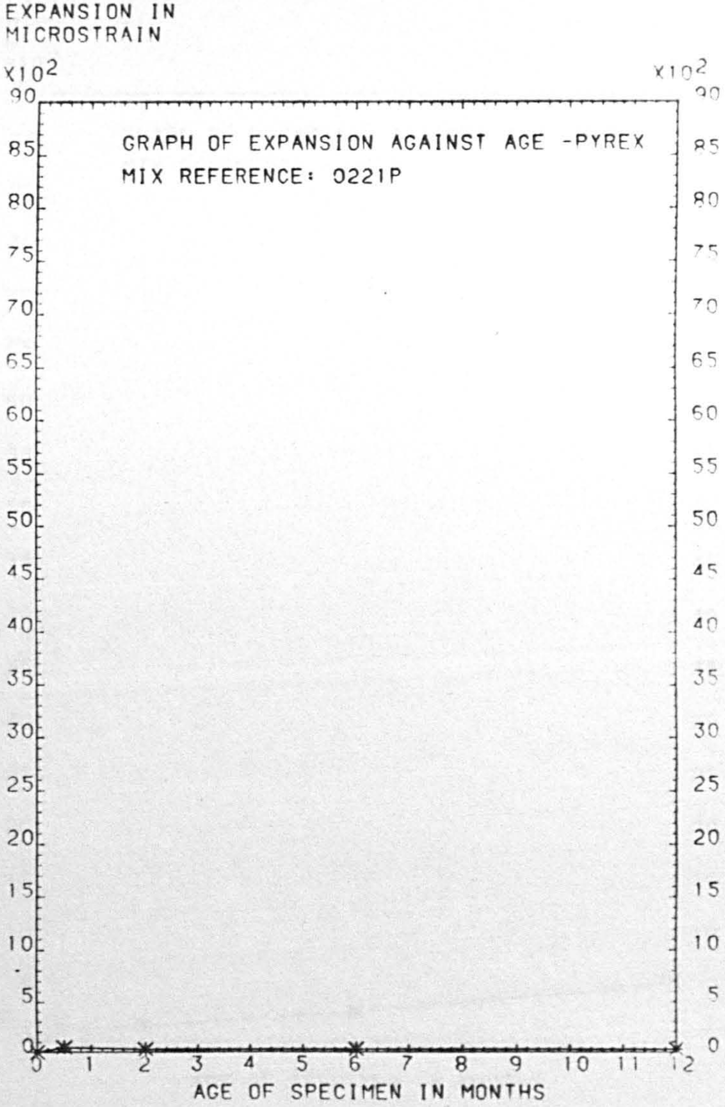

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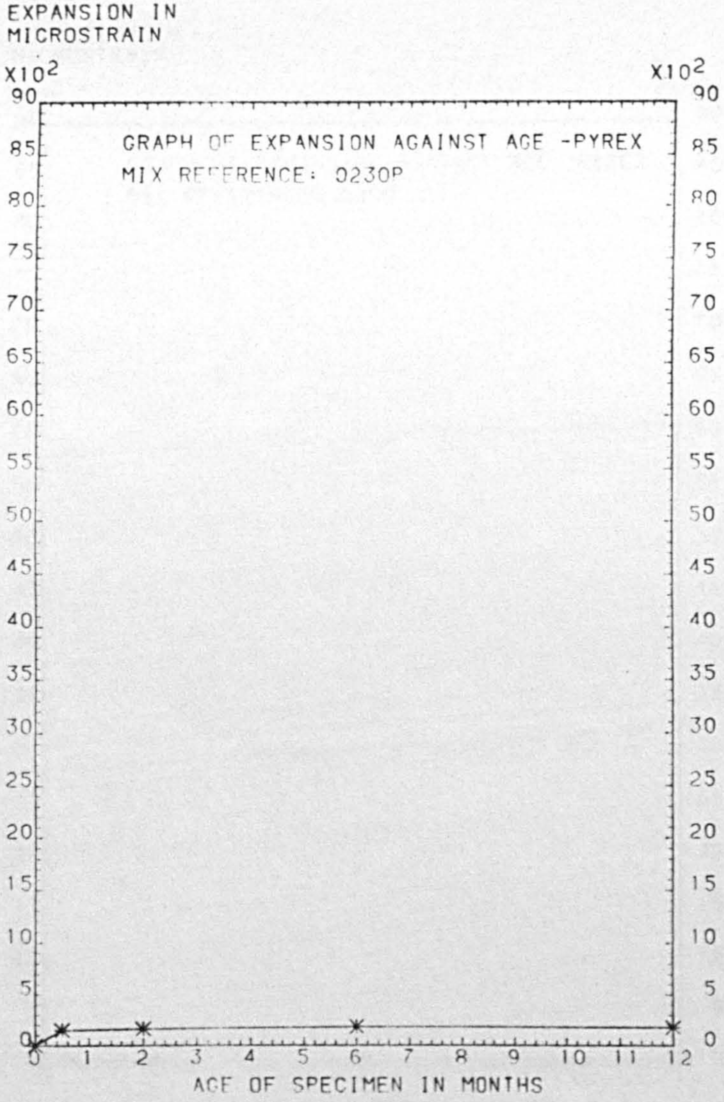

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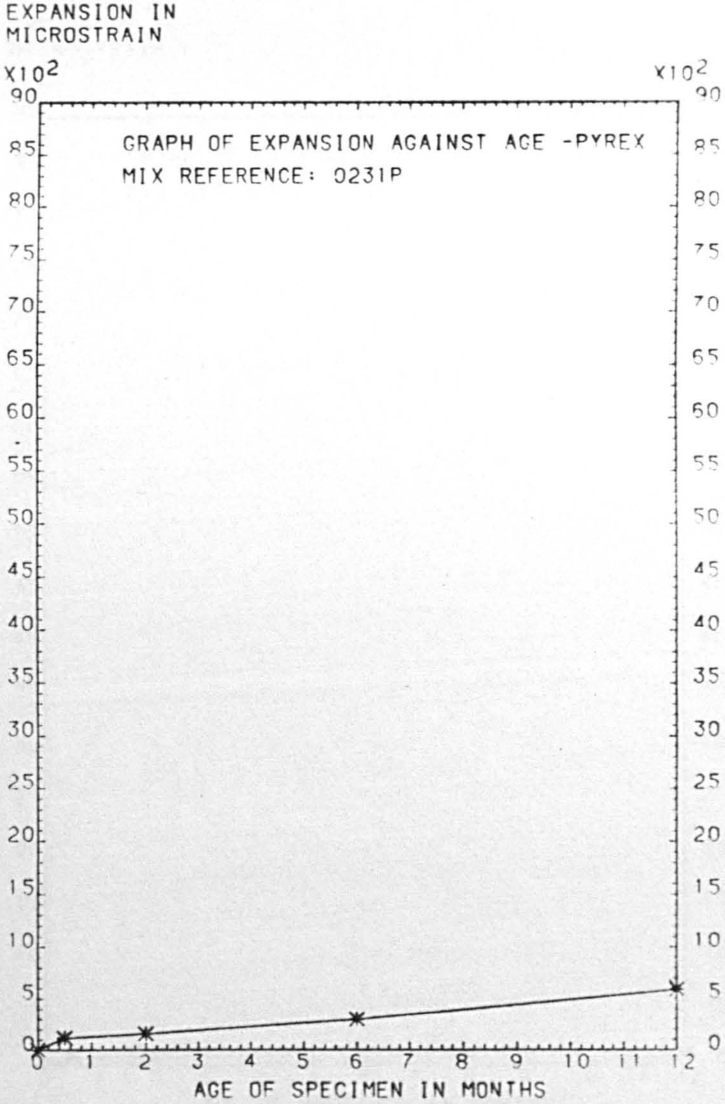

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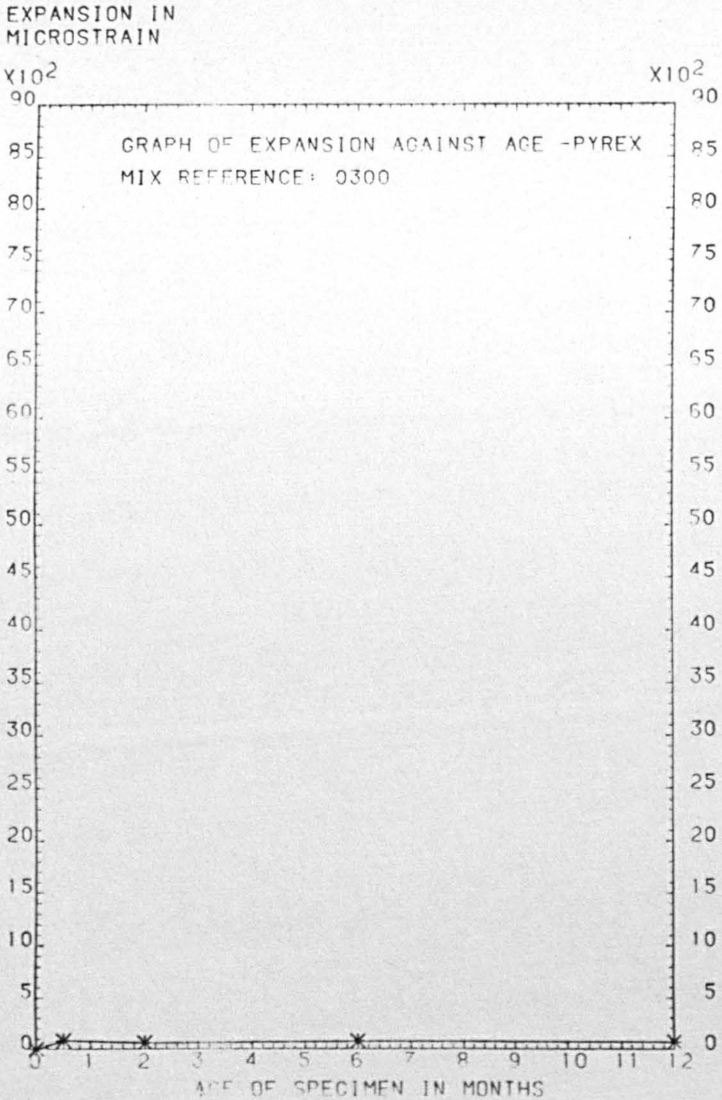

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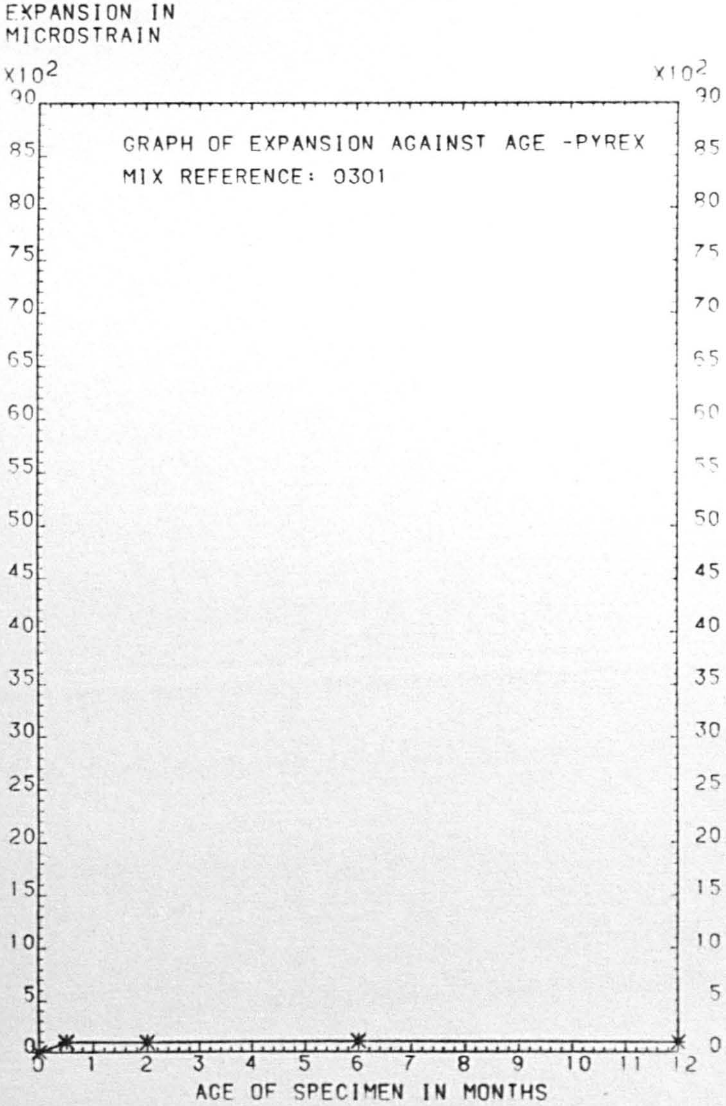

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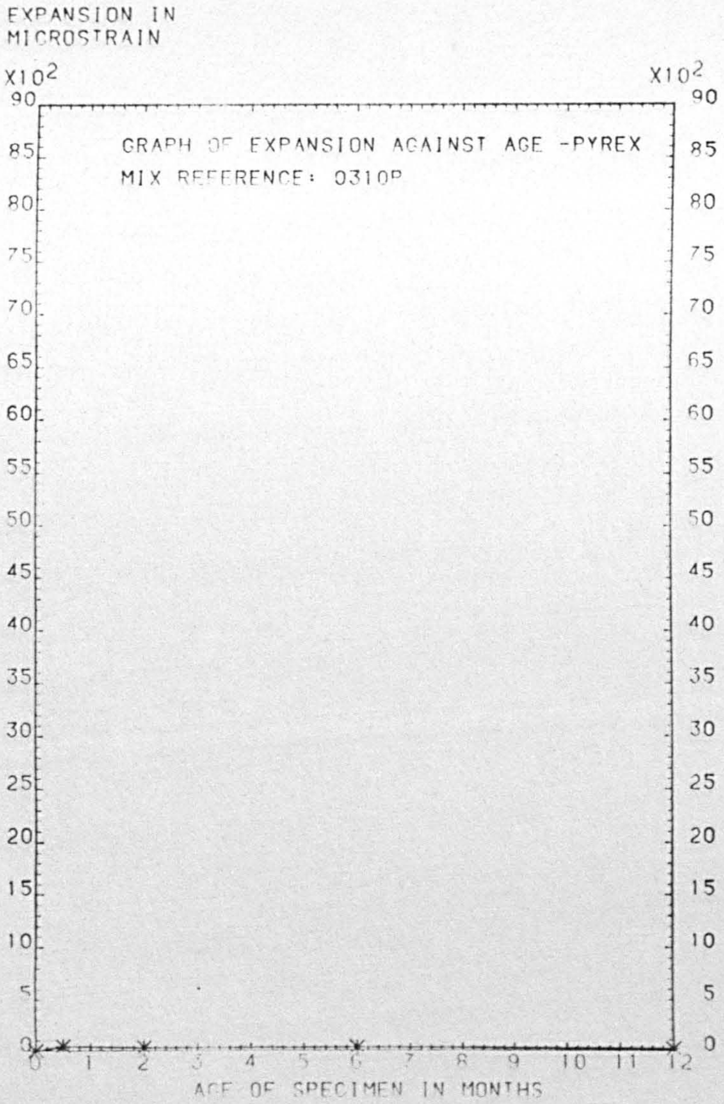

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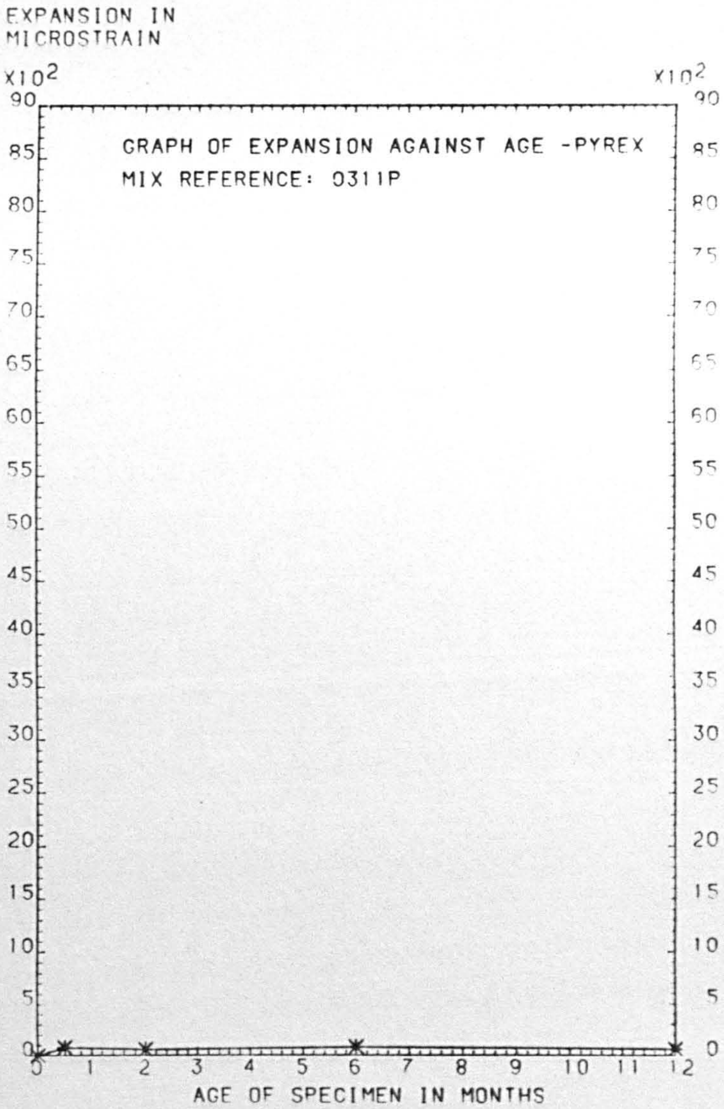

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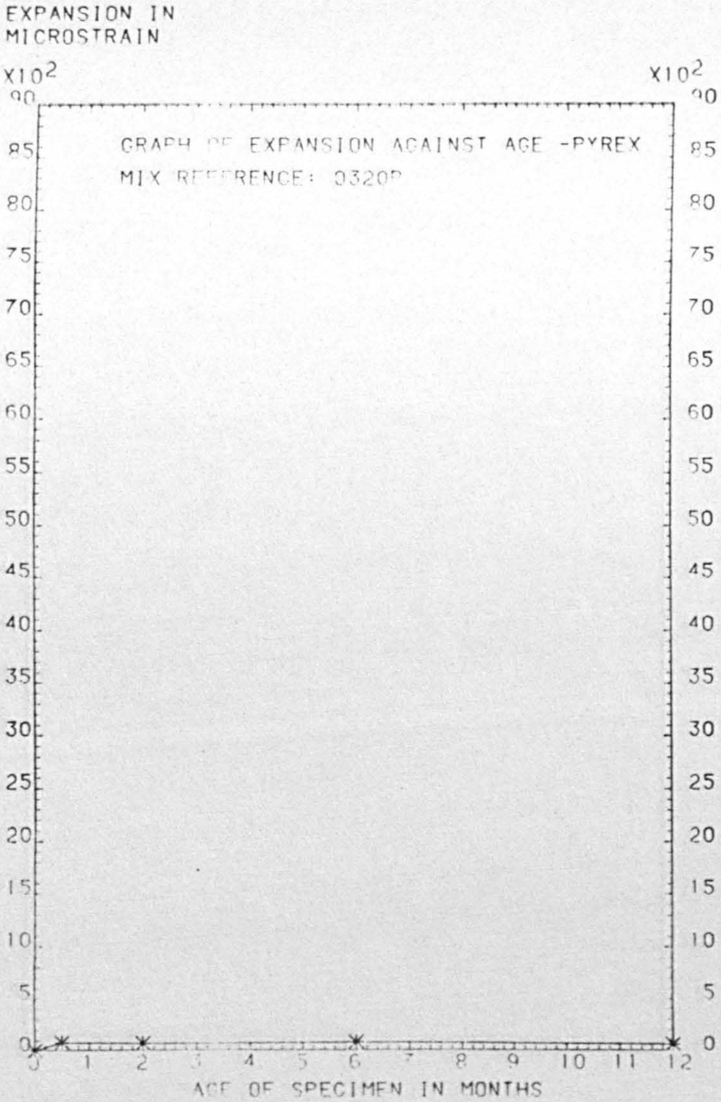

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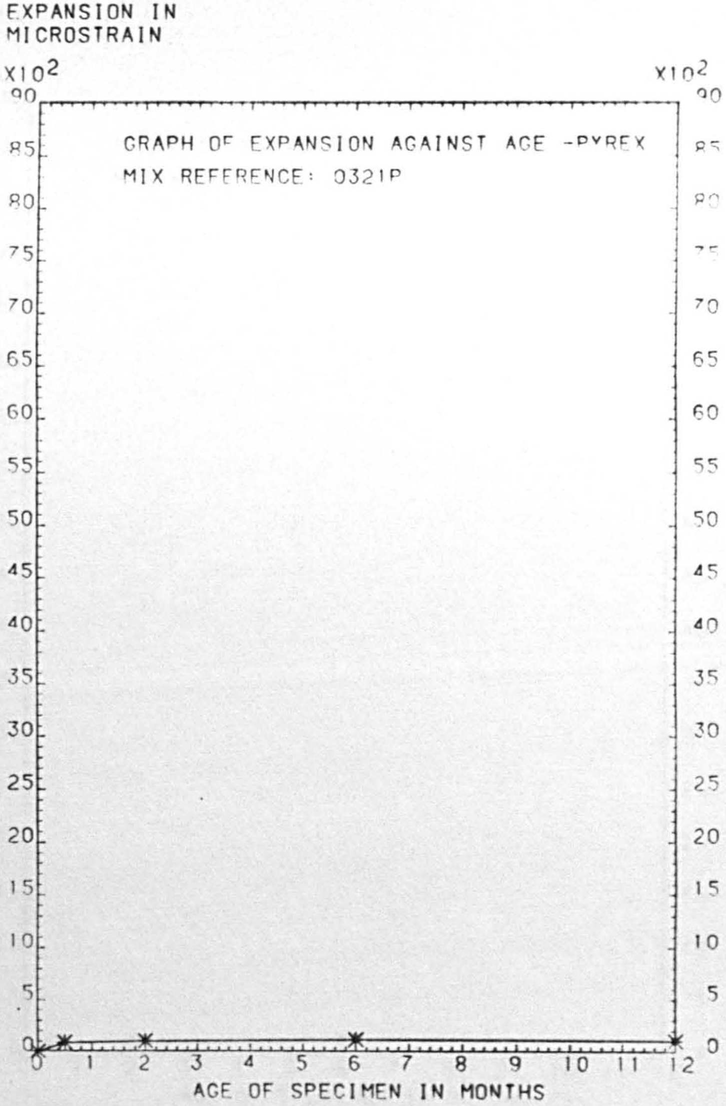

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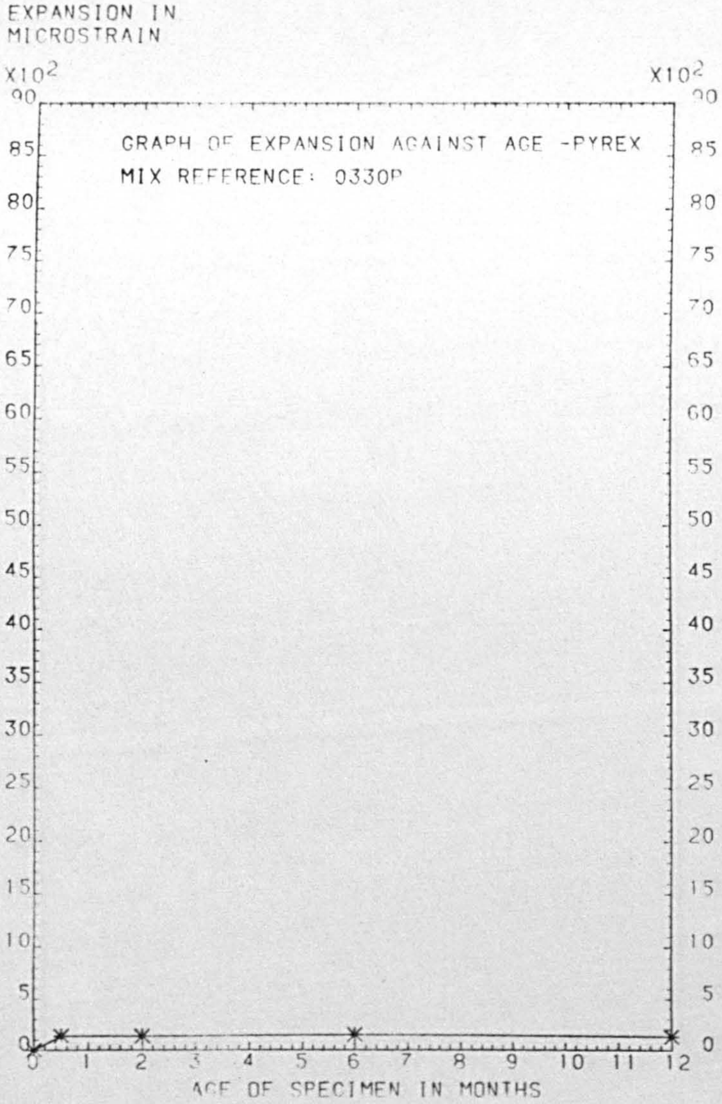

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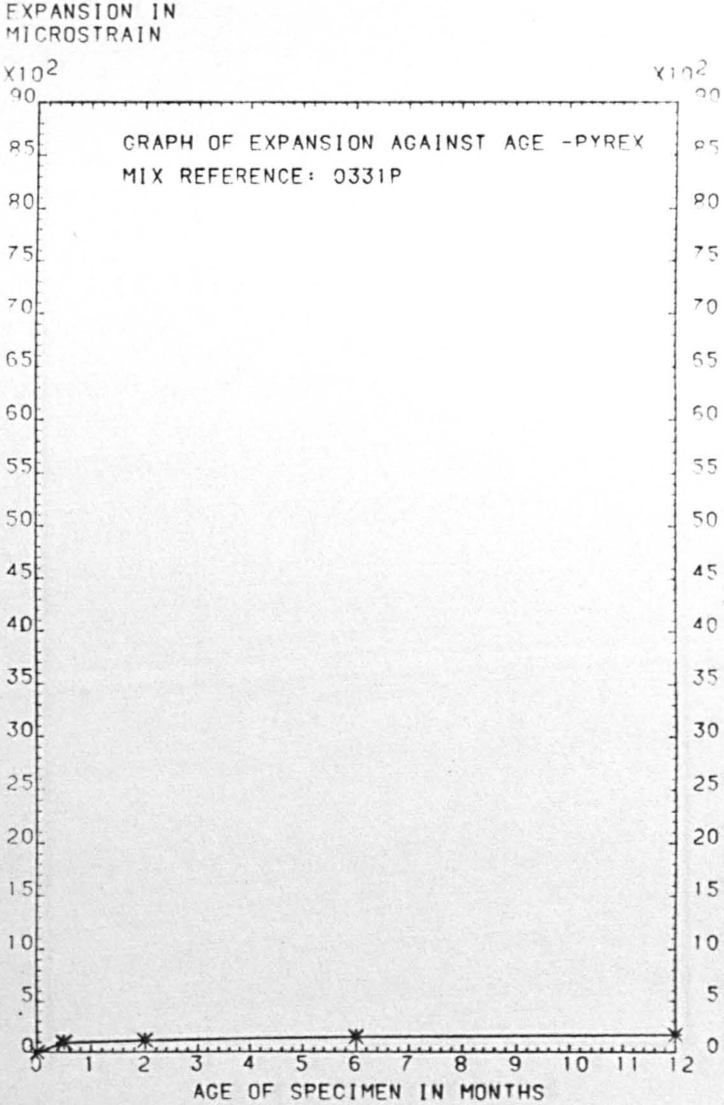

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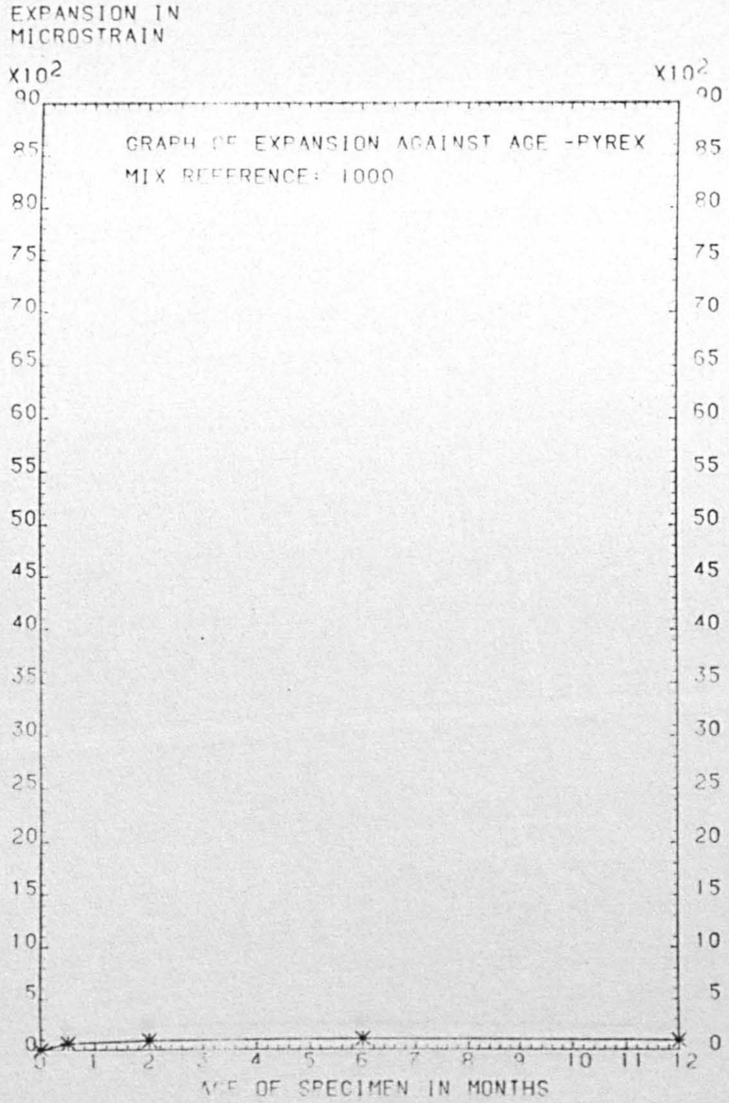

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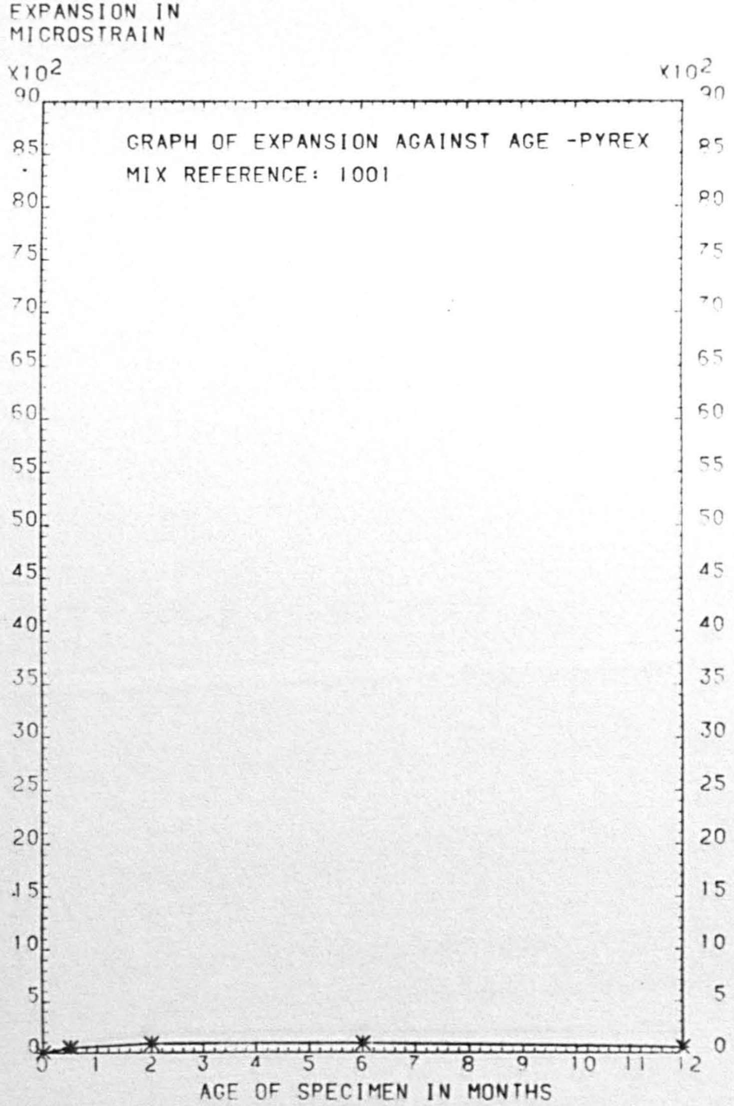

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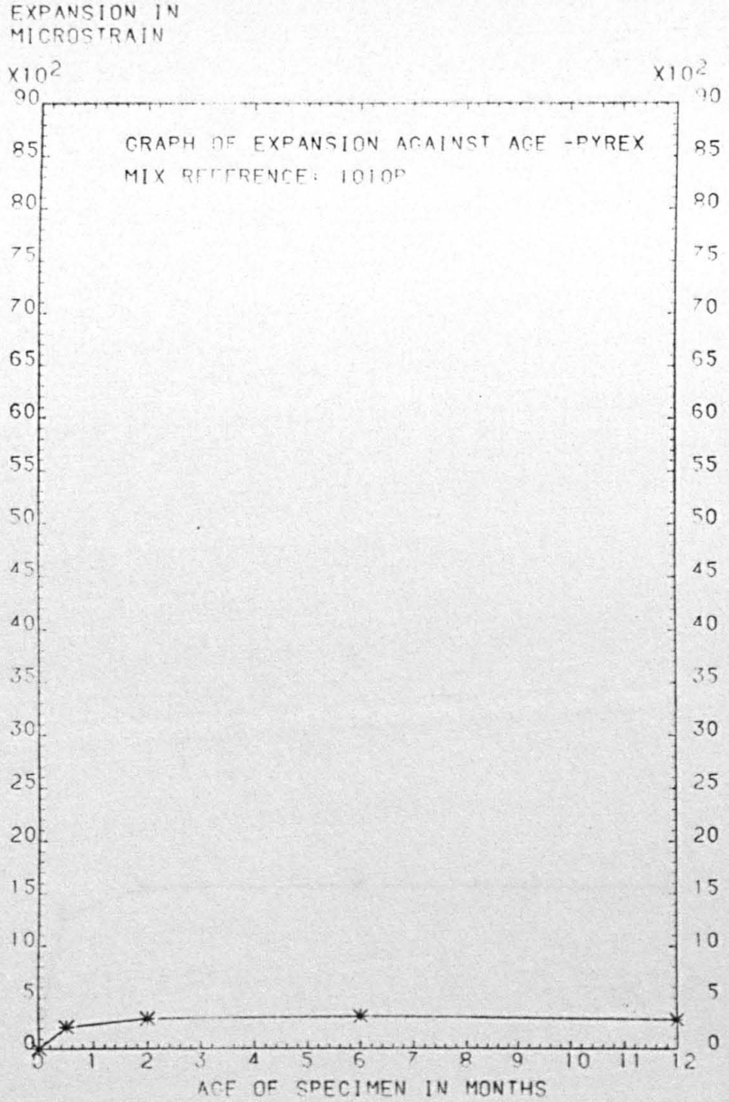

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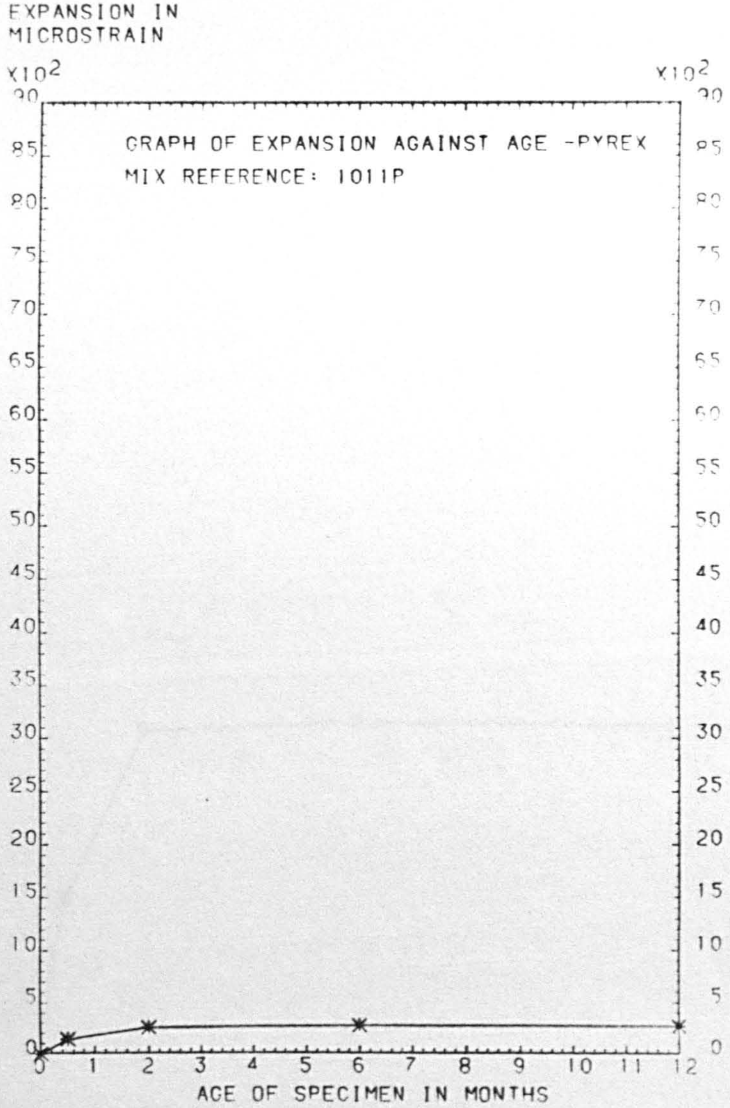

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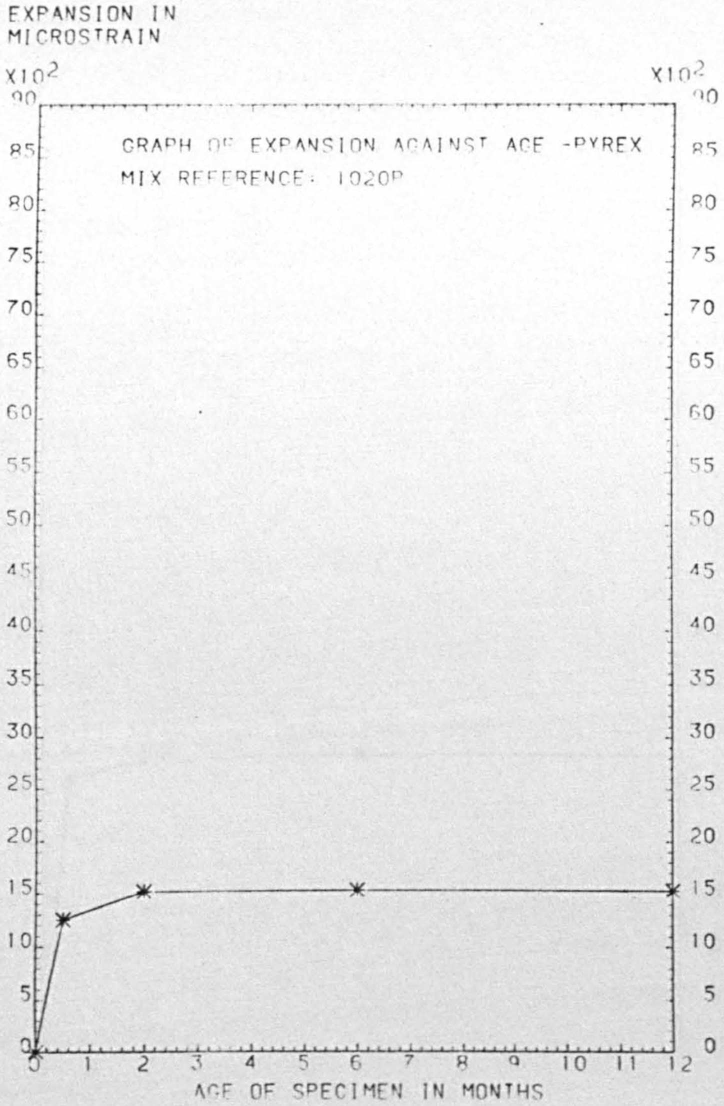

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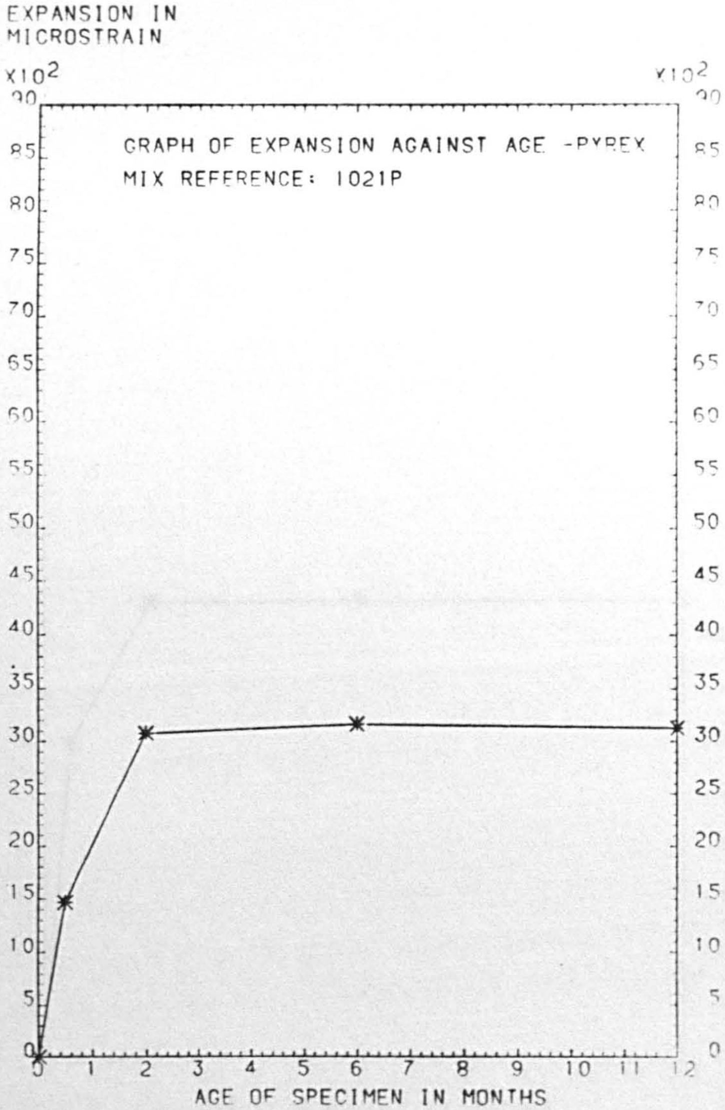

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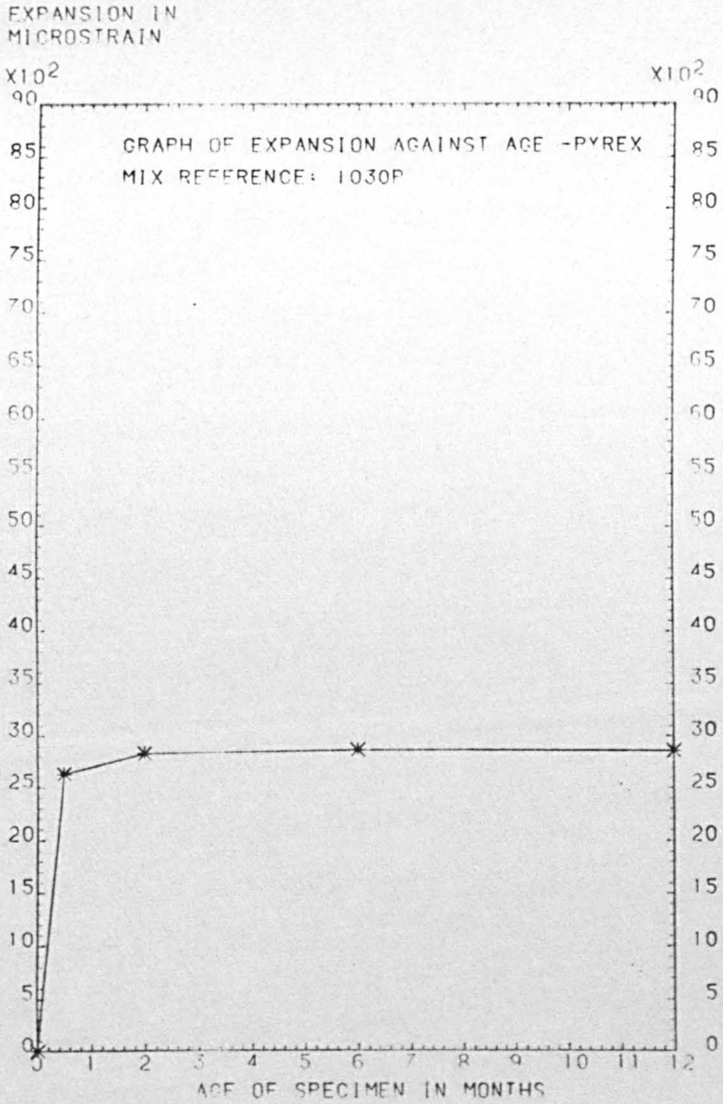

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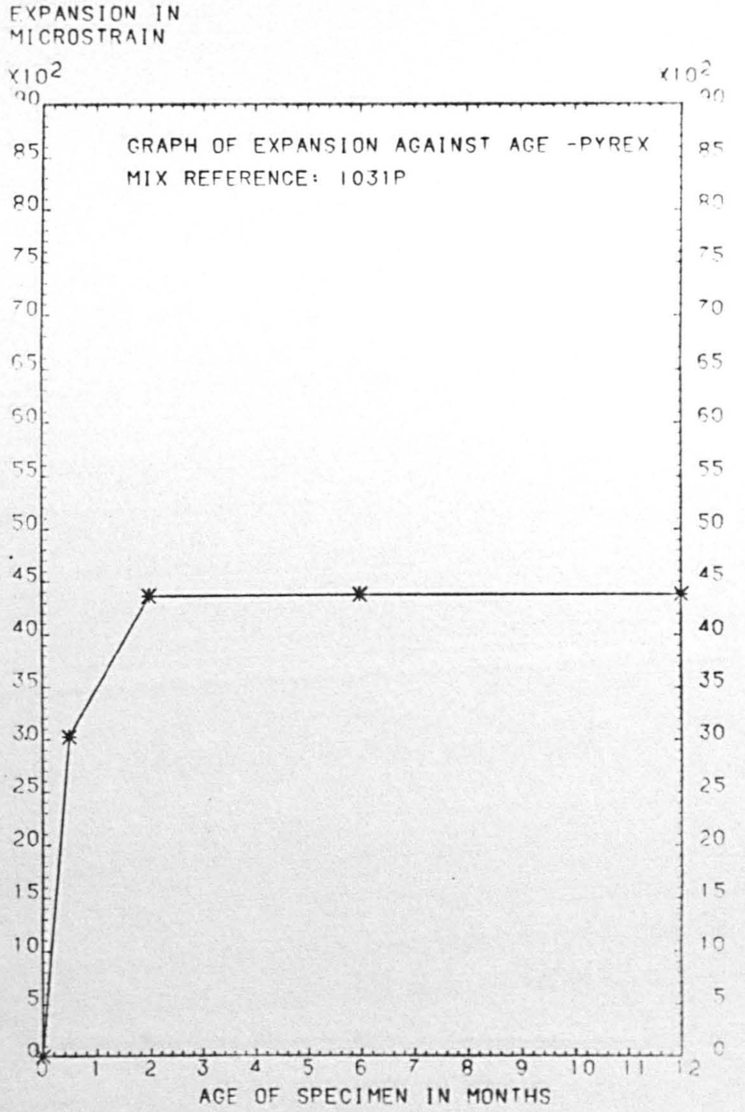

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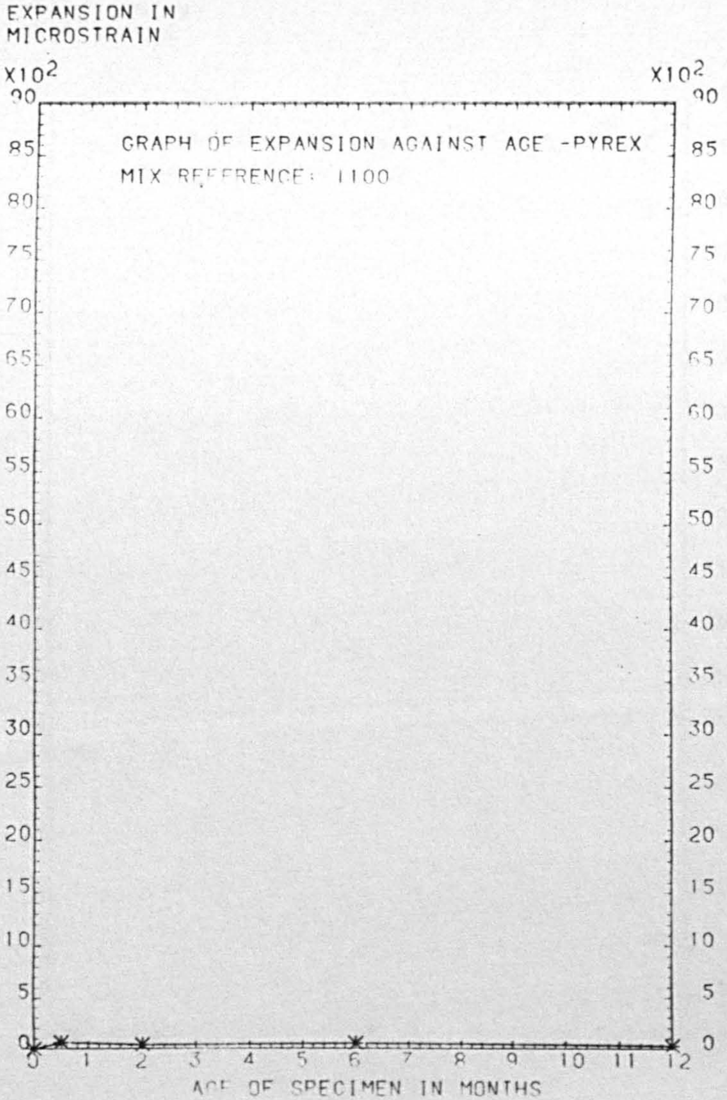

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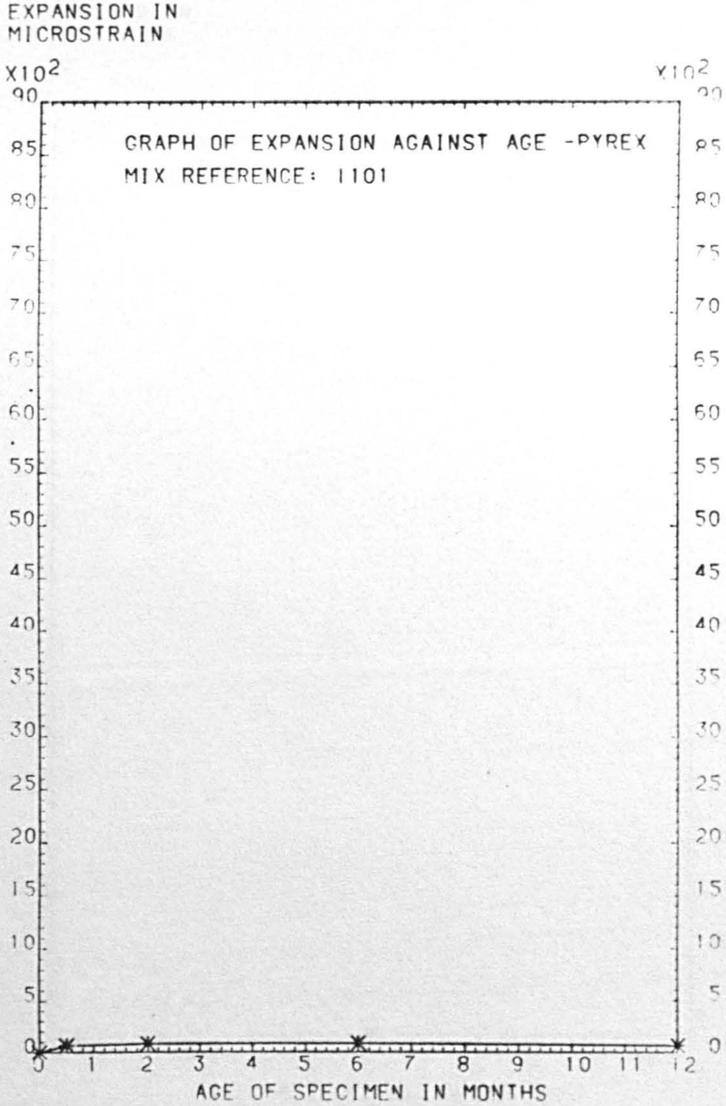

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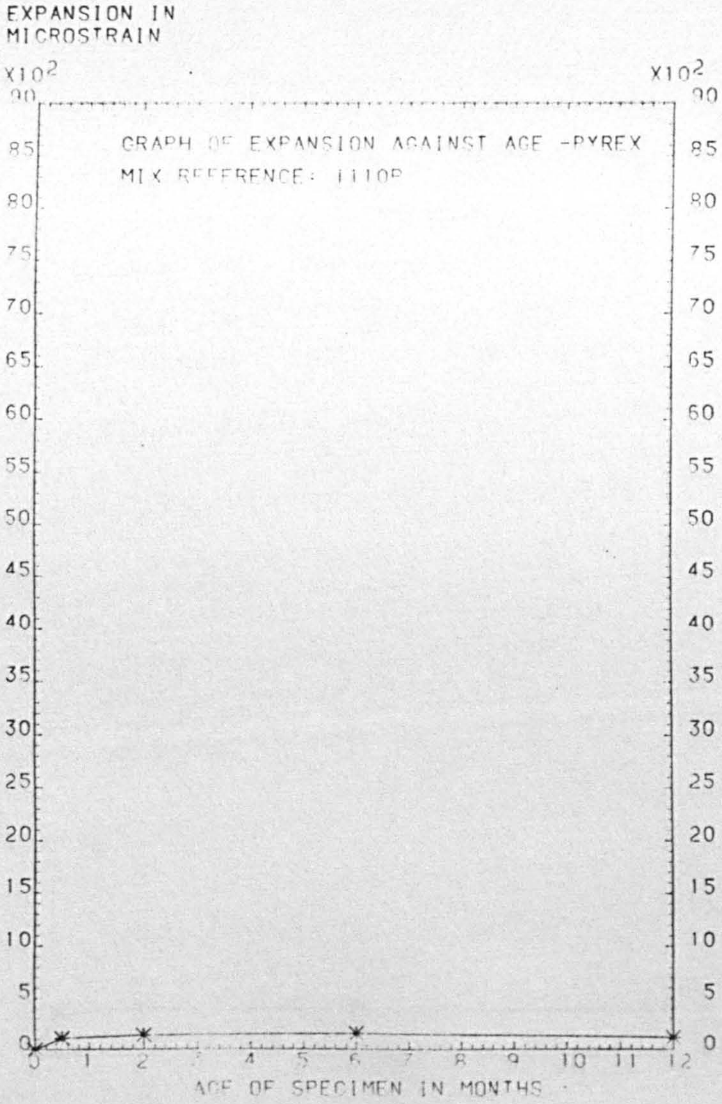

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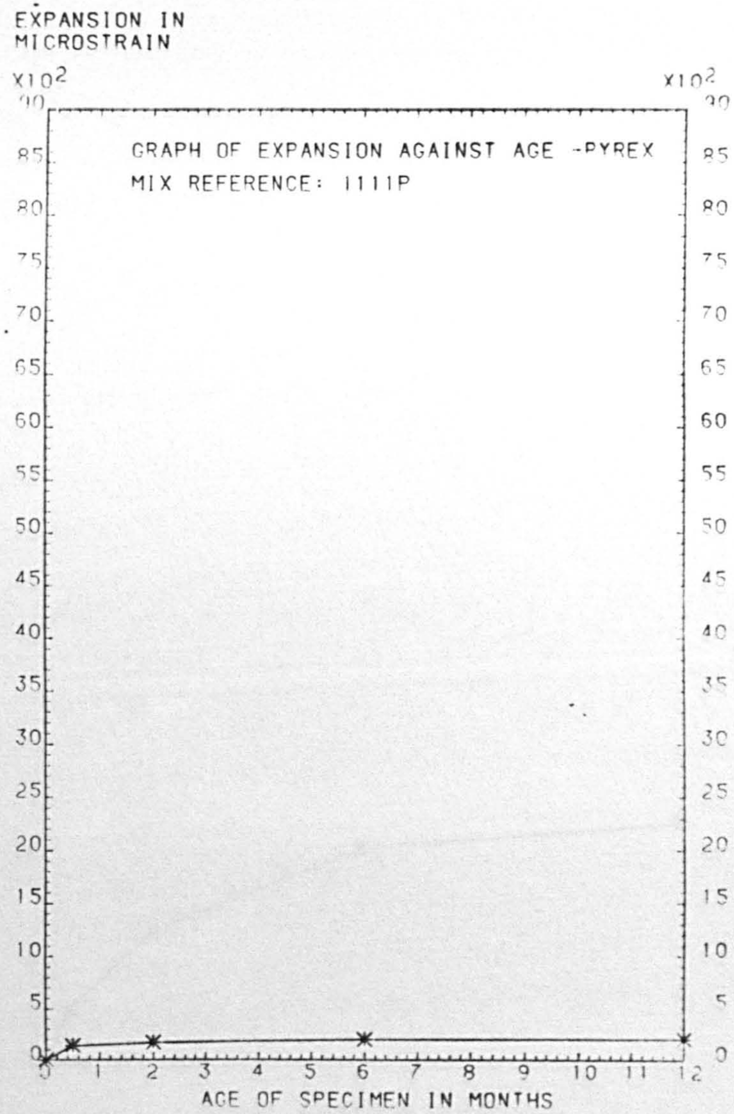

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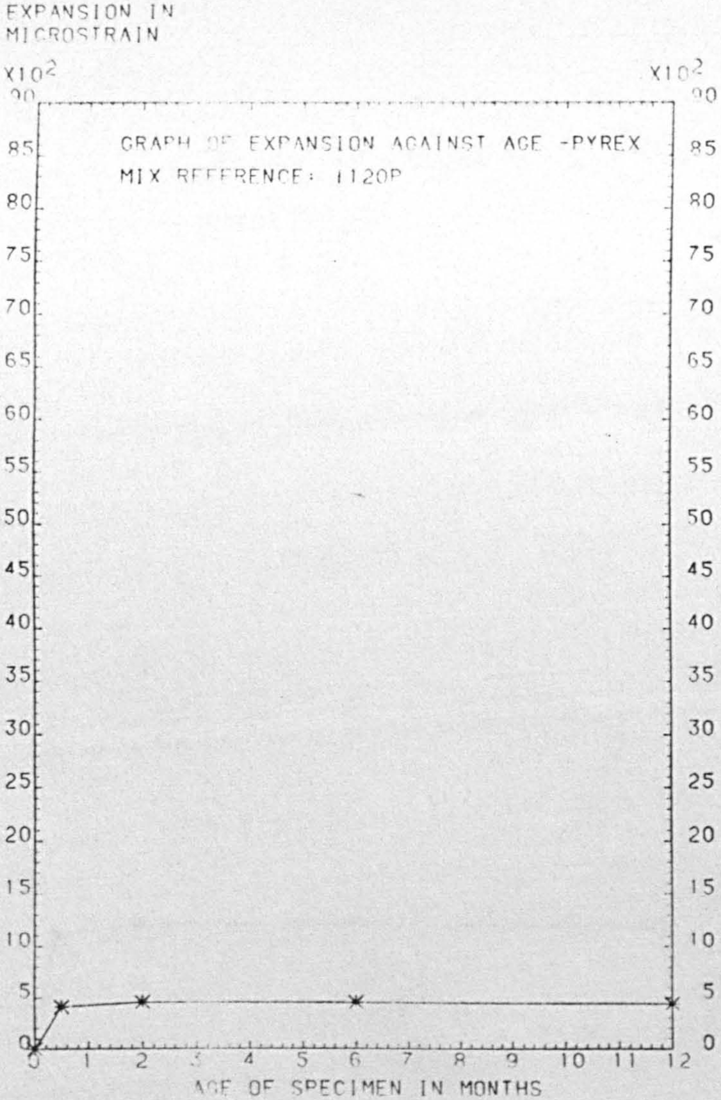

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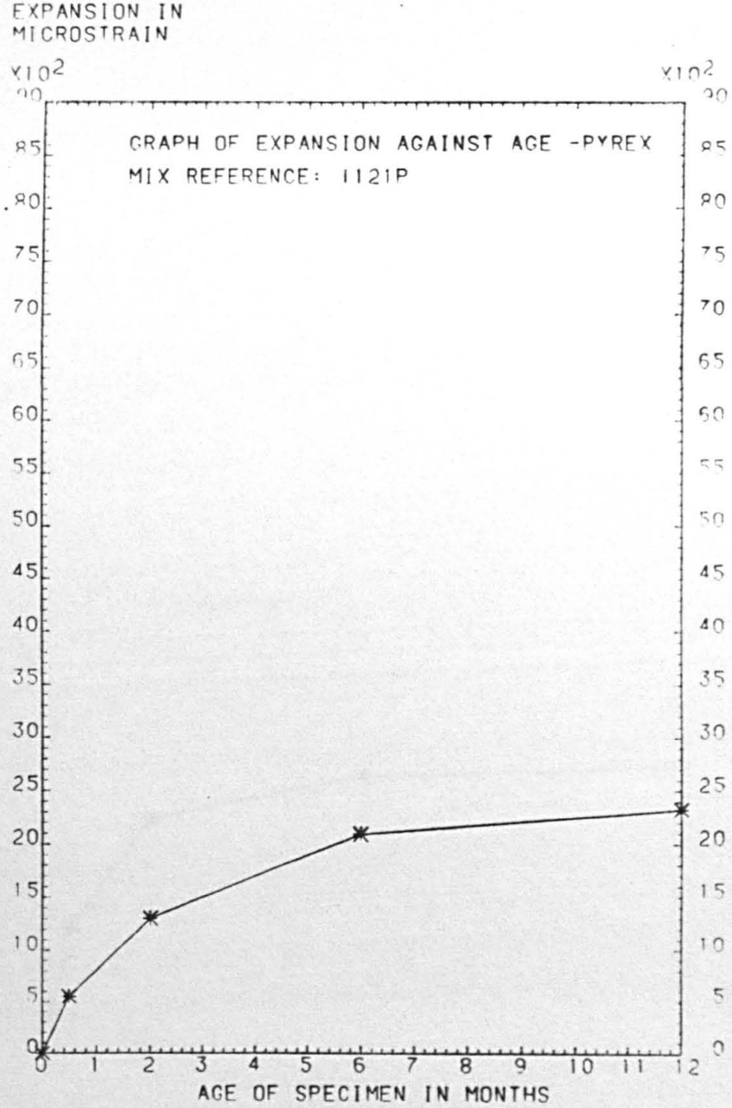

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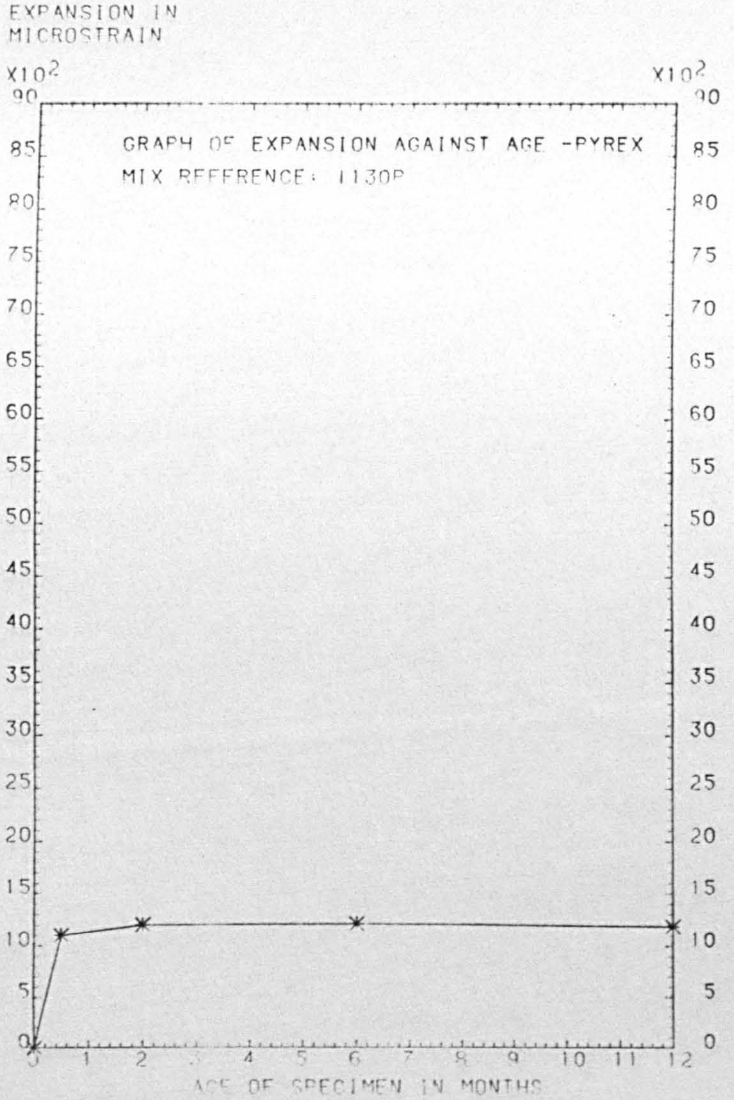

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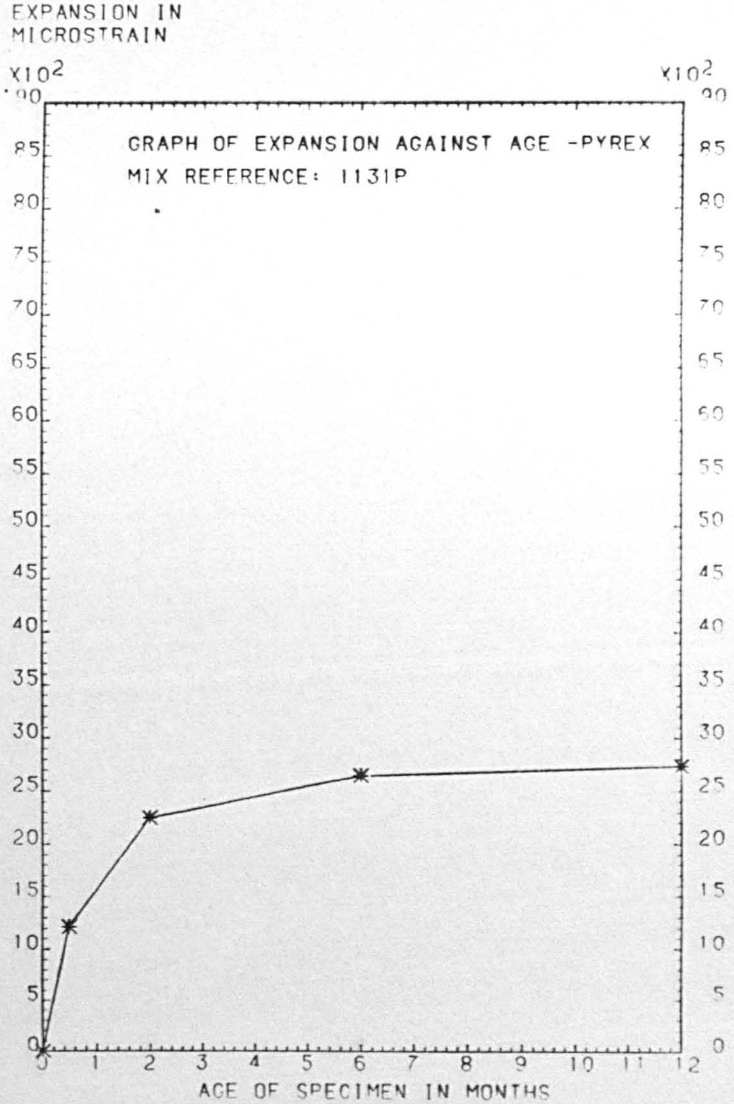

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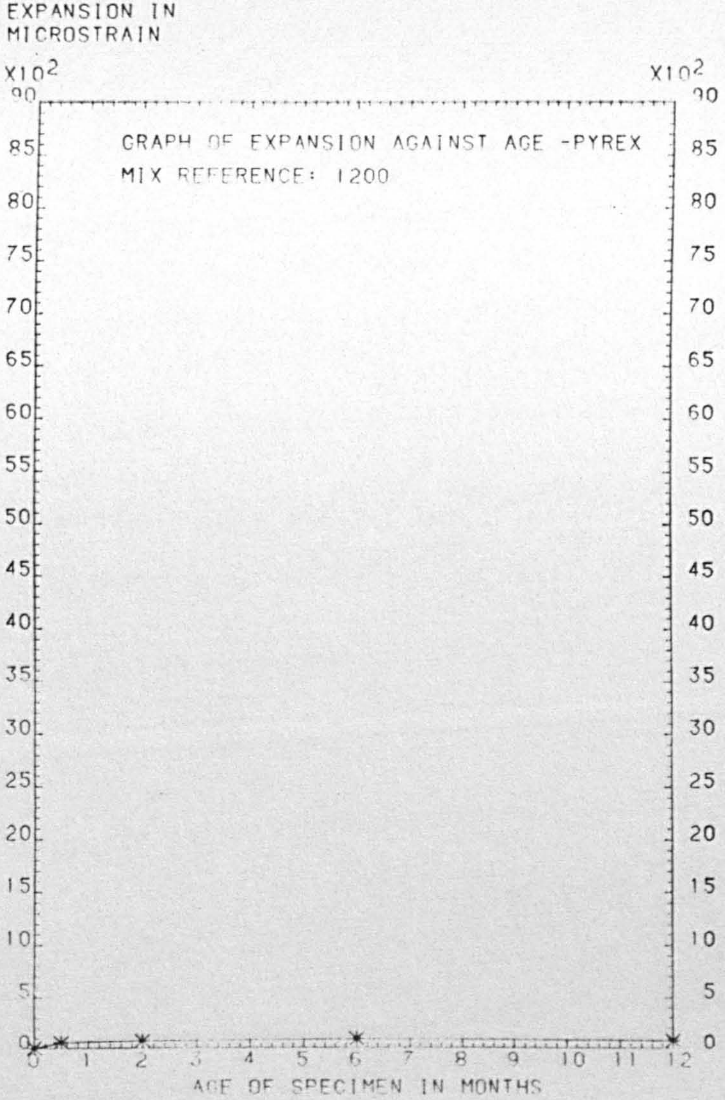

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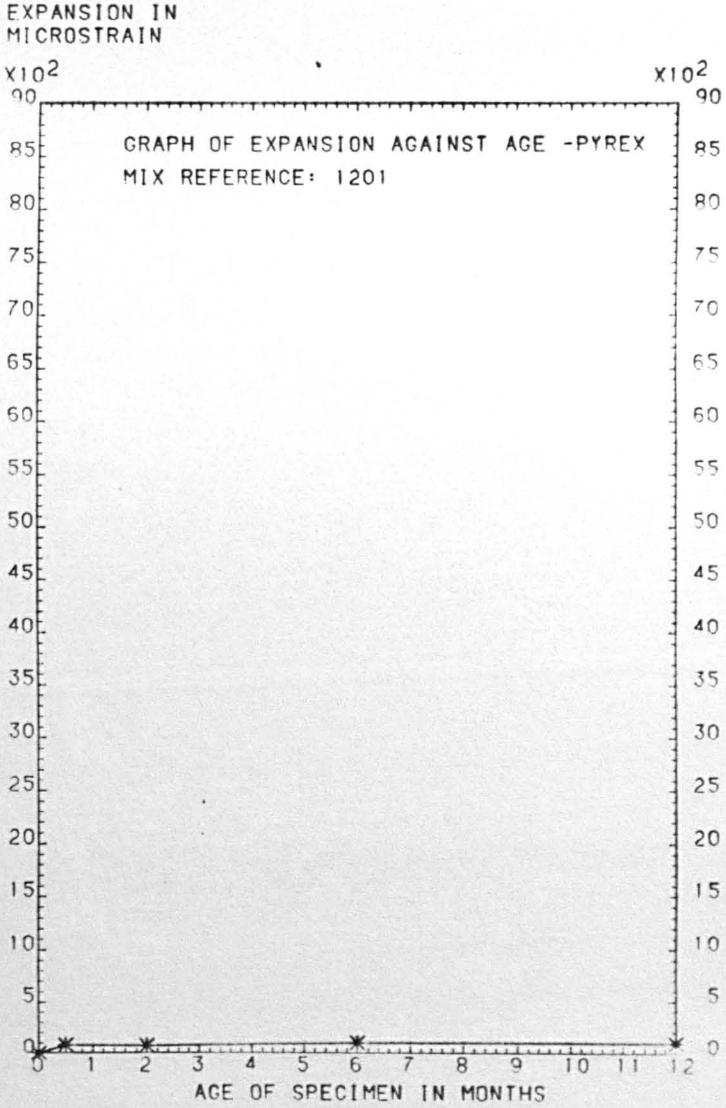

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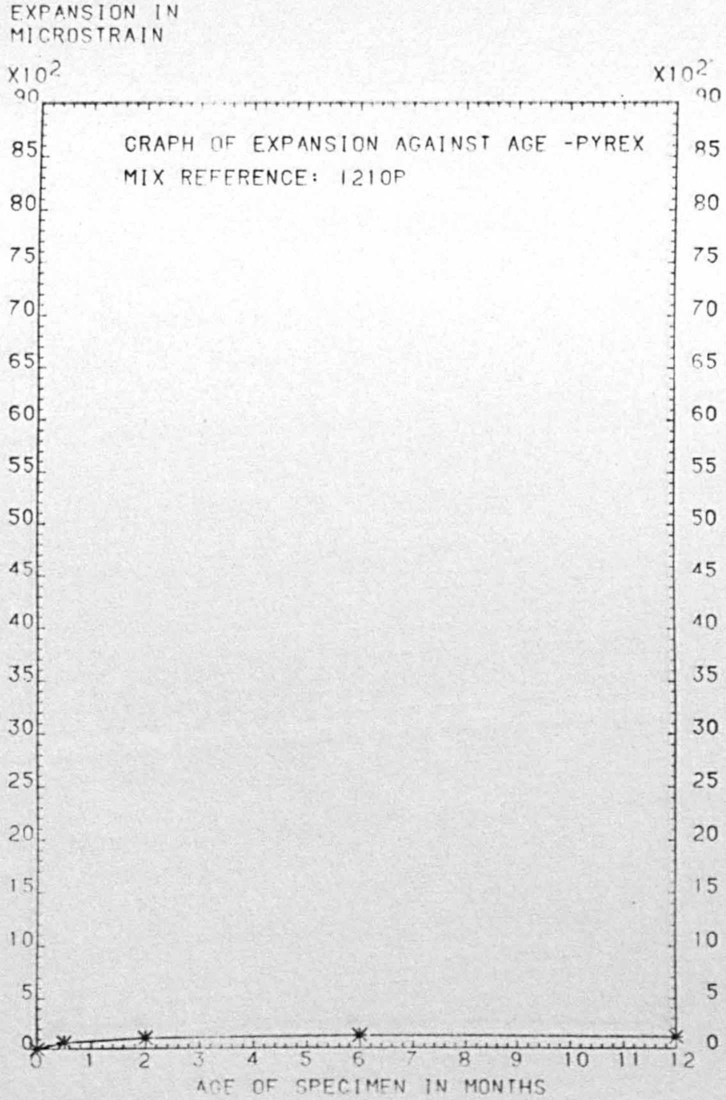

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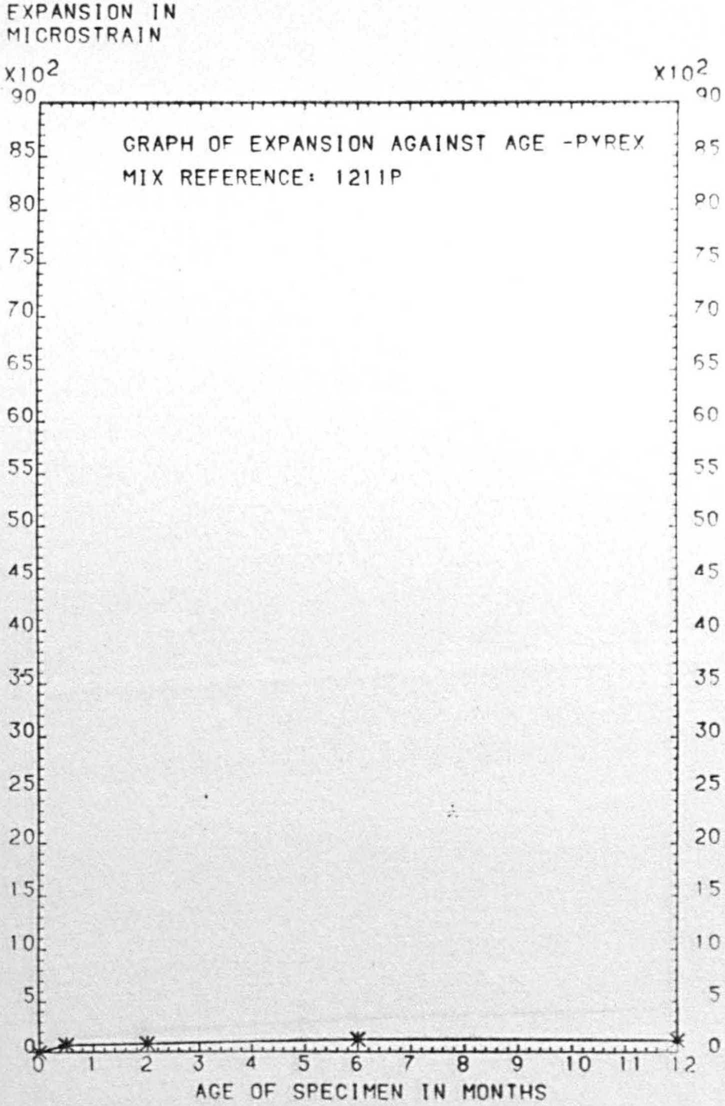

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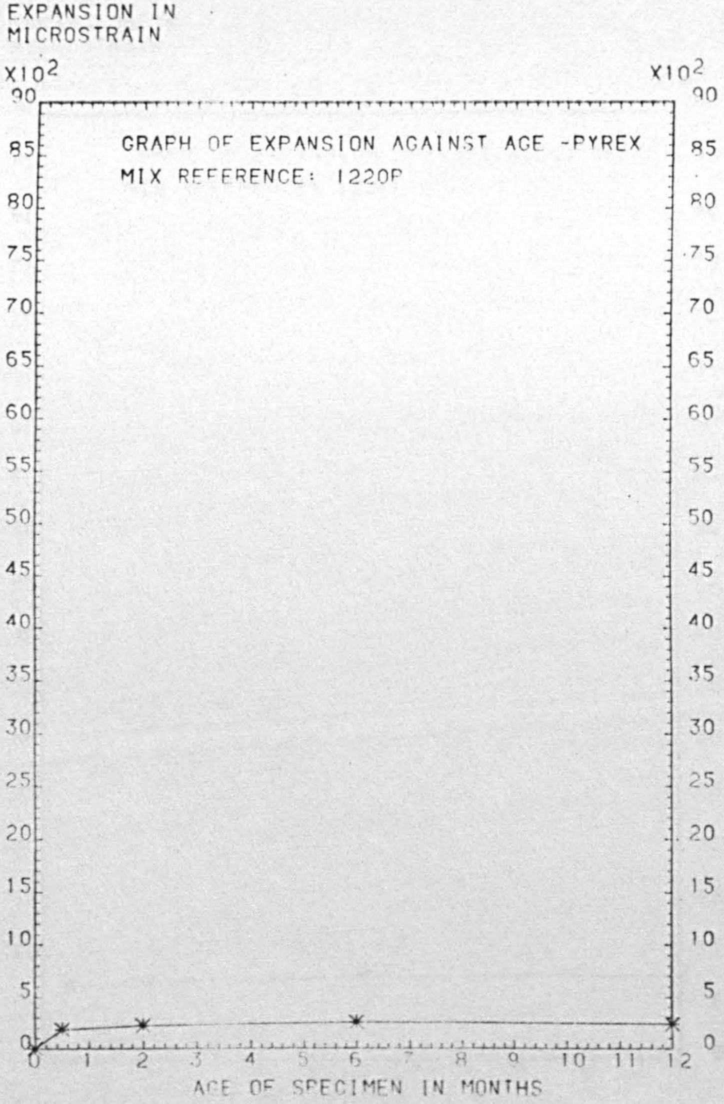

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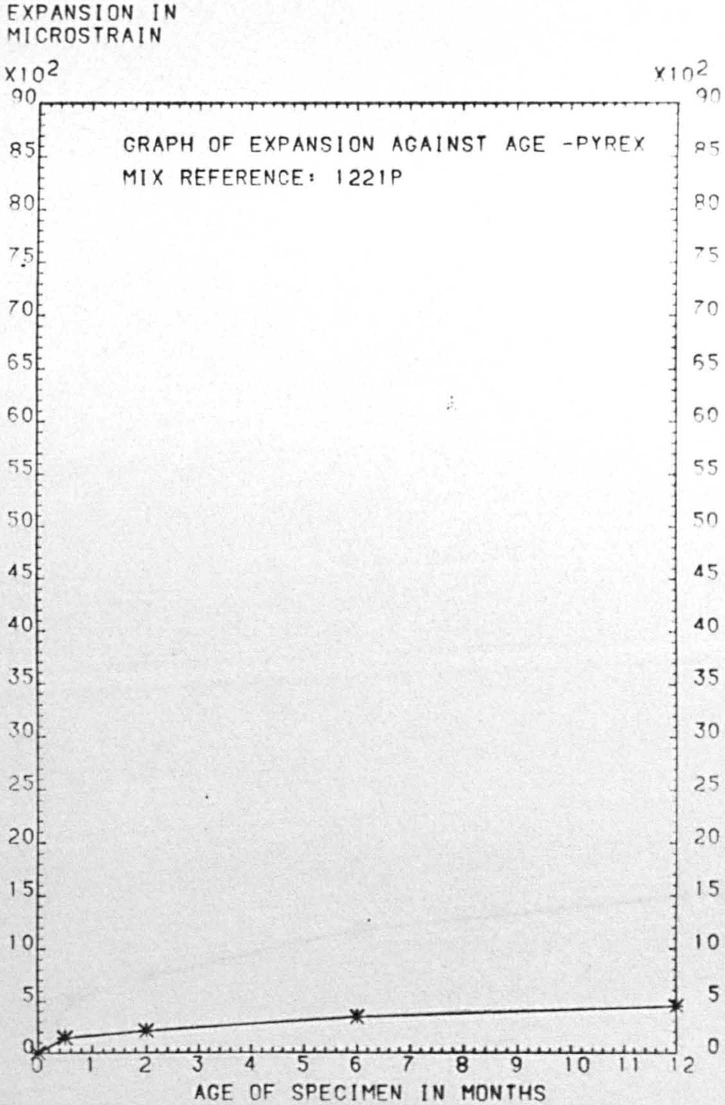

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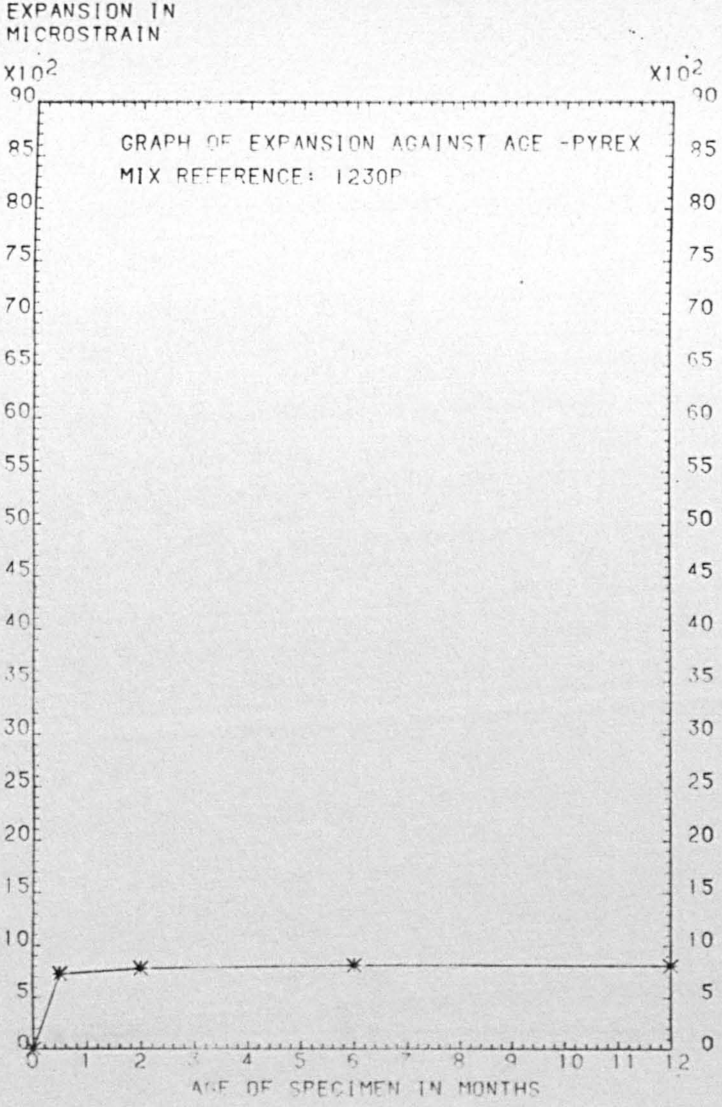

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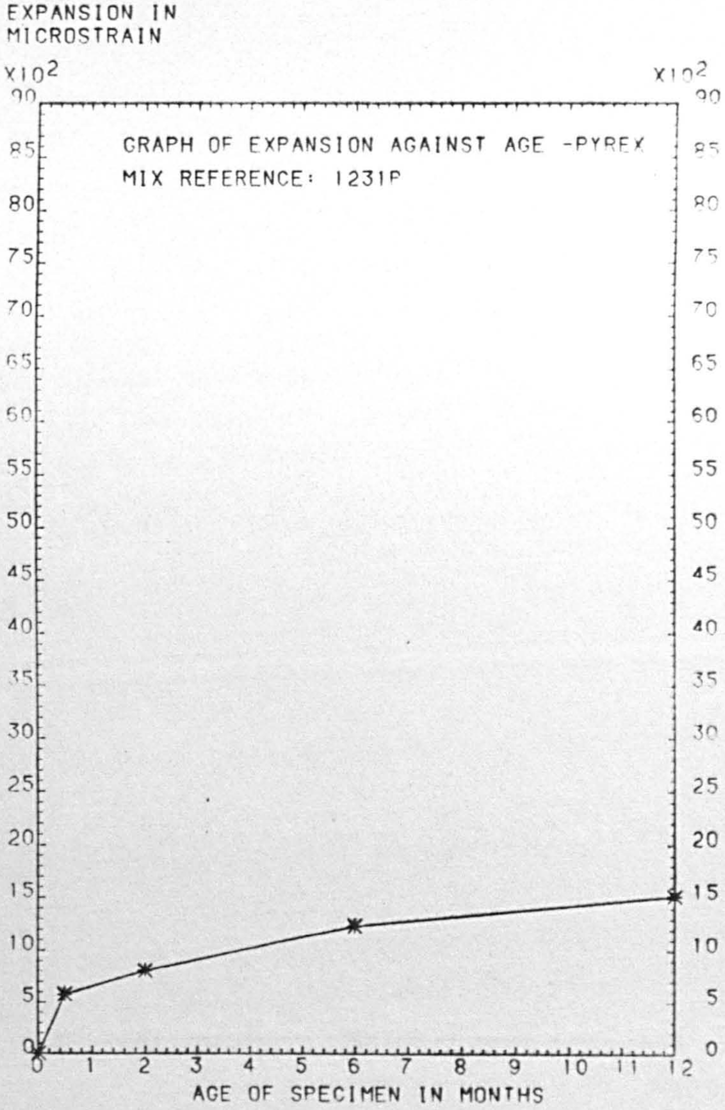

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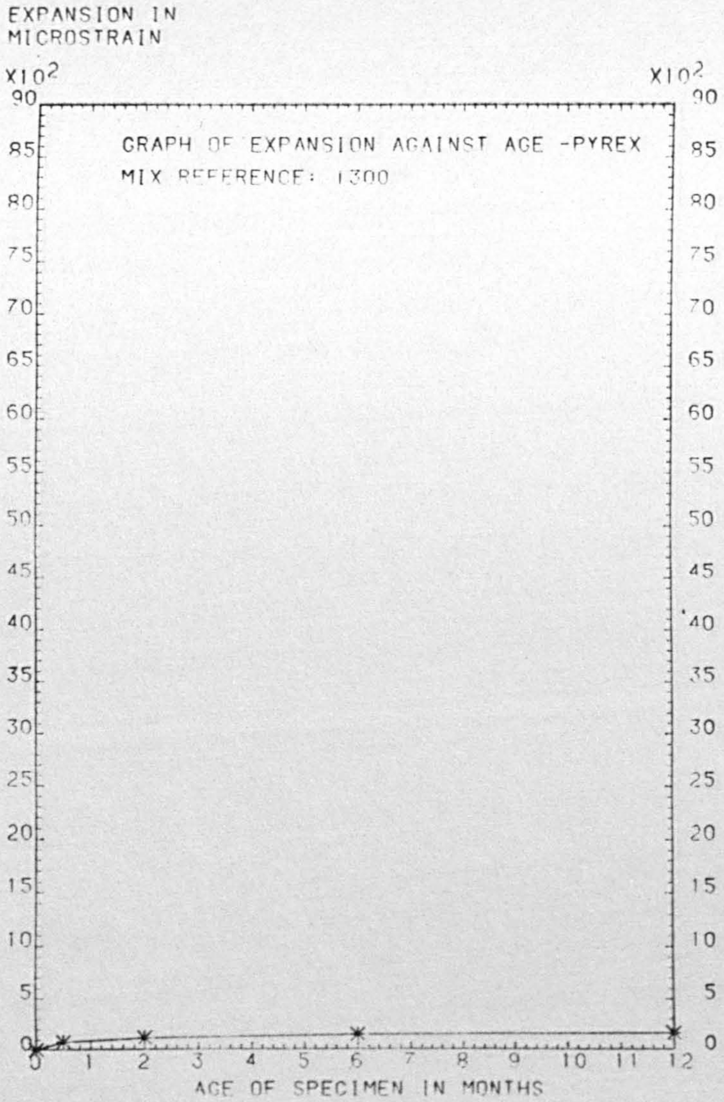

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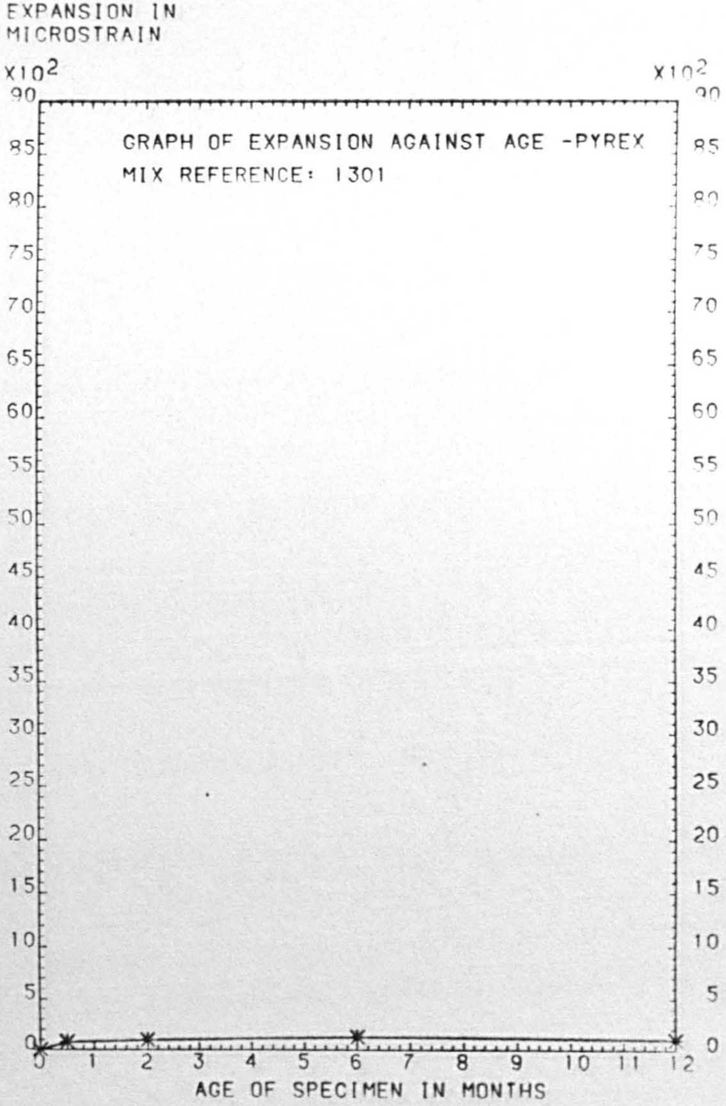

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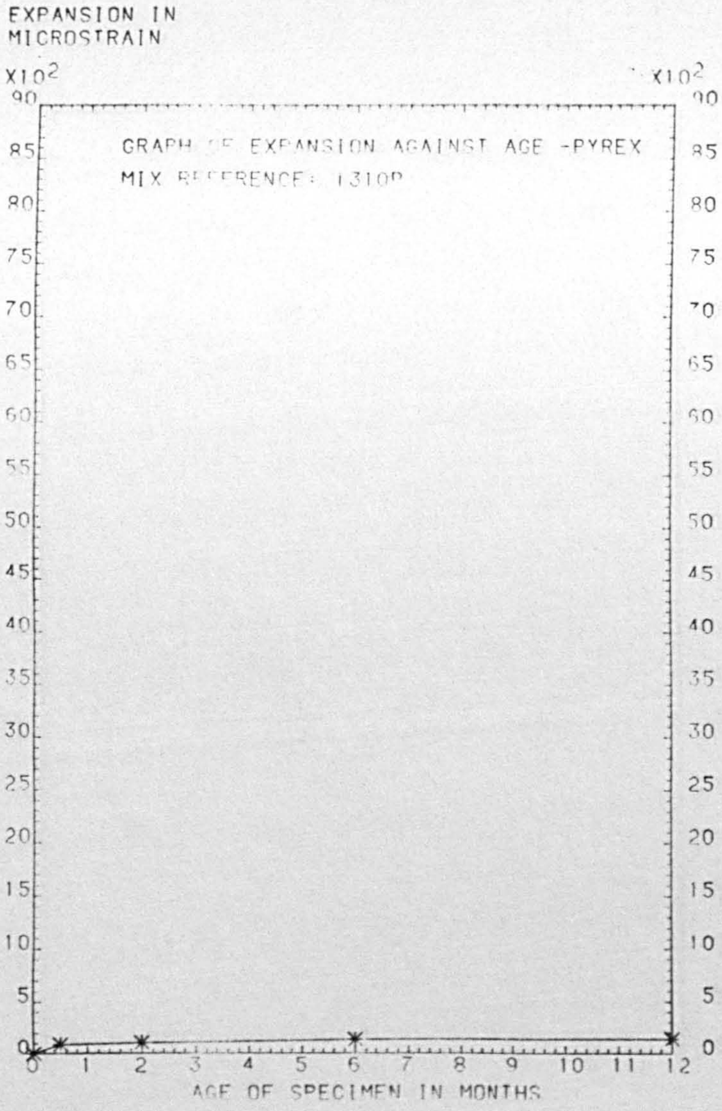

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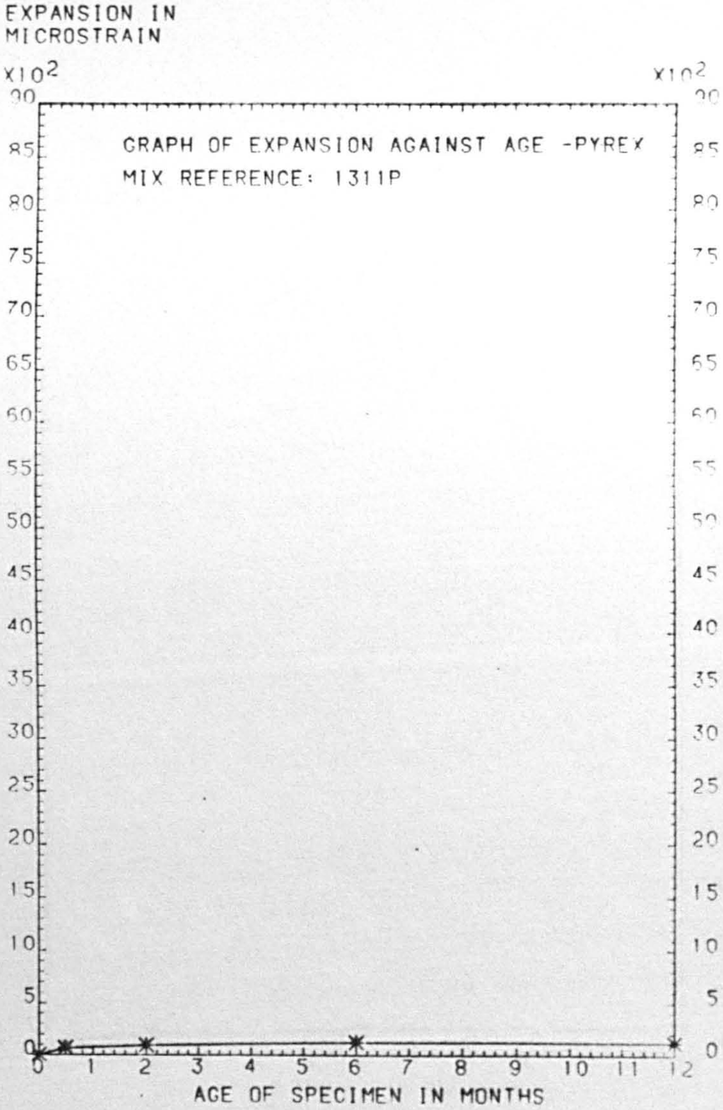

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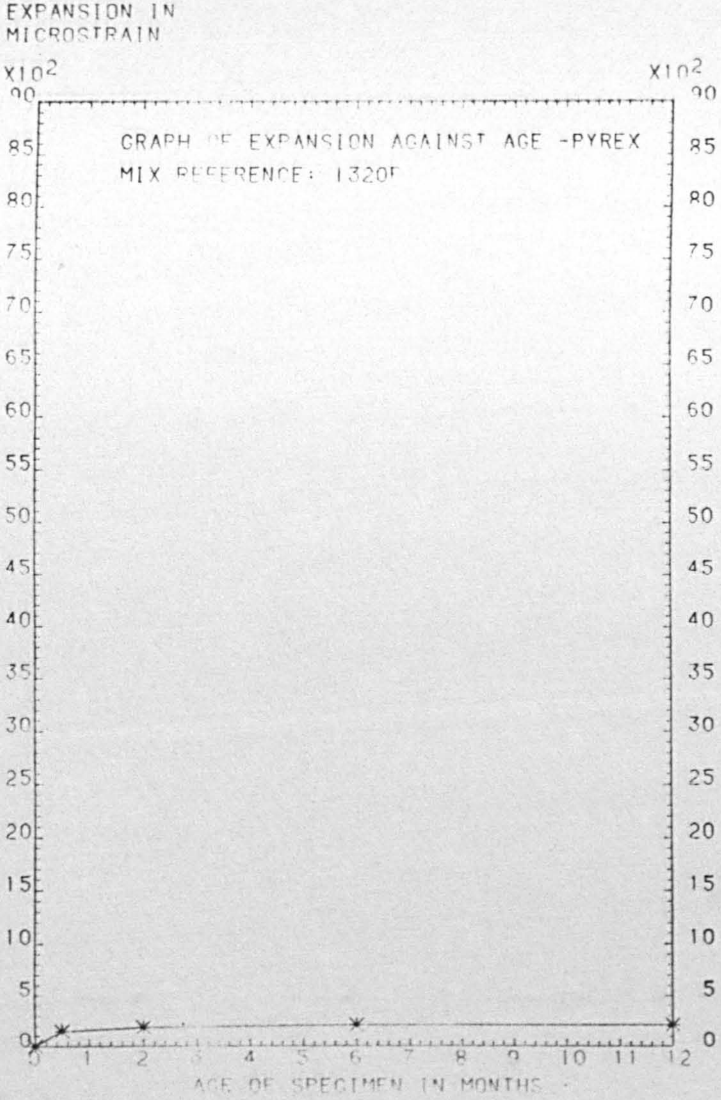

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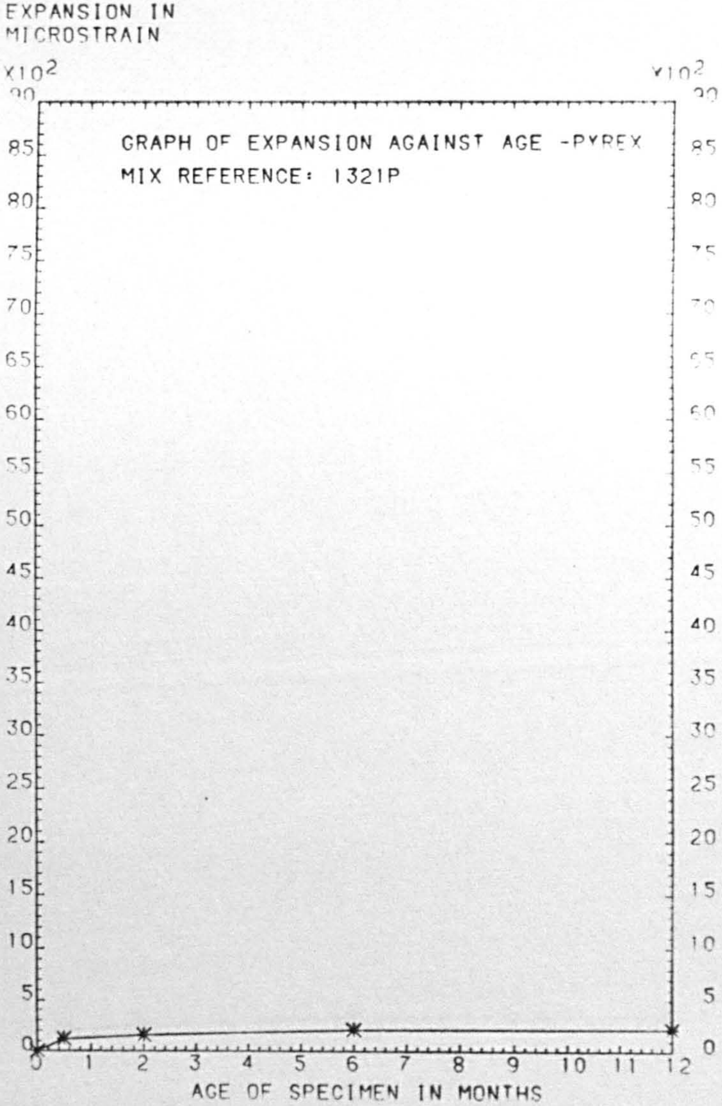

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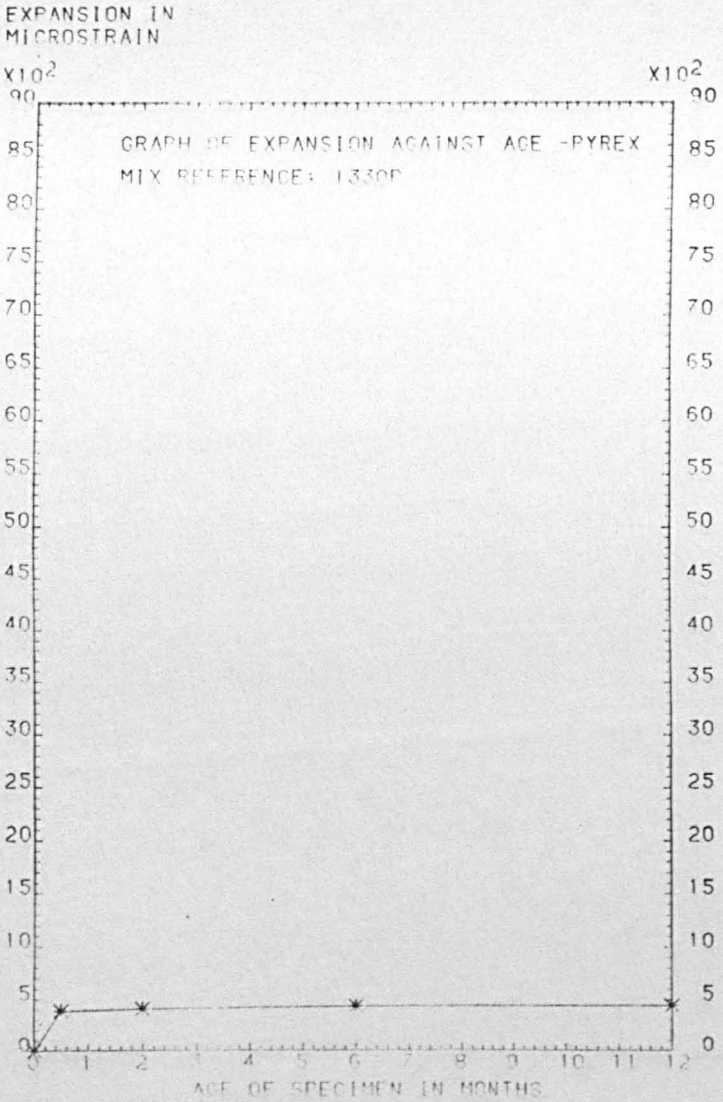

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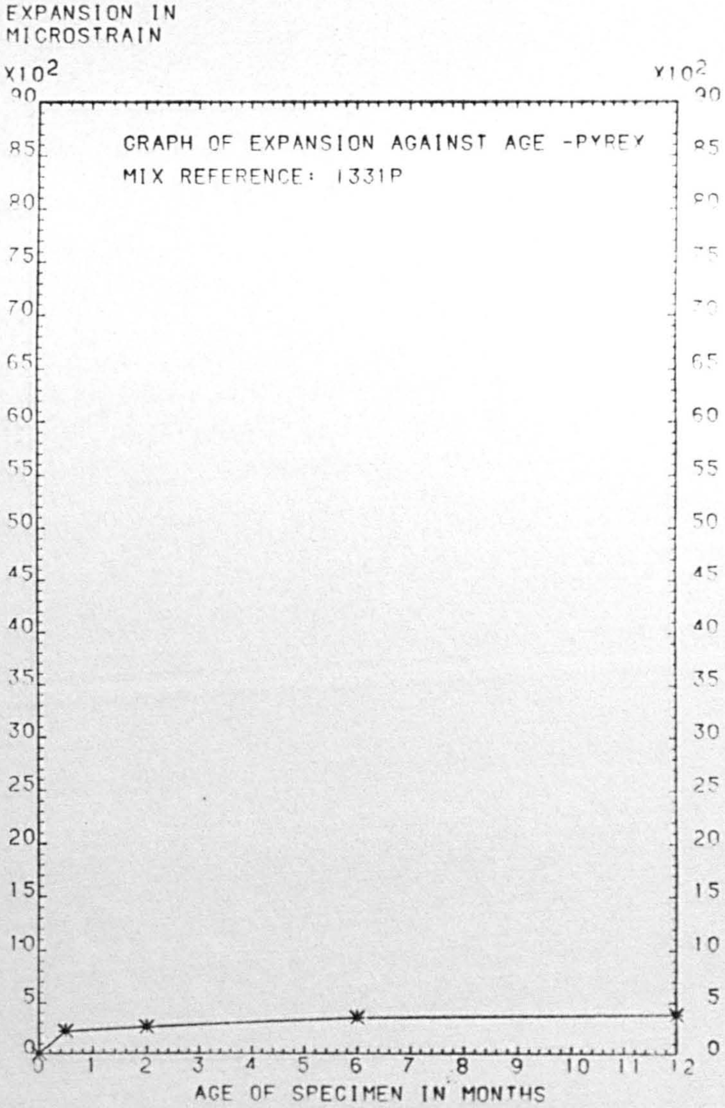

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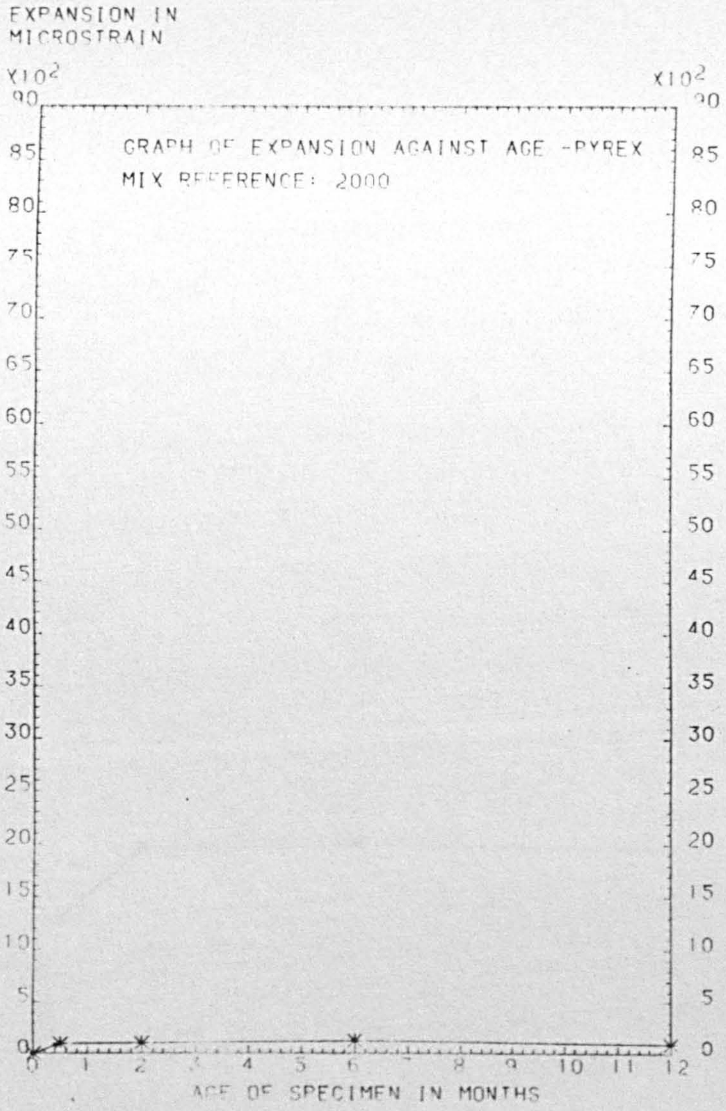

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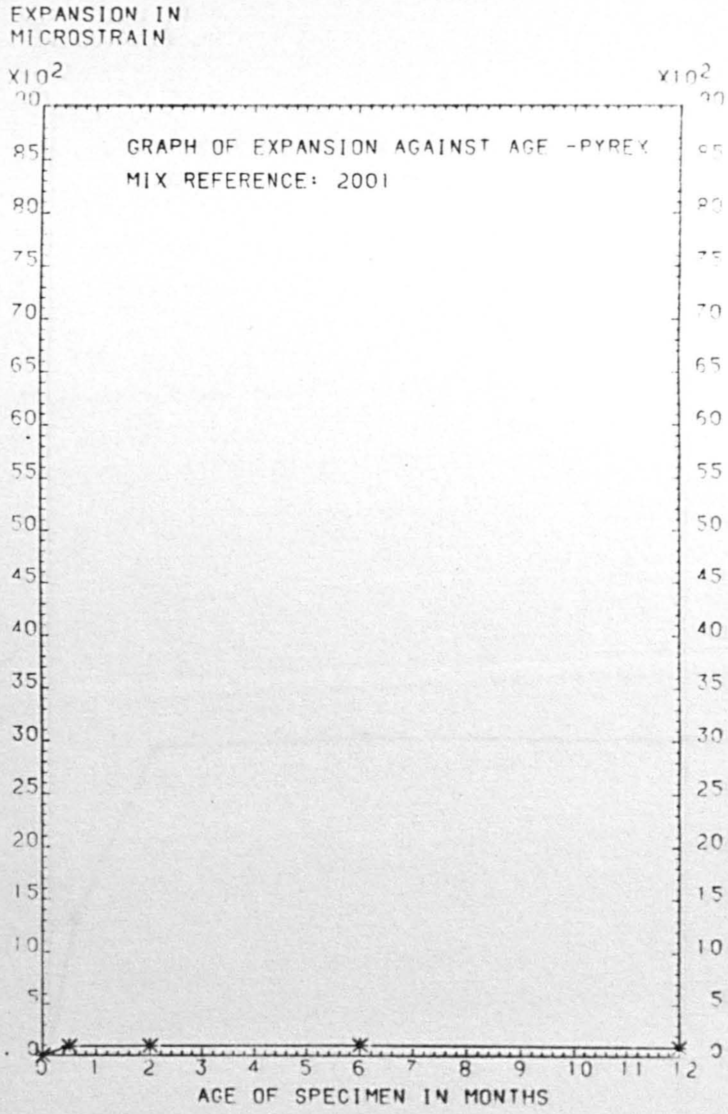

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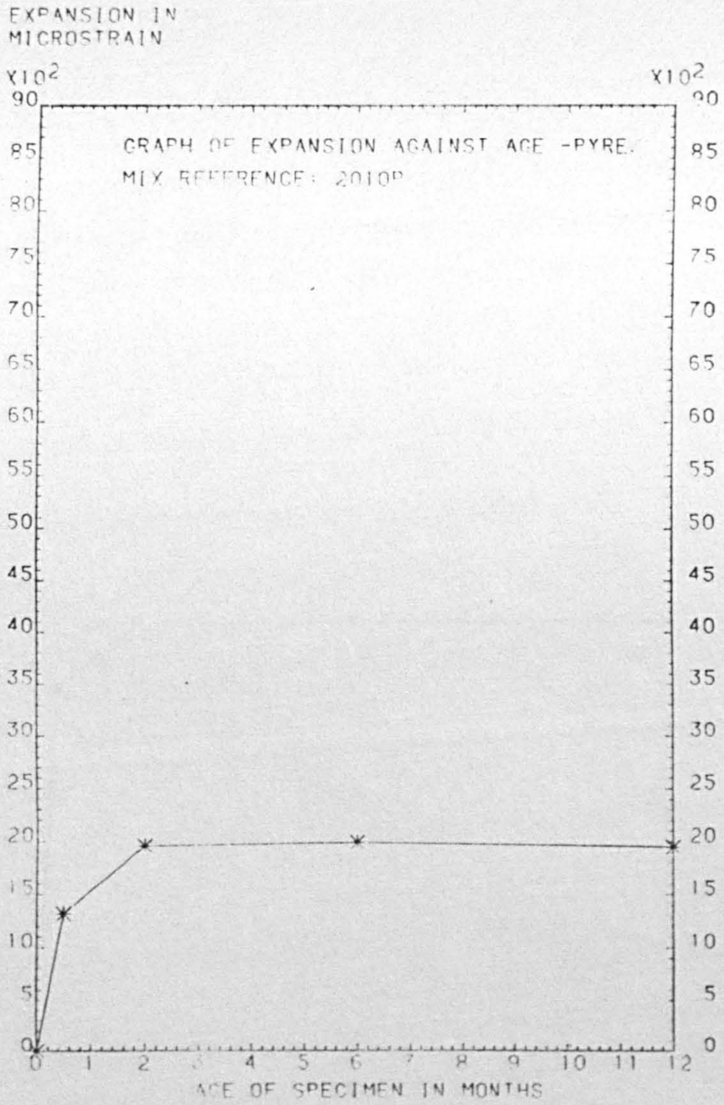

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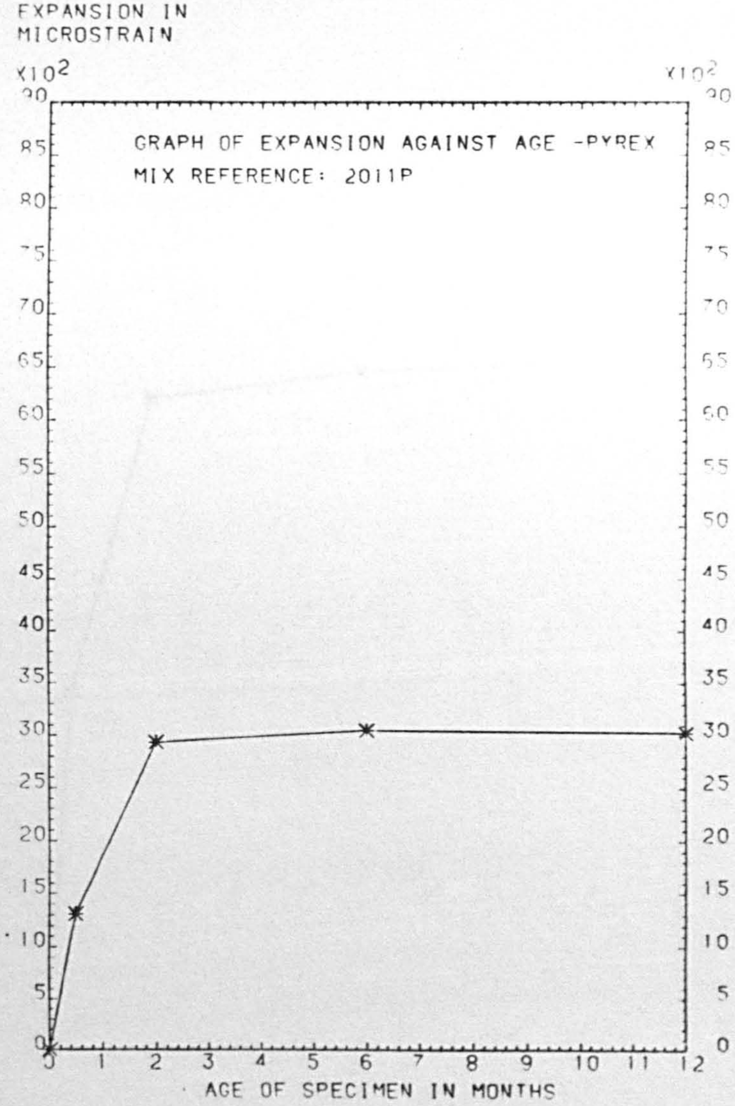

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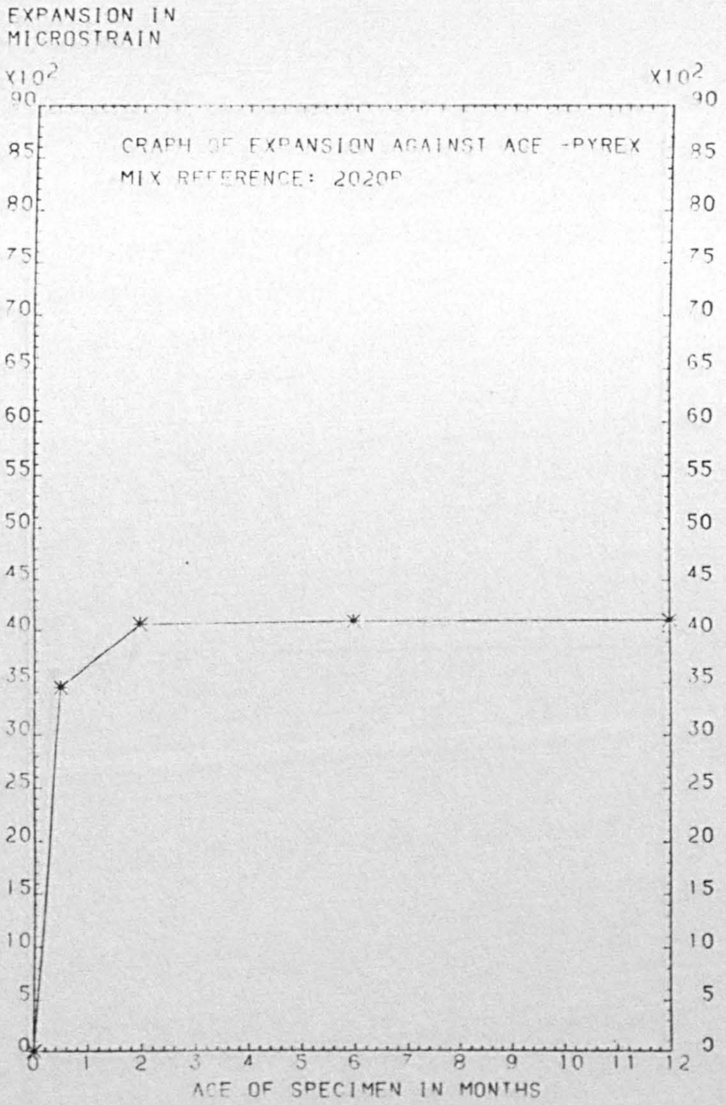

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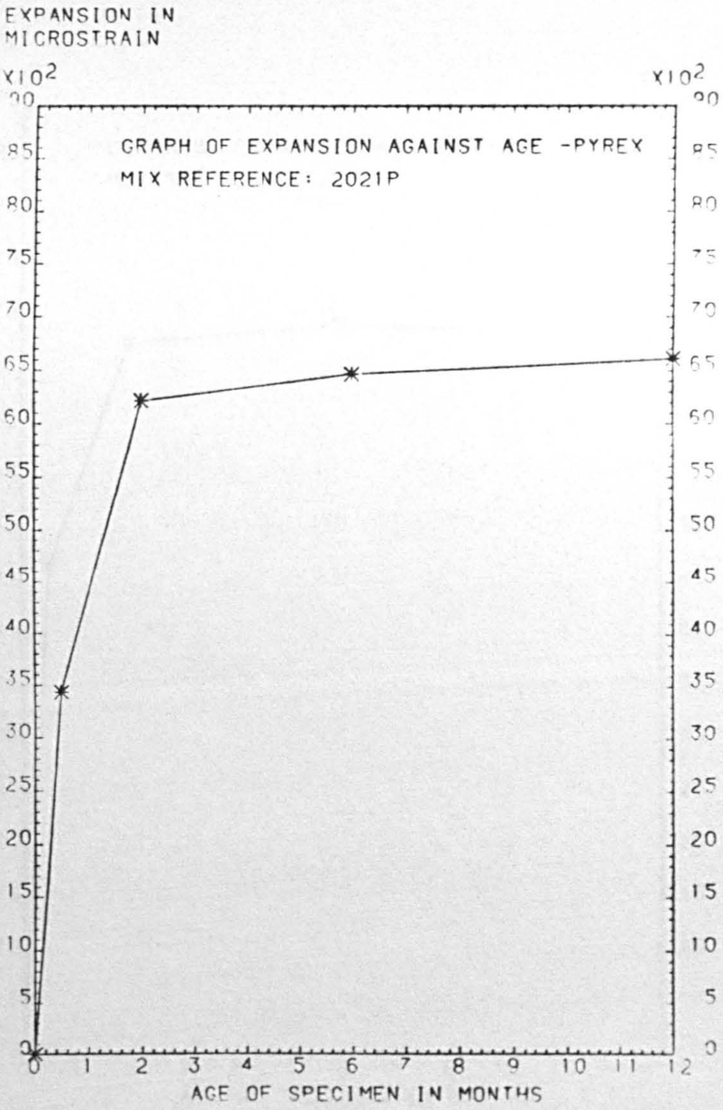

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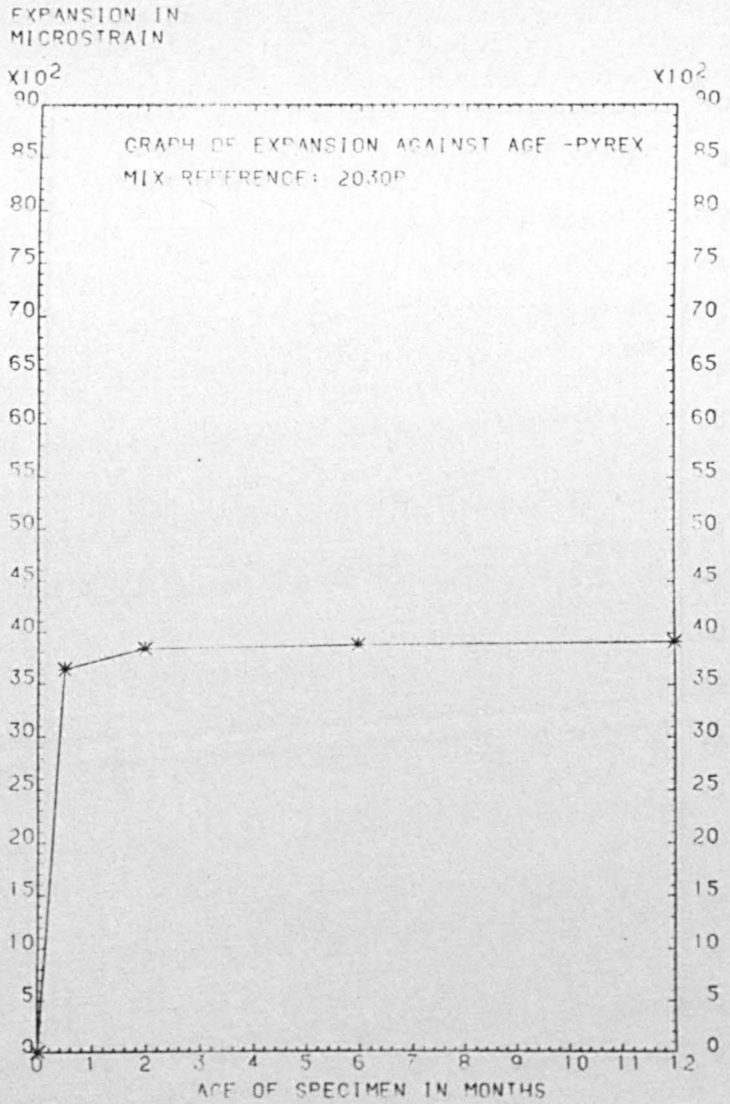

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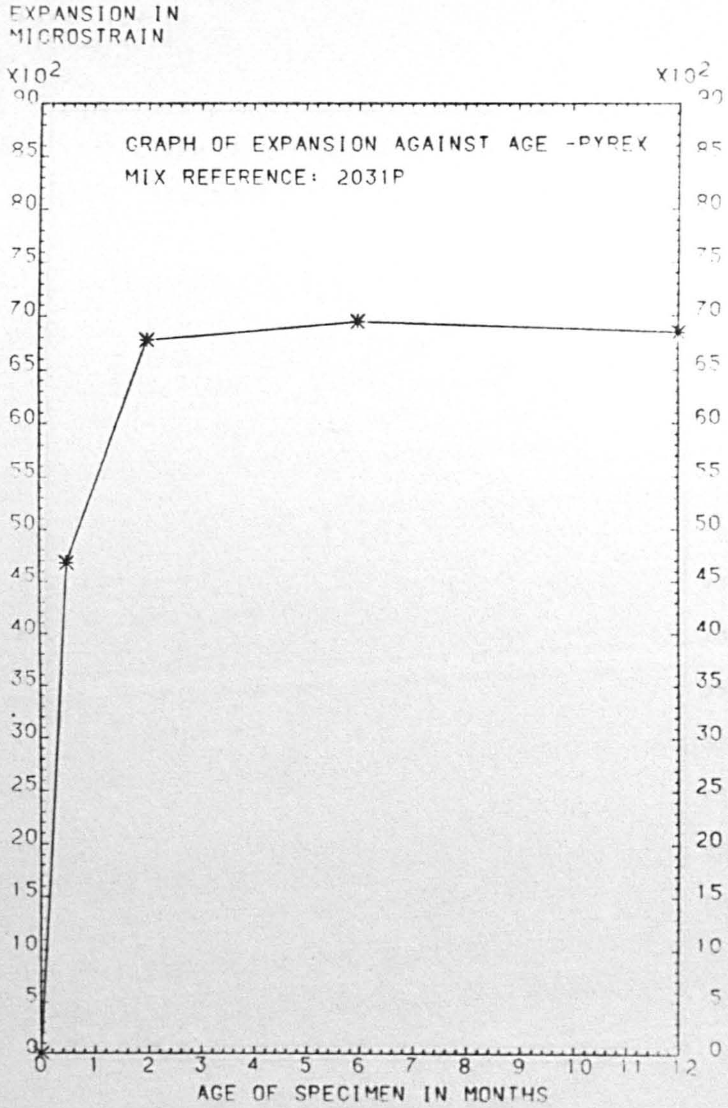

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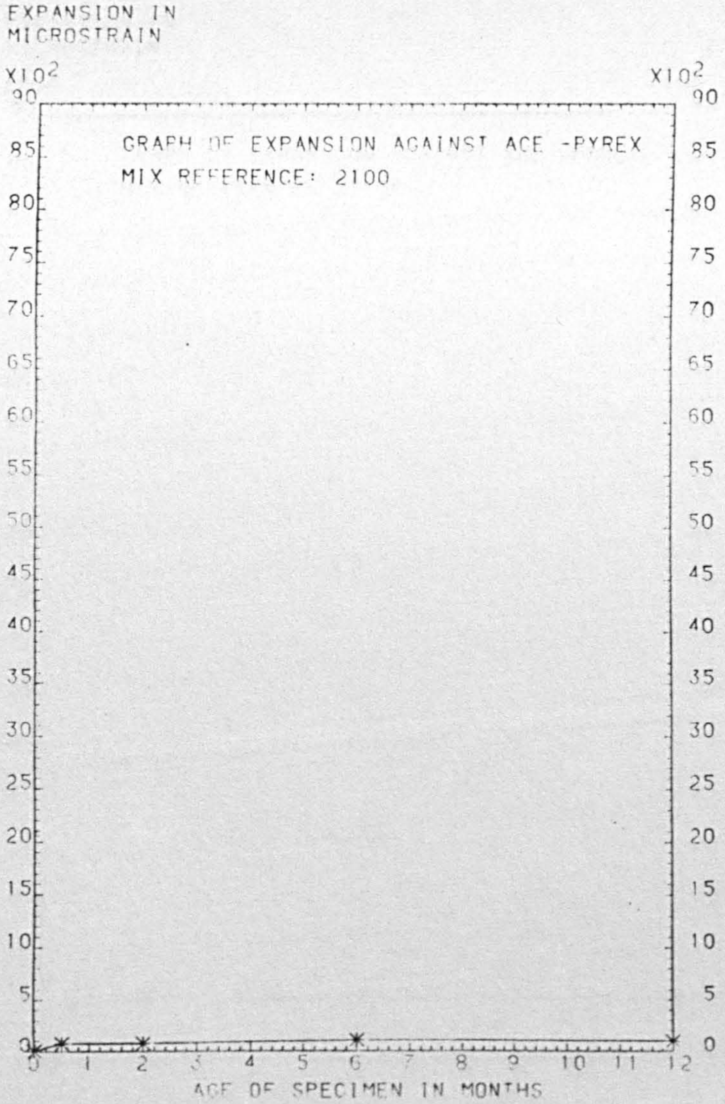

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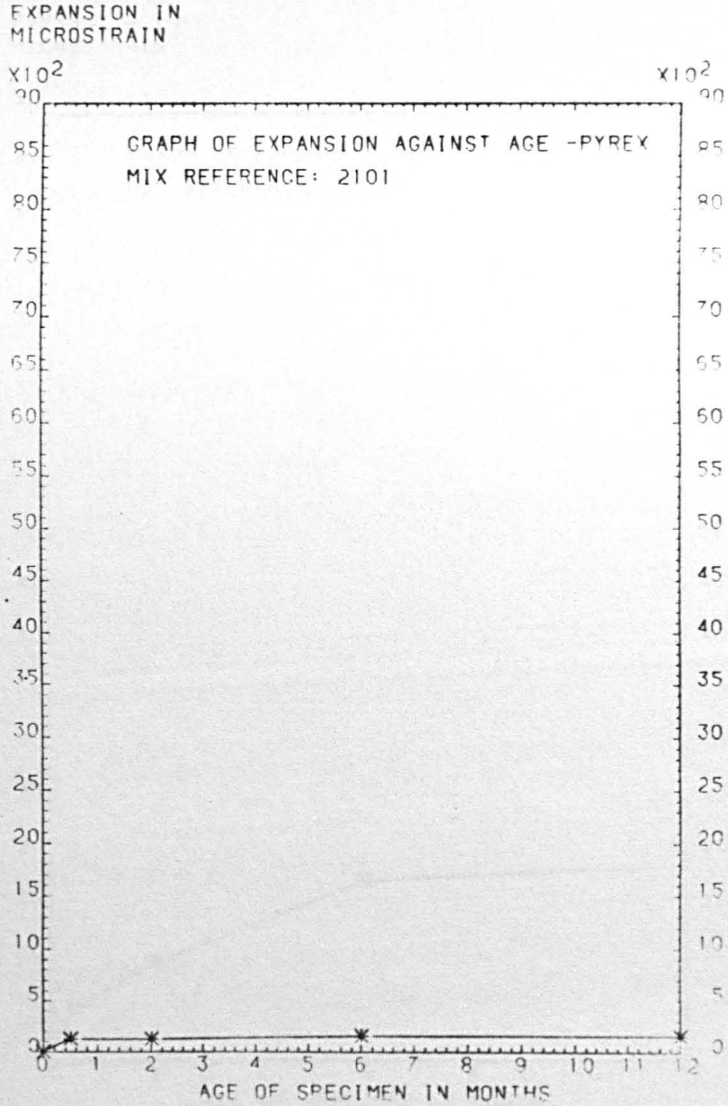

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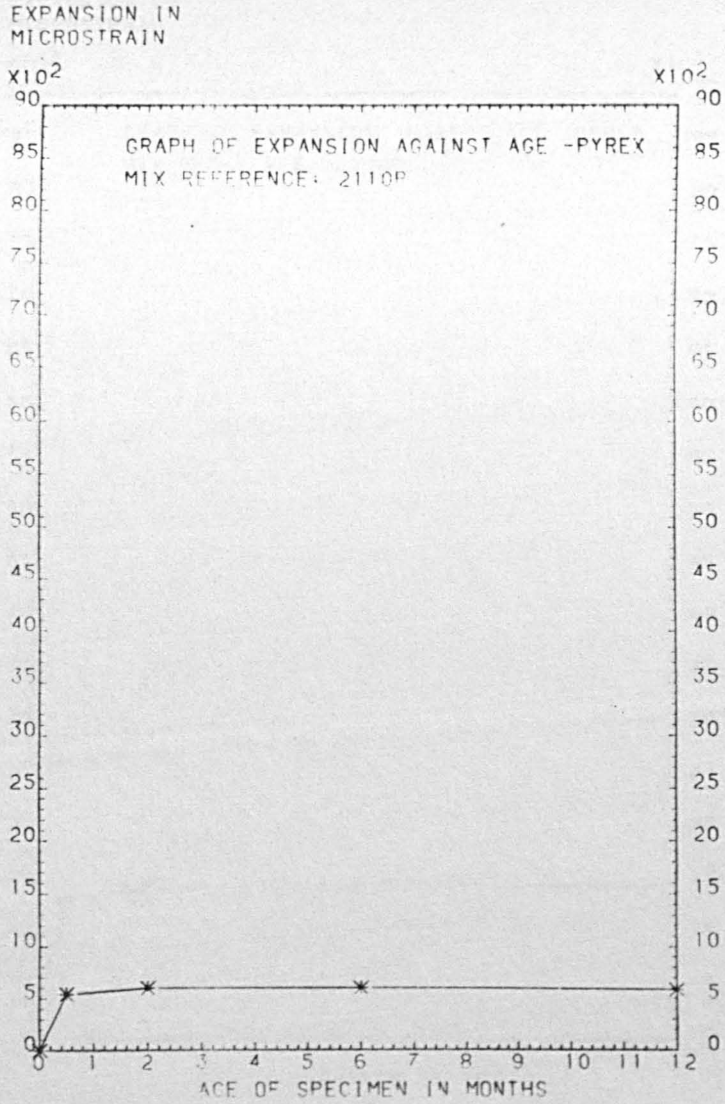

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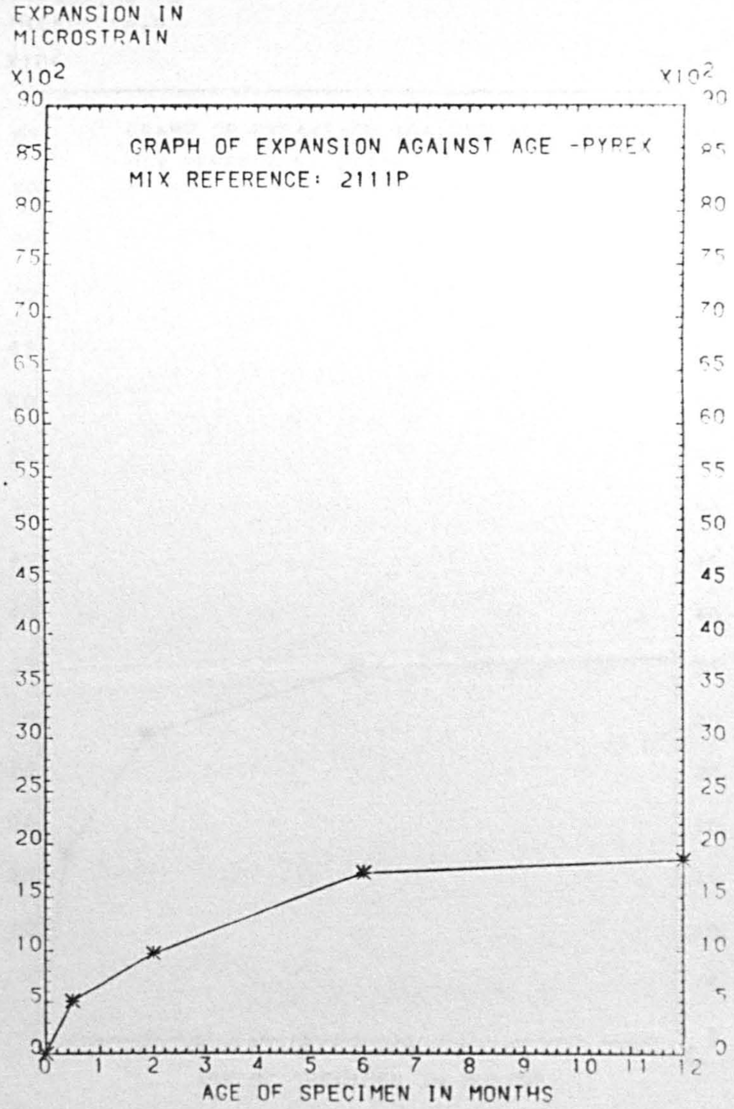

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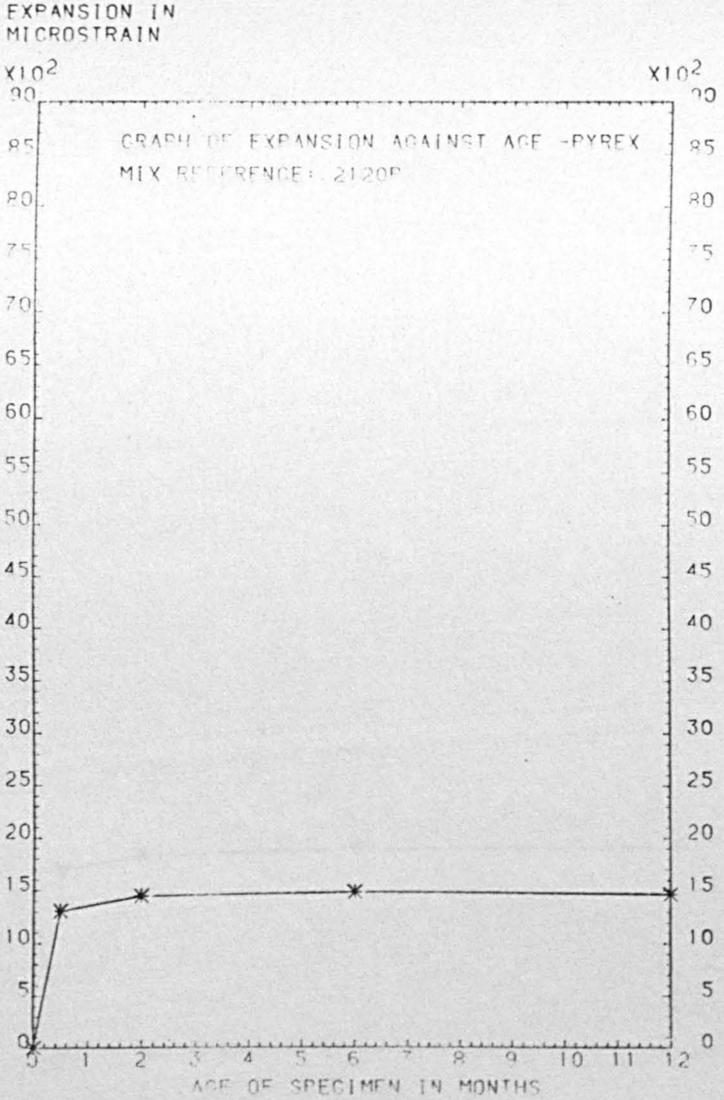

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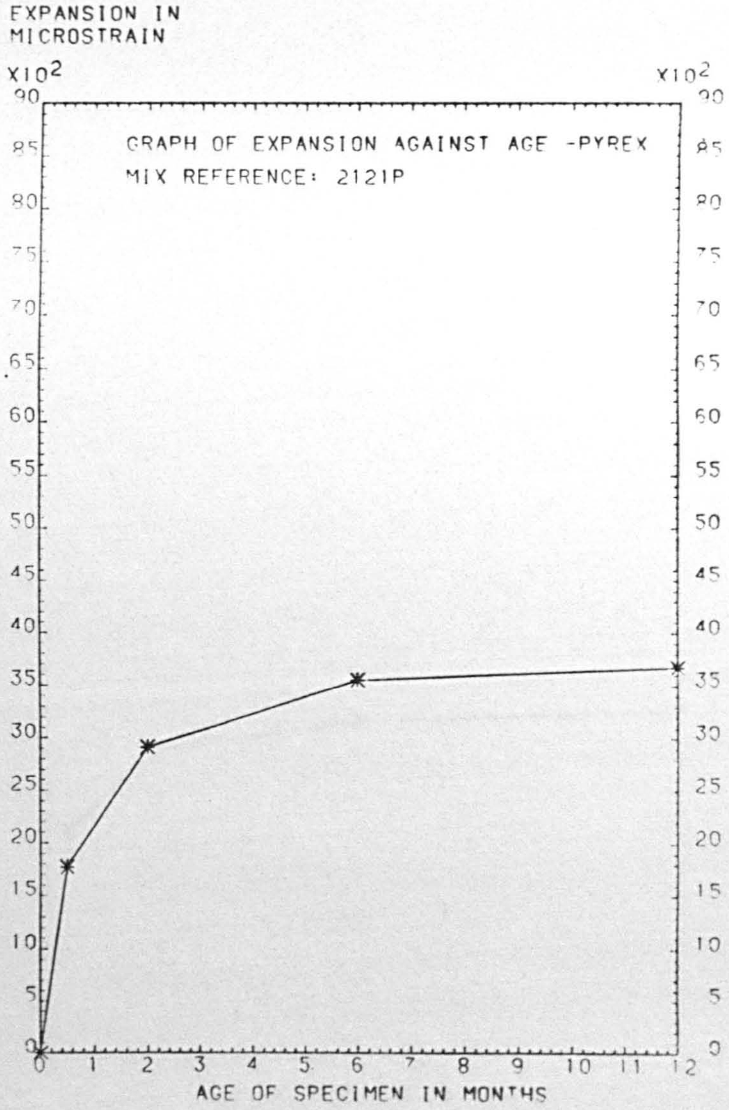

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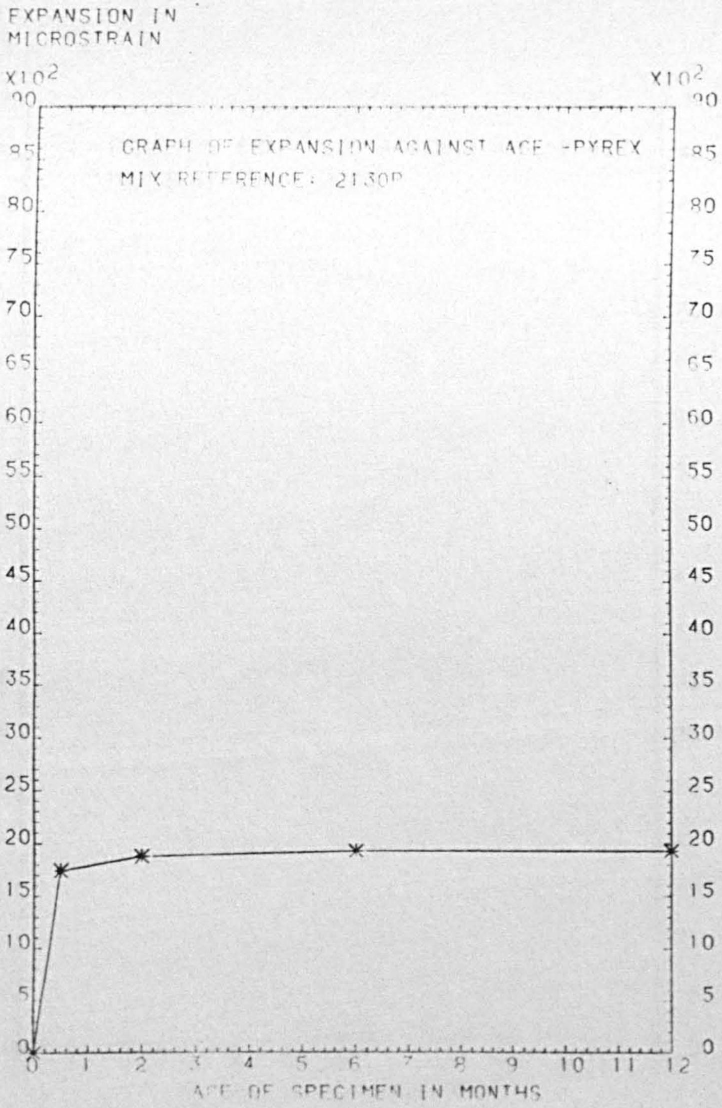

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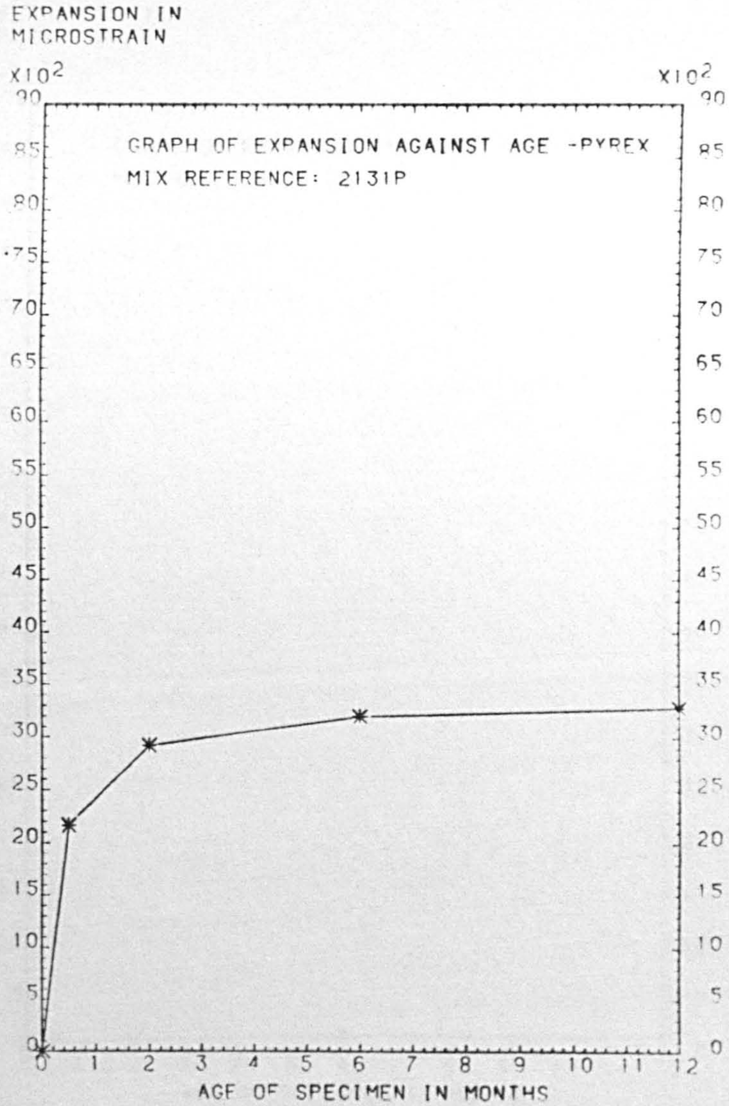

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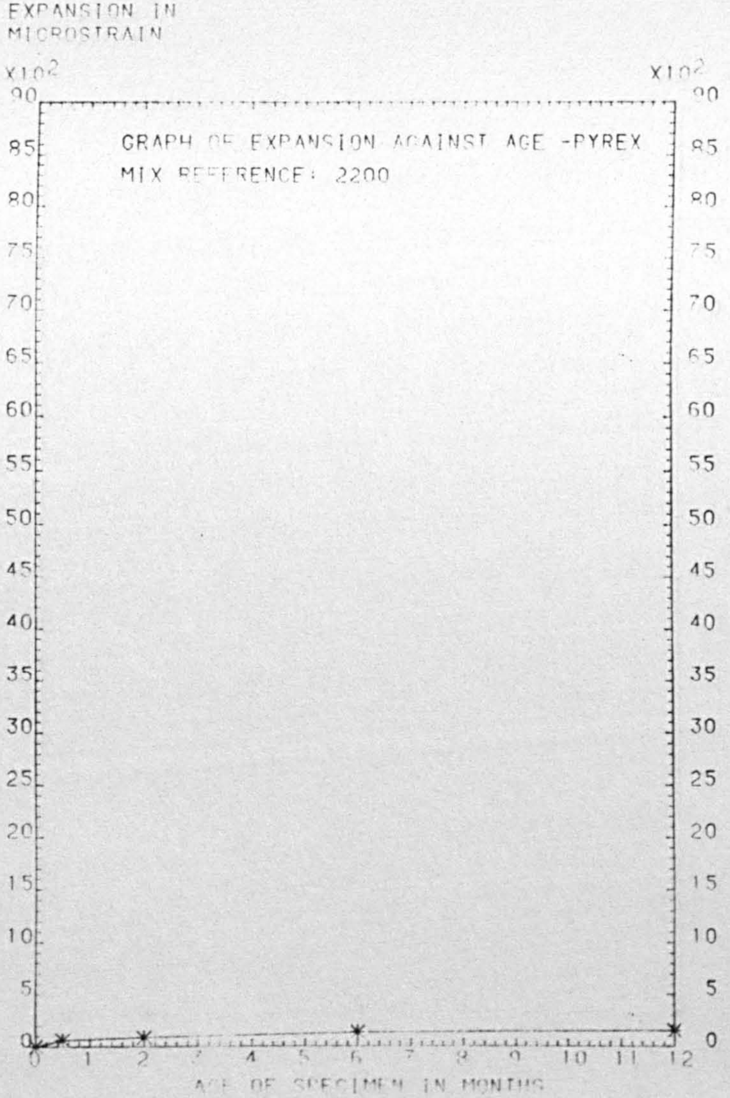

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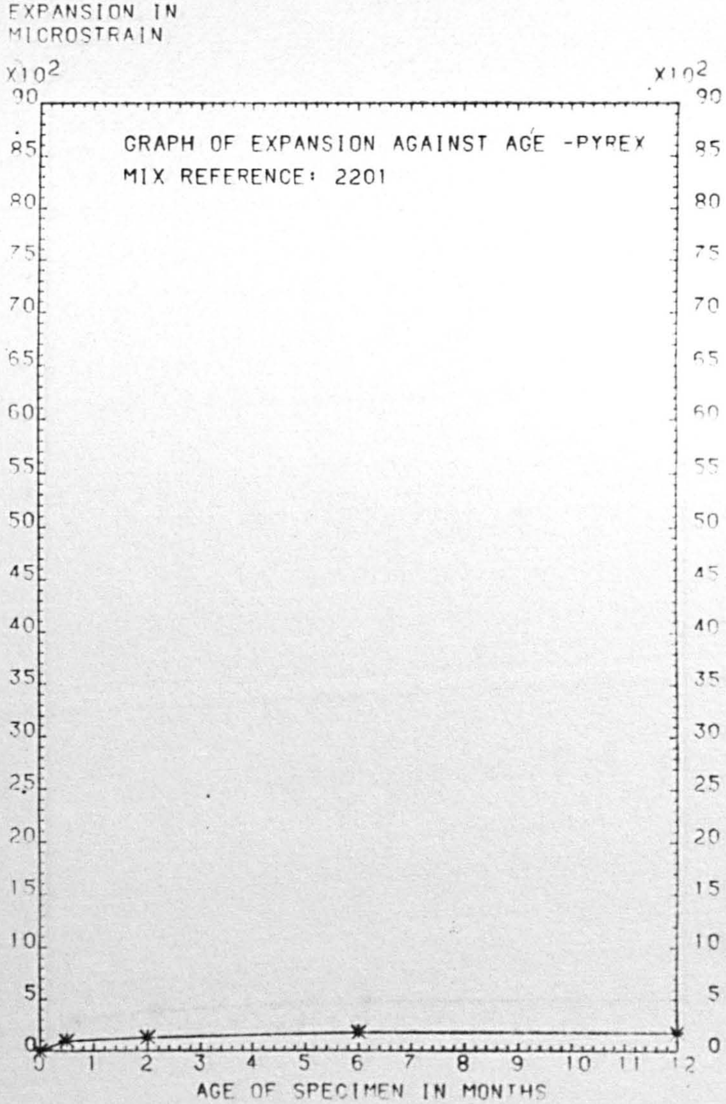

Figure D.82

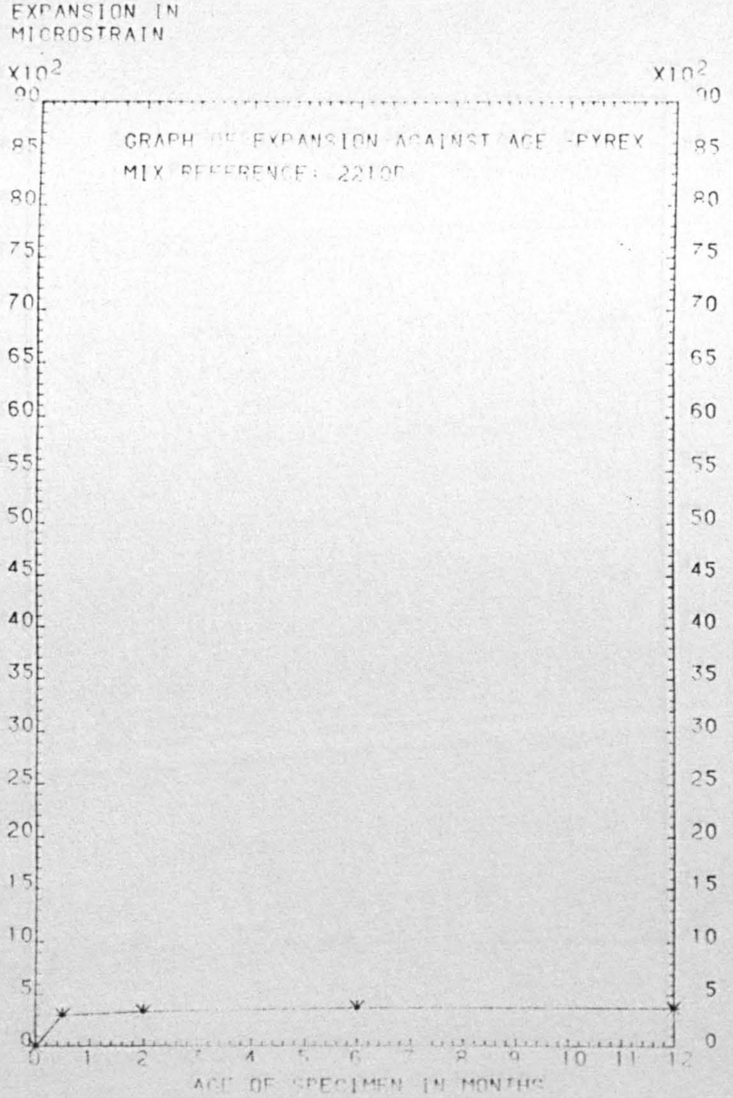

Figure D.83

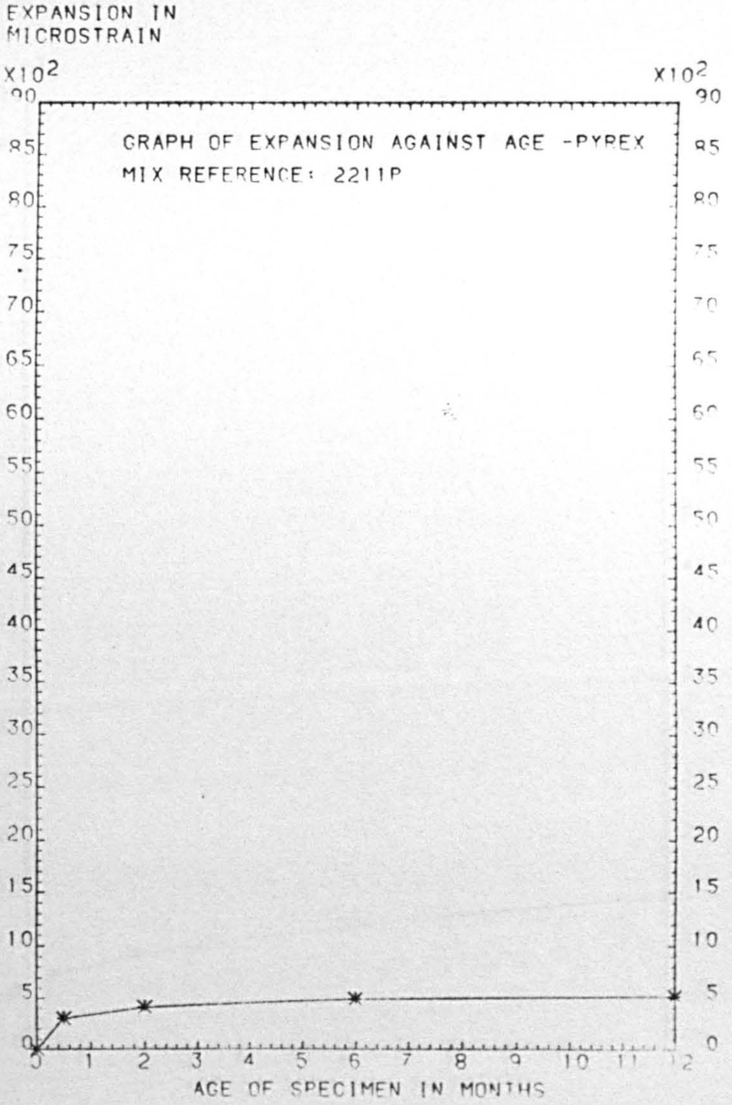

Figure D.84

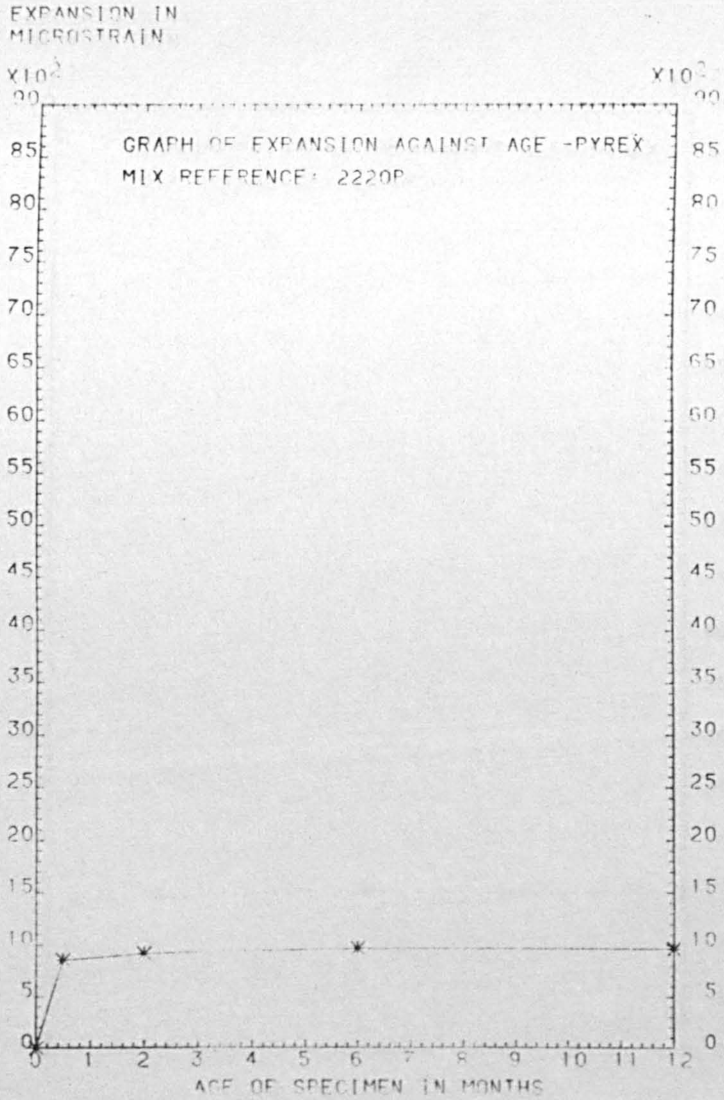

Figure D.85

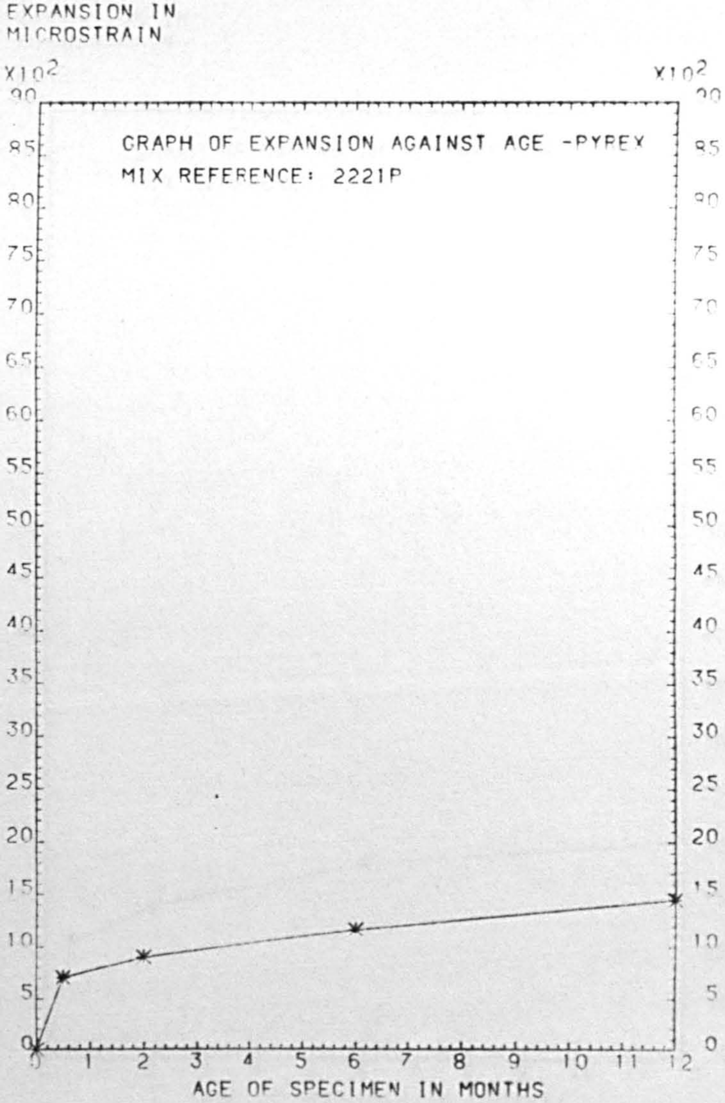

Figure D.86

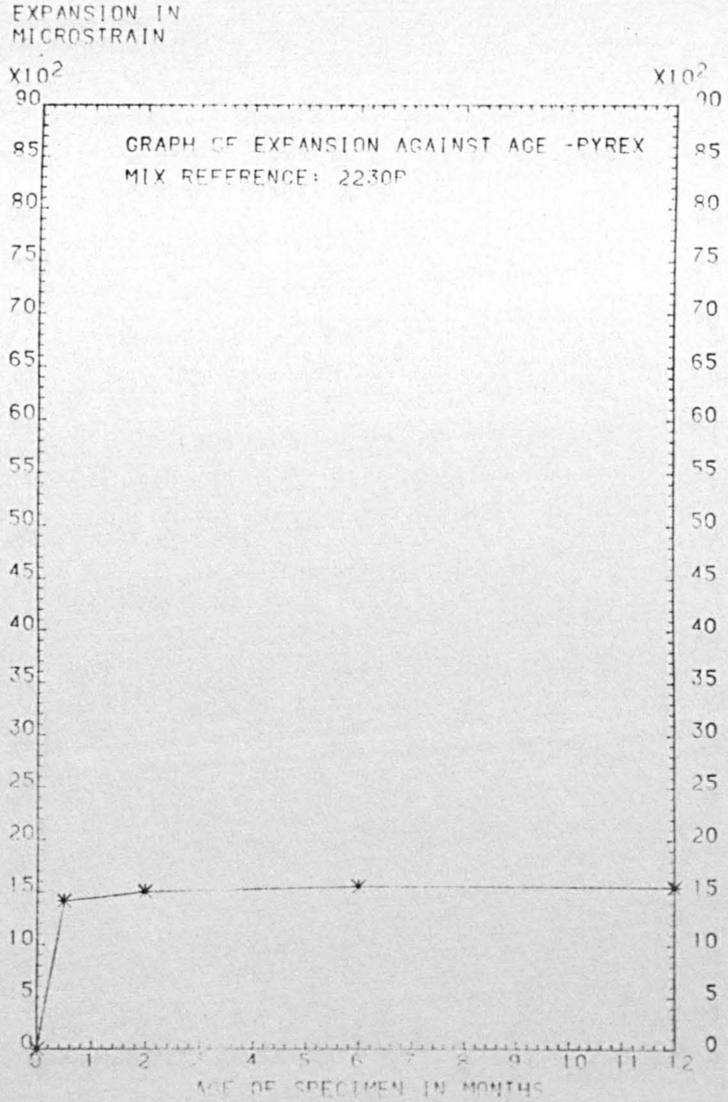

Figure D.87

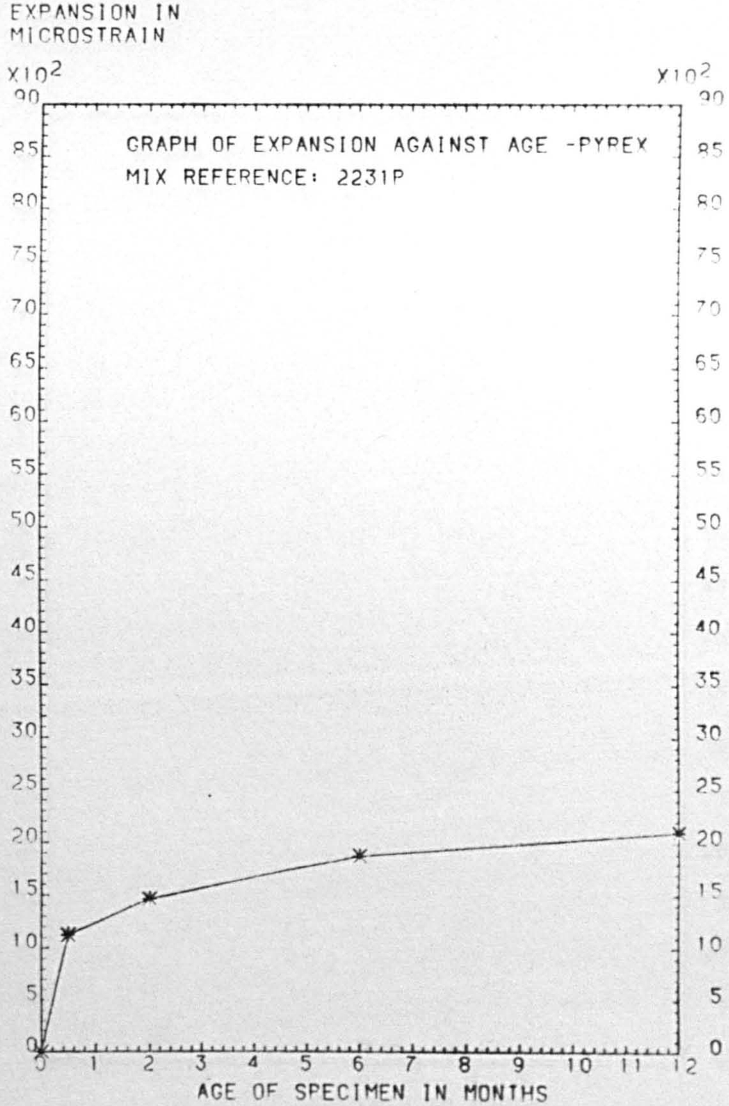

Figure D.88

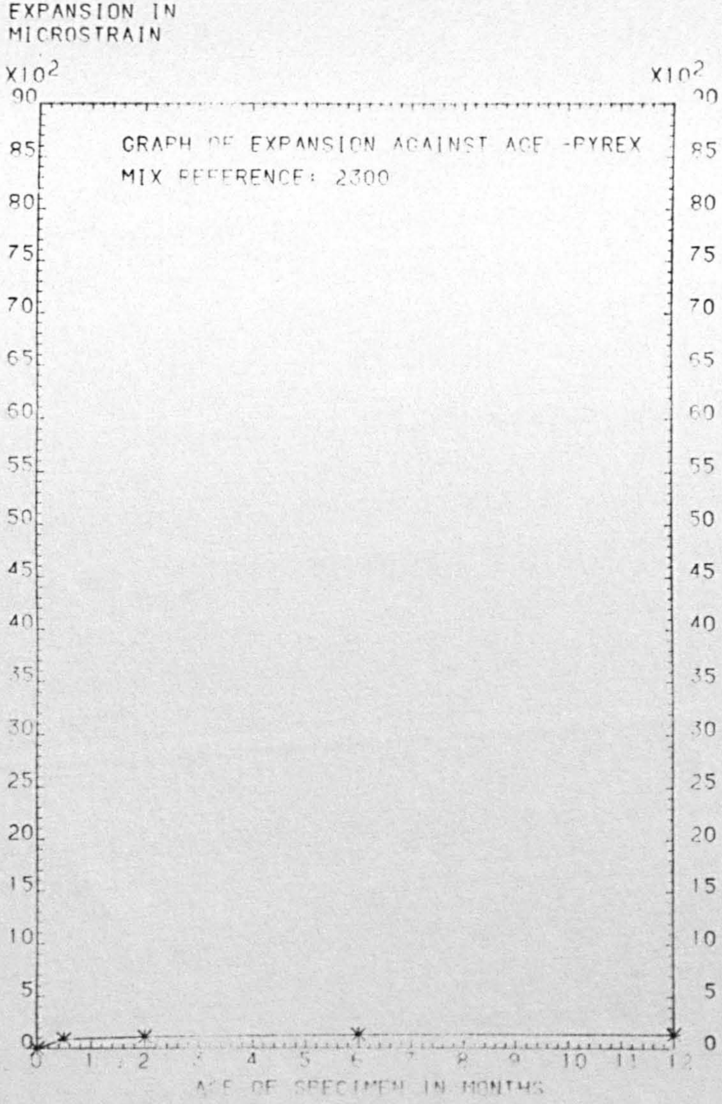

Figure D.89

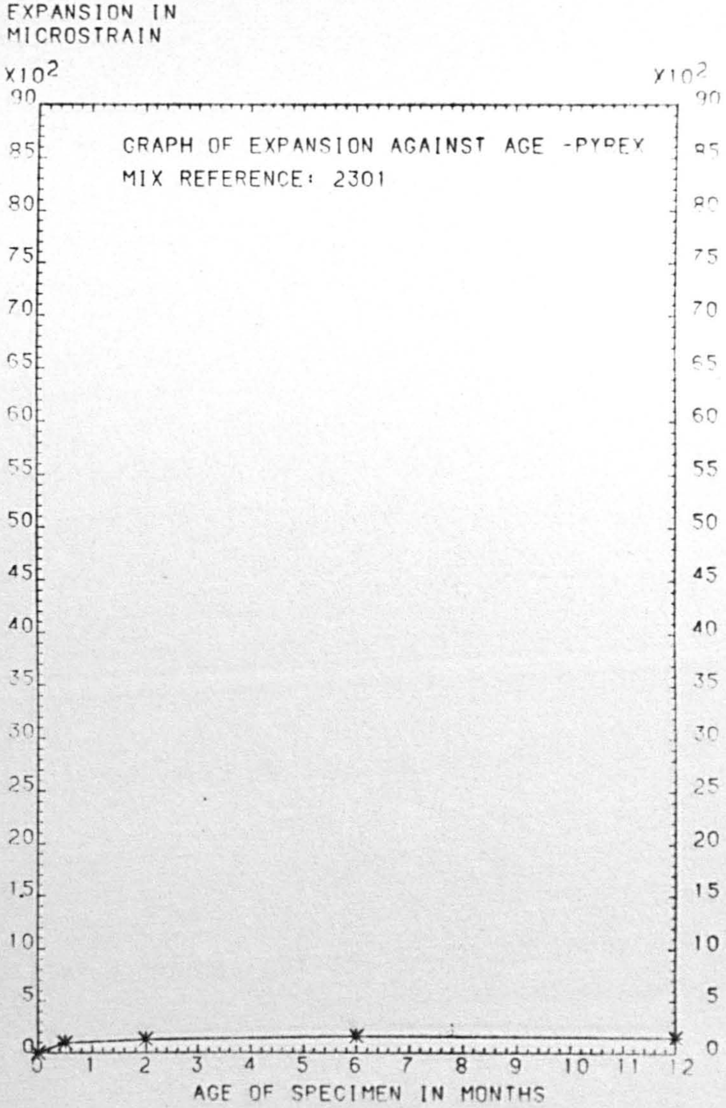

Figure D.90

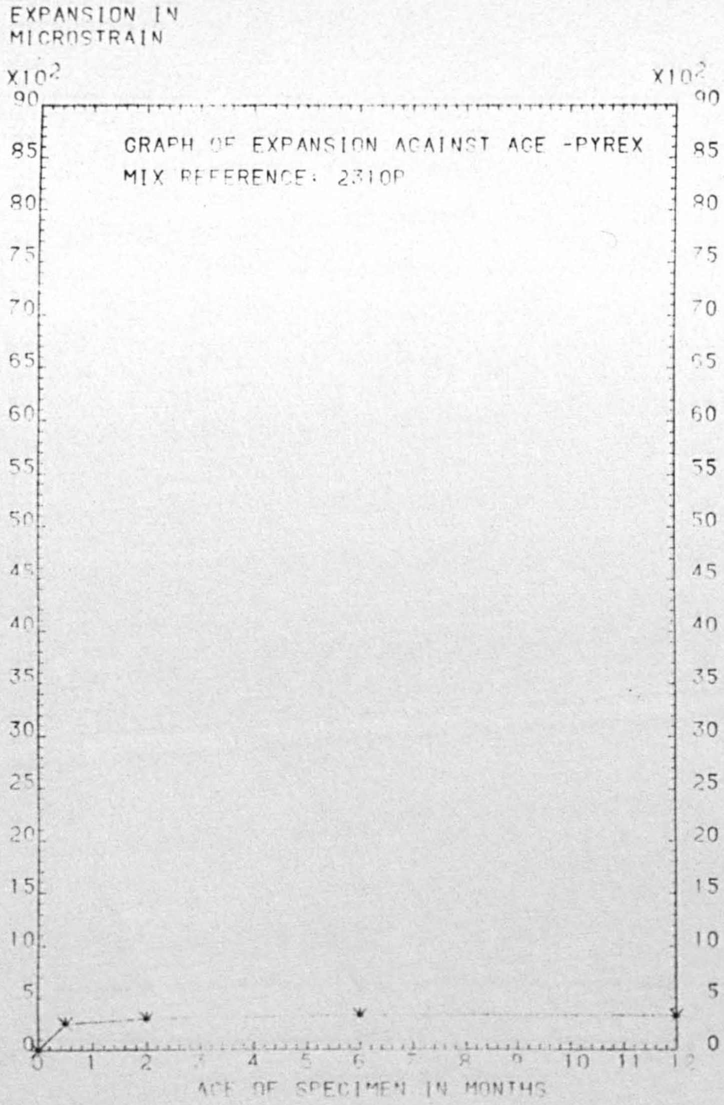

Figure D.91

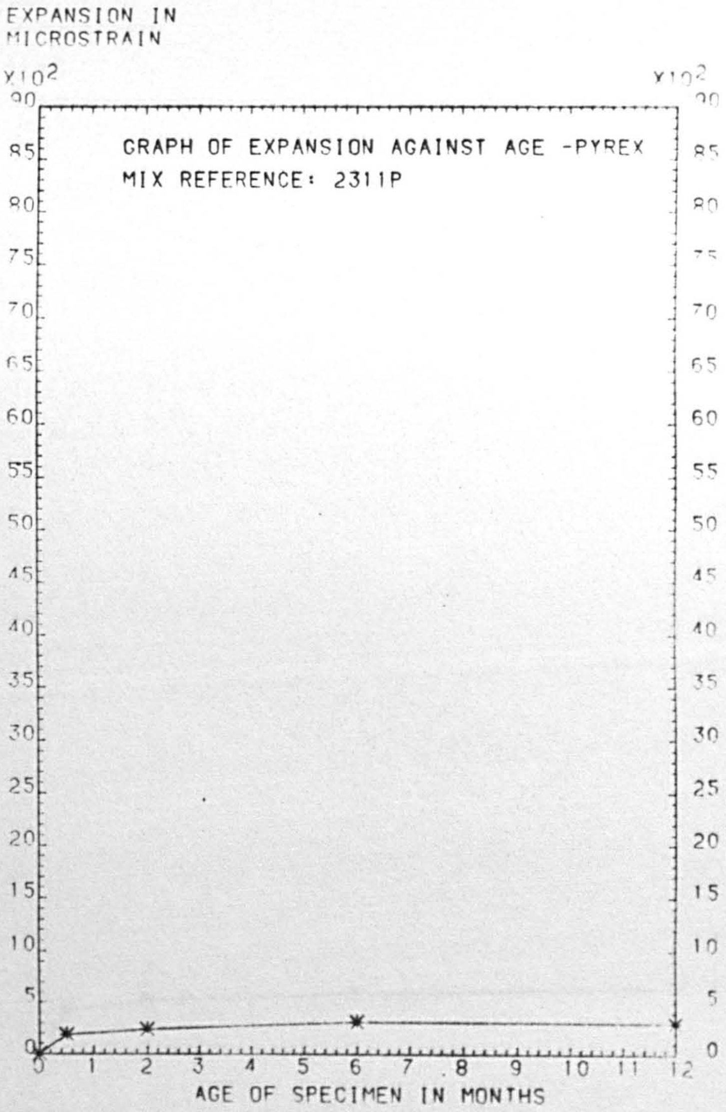

Figure D.92

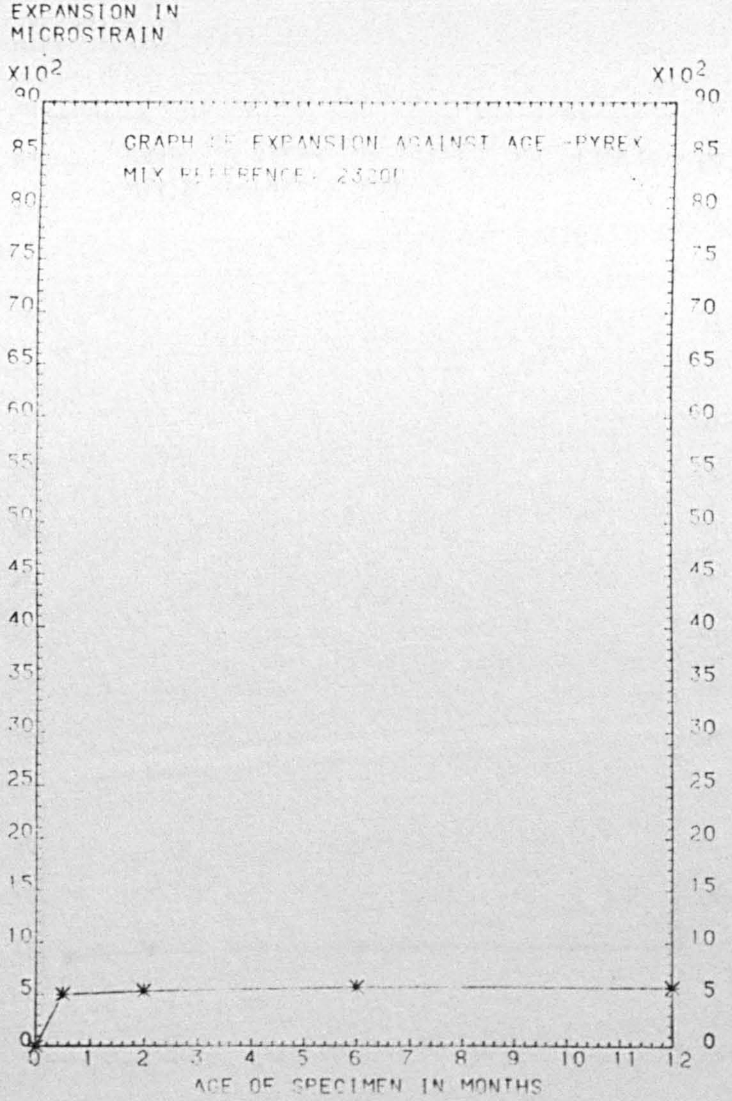

Figure D.93

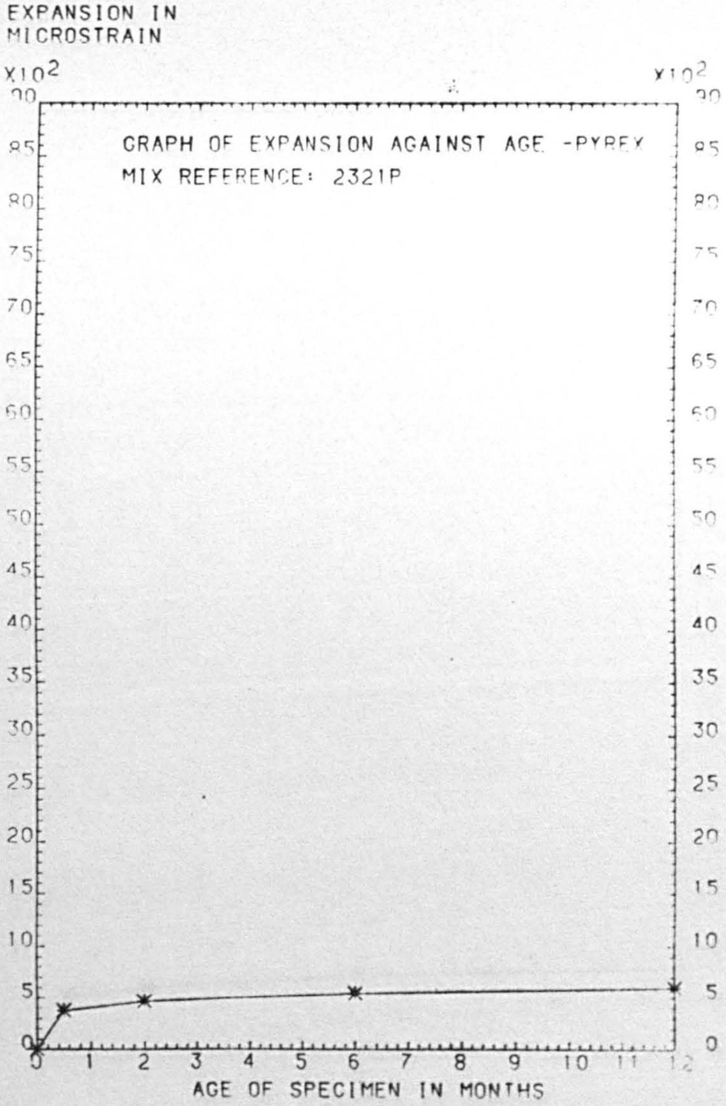

Figure D.94

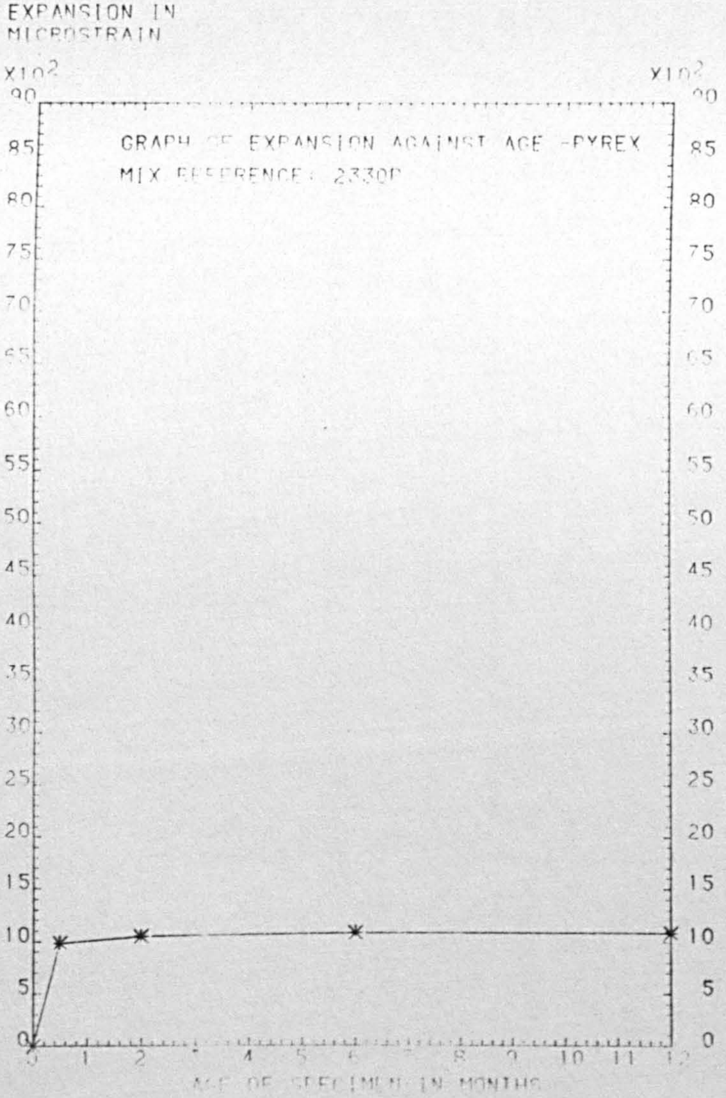

Figure D.95

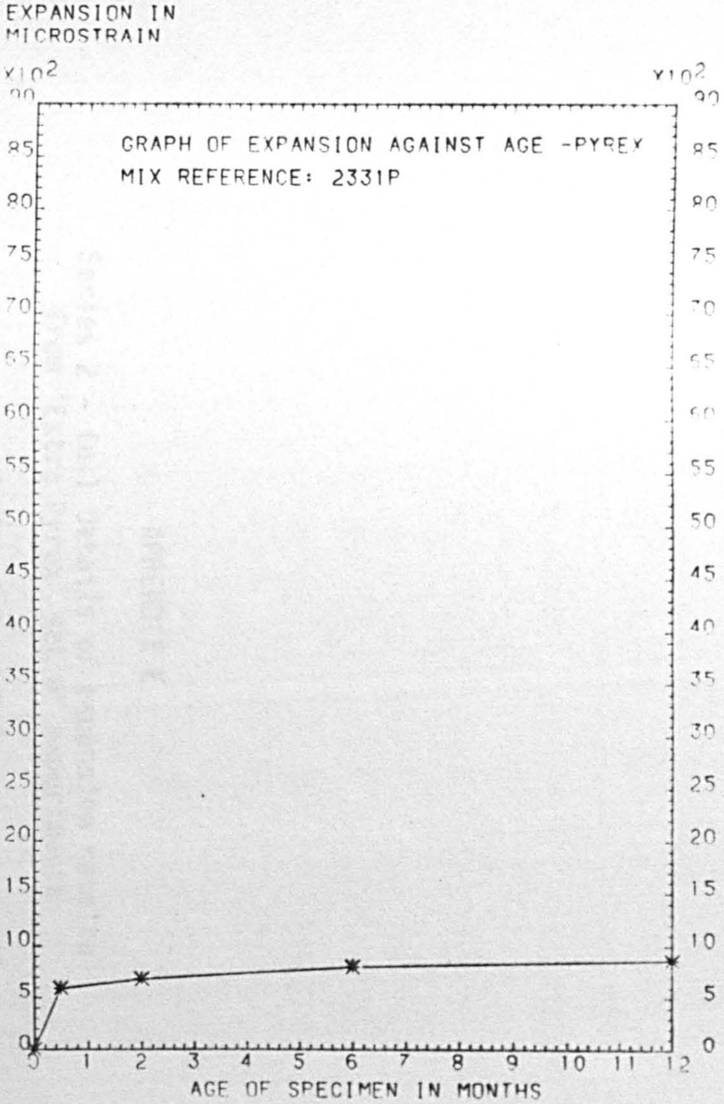

Figure D.96

APPENDIX E

Series 2 - full details of expansion results from 'Extra Pyrex' set of experiments

Tables E.1 to E.2

Figures E.1 to E.16

TABLE E.1 SERIES 2 MORTAR BAR EXPANSION RESULTS - EXTRA PYREX MIXES

MIX REF NO.	EXPANSION IN MICROSTRAIN (µε) AT AGE:-				'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	2 MONTHS	6 MONTHS	12 MONTHS	
2032 P	1573	1621	1663	1684	Harmful
2033 P	4865	5155	5223	5196	Harmful
2034 P	4903	5979	6088	6136	Harmful
2035 P	3669	6317	6468	6336	Harmful
2041 P	117	160	181*	192+	-
2051 P	153	228	245	232	-
2061 P	232	419	489	472	-
2071 P	372	705	771.	772	-
2431 P	3116	4480	4781	4824	Harmful
2531 P	1669	3221	3731	3888	Harmful
2631 P	1027	2204	2919	3228	Harmful
2731 P	792	1680	2483	2780	Harmful
3031 P	136	153	172	168	-
4031 P	288	305	328	324	-
5031 P	665	701	712	724	-
6031 P	2481	2627	2631	2652	Harmful

Note:- * denotes result from 1 bar failed the ASTM repeatability test
+ denotes results from 2 bars failed the ASTM repeatability test
** denotes based on limit referred to in ASTM C227

TABLE E.2(a) REPEATABILITY COMPUTATIONS FOR SERIES 2 'E.P.' SET, 14 DAY RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION      STRAIN
 2032P      0.393       1573.
 2033P      1.216       4865.
 2034P      1.226       4903.
 2035P      0.917       3669.
 2041P      0.029        117.
 2051P      0.038        153.
 2061P      0.058        232.
 2071P      0.093        372.
 2431P      0.779       3116.
 2531P      0.417       1669.
 2631P      0.257       1027.
 2731P      0.198        792.
 3031P      0.034        136.
 4031P      0.072        288.
 5031P      0.166        665.
 6031P      0.620       2481.
```

```
Mix Ref. EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
 No.                                                 DIFFERENCE
 2032P       0.011         0.003        -0.013        0.059
 2033P      -0.017         0.004         0.014        0.132
 2034P      -0.025         0.012         0.012        0.134
 2035P      -0.035         0.006         0.030        0.133
 2041P       0.001        -0.001         0.001        0.003
 2051P      -0.001        -0.000         0.002        0.003
 2061P       0.002        -0.002         0.           0.009
 2071P      -0.004        -0.001         0.005        0.014
 2431P       0.009        -0.017         0.008        0.117
 2531P       0.011        -0.001        -0.009        0.063
 2631P      -0.003        -0.015         0.017        0.039
 2731P      -0.006    .    0.002         0.004        0.030
 3031P      -0.000         0.000         0.           0.003
 4031P       0.002        -0.002         0.000        0.011
 5031P      -0.000         0.003        -0.002        0.025
 6031P       0.019        -0.012        -0.006        0.093
```

```
Mix Ref.
 No.    CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2032P         0.048         0.056         0.046
 2033P         0.165         0.179         0.169
 2034P         0.159         0.172         0.172
 2035P         0.102         0.132         0.108
 2041P         0.002         0.002         0.002
 2051P         0.002         0.003         0.001
 2061P         0.007         0.007         0.009
 2071P         0.010         0.013         0.009
 2431P         0.108         0.100         0.109
 2531P         0.052         0.061         0.053
 2631P         0.036         0.024         0.021
 2731P         0.024         0.028         0.026
 3031P         0.003         0.003         0.003
 4031P         0.009         0.009         0.011
 5031P         0.025         0.022         0.023
 6031P         0.074         0.081         0.087
```

TABLE E.2(b) REPEATABILITY COMPUTATIONS FOR SERIES 2 'E.P.' SET, 2 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
        Mix Ref.   AVERAGE    MICRO-
          No.     EXPANSION     STRAIN
         2032P      0.405      1621.
         2033P      1.289      5155.
         2034P      1.495      5979.
         2035P      1.579      6317.
         2041P      0.040       160.
         2051P      0.057       228.
         2061P      0.105       419.
         2071P      0.176       705.
         2431P      1.120      4480.
         2531P      0.805      3221.
         2631P      0.551      2204.
         2731P      0.420      1680.
         3031P      0.038       153.
         4031P      0.076       305.
         5031P      0.175       701.
         6031P      0.657      2627.


   Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
     No.                                                DIFFERENCE
    2032P        0.011         0.004        -0.014        0.061
    2033P       -0.017        -0.001         0.017        0.193
    2034P       -0.032         0.050        -0.019        0.224
    2035P        0.011        -0.036         0.026        0.237
    2041P       -0.000        -0.002         0.002        0.003
    2051P        0.            0.            0.           0.009
    2061P        0.003        -0.009         0.005        0.016
    2071P       -0.012         0.015        -0.002        0.026
    2431P       -0.004        -0.005         0.009        0.168
    2531P        0.017        -0.007        -0.009        0.121
    2631P        0.003        -0.028         0.025        0.083
    2731P       -0.010        -0.000         0.010        0.063
    3031P        0.001        -0.000        -0.000        0.003
    4031P        0.003        -0.000        -0.002        0.011
    5031P        0.001         0.003        -0.003        0.026
    6031P        0.017        -0.011        -0.007        0.099

Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2032P           0.050         0.057         0.046
 2033P           0.177         0.193         0.176
 2034P           0.193         0.174         0.206
 2035P           0.226         0.201         0.211
 2041P           0.003         0.001         0.001
 2051P           0.009         0.009         0.009
 2061P           0.012         0.007         0.010
 2071P           0.014         0.012         0.024
 2431P           0.164         0.163         0.159
 2531P           0.104         0.113         0.111
 2631P           0.080         0.055         0.058
 2731P           0.053         0.063         0.053
 3031P           0.002         0.003         0.003
 4031P           0.009         0.011         0.009
 5031P           0.026         0.024         0.023
 6031P           0.081         0.083         0.092
```

TABLE E.2(c) REPEATABILITY COMPUTATIONS FOR SERIES 2 'E.P.' SET, 6 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE    MICRO-
  No.     EXPANSION    STRAIN
 2032P      0.416      1663.
 2033P      1.306      5223.
 2034P      1.522      6088.
 2035P      1.617      6468.
 2041P      0.045       181.
 2051P      0.061       245.
 2061P      0.122       489.
 2071P      0.193       771.
 2431P      1.195      4781.
 2531P      0.933      3731.
 2631P      0.730      2919.
 2731P      0.621      2483.
 3031P      0.043       172.
 4031P      0.032       328.
 5031P      0.178       712.
 6031P      0.658      2631.
```

```
Mix Ref.  EXPN(1)-AVGE   EXPN(2)-AVGE   EXPN(3)-AVGE   ALLOWABLE
  No.                                                   DIFFERENCE
 2032P        0.010          0.003         -0.014          0.062
 2033P       -0.018          0.000          0.017          0.196
 2034P       -0.022          0.037         -0.015          0.223
 2035P        0.023         -0.054          0.031          0.243
 2041P       -0.001         -0.002          0.004          0.003
 2051P       -0.000          0.001         -0.000          0.009
 2061P        0.014         -0.017          0.004          0.018
 2071P       -0.015          0.023         -0.009          0.029
 2431P       -0.002         -0.006          0.009          0.179
 2531P        0.019         -0.011         -0.009          0.140
 2631P        0.014         -0.035          0.020          0.109
 2731P       -0.001         -0.009          0.009          0.093
 3031P        0.000          0.001         -0.001          0.003
 4031P        0.002          0.000         -0.002          0.012
 5031P        0.002          0.000         -0.002          0.027
 6031P        0.016         -0.011         -0.006          0.099
```

```
Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2032P           0.052          0.059          0.049
 2033P           0.178          0.196          0.179
 2034P           0.206          0.191          0.213
 2035P           0.220          0.189          0.212
 2041P           0.002          0.001         -0.001
 2051P           0.009          0.009          0.009
 2061P           0.005          0.001          0.015
 2071P           0.014          0.006          0.020
 2431P           0.177          0.173          0.171
 2531P           0.121          0.129          0.131
 2631P           0.095          0.075          0.089
 2731P           0.092          0.084          0.084
 3031P           0.003          0.002          0.002
 4031P           0.010          0.012          0.010
 5031P           0.025          0.027          0.025
 6031P           0.032          0.088          0.093
```

TABLE E.2(d) REPEATABILITY COMPUTATIONS FOR SERIES 2 'E.P.' SET, 12 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION     STRAIN
 2032P      0.421      1684.
 2033P      1.299      5196.
 2034P      1.534      6136.
 2035P      1.584      6336.
 2041P      0.048       192.
 2051P      0.058       232.
 2061P      0.118       472.
 2071P      0.193       772.
 2431P      1.206      4824.
 2531P      0.972      3888.
 2631P      0.807      3228.
 2731P      0.695      2780.
 3031P      0.042       168.
 4031P      0.081       324.
 5031P      0.181       724.
 6031P      0.663      2652.


Mix Ref. EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
 No.                                                DIFERENCE
 2032P      0.011        -0.000        -0.011        0.063
 2033P     -0.009         0.009        0.            0.195
 2034P      0.000         0.026        -0.026        0.230
 2035P      0.058        -0.058        -0.000        0.238
 2041P     -0.000        -0.004         0.004        0.003
 2051P     -0.000        -0.000         0.000        0.009
 2061P      0.014        -0.014        -0.000        0.018
 2071P     -0.017         0.017        0.            0.029
 2431P      0.000        -0.010         0.010        0.181
 2531P      0.016        -0.016         0.000        0.146
 2631P     -0.003        0.             0.003        0.121
 2731P     0.            -0.009         0.009        0.104
 3031P     -0.002         0.002        -0.000        0.003
 4031P      0.003         0.000        -0.003        0.012
 5031P      0.003         0.000        -0.003        0.027
 6031P      0.013        -0.013        0.            0.099

Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2032P            0.052        0.063        0.052
 2033P            0.186        0.186        0.195
 2034P            0.230        0.204        0.204
 2035P            0.180        0.180        0.238
 2041P            0.003       -0.001       -0.001
 2051P            0.009        0.009        0.009
 2061P            0.004        0.004        0.018
 2071P            0.012        0.012        0.029
 2431P            0.181        0.171        0.171
 2531P            0.130        0.130        0.146
 2631P            0.118        0.121        0.118
 2731P            0.104        0.095        0.095
 3031P            0.001        0.001        0.003
 4031P            0.009        0.012        0.009
 5031P            0.024        0.027        0.024
 6031P            0.086        0.086        0.099
```

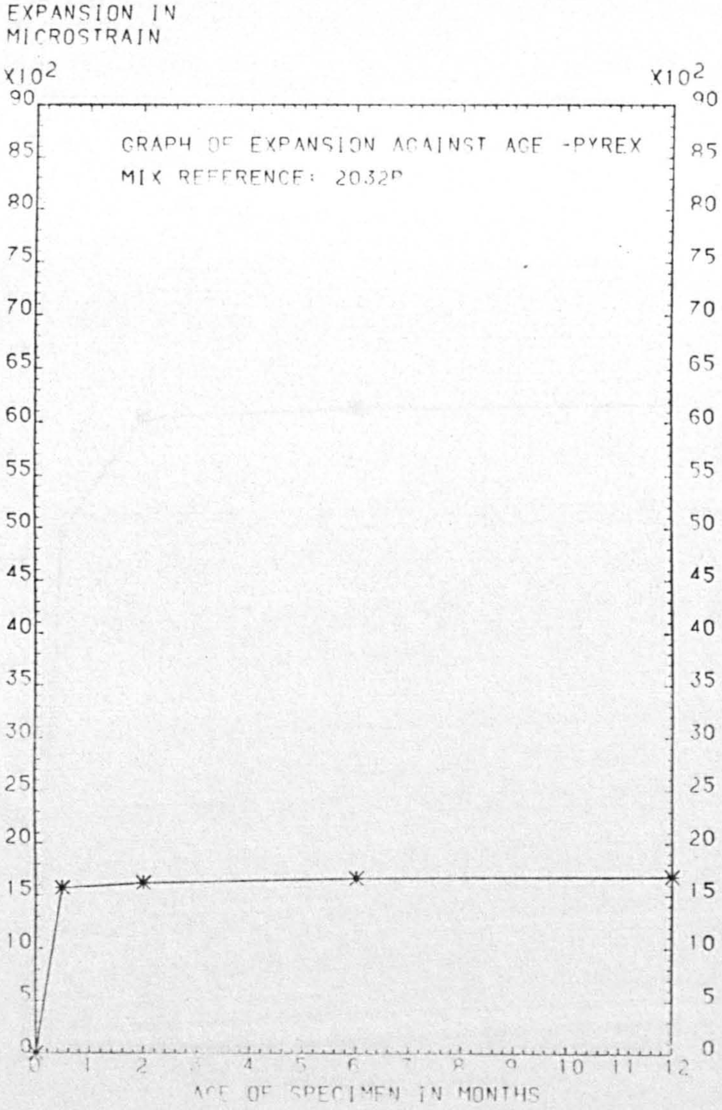

Figure E.1

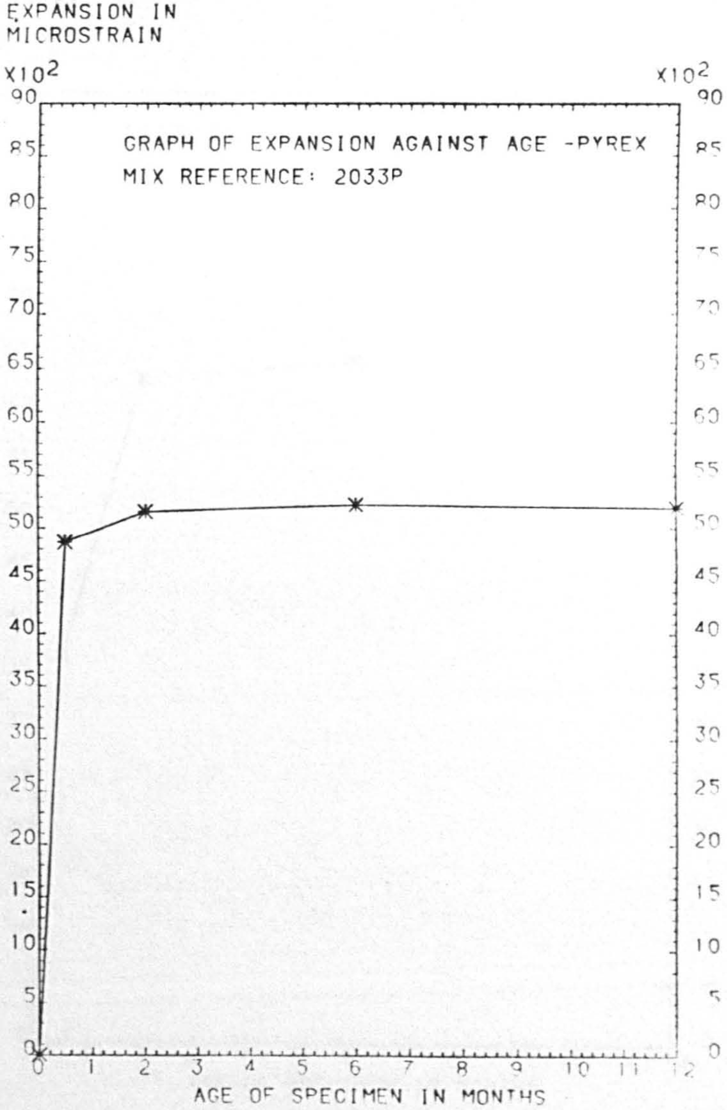

Figure E.2

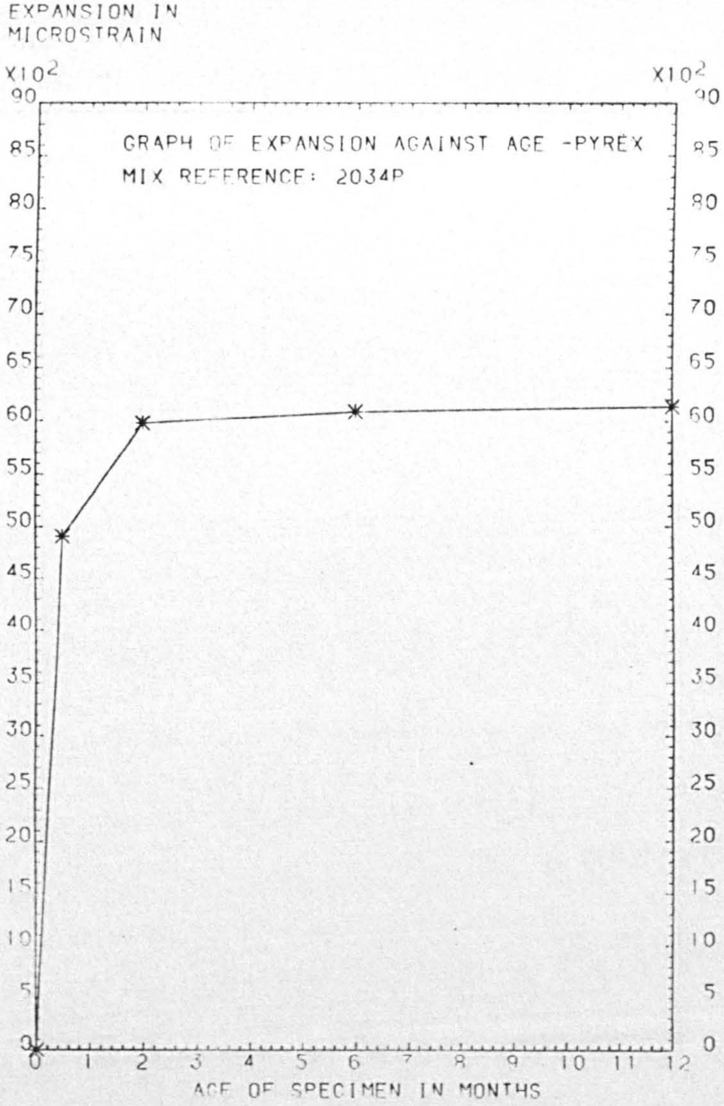

Figure E.3

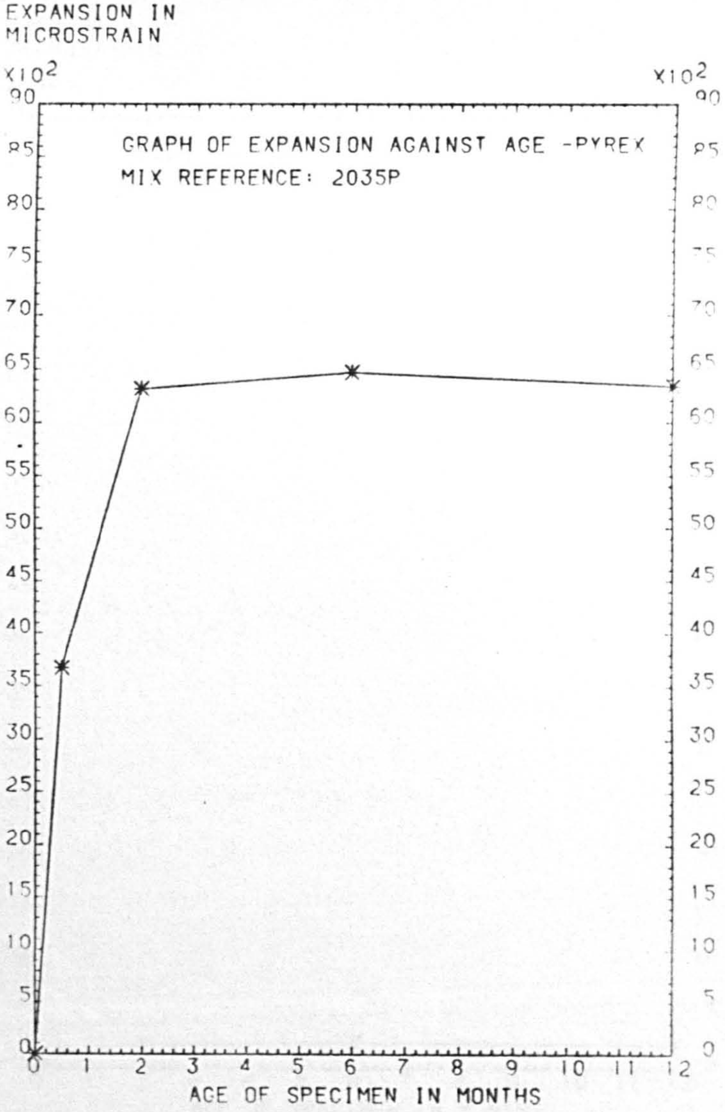

Figure E.4

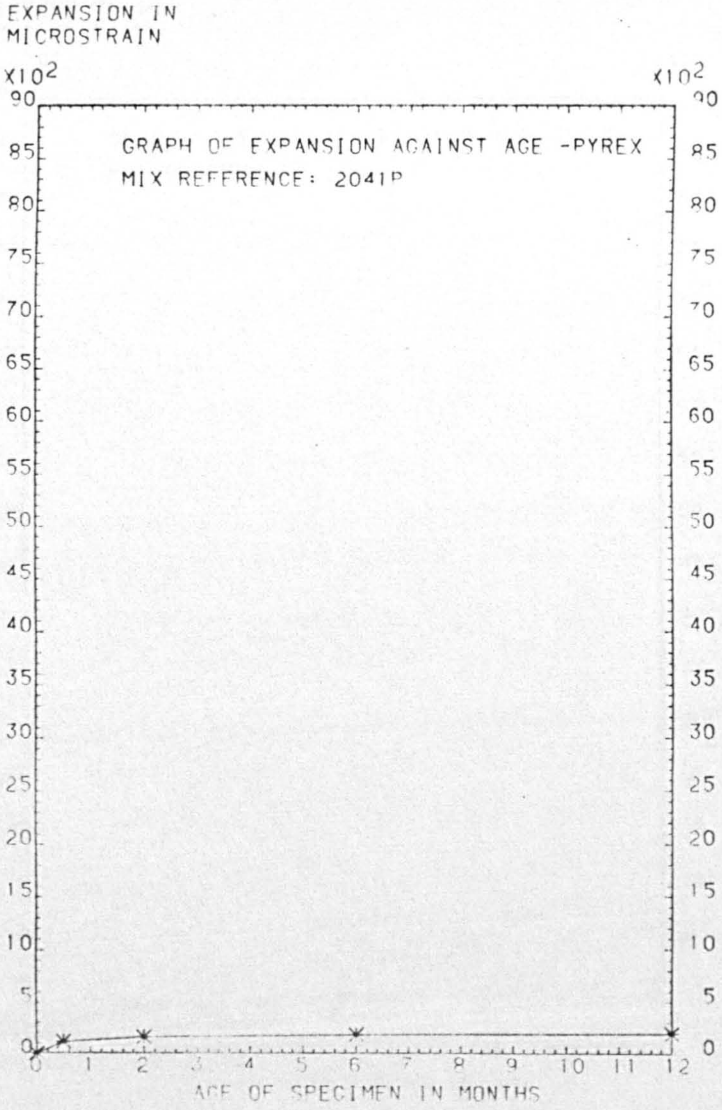

Figure E.5

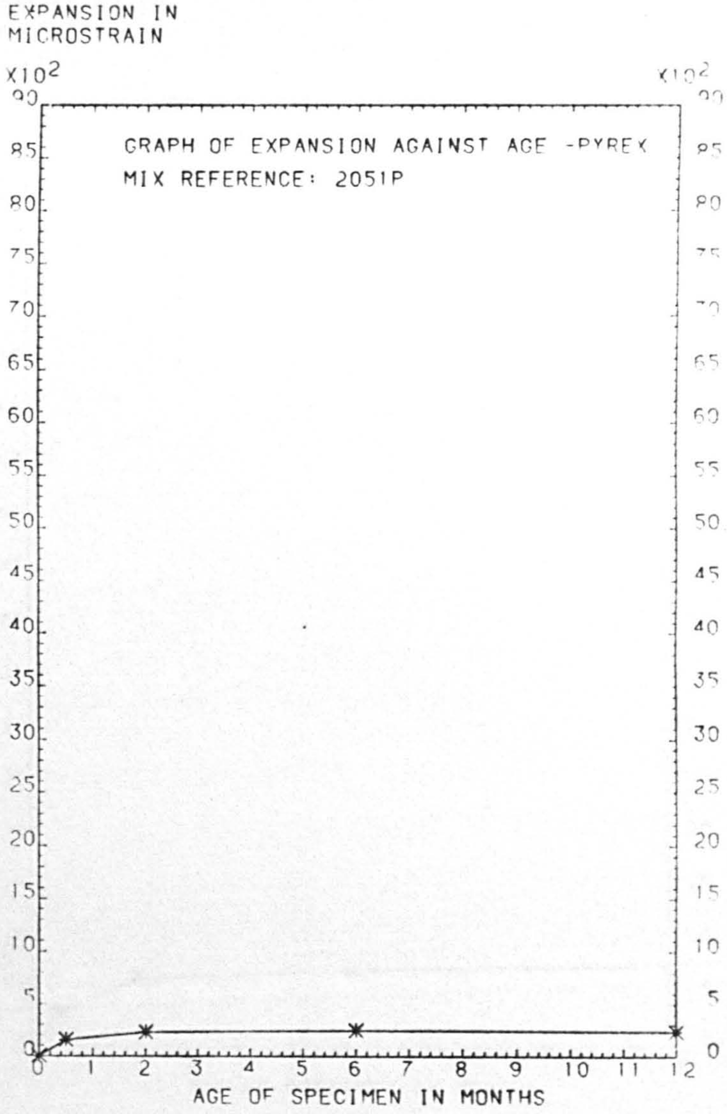

Figure E.6

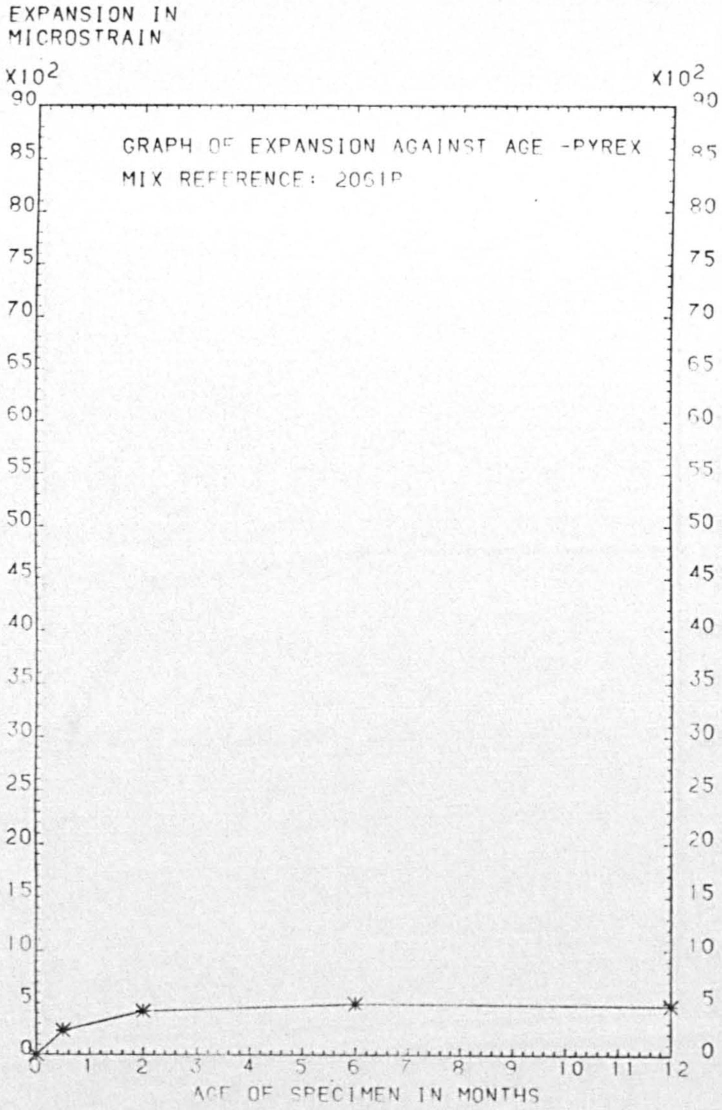

Figure E.7

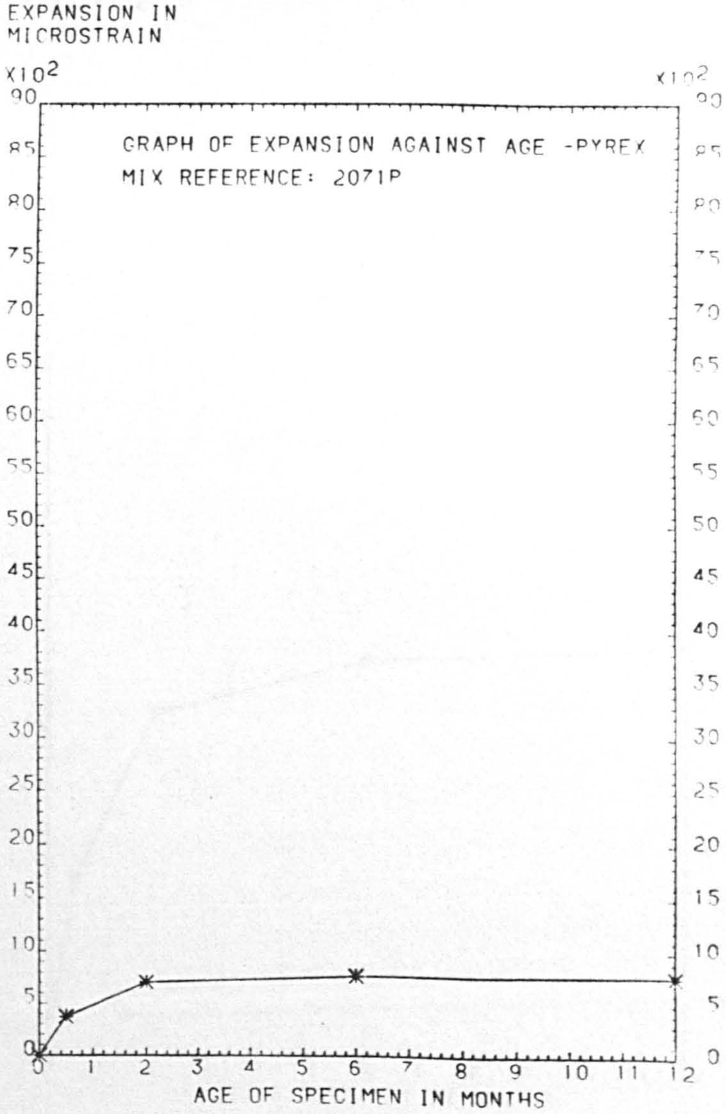

Figure E.8

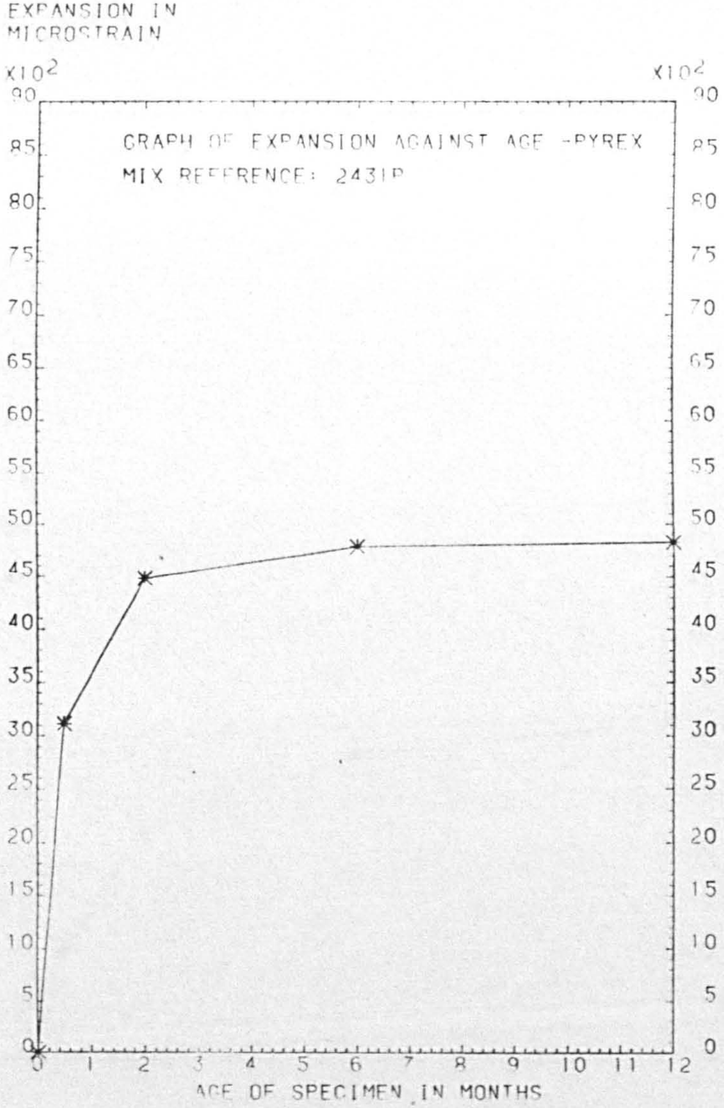

Figure E.9

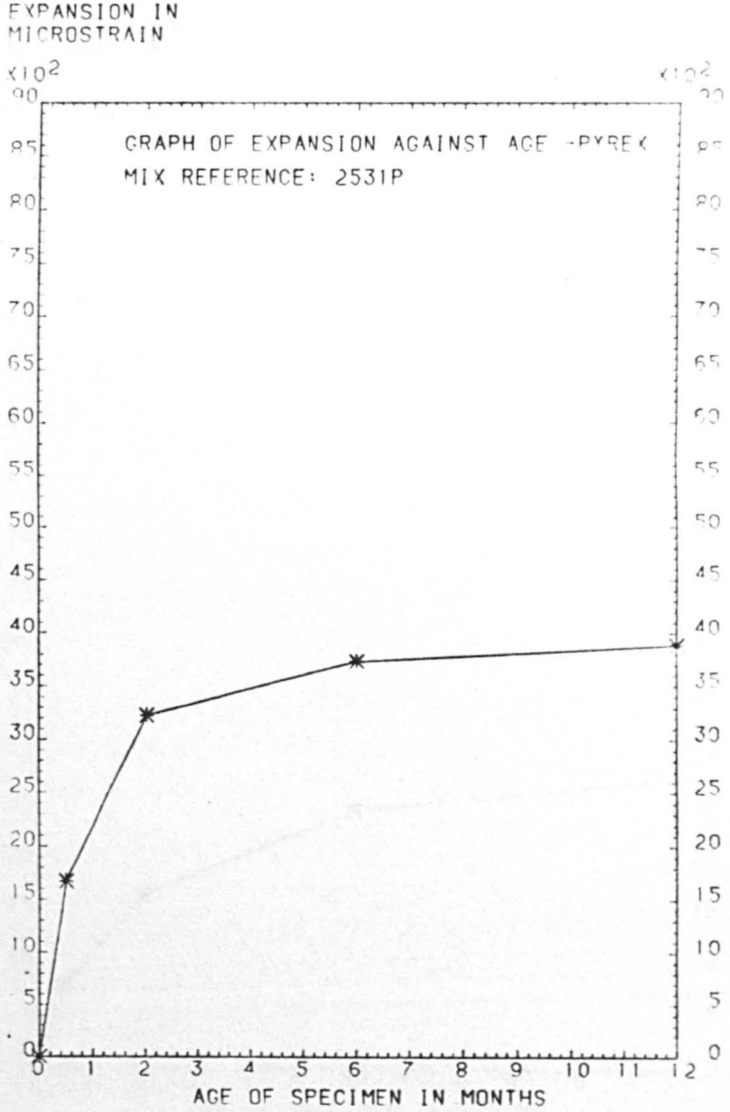

Figure E.10

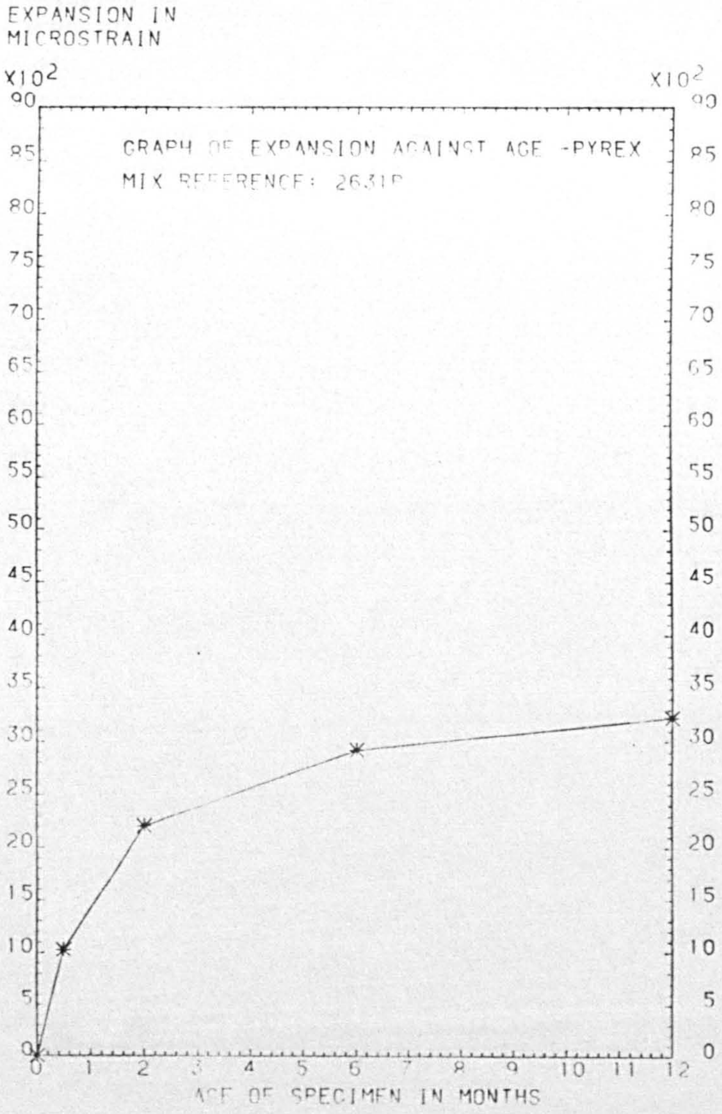

Figure E.11

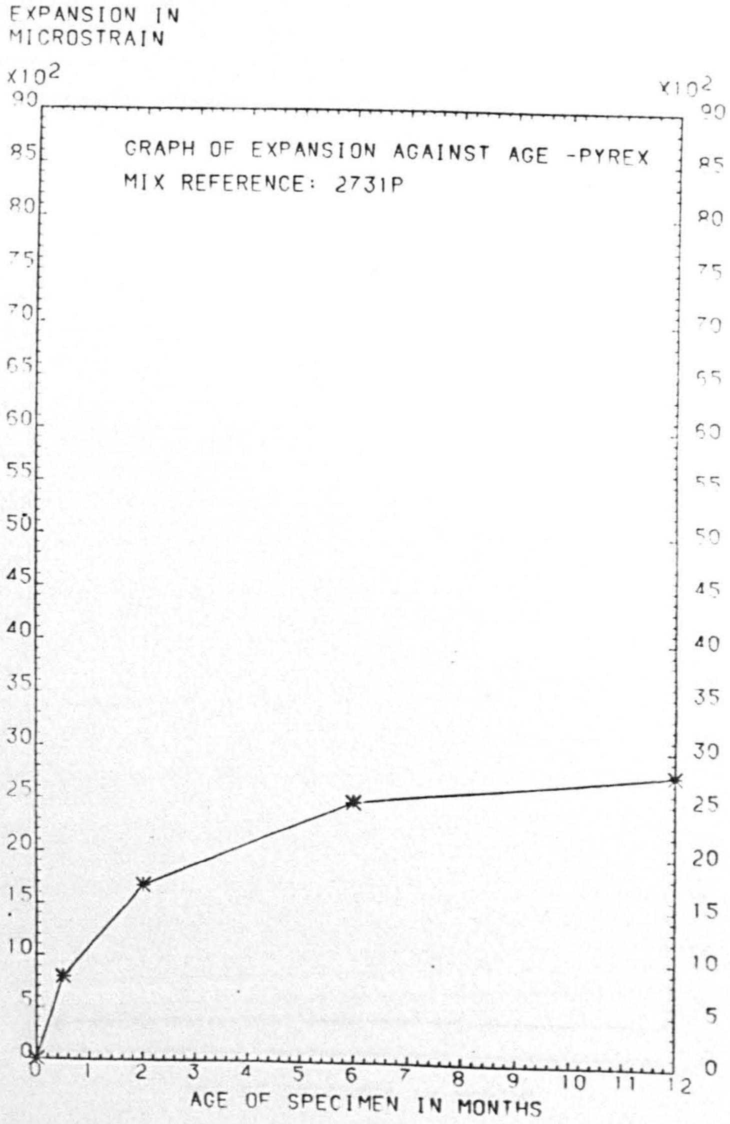

Figure E.12

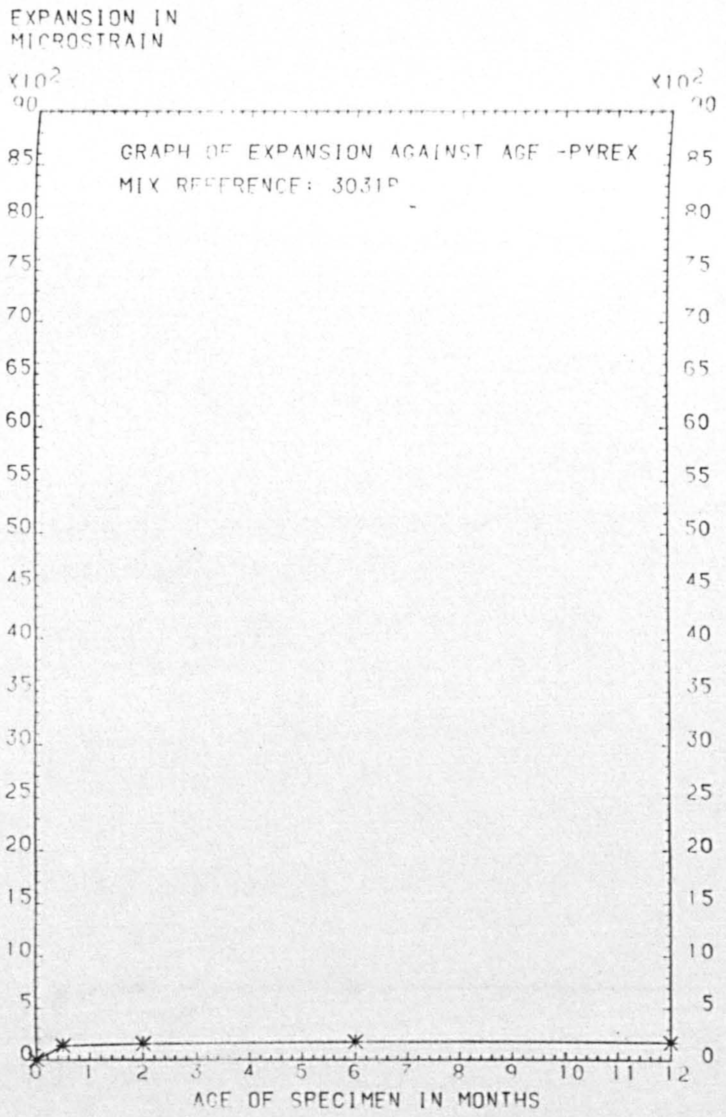

Figure E.13

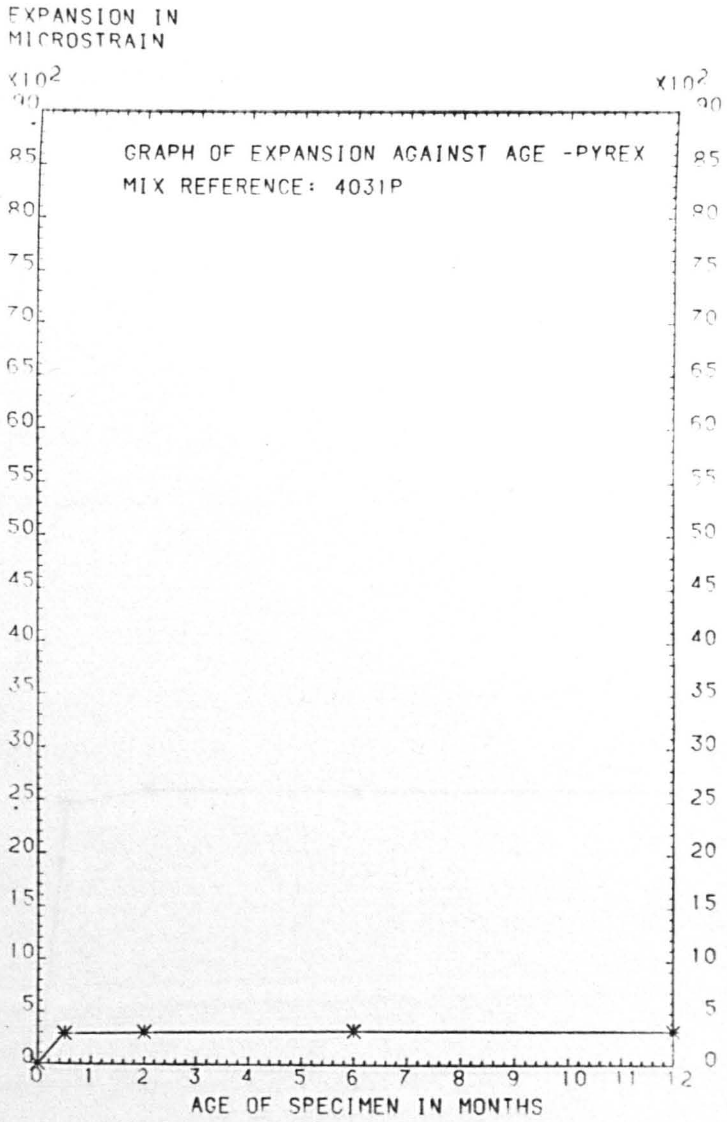

Figure E.14

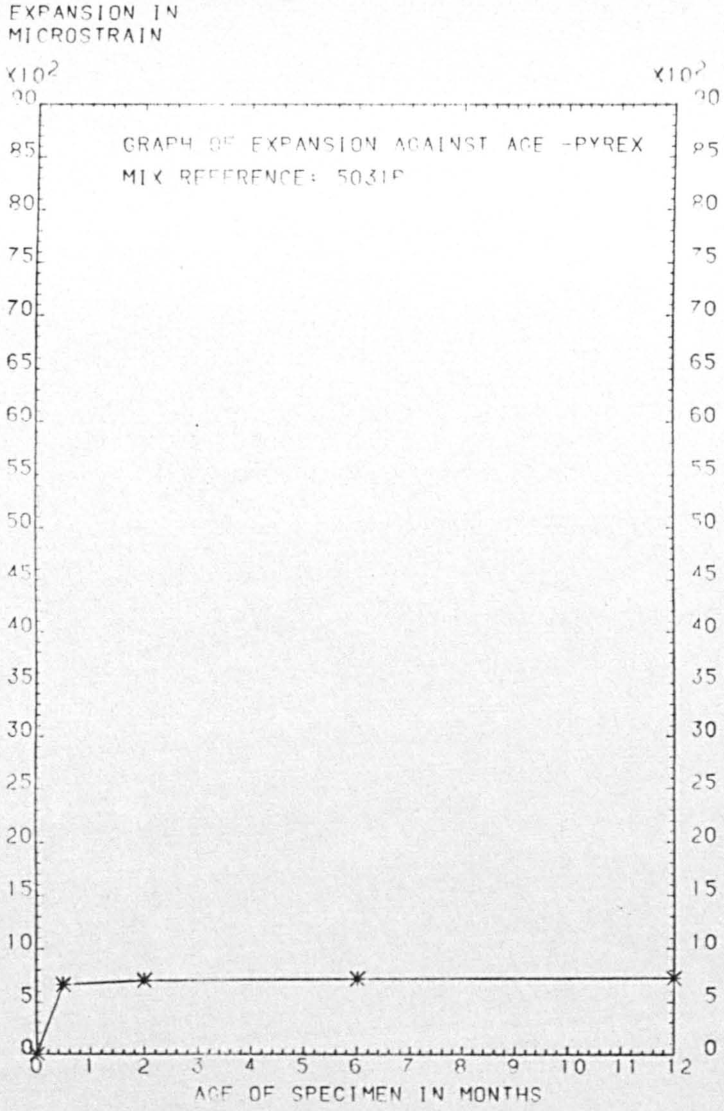

Figure E.15

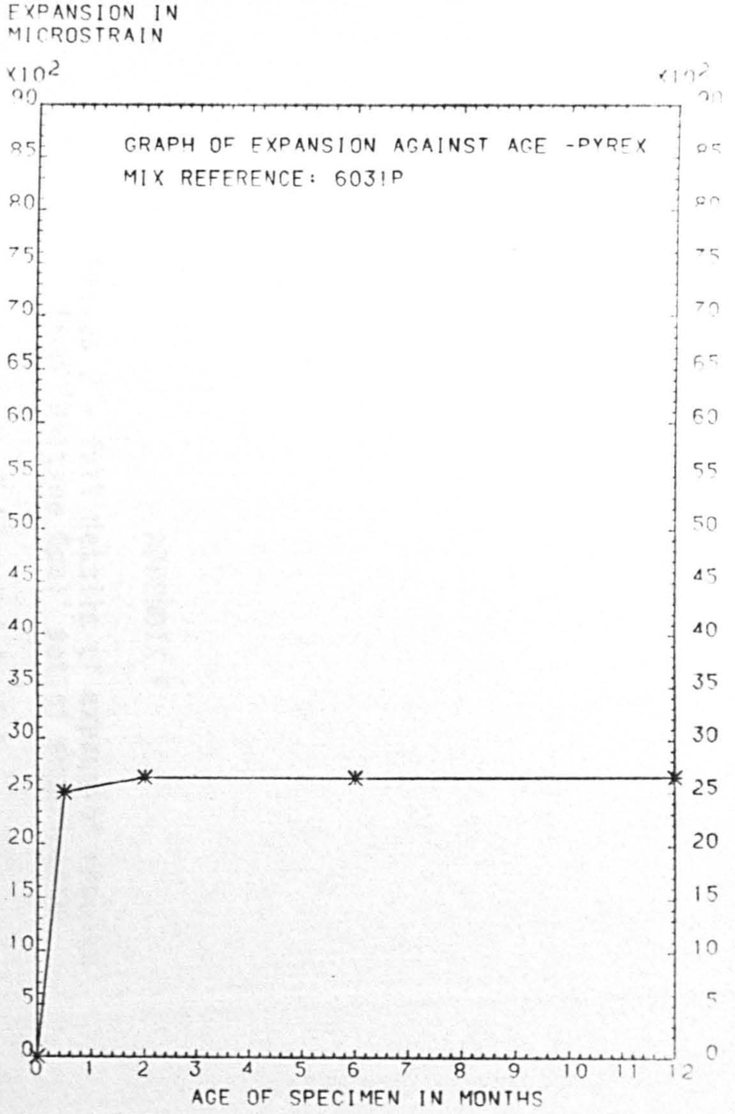

Figure E.16

APPENDIX F

Series 2 - full details of expansion results from 'Beltane Opal' set of experiments

Tables F.1 to F.2

Figures F.1 to F.16

TABLE F.1 SERIES 2 MORTAR BAR EXPANSION RESULTS - BELTANE OPAL MIXES

MIX REF NO.	EXPANSION IN MICROSTRAIN ($\mu\varepsilon$) AT AGE:-				'HARMFUL' REACTIVITY DESIGNATION **
	14 DAYS	2 MONTHS	6 MONTHS	12 MONTHS	
2001	92	95	105	88	-
2011 B	88	139	164	172	-
2021 B	84	172	216	236	-
2031 B	120	235	295	304	-
2041 B	276	493	529	528	-
2051 B	144	189*	200*	200	-
2061 B	112*	152+	159*	152	-
2071 B	109	140	153	156	-
2301	96	131	163	152	-
2311 B	112	238	262	264	-
2321 B	127	293	337	324	-
2331 B	160+	368	416	396	-
2341 B	129	219	251	268	-
2351 B	121*	223	247	248	-
2361 B	109	224	239	244	-
2371 B	92	223	252	224	-

Note:- * denotes result from 1 bar failed the ASTM repeatability test
+ denotes results from 2 bars failed the ASTM repeatability test
** denotes based on limit referred to in ASTM C227

TABLE F.2(a) REPEATABILITY COMPUTATIONS FOR SERIES 2 'B.O.' SET, 14 DAY RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
          Mix Ref.   AVERAGE     MICRO-
            No.     EXPANSION     STRAIN
          2041B      0.069        276.
          2051B      0.036        144.
          2061B      0.023        112.
          2071B      0.027        109.
          2011B      0.022         88.
          2021B      0.021         84.
          2031B      0.030        120.
          2341B      0.032        129.
          2351B      0.030        121.
          2361B      0.027        109.
          2371B      0.023         92.
          2311B      0.023        112.
          2321B      0.032        127.
          2331B      0.040        160.

 Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
  No.                                                 DIFFERENCE
 2041B        0.001         0.003        -0.004        0.010
 2051B       -0.002         0.000         0.002        0.003
 2061B       -0.004         0.002         0.002        0.003
 2071B       -0.001         0.001         0.001        0.003
 2011B       -0.002         0.000         0.002        0.003
 2021B        0.001        -0.001         0.000        0.003
 2031B        0.            0.            0.           0.003
 2341B       -0.001         0.002        -0.000        0.003
 2351B        0.004        -0.001        -0.002        0.003
 2361B        0.001        -0.001         0.001        0.003
 2371B       -0.001         0.001         0.000        0.003
 2311B       -0.000        -0.002         0.002        0.003
 2321B       -0.002         0.001         0.000        0.003
 2331B       -0.004         0.006        -0.002        0.003

Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
2041B         0.009         0.007         0.006
2051B         0.001         0.003         0.001
2061B        -0.001         0.001         0.001
2071B         0.002         0.002         0.002
2011B         0.001         0.003         0.001
2021B         0.002         0.002         0.003
2031B         0.003         0.003         0.003
2341B         0.002         0.001         0.003
2351B        -0.001         0.002         0.001
2361B         0.002         0.002         0.002
2371B         0.002         0.002         0.003
2311B         0.003         0.001         0.001
2321B         0.001         0.002         0.003
2331B        -0.001        -0.003         0.001
```

TABLE F.2(b) REPEATABILITY COMPUTATIONS FOR SERIES 2 'B.O.' SET, 2 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE    MICRO-
  No.     EXPANSION    STRAIN
 2041B      0.123       493.
 2051B      0.047       189.
 2061B      0.038       152.
 2071B      0.035       140.
 2011B      0.035       139.
 2021B      0.043       172.
 2031B      0.059       235.
 2341B      0.055       219.
 2351B      0.056       223.
 2361B      0.056       224.
 2371B      0.056       223.
 2311B      0.059       238.
 2321B      0.073       293.
 2331B      0.092       368.


Mix Ref. EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
  No.                                               DIFFERENCE
 2041B      -0.005         0.013        -0.007        0.019
 2051B      -0.005         0.003         0.003        0.003
 2061B      -0.004         0.004         0.           0.003
 2071B      -0.001         0.001        -0.000        0.003
 2011B      -0.001         0.001        -0.001        0.003
 2021B       0.001        -0.001         0.000        0.003
 2031B       0.000        -0.001         0.000        0.009
 2341B      -0.002         0.003        -0.002        0.008
 2351B       0.004        -0.001        -0.004        0.008
 2361B       0.002         0.           -0.002        0.008
 2371B       0.000         0.000        -0.001        0.008
 2311B       0.000         0.000        -0.001        0.009
 2321B       0.001        -0.000        -0.000        0.011
 2331B      -0.004         0.006        -0.002        0.014

Mix Ref.
 No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2041B            0.013         0.006         0.011
 2051B           -0.002         0.000         0.000
 2061B           -0.001        -0.001         0.003
 2071B            0.002         0.002         0.003
 2011B            0.002         0.002         0.002
 2021B            0.002         0.002         0.003
 2031B            0.003         0.008         0.008
 2341B            0.007         0.005         0.007
 2351B            0.004         0.008         0.005
 2361B            0.006         0.008         0.006
 2371B            0.003         0.008         0.008
 2311B            0.009         0.008         0.008
 2321B            0.010         0.011         0.011
 2331B            0.010         0.008         0.012
```

TABLE F.2(c) REPEATABILITY COMPUTATIONS FOR SERIES 2 'B.O.' SET, 6 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
Mix Ref.   AVERAGE     MICRO-
  No.     EXPANSION      STRAIN
 2041B      0.132        529.
 2051B      0.050        200.
 2061B      0.040        159.
 2071B      0.038        153.
 2011B      0.041        164.
 2021B      0.054        216.
 2031B      0.074        295.
 2341B      0.063        251.
 2351B      0.062        247.
 2361B      0.060        239.
 2371B      0.063        252.
 2311B      0.065        262.
 2321B      0.034        337.
 2331B      0.104        416.
```

```
Mix Ref.  EXPN(1)-AVGE   EXPN(2)-AVGE   EXPN(3)-AVGE   ALLOWABLE
  No.                                                   DIFFERENCE
 2041B       -0.008          0.012         -0.003         0.020
 2051B       -0.003          0.002          0.001         0.003
 2061B       -0.004          0.003          0.000         0.003
 2071B        0.001         -0.001          0.001         0.003
 2011B       -0.002          0.001          0.001         0.003
 2021B        0.002         -0.001         -0.001         0.008
 2031B        0.000         -0.001          0.000         0.011
 2341B       -0.004          0.003          0.000         0.009
 2351B        0.005         -0.002         -0.004         0.009
 2361B        0.000         -0.001          0.000         0.009
 2371B       -0.001          0.003         -0.002         0.009
 2311B        0.000          0.000         -0.000         0.010
 2321B       -0.000         -0.000          0.001         0.013
 2331B       -0.007          0.007          0.             0.016
```

```
Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
 2041B         0.012          0.008          0.017
 2051B        -0.000          0.001          0.002
 2061B        -0.001         -0.000          0.003
 2071B         0.002          0.002          0.002
 2011B         0.001          0.002          0.002
 2021B         0.006          0.007          0.007
 2031B         0.011          0.010          0.011
 2341B         0.006          0.006          0.009
 2351B         0.004          0.008          0.006
 2361B         0.009          0.008          0.009
 2371B         0.008          0.006          0.007
 2311B         0.010          0.009          0.009
 2321B         0.012          0.012          0.012
 2331B         0.009          0.009          0.016
```

TABLE F.2(d) REPEATABILITY COMPUTATIONS FOR SERIES 2 'B.O.' SET, 12 MONTH RESULTS - OUTPUT FROM PROGRAM 'CHECK'

```
              Mix Ref.   AVERAGE    MICRO-
                No.     EXPANSION    STRAIN
               2041B      0.132      528.
               2051B      0.050      200.
               2061B      0.038      152.
               2071B      0.039      156.
               2011B      0.043      172.
               2021B      0.059      236.
               2031B      0.076      304.
               2341B      0.067      268.
               2351B      0.062      248.
               2361B      0.061      244.
               2371B      0.056      224.
               2311B      0.066      264.
               2321B      0.081      324.
               2331B      0.099      396.

    Mix Ref.  EXPN(1)-AVGE  EXPN(2)-AVGE  EXPN(3)-AVGE  ALLOWABLE
     No.                                                 DIFERENCE
     2041B        0.             0.004        -0.004       0.020
     2051B       -0.002          0.002         0.000       0.003
     2061B       -0.000          0.000         0.          0.003
     2071B        0.003         -0.003        -0.000       0.003
     2011B       -0.000          0.003        -0.003       0.003
     2021B        0.003         -0.003         0.000       0.009
     2031B        0.002         -0.002         0.          0.011
     2341B       -0.003          0.003         0.          0.010
     2351B        0.008         -0.008         0.          0.009
     2361B        0.001         -0.001         0.000       0.009
     2371B       -0.002          0.002         0.000       0.008
     2311B        0.000          0.             0.          0.010
     2321B       -0.001          0.001        -0.000       0.012
     2331B       -0.007          0.007         0.000       0.015

 Mix Ref.
  No.     CHECK , IF OKAY ALL VALUES SHOULD BE POSITIVE
  2041B            0.020          0.016          0.016
  2051B            0.001          0.001          0.003
  2061B            0.003          0.003          0.003
  2071B            0.000          0.000          0.003
  2011B            0.003         -0.000          0.000
  2021B            0.006          0.006          0.009
  2031B            0.009          0.009          0.011
  2341B            0.007          0.007          0.010
  2351B            0.001          0.001          0.009
  2361B            0.003          0.008          0.009
  2371B            0.006          0.006          0.008
  2311B            0.010          0.010          0.010
  2321B            0.011          0.011          0.012
  2331B            0.008          0.008          0.015
```

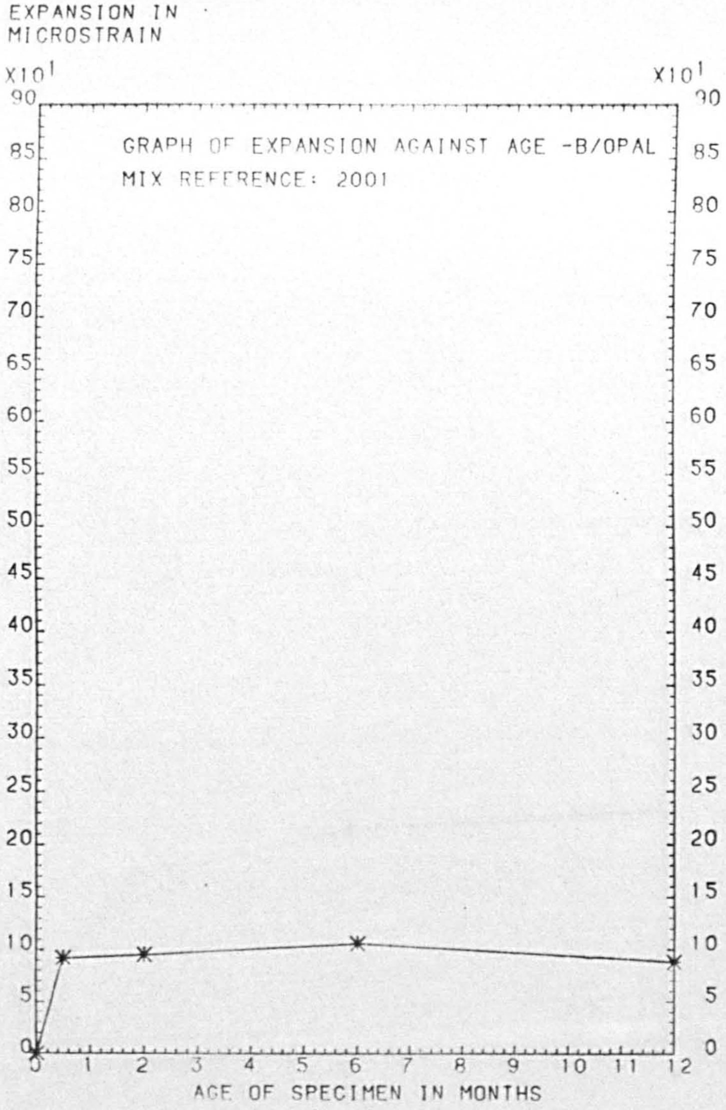

Figure F.1

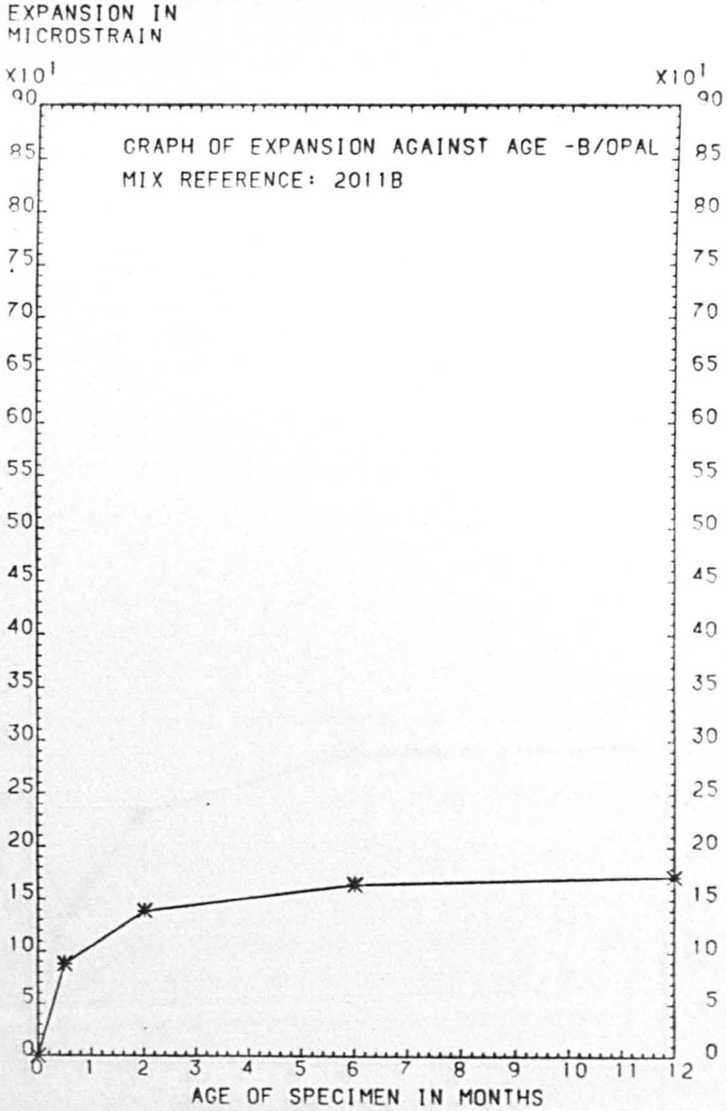

Figure F.2

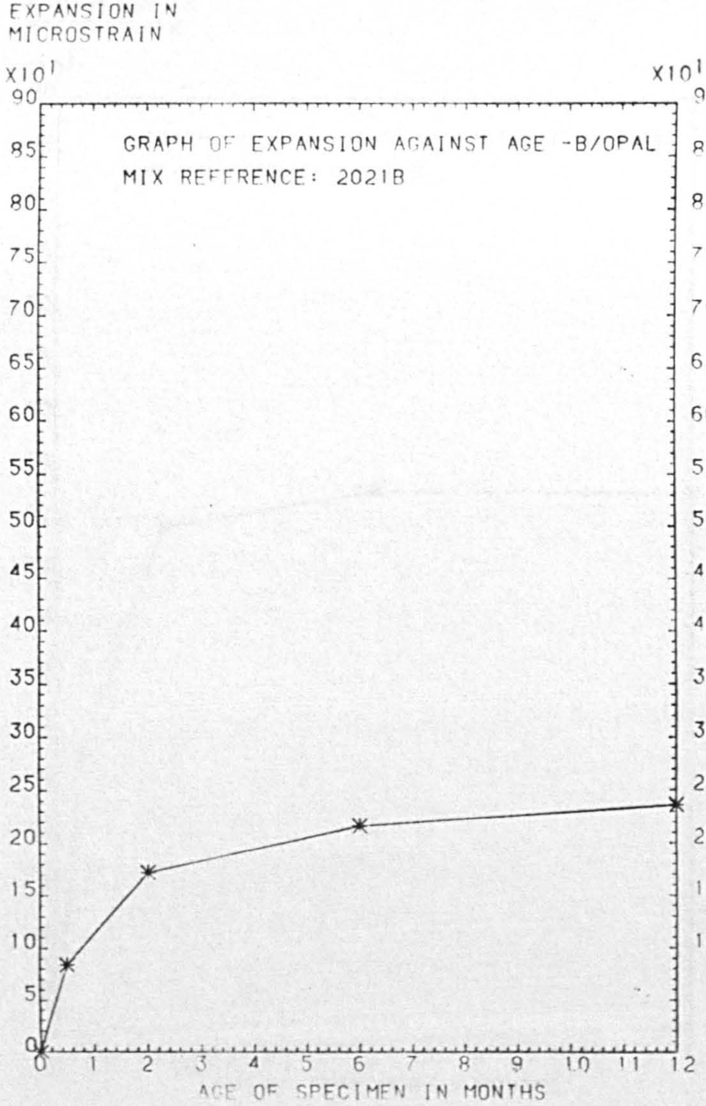

Figure F.3

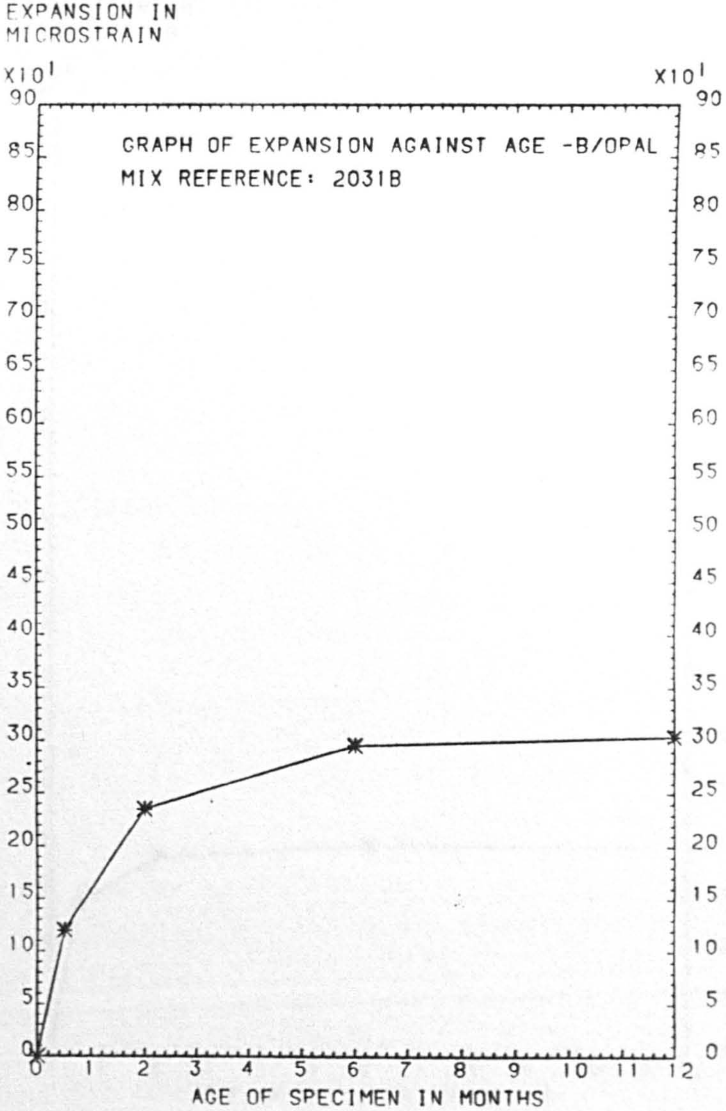

Figure F.4

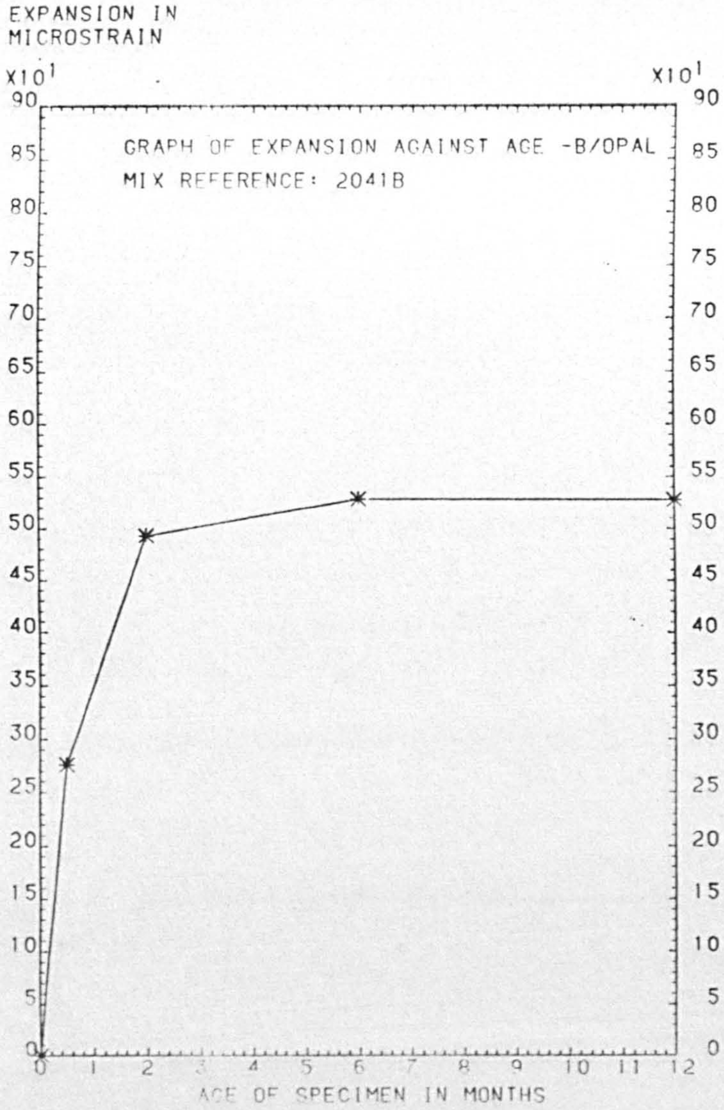

Figure F.5

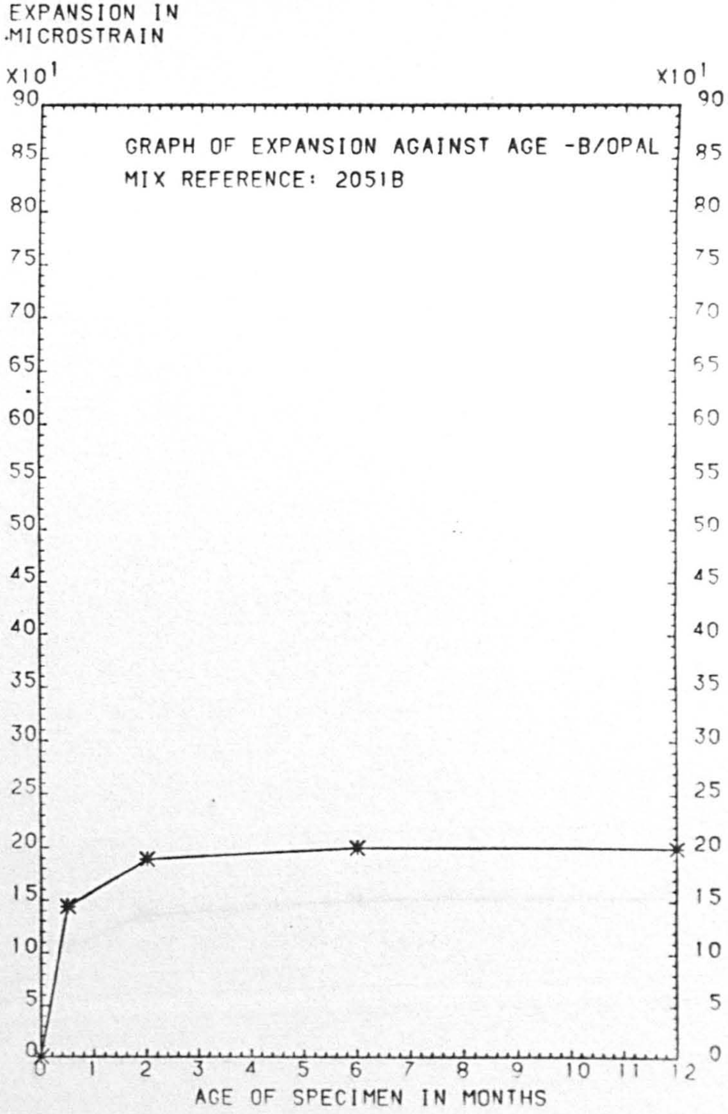

Figure F.6

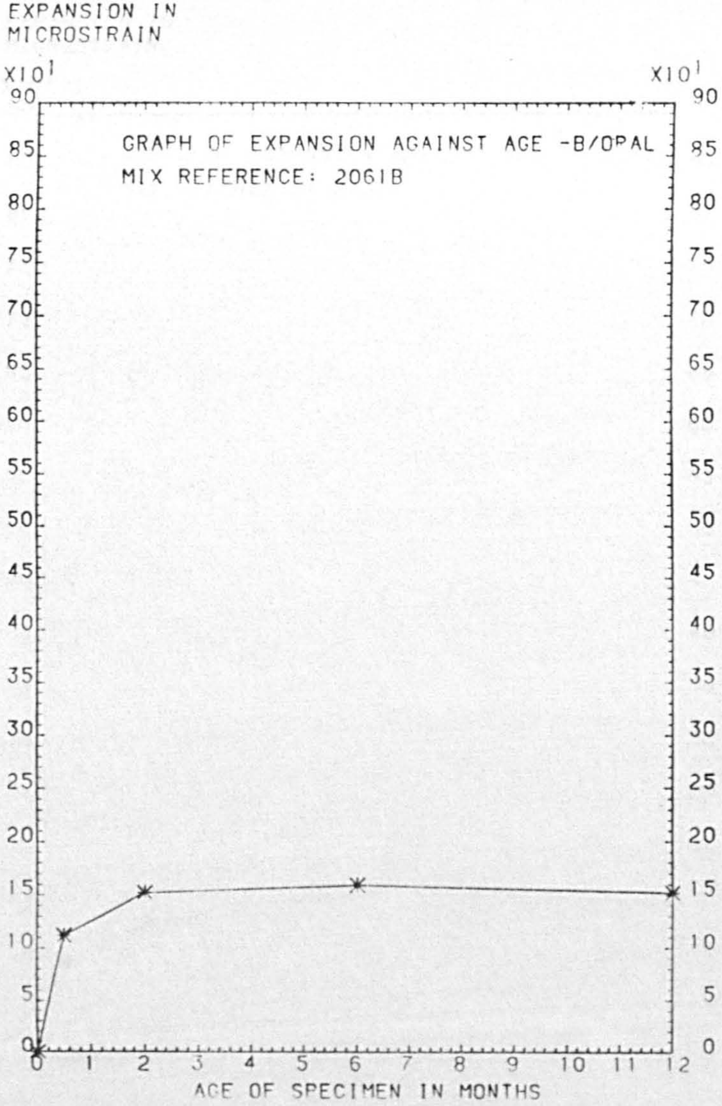

Figure F.7

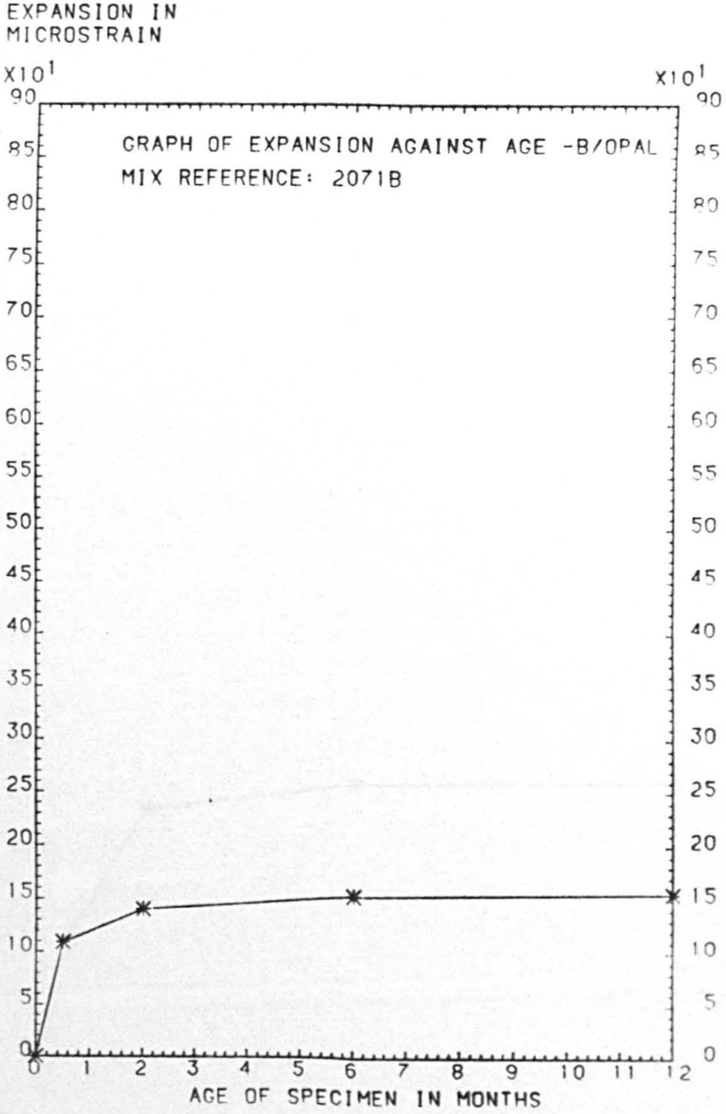

Figure F.8

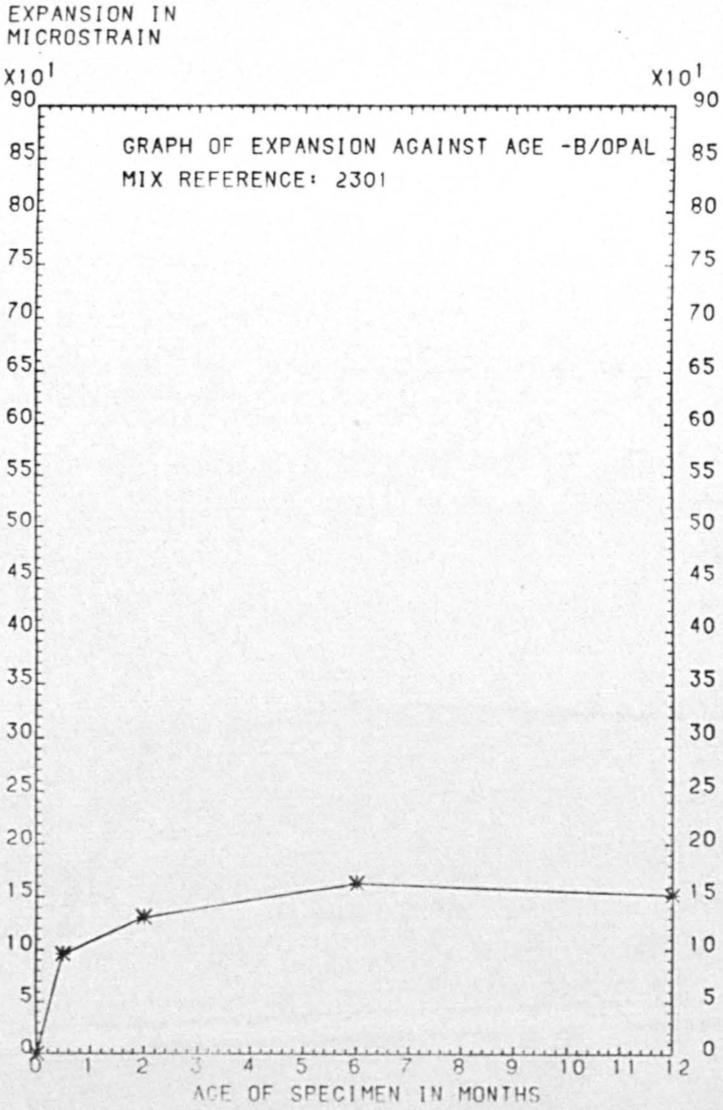

Figure F.9

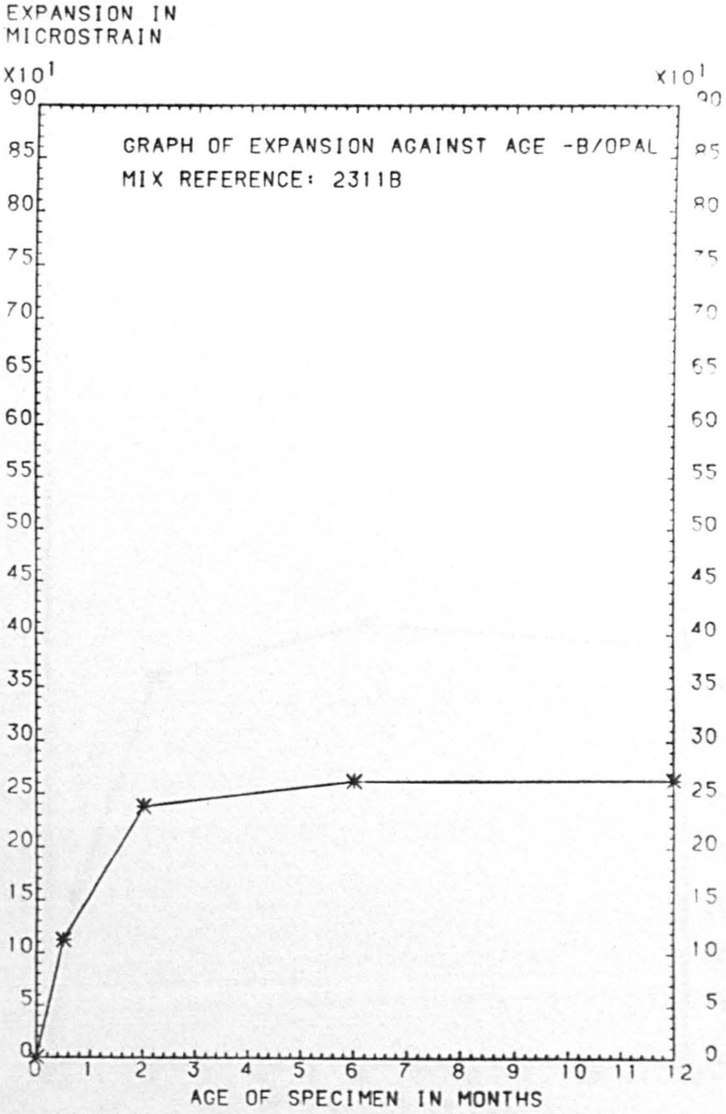

Figure F.10

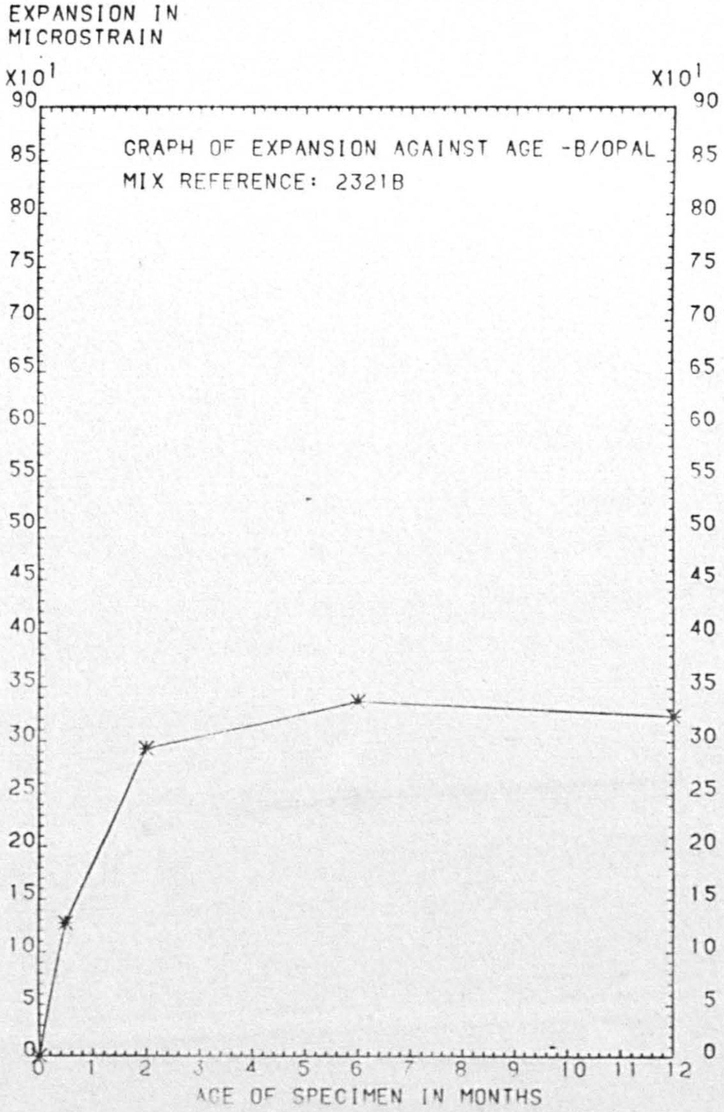

Figure F.11

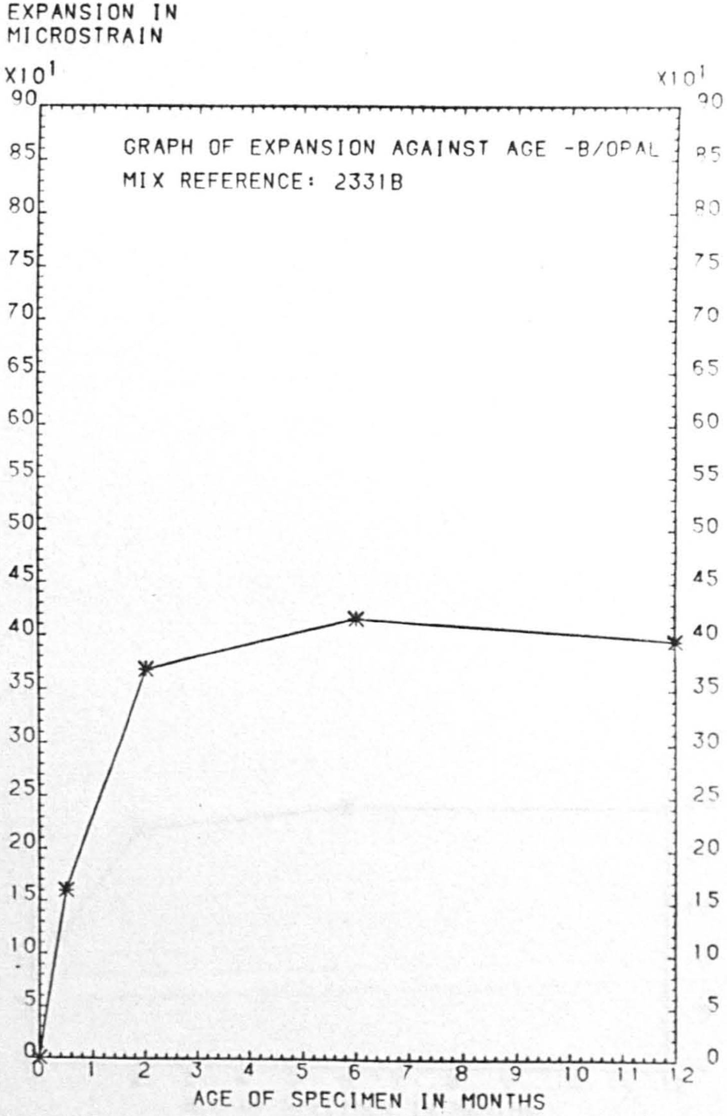

Figure F.12

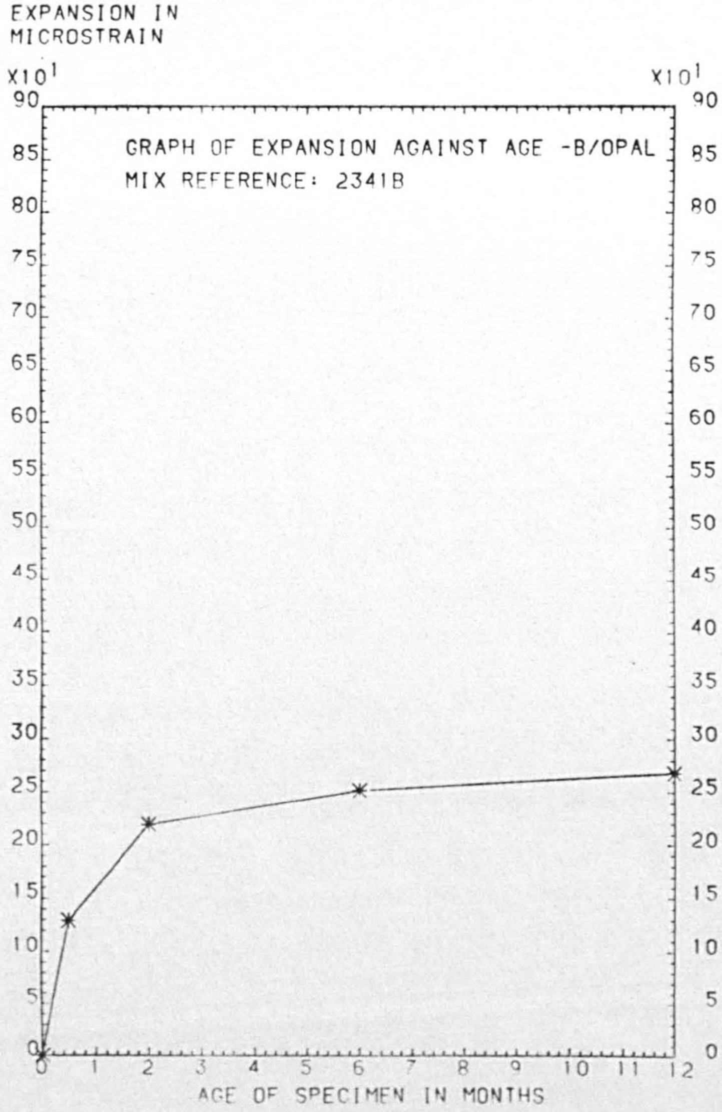

Figure F.13

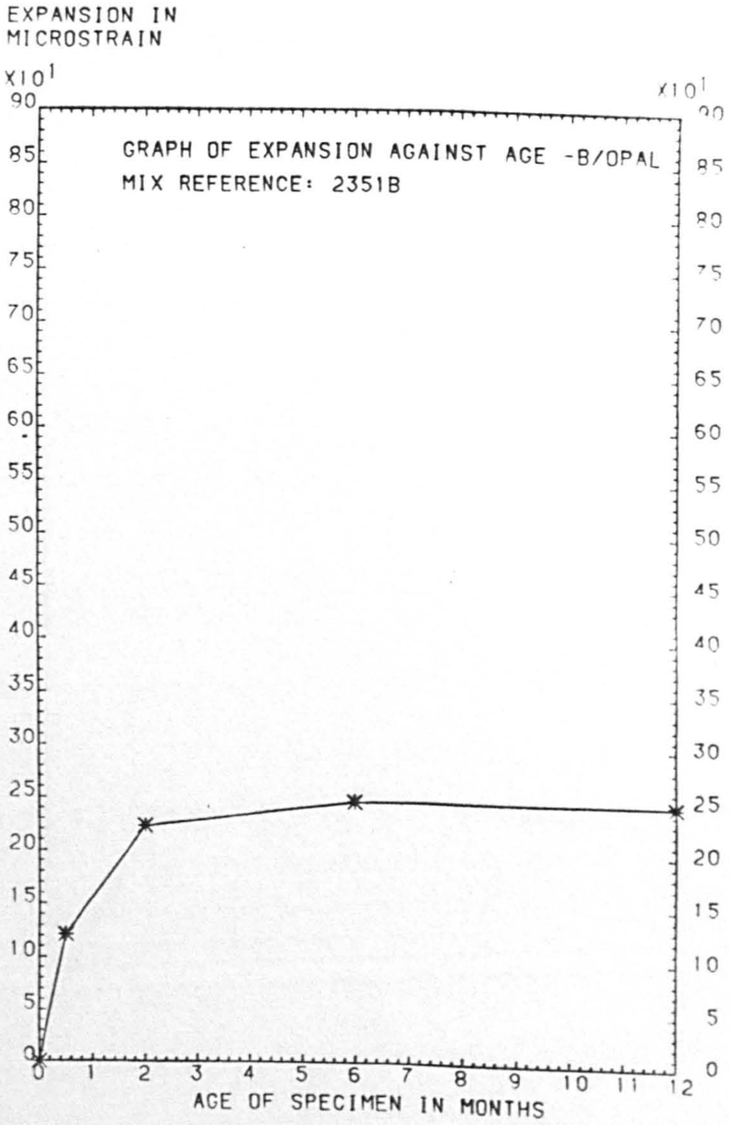

Figure F.14

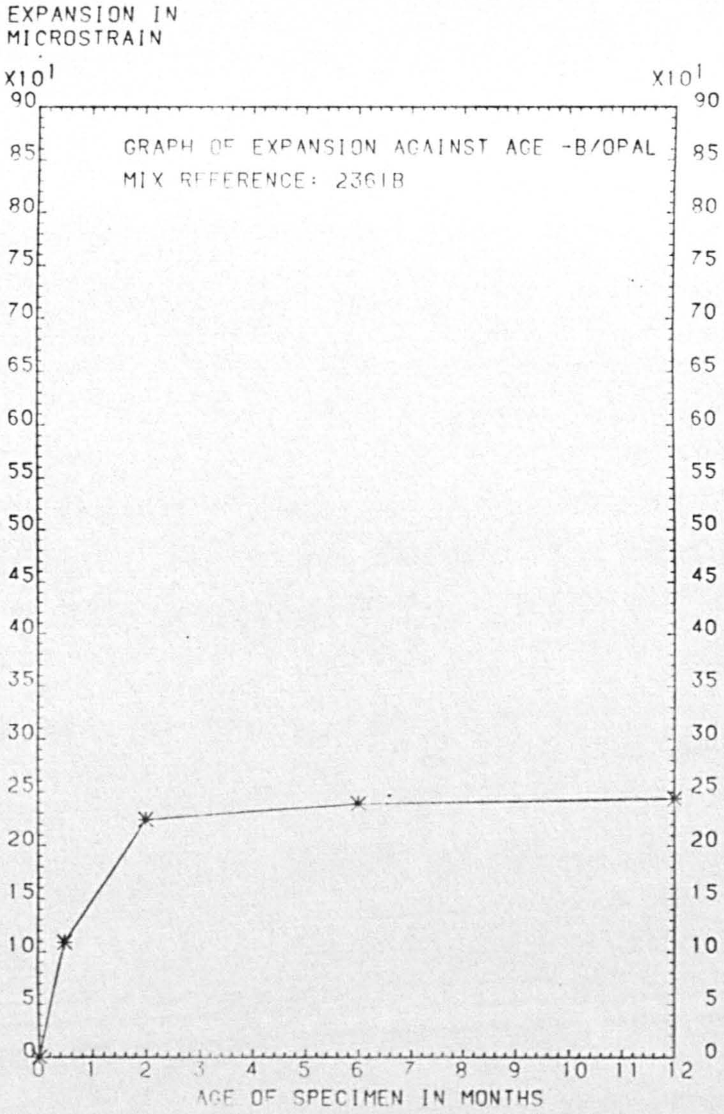

Figure F.15

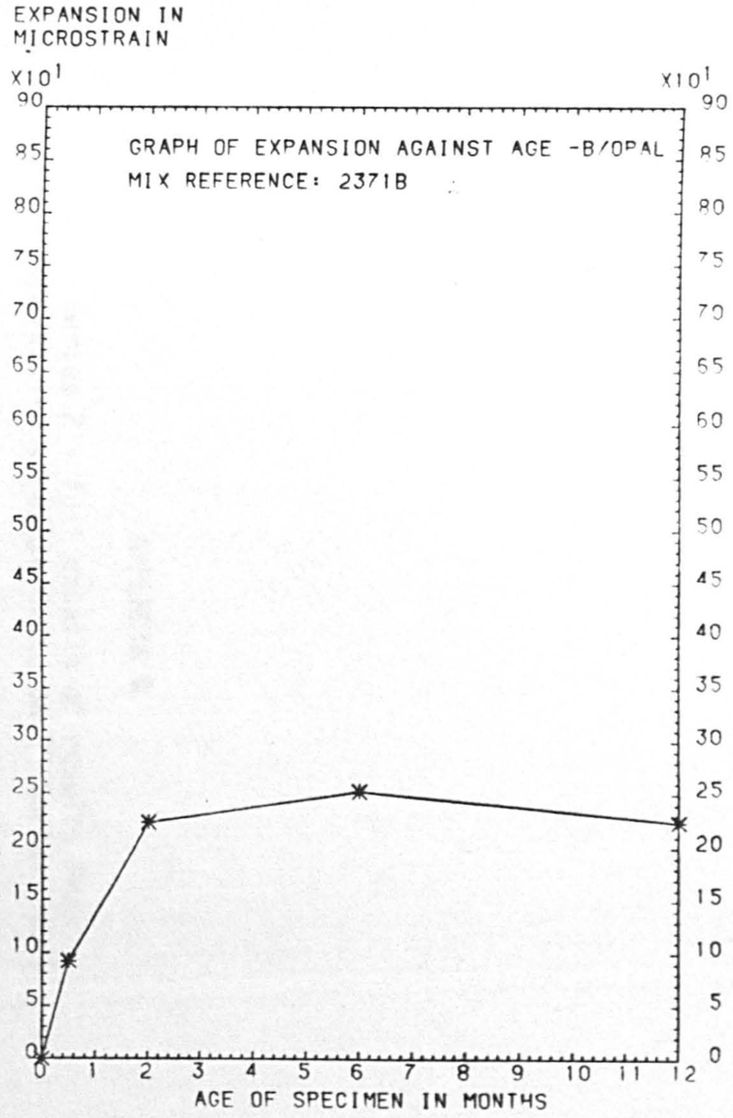

Figure F.16

APPENDIX G

Series 2 - full details of results from compressive strength tests on 2" mortar cubes

Table G.1

TABLE G.1 RESULTS OF COMPRESSION TESTS ON 2" CUBE SPECIMENS

MIX REF NO.	COMPRESSIVE STRENGTH (N/mm^2)				MEASURED WET DENSITY (kg/m^3)
	28 DAYS *	14 DAYS $^+$	2 MONTHS $^+$	6 MONTHS$^+$	
2000	51.4	48.5	48.8	44.0	2225
2001	40.1	25.7	34.2	30.7	2135
2010 P	50.6	49.2	56.5	50.4	2155
2011 P	35.6	31.3	36.1	33.3	2080
2020 P	44.1	45.2	52.9	45.7	2100
2021 P	35.9	29.5	35.7	33.4	2020
2030 P	38.6	43.8	51.8	45.0	2040
2031 P	29.0	31.5	37.8	34.0	1965
2100	56.8	59.4	66.6	60.7	2235
2101	38.9	38.0	45.7	43.5	2145
2110 P	46.2	57.7	67.2	60.5	2140
2111 P	35.9	40.1	48.2	43.0	2080
2120 P	43.2	57.2	64.2	58.8	2085
2121 P	33.7	39.1	49.5	44.9	2040
2130 P	37.5	52.8	59.9	55.2	2020
2131 P	29.5	40.1	48.3	44.2	2005
2200	45.2	60.7	72.5	69.8	2210
2201	26.4	40.7	52.5	50.9	2160
2210 P	40.3	59.5	68.4	63.9	2140
2211 P	25.9	39.4	51.8	49.0	2095
2220 P	37.0	57.8	65.4	59.3	2075
2221 P	26.6	40.8	52.2	50.0	2075
2230 P	34.9	55.8	62.0	57.2	2030
2231 P	24.5	41.3	50.2	47.6	2005
2300	31.0	49.7	62.3	59.5	2175
2301	19.2	30.4	39.9	41.0	2135
2310 P	33.1	52.3	61.5	60.4	2141
2311 P	18.1	33.1	42.9	41.7	2095
2320 P	28.5	49.2	56.4	56.5	2060
2321 P	19.7	32.4	41.7	41.6	2050
2330 P	27.7	48.1	56.0	53.1	2010
2331 P	18.6	31.5	38.5	37.5	1995

Note:- * cured at 20°C under water
+ cured at 37.8°C enclosed in polythene film

APPENDIX H

Computational procedures used in the analysis of variance for the results from a factorial experiment

APPENDIX H COMPUTATIONAL PROCEDURES USED IN THE ANALYSIS OF THE RESULTS FROM A FACTORIAL EXPERIMENT

H.1 INTRODUCTION TO COMPUTATIONS

In Chapter 4, Section 4.2.3, the theory underlying the analysis of variance (ANOVA) for a set of results from a factorial experiment was outlined. In particular, for the two factor experiment of dimensions p x q with n observations per cell which was chosen to demonstrate the theory, the following mean squares were defined:-

Mean Square due to the main effects of factor A

$$MS_a = \frac{SS_a}{df} = \frac{nq \sum_i (\bar{A}_i - \bar{G})^2}{p - 1}$$

Mean Square due to the main effects of factor B

$$MS_b = \frac{SS_b}{df} = \frac{np \sum_j (\bar{B}_j - \bar{G})^2}{q - 1}$$

Mean Square due to the interaction effects

$$MS_{ab} = \frac{SS_{ab}}{df} = \frac{n\sum_{ij}\sum(\overline{AB}_{ij} - \bar{A}_i - \bar{B}_j + \bar{G})^2}{(p - 1)(q - 1)}$$

Within cell Mean Square (error)

$$MS_{w.cell} = \frac{SS_{w.cell}}{df} = \frac{\sum_{ijk}\sum\sum(X_{ijk} - \overline{AB}_{ij})^2}{pq(n-1)}$$

where the notation is as described in Chapter 4.

It can be seen that each mean square can be separated into two components, namely a sum of squares divided by the degrees of freedom. This enables the analysis method to be simplified by calculating the two components individually before combining them to obtain the required mean squares. To this end the computations are carried out in a step-by-step fashion in order to complete the following ANOVA table:-

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	'F' (Mean Square ratio)	Sig Level or Tail Prob
A	SS_a	(p-1)	$MS_a=\frac{SS_a}{(p-1)}$	$\frac{MS_a}{MS_{w.cell}}$	
B	SS_b	(q-1)	$MS_b=\frac{SS_b}{(q-1)}$	$\frac{MS_b}{MS_{w.cell}}$	
AB	SS_{ab}	(p-1)(q-1)	$MS_{ab}=\frac{SS_{ab}}{(p-1)(q-1)}$	$\frac{MS_{ab}}{MS_{w.cell}}$	
Exp.Error (Within Cell)	$SS_{w.cell}$	pq(n-1)	$MS_{w.cell} =\frac{SS_{w.cell}}{pq(n-1)}$		
Total	SS_{total}	pqn-1			

H.2 COMPUTATIONAL SYMBOLS AND FORMULAE

The definitions of the sums of squares incorporated in the mean squares given above are presented in their most concise form. However, for computation purposes a more suitable algebraic form is available. Take, for example, the sum of squares due to the main effects of factor A:-

$$SS_a = nq\sum_i (\bar{A}_i - \bar{G})^2$$

It can easily be shown that an equivalent definition is:-

$$SS_a = \frac{\sum_i A_i^2}{nq} - \frac{G^2}{npq}$$

similarly

$$SS_b = \frac{\sum_j B_j^2}{np} - \frac{G^2}{npq}$$

$$SS_{ab} = \frac{\sum_{ij}\sum (AB_{ij})^2}{n} - \frac{G^2}{npq} - SS_a - SS_b$$

$$SS_{w.cell} = \sum_{ijk}\sum\sum X_{ijk}^2 - \frac{\sum_{ij}\sum (AB_{ij})^2}{n}$$

$$SS_{total} = \sum_{ijk}\sum\sum X_{ijk}^2 - \frac{G^2}{npq}$$

The alternative forms for the definitions of the sums of squares involve the use of summation terms rather than mean values. This has practical advantages for the step-by-step approach to the computations. The following COMPUTATIONAL SYMBOLS can now be defined:-

$$(1) = \frac{G^2}{npq} \qquad (2) = \sum X_{ijk}^2$$

$$(3) = \frac{\sum A_i^2}{nq} \qquad (4) = \frac{\sum B_j^2}{np}$$

$$(5) = \frac{\sum (AB_{ij})^2}{n}$$

where the summations are over all available suffices and the divisors represent the number of basic observations incorporated in the numerator term.

Using these symbols the formulae for the sums of squares can be re-written as COMPUTATION FORMULAE, as follows:-

$SS_a = (3) - (1)$

$SS_b = (4) - (1)$

$SS_{ab} = (5) - (1)$

$SS_{w.cell} = (2) - (5)$

$SS_{total} = (2) - (1)$

For this particular example the values of the computational symbols can be calculated using a single 2-way summary table showing the sums of all the measurements under each treatment combination ab_{ij}. However, in a more complex experimental field of more than two factors a series of summary tables would be required which would include 2-way summary tables, 3-way tables, etcetera, up to the number of factors involved.

Once the sums of squares are evaluated the degrees of freedom are used to obtain the mean squares which can then be utilized in the variance-ratio tests ('F'-tests) to determine the level of significance of the attributable effects.

H.3 HAND CALCULATION OF ANOVA TABLE

In this section a full set of computations are presented in order to demonstrate the production of an ANOVA table. For this purpose an actual set of experimental results is used from the Series 2, 3x4x4x2 factorial set of experiments described in

Chapters 5 and 6 of this thesis. The expansion measurements taken at an age of 6 months were selected, and these are tabulated below, using the notation unique to the experimental section of the work.

MIX REF. NO.	EXPANSION AT 6 MONTHS	MIX REF. NO.	EXPANSION AT 6 MONTHS	MIX REF. NO.	EXPANSION AT 6 MONTHS
0000	91	1000	103	2000	125
0001	81	1001	87	2001	105
0010P	80	1010P	311	2010P	1993
0011P	91	1011P	271	2011P	3048
0020P	172	1020P	1533	2020P	4099
0021P	99	1021P	3164	2021P	6467
0030P	1425	1030P	2864	2030P	3887
0031P	2819	1031P	4385	2031P	6948
0100	52	1100	51	2100	104
0101	95	1101	84	2101	156
0110P	64	1110P	153	2110P	597
0111P	88	1111P	196	2111P	1732
0120P	93	1120P	452	2120P	1479
0121P	99	1121P	2092	2121P	3557
0130P	399	1130P	1208	2130P	1924
0131P	2023	1131P	2649	2131P	3201
0200	5	1200	91	2200	137
0201	48	1201	99	2201	177
0210P	63	1210P	128	2210P	371
0211P	78	1211P	124	2211P	491
0220P	60	1220P	251	2220P	964
0221P	29	1221P	340	2221P	1164
0230P	180	1230P	807	2230P	1556
0231P	297	1231P	1236	2231P	1872
0300	73	1300	148	2300	133
0301	99	1301	125	2301	163
0310P	31	1310P	137	2310P	331
0311P	79	1311P	123	2311P	309
0320P	79	1320P	208	2320P	568
0321P	115	1321P	209	2321P	543
0330P	147	1330P	433	2330P	1077
0331P	148	1331P	351	2331P	800

The notation and system used for the computations on the results from this four factor experiment are simply an extension of those described for the elementary two factor experiment. Hence, the required computation symbols are:-

$$(1) = \frac{G^2}{pqrsn} \qquad (2) = \Sigma X^2_{ijklm} \qquad (3)\ \frac{(\Sigma A_i{}^2)}{qrsn}$$

$$(4) = \frac{(\Sigma B_j{}^2)}{prsn} \qquad (5) = \frac{(\Sigma C_k{}^2)}{pqsn} \qquad (6) = \frac{(\Sigma D_l{}^2)}{pqrn}$$

$$(7) = \frac{[\Sigma(AB_{ij})^2]}{rsn} \qquad (8) = \frac{[\Sigma(AC_{ik})^2]}{qsn} \qquad (9) = \frac{[\Sigma(BC_{jk})^2]}{psn}$$

$$(10) = \frac{[\Sigma(AD_{il})^2]}{qrn} \qquad (11) = \frac{[\Sigma(BD_{jl})^2]}{prn} \qquad (12) = \frac{[\Sigma(CD_{kl})^2]}{pqn}$$

$$(13) = \frac{[\Sigma(ABC_{ijk})^2]}{sn} \qquad (14) = \frac{[\Sigma(ABD_{ijl})^2]}{rn} \qquad (15) = \frac{[\Sigma(ACD_{ikl})^2]}{qn}$$

$$(16) = \frac{[\Sigma(BCD_{jkl})^2]}{pn} \qquad (17) = \frac{[\Sigma(ABCD_{ijkl})^2]}{n}$$

For the four factor experiment of dimensions 3x4x4x2 we have

$p = 3$

$q = 4$

$r = 4$

$s = 2$

In addition, $n = 1$ since there is only 1 result per cell of the experiment.

The term for the nth observation under the general treatment combination $abcd_{ijkl}$ of the experiment is X_{ijklm}

where $i = 1$ to p

$j = 1$ to q

$k = 1$ to r

$l = 1$ to s

$m = 1$ to n

The link between the Mix Reference Numbers and the notation used in the ANOVA for the treatment combinations is explained by the following examples:-

Mix Ref. No. 0000 = Treatment Combination $abcd_{1111}$

Mix. Ref. No. 0231P = Treatment Combination $abcd_{1342}$

Mix. Ref. No. 2300P = Treatment Combination $abcd_{3411}$

The relevant 4-way, 3-way and 2-way summary tables may now be written down. The 4-way table is simply the experimental results table given above written in a different order using the computational notation.

ABCD SUMMARY TABLE

			a_1	a_2	a_3	TOTAL
d_1	c_1	b_1	$ABCD_{1111}$ 91	$ABCD_{2111}$ 103	$ABCD_{3111}$ 125	BCD_{111} 319
		b_2	$ABCD_{1211}$ 52	$ABCD_{2211}$ 51	$ABCD_{3211}$ 104	BCD_{211} 207
		b_3	$ABCD_{1311}$ 5	$ABCD_{2311}$ 91	$ABCD_{3311}$ 137	BCD_{311} 233
		b_4	$ABCD_{1411}$ 73	$ABCD_{2411}$ 148	$ABCD_{3411}$ 133	BCD_{411} 354
	c_2	b_1	$ABCD_{1121}$ 80	$ABCD_{2121}$ 311	$ABCD_{3121}$ 1993	BCD_{121} 2384
		b_2	$ABCD_{1221}$ 64	$ABCD_{2221}$ 153	$ABCD_{3221}$ 547	BCD_{221} 814
		b_3	$ABCD_{1321}$ 63	$ABCD_{2321}$ 128	$ABCD_{3321}$ 371	BCD_{321} 562
		b_4	$ABCD_{1421}$ 31	$ABCD_{2421}$ 137	$ABCD_{3421}$ 331	BCD_{421} 499
	c_3	b_1	$ABCD_{1131}$ 172	$ABCD_{2131}$ 1533	$ABCD_{3131}$ 4099	BCD_{131} 5804
		b_2	$ABCD_{1231}$ 93	$ABCD_{2231}$ 452	$ABCD_{3231}$ 1479	BCD_{231} 2024
		b_3	$ABCD_{1331}$ 60	$ABCD_{2331}$ 251	$ABCD_{3331}$ 964	BCD_{331} 1275
		b_4	$ABCD_{1431}$ 79	$ABCD_{2431}$ 208	$ABCD_{3431}$ 568	BCD_{431} 855
	c_4	b_1	$ABCD_{1141}$ 1425	$ABCD_{2141}$ 2864	$ABCD_{3141}$ 3887	BCD_{141} 8176
		b_2	$ABCD_{1241}$ 399	$ABCD_{2241}$ 1208	$ABCD_{3241}$ 1924	BCD_{241} 3531
		b_3	$ABCD_{1341}$ 180	$ABCD_{2341}$ 807	$ABCD_{3341}$ 1556	BCD_{341} 2543
		b_4	$ABCD_{1441}$ 147	$ABCD_{2441}$ 433	$ABCD_{3441}$ 1077	BCD_{441} 1657
d_2	c_1	b_1	$ABCD_{1112}$ 81	$ABCD_{2112}$ 87	$ABCD_{3112}$ 105	BCD_{112} 273
		b_2	$ABCD_{1212}$ 95	$ABCD_{2212}$ 84	$ABCD_{3212}$ 156	BCD_{212} 335
		b_3	$ABCD_{1312}$ 48	$ABCD_{2312}$ 99	$ABCD_{3312}$ 177	BCD_{312} 324
		b_4	$ABCD_{1412}$ 99	$ABCD_{2412}$ 125	$ABCD_{3412}$ 163	BCD_{412} 387
	c_2	b_1	$ABCD_{1122}$ 91	$ABCD_{2122}$ 271	$ABCD_{3122}$ 3048	BCD_{122} 3410
		b_2	$ABCD_{1222}$ 88	$ABCD_{2222}$ 196	$ABCD_{3222}$ 1732	BCD_{222} 2016
		b_3	$ABCD_{1322}$ 78	$ABCD_{2322}$ 124	$ABCD_{3322}$ 491	BCD_{322} 693
		b_4	$ABCD_{1422}$ 79	$ABCD_{2422}$ 123	$ABCD_{3422}$ 309	BCD_{422} 511
	c_3	b_1	$ABCD_{1132}$ 99	$ABCD_{2132}$ 3164	$ABCD_{3132}$ 6467	BCD_{132} 9730
		b_2	$ABCD_{1232}$ 99	$ABCD_{2232}$ 2092	$ABCD_{3232}$ 3557	BCD_{232} 5748
		b_3	$ABCD_{1332}$ 29	$ABCD_{2332}$ 340	$ABCD_{3332}$ 1164	BCD_{332} 1533
		b_4	$ABCD_{1432}$ 115	$ABCD_{2432}$ 209	$ABCD_{3432}$ 543	BCD_{432} 867
	c_4	b_1	$ABCD_{1142}$ 2819	$ABCD_{2142}$ 4385	$ABCD_{3142}$ 6948	BCD_{142} 14152
		b_2	$ABCD_{1242}$ 2023	$ABCD_{2242}$ 2649	$ABCD_{3242}$ 3201	BCD_{242} 7873
		b_3	$ABCD_{1342}$ 297	$ABCD_{2342}$ 1236	$ABCD_{3342}$ 1872	BCD_{342} 3405
		b_4	$ABCD_{1442}$ 148	$ABCD_{2442}$ 351	$ABCD_{3442}$ 800	BCD_{442} 1299
TOTAL			A_1 9302	A_2 24413	A_3 50078	G 83793

ABC SUMMARY TABLE

		a_1	a_2	a_3	TOTAL
c_1	b_1	ABC_{111} 172	ABC_{211} 190	ABC_{311} 230	BC_{11} 592
	b_2	ABC_{121} 147	ABC_{221} 135	ABC_{321} 260	BC_{21} 542
	b_3	ABC_{131} 53	ABC_{231} 190	ABC_{331} 314	BC_{31} 557
	b_4	ABC_{141} 172	ABC_{241} 273	ABC_{341} 296	BC_{41} 741
c_2	b_1	ABC_{112} 171	ABC_{212} 582	ABC_{312} 5041	BC_{12} 5794
	b_2	ABC_{122} 152	ABC_{222} 349	ABC_{322} 2329	BC_{22} 2830
	b_3	ABC_{132} 141	ABC_{232} 252	ABC_{332} 862	BC_{32} 1255
	b_4	ABC_{142} 110	ABC_{242} 260	ABC_{342} 640	BC_{42} 1010
c_3	b_1	ABC_{113} 271	ABC_{213} 4697	ABC_{313} 10566	BC_{13} 15534
	b_2	ABC_{123} 192	ABC_{223} 2544	ABC_{323} 5036	BC_{23} 7772
	b_3	ABC_{133} 89	ABC_{233} 591	ABC_{333} 2128	BC_{33} 2808
	b_4	ABC_{143} 194	ABC_{243} 417	ABC_{343} 1111	BC_{43} 1722
c_4	b_1	ABC_{114} 4244	ABC_{214} 7249	ABC_{314} 10835	BC_{14} 22328
	b_2	ABC_{124} 2422	ABC_{224} 3857	ABC_{324} 5125	BC_{24} 11404
	b_3	ABC_{134} 477	ABC_{234} 2043	ABC_{334} 3428	BC_{34} 5948
	b_4	ABC_{144} 295	ABC_{244} 784	ABC_{344} 1877	BC_{44} 2956
TOTAL		A_1 9302	A_2 24413	A_3 50078	G 83793

ABD SUMMARY TABLE

		a_1	a_2	a_3	TOTAL
d_1	b_1	ABD_{111} 1768	ABD_{211} 4811	ABD_{311} 10104	BD_{11} 16683
	b_2	ABD_{121} 608	ABD_{221} 1864	ABD_{321} 4104	BD_{21} 6576
	b_3	ABD_{131} 308	ABD_{231} 1277	ABD_{331} 3028	BD_{31} 4613
	b_4	ABD_{141} 330	ABD_{241} 926	ABD_{341} 2109	BD_{41} 3365
d_2	b_1	ABD_{112} 3090	ABD_{212} 7907	ABD_{312} 16568	BD_{12} 27565
	b_2	ABD_{122} 2305	ABD_{222} 5021	ABD_{322} 8646	BD_{22} 15972
	b_3	ABD_{132} 452	ABD_{232} 1799	ABD_{332} 3704	BD_{32} 5955
	b_4	ABD_{142} 441	ABD_{242} 808	ABD_{342} 1815	BD_{42} 3064
TOTAL		A_1 9302	A_2 24413	A_3 50078	G 83793

ACD SUMMARY TABLE

		d_1	d_2	TOTAL
c_1	a_1	ACD_{111} 221	ACD_{112} 323	AC_{11} 544
	a_2	ACD_{211} 393	ACD_{212} 395	AC_{21} 788
	a_3	ACD_{311} 499	ACD_{312} 601	AC_{31} 1100
c_2	a_1	ACD_{121} 238	ACD_{122} 336	AC_{12} 574
	a_2	ACD_{221} 729	ACD_{222} 714	AC_{22} 1443
	a_3	ACD_{321} 3292	ACD_{322} 5580	AC_{32} 8872
c_3	a_1	ACD_{131} 404	ACD_{132} 342	AC_{13} 746
	a_2	ACD_{231} 2444	ACD_{232} 5805	AC_{23} 8249
	a_3	ACD_{331} 7110	ACD_{332} 11731	AC_{33} 18841
c_4	a_1	ACD_{141} 2151	ACD_{142} 5287	AC_{14} 7438
	a_2	ACD_{241} 5312	ACD_{242} 8621	AC_{24} 13933
	a_3	ACD_{341} 8444	ACD_{342} 12821	AC_{34} 21265
TOTAL		D_1 31237	D_2 52556	G 83793

BCD SUMMARY TABLE

		b_1	b_2	b_3	b_4	TOTAL
d_1	c_1	BCD_{111} 319	BCD_{211} 207	BCD_{311} 233	BCD_{411} 354	CD_{11} 1113
	c_2	BCD_{121} 2384	BCD_{221} 814	BCD_{321} 562	BCD_{421} 499	CD_{21} 4259
	c_3	BCD_{131} 5804	BCD_{231} 2024	BCD_{331} 1275	BCD_{431} 855	CD_{31} 9958
	c_4	BCD_{141} 8176	BCD_{241} 3531	BCD_{341} 2543	BCD_{441} 1657	CD_{41} 15907
d_2	c_1	BCD_{112} 273	BCD_{212} 335	BCD_{312} 324	BCD_{412} 387	CD_{12} 1319
	c_2	BCD_{122} 3410	BCD_{222} 2016	BCD_{322} 693	BCD_{422} 511	CD_{22} 6630
	c_3	BCD_{132} 9730	BCD_{232} 5748	BCD_{332} 1533	BCD_{432} 867	CD_{32} 17878
	c_4	BCD_{142} 14152	BCD_{242} 7873	BCD_{342} 3405	BCD_{442} 1299	CD_{42} 26729
TOTAL		B_1 44248	B_2 22548	B_3 10568	B_4 6429	G 83793

AB SUMMARY TABLE

	b_1	b_2	b_3	b_4	TOTAL
a_1	AB_{11} 4858	AB_{12} 2913	AB_{13} 760	AB_{14} 771	A_1 9301
a_2	AB_{21} 12718	AB_{22} 6885	AB_{23} 3076	AB_{24} 1734	A_2 24413
a_3	AB_{31} 26672	AB_{32} 12750	AB_{33} 6732	AB_{34} 3924	A_3 50078
TOTAL	B_1 44248	B_2 22548	B_3 10568	B_4 6429	G 83793

AC SUMMARY TABLE

	c_1	c_2	c_3	c_4	TOTAL
a_1	AC_{11} 544	AC_{12} 574	AC_{13} 746	AC_{14} 7438	A_1 9302
a_2	AC_{21} 788	AC_{22} 1443	AC_{23} 8249	AC_{24} 13933	A_2 24413
a_3	AC_{31} 1100	AC_{32} 8872	AC_{33} 18841	AC_{34} 21265	A_3 50078
TOTAL	C_1 2432	C_2 10889	C_3 27836	C_4 42636	G 83793

BC SUMMARY TABLE

	c_1	c_2	c_3	c_4	TOTAL
b_1	BC_{11} 592	BC_{12} 5794	BC_{13} 15534	BC_{14} 22328	B_1 44248
b_2	BC_{21} 542	BC_{22} 2830	BC_{23} 7772	BC_{24} 11404	B_2 22548
b_3	BC_{31} 557	BC_{32} 1255	BC_{33} 2808	BC_{34} 5948	B_3 10568
b_4	BC_{41} 741	BC_{42} 1010	BC_{43} 1722	BC_{44} 2956	B_4 6429
TOTAL	C_1 2432	C_2 10889	C_3 27836	C_4 42636	G 83793

AD SUMMARY TABLE

	d_1	d_2	TOTAL
a_1	AD_{11}	AD_{12}	A_1
	3014	6288	9302
a_2	AD_{21}	AD_{22}	A_2
	8878	15535	24413
a_3	AD_{31}	AD_{32}	A_3
	19345	30733	50078
TOTAL	D_1	D_2	G
	31237	52556	83793

BD SUMMARY TABLE

	d_1	d_2	TOTAL
b_1	BD_{11}	BD_{12}	B_1
	16683	27565	44248
b_2	BD_{21}	BD_{22}	B_2
	6576	15972	22548
b_3	BD_{31}	BD_{32}	B_3
	4613	5955	10568
b_4	BD_{41}	BD_{42}	B_4
	3365	3064	6429
TOTAL	D_1	D_2	G
	31237	52556	83793

CD SUMMARY TABLE

	d_1	d_2	TOTAL
c_1	CD_{11}	CD_{12}	C_1
	1113	1319	2432
c_2	CD_{21}	CD_{22}	C_2
	4259	6630	10889
c_3	CD_{31}	CD_{32}	C_3
	9958	17878	27836
c_4	CD_{41}	CD_{42}	C_4
	15907	26729	42636
TOTAL	D_1	D_2	G
	31237	52556	83793

From the column and row totals in these summary tables the values of the computational symbols may be obtained.

SYMBOL	FORMULA	COMPUTED VALUE
(1)	$\frac{G^2}{pqrsn}$	73138196.3
(2)	ΣX^2_{ijklm}	249328849.0
(3)	$\frac{(\Sigma A_i{}^2)}{qrsn}$	99697745.5
(4)	$\frac{(\Sigma B_j{}^2)}{prsn}$	109138019.7
(5)	$\frac{(\Sigma C_k{}^2)}{pqsn}$	113214847.4
(6)	$\frac{(\Sigma D_l{}^2)}{pqrn}$	77872568.9
(7)	$\frac{[\Sigma(AB_{ij})^2]}{rsn}$	148694092.4
(8)	$\frac{[\Sigma(AC_{ik})^2}{qsn}$	151061168.1
(9)	$\frac{[\Sigma(BC_{jk})^2]}{psn}$	171824124.5
(10)	$\frac{[\Sigma(AD_{il})^2]}{qrn}$	105470285.2
(11)	$\frac{[\Sigma(BD_{jl})^2]}{prn}$	117829449.1
(12)	$\frac{[\Sigma(CD_{kl})^2]}{pqn}$	120944270.8
(13)	$\frac{[\Sigma(ABC_{ijk})^2]}{sn}$	230269599.5
(14)	$\frac{[\Sigma(ABD_{ijl})^2]}{rn}$	159626004.3

SYMBOL	FORMULA	COMPUTED VALUE
(15)	$\frac{[\Sigma(ACD_{ikl})^2]}{qn}$	160793855.3
(16)	$\frac{[\Sigma(BCD_{jkl})^2]}{pn}$	186378751.7
(17)	$\frac{[\Sigma(ABCD_{ijkl})^2]}{n}$	249328849.0

The computational formulae for the sums of squares, again an extension of the system defined for the simpler 2-factor experiment, and their calculated values can then be written as follows:-

SS_a	(3)-(1)	26559549.2
SS_b	(4)-(1)	35999823.4
SS_c	(5)-(1)	40076651.0
SS_d	(6)-(1)	4734372.5
SS_{ab}	(7)-(3)-(4)+(1)	12996523.5
SS_{ac}	(8)-(3)-(5)+(1)	11286771.5
SS_{bc}	(9)-(4)-(5)+(1)	22609453.7
SS_{ad}	(10)-(3)-(6)+(1)	1038167.2
SS_{bd}	(11)-(4)-(6)+(1)	3957056.9
SS_{cd}	(12)-(5)-(6)+(1)	2995050.9
SS_{abc}	(13)-(7)-(8)-(9)+(3)+(4)+(5)-(1)	7602630.8
SS_{abd}	(14)-(7)-(10)-(11)+(3)+(4)+(6)-(1)	1202315.3
SS_{acd}	(15)-(8)-(10)-(12)+(3)+(5)+(6)-(1)	965096.6
SS_{bcd}	(16)-(9)-(11)-(12)+(4)+(5)+(6)-(1)	2868146.9
SS_{abcd}	(17)-(13)-(14)-(15)-(16)+(7)+(8)+(9) +(10)+(11)+(12)-(3)-(4)-(5)-(6)+(1)	1299043.2
$SS_{w.cell}$	(2)-(17)	0.0
SS_{total}	(2)-(1)	176190652.7

Using these values of the sums of squares and the appropriate degrees of freedom the complete ANOVA table can be compiled. Since the within cell experimental error cannot be measured with the inclusion of only 1 result per treatment combination the highest order interaction is removed from the analysis and used as the error term.

ANOVA TABLE

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Square (MS)	F	Tail Prob.(α)
A (Cement Alkali Content)	26559549.2	(p-1) = 2	13279774.6	184.01	$0.00<\alpha<0.01$
B (Pfa as cement replacement)	35999823.4	(q-1) = 3	11999941.1	166.28	$0.00<\alpha<0.01$
C (Reactive Aggregate Content)	40076651.0	(r-1) = 3	13358883.7	185.11	$0.00<\alpha<0.01$
D (Free water content)	4734372.5	(s-1) = 1	4734372.5	65.60	$0.00<\alpha<0.01$
AB	12996523.5	(p-1)(q-1) = 6	2166087.3	30.01	$0.00<\alpha<0.01$
AC	11286771.5	(p-1)(r-1) = 6	1881128.6	26.07	$0.00<\alpha<0.01$
BC	22609453.7	(q-1)(r-1) = 9	2512161.5	34.81	$0.00<\alpha<0.01$
AD	1038167.2	(p-1)(s-1) = 2	519083.6	7.19	$0.00<\alpha<0.01$
BD	3957056.9	(q-1)(s-1) = 3	1319019.0	18.28	$0.00<\alpha<0.01$
CD	2995050.9	(r-1)(s-1) = 3	998350.3	13.83	$0.00<\alpha<0.01$
ABC	7602630.8	(p-1)(q-1)(r-1) =18	422368.4	5.85	$0.00<\alpha<0.01$
ABD	1202315.3	(p-1)(q-1)(s-1) = 6	200385.9	2.78	$0.01<\alpha<0.05$
ACD	965096.6	(p-1)(r-1)(s-1) = 6	160849.4	2.23	$0.05<\alpha<0.10$
BCD	2868146.9	(q-1)(r-1)(s-1) = 9	318683.0	4.42	$0.00<\alpha<0.01$
ABCD, used to represent the Within Cell (Experimental Error) term	1299043.2	(p-1)(q-1)(r-1)(s-1) =18	72169.1		
TOTAL	176190652.7	pqrsn-1 =95			

APPENDIX J

Full details of results from the chemical analysis of the mortar bars

Tables J.1 to J.3

TABLE J.1 SERIES 2 CHEMICAL ANALYSIS RESULTS - 3x4x4x2 FACTORIAL MIXES

Mix Ref No.	Age of Bar when sample taken (days)		Measured Density of bar	ANALYSIS OF BAR *				ANALYSIS OF LIQUID FROM STORAGE CONTAINER			
	Bar	Liquid	(kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq} (kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq}** (kg/m^3)
0000	463	463	2260	0.022	0.090	0.081	1.83	0.007	0.016	0.017	0.39
0001	525	525	2160	0.022	0.077	0.072	1.56	0.010	0.021	0.024	0.51
0010P	750	750	2280	0.047	0.088	0.105	2.39	0.017	0.023	0.032	0.73
0011P	691	691	2110	0.042	0.075	0.091	1.92	0.023	0.031	0.043	0.91
0020P	742	742	2180	0.088	0.105	0.157	3.41	0.024	0.026	0.041	0.88
0021P	673	673	2090	0.057	0.058	0.095	1.98	0.035	0.041	0.062	1.29
0030P	442	442	2100	0.093	0.096	0.156	3.28	0.020	0.014	0.030	0.62
0031P	467	467	1990	0.098	0.094	0.160	3.19	0.023	0.021	0.037	0.73
0100	743	743	2260	0.058	0.179	0.176	3.98	0.014	0.027	0.031	0.71
0101	673	673	2160	0.061	0.172	0.174	3.76	0.015	0.035	0.038	0.81
0110P	700	700	2240	0.081	0.184	0.202	4.53	0.015	0.029	0.034	0.76
0111P	699	699	2130	0.074	0.175	0.189	4.03	0.020	0.040	0.046	0.98
0120P	691	691	2150	0.102	0.187	0.225	4.84	0.018	0.033	0.040	0.87
0121P	756	756	2060	0.086	0.165	0.195	4.01	0.024	0.045	0.054	1.12
0130P	749	749	2130	0.132	0.199	0.263	5.60	0.012	0.018	0.024	0.51
0131P	755	755	2010	0.117	0.172	0.231	4.64	0.022	0.032	0.044	0.88
0200	748	748	2200	0.100	0.248	0.263	5.79	0.012	0.023	0.027	0.60
0201	687	687	2140	0.082	0.253	0.249	5.32	0.012	0.029	0.031	0.67
0210P	658	658	2230	0.101	0.255	0.269	6.00	0.012	0.024	0.028	0.63
0211P	679	679	2150	0.098	0.247	0.261	5.61	0.019	0.039	0.045	0.96
0220P	708	708	2140	0.121	0.248	0.285	6.09	0.012	0.022	0.026	0.56
0221P	750	750	2070	0.116	0.242	0.275	5.70	0.020	0.039	0.046	0.95
0230P	656	656	2040	0.139	0.241	0.297	6.07	0.014	0.019	0.026	0.54
0231P	673	673	2020	0.124	0.236	0.279	5.64	0.018	0.035	0.041	0.83
0300	701	701	2230	0.097	0.271	0.275	6.14	0.009	0.017	0.020	0.45
0301	659	659	2160	0.097	0.279	0.281	6.07	0.010	0.020	0.024	0.51
0310P	750	750	2170	0.108	0.277	0.290	6.30	0.010	0.017	0.021	0.46
0311P	686	686	2100	0.115	0.275	0.295	6.20	0.010	0.020	0.023	0.49
0320P	677	677	2150	0.123	0.261	0.295	6.34	0.010	0.018	0.022	0.47
0321P	707	707	2040	0.116	0.267	0.292	5.95	0.011	0.022	0.026	0.53
0330P	672	672	2060	0.139	0.263	0.312	6.42	0.011	0.017	0.022	0.46
0331P	685	685	1980	0.136	0.267	0.312	6.18	0.013	0.022	0.027	0.54

* by dilute hydrochloric acid extraction

** expressed with respect to the mass of the bar

TABLE J.1 continued

Mix Ref No.	Age of Bar when sample taken (days)		Measured Density of bar (kg/m^3)	ANALYSIS OF BAR *				ANALYSIS OF LIQUID FROM STORAGE CONTAINER			
	Bar	Liquid		%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq} (kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq}** (kg/m^3)
1000	460	460	2260	0.047	0.133	0.134	3.04	0.019	0.027	0.037	0.84
1001	454	454	2180	0.050	0.133	0.137	2.99	0.018	0.034	0.040	0.88
1010P	544	544	2200	0.081	0.133	0.168	3.70	0.018	0.026	0.035	0.77
1011P	564	564	2130	0.074	0.111	0.147	3.13	0.025	0.033	0.046	0.99
1020P	573	573	2130	0.140	0.161	0.246	5.25	0.016	0.015	0.027	0.57
1021P	628	628	2120	0.124	0.152	0.224	4.75	0.024	0.020	0.037	0.79
1030P	483	483	2110	0.159	0.166	0.268	5.66	0.013	0.010	0.020	0.42
1031P	482	482	2020	0.159	0.166	0.268	5.42	0.014	0.013	0.023	0.46
1100	586	586	2270	0.094	0.223	0.241	5.47	0.015	0.029	0.034	0.77
1101	543	543	2200	0.078	0.196	0.207	4.56	0.017	0.035	0.040	0.88
1110P	558	558	2260	0.121	0.225	0.270	6.09	0.015	0.026	0.033	0.74
1111P	592	592	2130	0.097	0.190	0.222	4.73	0.019	0.034	0.041	0.88
1120P	571	571	2150	0.147	0.225	0.295	6.35	0.016	0.021	0.030	0.64
1121P	544	544	2100	0.147	0.213	0.287	6.03	0.019	0.027	0.037	0.77
1130P	564	564	2070	0.183	0.245	0.344	7.13	0.015	0.017	0.026	0.54
1131P	635	635	2030	0.167	0.223	0.314	6.37	0.014	0.015	0.024	0.49
1200	543	543	2250	0.108	0.278	0.291	6.55	0.014	0.025	0.031	0.69
1201	564	564	2150	0.104	0.261	0.276	5.93	0.017	0.031	0.037	0.79
1210P	557	557	2170	0.129	0.259	0.300	6.51	0.015	0.024	0.031	0.68
1211P	579	579	2040	0.119	0.264	0.292	5.96	0.020	0.036	0.044	0.89
1220P	565	565	2130	0.160	0.283	0.347	7.38	0.014	0.021	0.028	0.59
1221P	578	578	2080	0.148	0.272	0.327	6.81	0.023	0.038	0.047	0.99
1230P	580	580	2080	0.187	0.283	0.374	7.77	0.014	0.016	0.024	0.51
1231P	627	627	1980	0.173	0.269	0.349	6.92	0.020	0.026	0.037	0.74
1300	570	570	2180	0.115	0.295	0.309	6.73	0.010	0.018	0.022	0.48
1301	635	635	2140	0.112	0.296	0.307	6.57	0.017	0.027	0.034	0.74
1310P	587	587	2180	0.127	0.278	0.310	6.75	0.015	0.023	0.030	0.65
1311P	634	634	2070	0.139	0.316	0.346	7.17	0.010	0.015	0.021	0.42
1320P	573	573	2100	0.160	0.288	0.350	7.35	0.018	0.026	0.035	0.73
1321P	545	545	2060	0.148	0.285	0.336	6.92	0.018	0.026	0.036	0.73
1330P	580	580	2030	0.182	0.285	0.370	7.51	0.016	0.019	0.028	0.57
1331P	642	642	1960	0.168	0.285	0.356	6.98	0.014	0.021	0.027	0.54

* by dilute hydrochloric acid extraction

** expressed with respect to the mass of the bar

TABLE J.1 continued

Mix Ref No.	Age of Bar when sample taken (days)		Measured Density of bar (kg/m^3)	ANALYSIS OF BAR *				ANALYSIS OF LIQUID FROM STORAGE CONTAINER			
	Bar	Liquid		%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq} (kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq}** (kg/m^3)
2000	168	501	2230	0.094	0.234	0.248	5.53	0.024	0.075	0.073	1.62
2001	168	516	2070	0.074	0.201	0.207	4.27	0.027	0.077	0.077	1.60
2010P	168	440	2220	0.137	0.266	0.313	6.94	0.018	0.037	0.042	0.93
2011P	168	515	2060	0.144	0.253	0.311	6.40	0.022	0.042	0.049	1.02
2020P	168	466	2140	0.105	0.301	0.303	6.49	0.015	0.021	0.029	0.62
2021P	168	417	2090	0.179	0.284	0.366	7.66	0.016	0.023	0.031	0.65
2030P	168	468	2110	0.102	0.313	0.309	6.51	0.013	0.017	0.024	0.51
2031P	168	467	2000	0.088	0.301	0.286	5.72	0.011	0.014	0.020	0.40
2100	168	446	2250	0.115	0.308	0.317	7.14	0.015	0.046	0.046	1.03
2101	168	459	2150	0.097	0.294	0.290	6.24	0.019	0.051	0.053	1.20
2110P	168	468	2210	0.102	0.349	0.332	7.34	0.016	0.033	0.038	0.83
2111P	168	501	2140	0.144	0.306	0.346	7.39	0.023	0.049	0.056	1.19
2120P	168	437	2120	0.191	0.343	0.417	8.85	0.012	0.023	0.027	0.58
2121P	168	432	2070	0.177	0.329	0.393	8.13	0.018	0.034	0.041	0.84
2130P	168	524	2060	0.202	0.337	0.424	8.74		LIQUID LOST		
2131P	168	461	2020	0.193	0.307	0.395	7.98	0.016	0.026	0.033	0.67
2200	168	453	2210	0.124	0.357	0.359	7.93	0.013	0.035	0.036	0.81
2201	168	452	2130	0.111	0.335	0.331	7.05	0.021	0.051	0.054	1.16
2210P	168	454	2140	0.143	0.343	0.369	7.89	0.015	0.034	0.037	0.80
2211P	168	431	2070	0.143	0.349	0.373	7.72	0.019	0.040	0.046	0.94
2220P	168	453	2120	0.167	0.345	0.394	8.35	0.016	0.028	0.034	0.72
2221P	168	439	2060	0.152	0.329	0.369	7.60	0.016	0.031	0.036	0.75
2230P	168	451	2030	0.190	0.332	0.409	8.30	0.014	0.024	0.030	0.61
2231P	168	446	2010	0.186	0.319	0.396	7.96	0.016	0.024	0.032	0.64
2300	168	515	2240	0.148	0.349	0.378	8.47	0.013	0.027	0.030	0.68
2301	168	508	2110	0.135	0.349	0.365	7.69	0.014	0.028	0.032	0.68
2310P	168	444	2170	0.146	0.311	0.350	7.60	0.008	0.019	0.021	0.45
2311P	168	452	2060	0.124	0.316	0.332	6.83	0.015	0.031	0.035	0.73
2320P	168	503	2110	0.175	0.361	0.413	8.71	0.013	0.022	0.027	0.58
2321P	168	440	2050	0.156	0.336	0.377	7.74	0.015	0.028	0.033	0.68
2330P	168	445	2040	0.183	0.316	0.391	7.98	0.011	0.019	0.023	0.47
2331P	168	503	1990	0.175	0.361	0.413	8.22	0.017	0.031	0.038	0.76

* by dilute hydrochloric acid extraction

** expressed with respect to the mass of the bar

TABLE J.2 SERIES 2 CHEMICAL ANALYSIS RESULTS - EXTRA PYREX MIXES

Mix Ref No.	Age of Bar when sample taken (days)		Measured Density of bar (kg/m^3)	ANALYSIS OF BAR *				ANALYSIS OF LIQUID FROM STORAGE CONTAINER			
	Bar	Liquid		%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq} (kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq}** (kg/m^3)
2032P	169	421	2110	0.208	0.295	0.402	8.48	0.013	0.023	0.029	0.60
2033P	167	426	2100	0.210	0.296	0.405	8.51	0.014	0.021	0.027	0.57
2034P	167	418	2110	0.205	0.285	0.393	8.29	0.011	0.017	0.022	0.46
2035P	167	426	2030	0.194	0.269	0.371	7.53	0.015	0.021	0.029	0.58
2041P	168	419	2180	0.071	0.196	0.201	4.37	0.024	0.067	0.067	1.47
2051P	167	418	2190	0.074	0.194	0.202	4.42	0.023	0.064	0.065	1.42
2061P	169	421	2170	0.089	0.217	0.232	5.03	0.028	0.068	0.073	1.58
2071P	168	420	2150	0.084	0.206	0.219	4.71	0.024	0.058	0.062	1.34
2431P	168	427	2070	0.235	0.331	0.453	9.37	0.012	0.021	0.026	0.54
2531P	167	426	2060	0.221	0.348	0.450	9.27	0.016	0.028	0.035	0.72
2631P	168	420	2020	0.214	0.373	0.460	9.29	0.022	0.037	0.046	0.93
2731P	168	420	2010	0.212	0.379	0.461	9.27	0.021	0.037	0.045	0.91
3031P	167	418	1720	0.136	0.134	0.224	3.85	0.020	0.019	0.032	0.55
4031P	168	419	1800	0.140	0.152	0.240	4.32	0.013	0.014	0.022	0.40
5031P	169	421	1840	0.162	0.184	0.283	5.21	0.017	0.017	0.028	0.52
6031P	168	419	2010	0.182	0.230	0.333	6.70	0.018	0.019	0.030	0.61

* by dilute hydrochloric acid extraction

** expressed with respect to the mass of the bar

TABLE J.3 SERIES 2 CHEMICAL ANALYSIS RESULTS - BELTANE OPAL MIXES

Mix Ref No.	Age of Bar when sample taken (days)		Measured Density of bar (kg/m^3)	ANALYSIS OF BAR *				ANALYSIS OF LIQUID FROM STORAGE CONTAINER			
	Bar	Liquid		%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq} (kg/m^3)	%Na_2O	%K_2O	%Na_2O_{eq}	Na_2O_{eq}** (kg/m^3)
2001	168	516	2070	0.074	0.201	0.207	4.27	0.0266	0.0771	0.0774	1.601
2011B	167	432	2070	0.113	0.314	0.320	6.63	0.0013	0.0051	0.0047	0.097
2021B	167	432	1980	0.109	0.295	0.303	6.01	0.0015	0.0043	0.0043	0.085
2031B	168	434	1910	0.098	0.285	0.286	5.47	0.0018	0.0067	0.0062	0.118
2041B	168	434	2180	0.089	0.251	0.254	5.53	0.0092	0.0253	0.0258	0.563
2051B	169	435	2180	0.098	0.279	0.282	6.15	0.0050	0.0141	0.0143	0.312
2061B	168	433	2170	0.102	0.275	0.283	6.14	0.0032	0.0085	0.0088	0.191
2071B	168	427	2160	0.105	0.292	0.297	6.41	0.0034	0.0086	0.0090	0.194
2301	168	508	2110	0.135	0.349	0.365	7.69	0.0139	0.0276	0.0320	0.676
2311B	168	427	2070	0.147	0.385	0.401	8.29	0.0039	0.0098	0.0104	0.215
2321B	168	433	1970	0.140	0.372	0.385	7.59	0.0026	0.0064	0.0068	0.134
2331B	167	432	1890	0.148	0.395	0.408	7.72	0.0017	0.0062	0.0058	0.109
2341B	169	435	2140	0.128	0.337	0.350	7.49	0.0090	0.0202	0.0223	0.476
2351B	168	433	2120	0.140	0.353	0.372	7.90	0.0054	0.0137	0.0145	0.307
2361B	169	435	2120	0.137	0.353	0.370	7.84	0.0056	0.0129	0.0140	0.297
2371B	168	434	2130	0.139	0.359	0.375	7.99	0.0046	0.0107	0.0117	0.249

* by dilute hydrochloric acid extraction

** expressed with respect to the mass of the bar

LIST OF REFERENCES

(1) STANTON, T E. Expansion of concrete through reaction between cement and aggregate. **Proceedings of the American Society for Civil Engineers.** Vol.66, Dec. 1940, pp 1781-1811.

(2) COOMBES, L.H. Val de la Mare Dam, Jersey, Channel Islands. **Proceedings of the 3rd International Symposium on the Effect of Alkalis on the Properties of Concrete, London.** Cement and Concrete Association, Slough, Sept. 1976, pp 357-370.

(3) WINNEY, M. Alkali-aggregate threatens future of Val de la Mare Dam. **New Civil Engineer.** 29th May 1980, pp 14-15.

(4) PALMER, D. Alkali-aggregate reaction, recent occurrences in the British Isles. **Proceedings of the 4th International Conference on the Effects of Alkalis in Cement and Concrete, Purdue University, West Lafayette.** Publication No. CE MAT 1-78, June 1978, pp 285-298.

(5) GUTT, W and NIXON, P.J. Alkali aggregate reactions in concrete in the U.K. Concrete, Journal of the Concrete Society. May 1979, Vol.13, No. 5, pp 19-21.

(6) ALLEN, R.T.L. Alkali-silica reaction in Great Britain - a review. **Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town.** National Building Research Institute, Pretoria, Paper S252/18, April 1981.

(7) PALMER, D. Alkali-aggregate reaction in Great Britain - the present position. Concrete - Journal of the Concrete Society. March 1981, Vol.15, No.3, pp 24-27.

(8) JONES, F.E. Reactions between aggregates and cement. Part II, Alkali-aggregate interaction : British Portland cements and British aggregates. Department of Scientific and Industrial Research, Building Research Station. National Building Studies Research Paper No.15, H.M.S.O., London, 1952.

(9) JONES, F.E. and TARLETON, R.D. Reactions between aggregates and cement. Part III, Alkali-aggregate interaction : The expansion bar test : Cements of medium alkali content. Department of Scientific and Industrial Research, Building Research Station. National Building Studies Research Paper No.17, H.M.S.O., London, 1952.

(10) JONES, F.E. and TARLETON, R.D. Reactions between aggregates and cement. Part IV, Alkali-aggregate interaction : The expansion bar test and its application to the examination of some British aggregates for possible expansive reaction with Portland cements of high alkali content. Department of Scientific and Industrial Research, Building Research Station. National Building Studies Research Paper No.20, H.M.S.O., London, 1958.

(11) CORISH, A.T. and JACKSON, P.J. Portland cement properties - past and present. Concrete, Journal of the Concrete Society. July 1982, Vol.12, No.7, pp 16-18.

(12) BUTTLER, F.G., MORGAN, S.R. and WALKER, E.J. Studies on the rate and extent of reaction between calcium hydroxide and pulverised fuel ash at 38°C. Proceedings of the 5th International Conference on Alkali-aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/38, April 1981.

(13) AMERICAN SOCIETY FOR TESTING AND MATERIALS. Standard recommended practice for petrographic examination of aggregates for concrete. ASTM C295-65 (reapproved 1973), 1980.

(14) JONES, F.E. and TARLETON, R.D. Reaction between aggregates and cement. Part V, Alkali-aggregate interaction : Effect on bending strength of mortars and the development of gelatinous reaction products and cracking. Part VI, Alkali-aggregate interaction : Experience with some forms of rapid and accelerated tests for alkali-aggregate reactivity : Recommended test procedures. Department of Scientific and Industrial Research, Building Research Station. National Building Studies Research Paper No.25, H.M.S.O., London, 1958.

(15) AMERICAN SOCIETY FOR TESTING AND MATERIALS. Standard test method for potential reactivity of aggregates (chemical method). ASTM C289-71 (reapproved 1976), 1980. [new edition ASTM C289-81].

(16) AMERICAN SOCIETY FOR TESTING AND MATERIALS. Standard test method for potential alkali reactivity of carbonate rocks for concrete aggregates (rock cylinder method). ASTM C586-69 (reapproved 1981).

(17) CANADIAN STANDARDS ASSOCIATION CAN3-A23.3-M77. Methods of test for concrete. CSA A23.2-14A, Alkali aggregate reaction.

(18) AMERICAN SOCIETY FOR TESTING AND MATERIALS. Standard test method for potential alkali reactivity of cement-aggregate combinations (mortar-bar method). ASTM C227-71 (reapproved 1976), 1980 [new edition ASTM C227-81]

(19) NIXON, P.J., COLLINS, R.J. and RAYMENT, P.L. The concentration of alkalis by moisture migration in concrete - a factor influencing alkali aggregate reaction. Cement and Concrete Research. Vol.9, No.4, pp 417-423.

(20) SIMS, I. The application and reliability of standard testing procedures for potential alkali-reactivity. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/13, April 1981.

(21) HOBBS, D.W. Influence of mix proportions and cement alkali content upon expansion due to the alkali-silica reaction. Cement and Concrete Association, Slough. Technical Report No.534, June 1980.

(22) JONES, F.E. Reactions between aggregates and cement. Part 1, Alkali-aggregate interaction : General. Department of Scientific and Industrial Research, Building Research Station. National Building Studies Research Paper No.14, H.M.S.O., London 1952.

(23) SULLIVAN, P.J.E., NEWMAN, J.B. and McLEISH, A. The Durability of concrete in marine applications - a factorial approach to experimentation. Proceedings of the 2nd International Conference on the Behaviour of Off-Shore Structures (BOSS'79), Imperial College, London. Paper No.44, August 1979, pp 581-590.

(24) AMERICAN SOCIETY FOR TESTING AND MATERIALS. Standard test method for effectiveness of mineral admixtures in preventing excessive expansion of concrete due to the alkali-aggregate reaction. ASTM C441-69 (reapproved 1975), 1980. [new edition ASTM C441-81]

(25) COOMBES, L.H., COLE, R.G. and CLARKE, R.M. Remedial measures to Val de la Mare Dam, Jersey, Channel Islands, following alkali-aggregate reactivity. Paper presented at BNCOLD/Newcastle University Symposium, Newcastle-upon-Tyne. Paper No.3.3, September 1975.

(26) COOMBES, L.H. Written contribution to Paper No.3.3 presented at BNCOLD/Newcastle University Symposium, Newcastle-upon-Tyne, September 1975. Unpublished.

(27) COLE, R.G. Written contribution to Paper No.3.3 presented at BNCOLD/Newcastle University Symposium, Newcastle-upon-Tyne, September 1975. Unpublished.

(28) FIGG, J.W. Quality decrease brings need for more caution. New Civil Engineer. 17th November 1977, pp 58-60.

(29) NEWS : Concrete problems burst forth. New Civil Engineer. 24th April 1980, p 4.

(30) NEWS : Devon viaduct faces early end to lifespan. New Civil Engineer. 31st March 1983, p 4.

(31) NEWS : Alkali-aggregate hits Derbyshire bridges. New Civil Engineer. 12th February 1981, p 4.

(32) NEWS : Authorities clam up on M1 repairs. New Civil Engineer. 17th May 1984, p 6.

(33) NEWS : Supports needed for AAR hit bridge. New Civil Engineer. 11th October 1984, p 7.

(34) NEWS : Push for low alkali cement. New Civil Engineer. 13th September 1984, p 6.

(35) WINNEY, M. Alkali-aggregate cracks up Plymouth car park. New Civil Engineer. 24th April 1980, pp 22-23.

(36) NEWS : Alkali hit hospital awaits repair method. New Civil Engineer. 12th January 1984, p 6.

(37) SHARP, R. Alkali-aggregate advice. New Civil Engineer. Letters to the Editor, 22nd May 1980, p 60.

(38) HOBBS, D.W. Influence of pulverized-fuel ash and granulated blastfurnace slag upon expansion caused by the alkali-silica reaction. Magazine of Concrete Research. Vol.34, No.119, June 1982, pp 83-94.

(39) NEWS : AAR problem now feared widespread. New Civil Engineer. 2nd August 1984, p 9.

(40) NEWS : AAR problem now countrywide says DTp. New Civil Engineer. 9th August 1984, p 6.

(41) NIXON, P.J. CS working party comments on changes in ordinary Portland cement. Concrete, Journal of the Concrete Society. September 1984, Vol.18, No.9, pp 38-39.

(42) CONCRETE SOCIETY WORKING PARTY Interim report on the changes in cement properties and their effects on concrete. Presented at a half-day seminar at the Royal Aeronautical Society, 4 Hamilton Place, London. 23rd October 1984, unpublished draft.

(43) NEWS : Tarbela latest victim of concrete 'cancer'. New Civil Engineer. 26th April 1984, p 4.

(44) DIAMOND, S. A review of alkali-silica reaction and expansion mechanisms. 1. Alkalis in cements and in concrete pore solutions. Cement and Concrete Research. Vol.5, No.4, 1975, pp 329-346.

(45) HOBBS, D.W. Expansion of concrete due to alkali-silica reaction : an explanation. Magazine of Concrete Research. Vol.30, No.105, December 1978, pp 215-220.

(46) DENT-GLASSER, L.S. and KATAOKA, N. The chemistry of alkali-aggregate reactions. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/23, April 1981.

(47) FIGG, J.W. Concrete durability and alkali reactions. Concrete, Journal of the Concrete Society. August 1981, Vol.15, No.8, pp 18-22.

(48) HAWKINS, M.R. et alia. Minimising the risk of alkali-silica reaction, Guidance Notes. Report of a Working Party, September 1983.

(49) DIAMOND, S. On the physics and chemistry of alkali-silica reactions. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/22, April 1981.

(50) DENT-GLASSER, L.S. Osmotic pressure and the swelling of gels. Cement and Concrete Research. Vol.9, No.4, 1979, pp 515-517.

(51) OBERHOLSTER, R.E. and WESTRA, W.B. The effectiveness of mineral admixtures in reducing expansion due to alkali-aggregate reaction with Malmesbury group aggregates. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/31, April 1981.

(52) DIAMOND, S. and THAULOW, N. A study of expansion due to alkali-silica reaction as conditioned by the grain size of the reactive aggregate. Cement and Concrete Research. Vol.4, No.4, 1979, pp 591-607.

(53) HOBBS, D.W. and GUTTERIDGE, W.A. Particle size of aggregate and its influence upon the expansion caused by the alkali-silica reaction. Magazine of Concrete Research. Vol.31, No.109, December 1979, pp 235-242.

(54) LUDWIG, U. Theoretical and practical research on the alkali-silica reaction. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/24, April 1981.

(55) LENZNER, D. Influence of the amount of mixing water on the alkali-silica reaction. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/26, April 1981.

(56) DAHMS, J. Influences on the alkali-aggregate reaction under field conditions. Proceedings of the 3rd International Symposium on the Effect of Alkalis on the Properties of Concrete, London. Cement and Concrete Association, Slough, September 1976, pp 277-290.

(57) GUTTERIDGE, W.A. and HOBBS, D.W. Some chemical and physical properties of Beltane opal rock and its gelatinous alkali silica reaction product. Cement and Concrete Research. Vol.10, No.2, 1980, pp 183-193.

(58) GAZE, M.E. and NIXON, P.J. The effect of p.f.a. upon alkali-aggregate reaction. Magazine of Concrete Research. Vol.35, No.123, June 1983, pp 107-110.

(59) FIGG, J.W. Reaction between cement and artificial glass in concrete. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/7, April 1981.

(60) NIXON, P.J. and GAZE, M.E. The use of flyash and granulated blastfurnace slag to reduce expansion due to alkali-aggregate reaction. Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town. National Building Institute, Pretoria, Paper No.S252/32, April 1981.

(61) HOBBS, D.W. Expansion due to alkali-silica reaction and the influence of pulverised fuel ash. **Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town.** National Building Institute, Pretoria, Paper No.S252/30, April 1981.

(62) GRATTAN-BELLEW, P.E. A review of test methods for alkali-expansivity of concrete aggregates. **Proceedings of the 5th International Conference on Alkali-Aggregate Reaction in Concrete, Cape Town.** National Building Institute, Pretoria, Paper No.S252/9, April 1981.

(63) FERGUSON, H. Shifting sand relieves site shortage. **New Civil Engineer.** 28th June 1984, pp 20-21.

(64) NEWS : Silica fume may be concrete cureall. **New Civil Engineer.** 10th June 1982, p 4.

(65) GUOMUNDSSON, G. and ASGEIRSSON, H. Parameters affecting alkali expansion in Icelandic concretes. **Proceedings of the 6th International Conference on Alkalis in Concrete - Research and Practice, Copenhagen.** Danish Concrete Association, June 1983, pp 217-221.

(66) TANG MING-SHU, YE YU-FENG, YUAN MEI-QU and ZHEN SHI-HUA. The preventative effect of mineral admixtures on alkali-silica reaction and its mechanism. **Cement and Concrete Research.** Vol.13, No.2, 1983, pp 171-176.

(67) SPRUNG, S. and ADABIAN, M. The effect of admixtures on alkali-aggregate reaction in concrete. Proceedings of the 3rd International Symposium on the Effect of Alkalis on the Properties of Concrete, London. Cement and Concrete Association, Slough, September 1976, pp 125-137.

(68) OWENS, P.L. The use of pulverized fuel ash (p.f.a.) as a method of controlling alkali-silica reaction (ASR) Submission to B.S.I. committee CAB 1/3. July 1980, unpublished.

(69) BUTTLER, F.G., NEWMAN, J.B. and OWENS, P.L. PFA and the alkali-silica reaction. Consulting Engineer. Vol.44, No.11, November 1980, pp 57-62.

(70) HOBBS, D.W. The effect of pulverized-fuel ash upon the workability of cement paste and concrete. Magazine of Concrete Research. Vol.32, No.113, December 1980, pp 219-226.

(71) HOBBS, D.W. Possible influence of small additions of pfa, gbfs and limestone flour upon expansion caused by the alkali-silica reaction. Magazine of Concrete Research. Vol.35, No.122, March 1983, pp 55-58.

(72) B.R.E. DIGEST. Alkali-aggregate reactions in concrete. Building Research Establishment, Garston. Digest 258, February 1982.

(73) CONFERENCE PROCEEDINGS. 6th International Conference on Alkalis in Concrete - Research and Practice, Copenhagen. Danish Concrete Association. June 1983.

(74) OWENS, P.L. Fly ash and its usage in concrete. Concrete, Journal of the Concrete Society. July 1979, Vol.13, No.7, pp 21-26.

(75) OWENS, P.L. Pulverised fuel ash - Part 1,2 and 3. Concrete, Journal of the Concrete Society. Part 1, July 1980, Vol.10, No.7, pp 35-36. Part 2, October 1980, Vol.10, No.10, pp 33-34. Part 3, June 1982, Vol.12, No.6, pp 39-41.

(76) BUTTLER, F.G. Effect of reaction between pulverised fuel ash and calcium hydroxide on concrete properties. Proceedings of Symposium N on the Effects of Flyash incorporation in Cement and Concrete, Boston. Materials Research Society, Annual Meeting, Nov 1981, pp 155-165.

(77) WINER, B.J. Statistical Principles in Experimental Design. McGraw-Hill Book Company, 2nd edition, 1971.

(78) JOHN, J.A. and QUENOUILLE, M.H. Experiments : Design and Analysis. Charles Griffin and Co. Ltd., London, 2nd edition, 1977.

(79) COX, D.R. Planning of Experiments. John Wiley and Sons Ltd., London, 1958.

(80) DAVIES, O.L. et alia. The Design and Analysis of Industrial Experiments. Longman Group Ltd. (for I.C.I. Ltd.), London, 2nd edition, 1979.

(81) DEPARTMENT OF THE ENVIRONMENT. Design of Normal Concrete Mixes. H.M.S.O., London, 1975.

(82) FISHER, R.A. The Design of Experiments. Oliver and Boyd Ltd., London, 7th edition, 1960.

(83) BRITISH STANDARDS INSTITUTION. Methods of testing cement. Part 2, Chemical tests. BS 4550 : Part 2 : 1970.

(84) BRITISH STANDARDS INSTITUTION. Methods of testing cement. Part 3, Physical tests. BS 4550 : Part 3 : 1978.

(85) BRITISH STANDARDS INSTITUTION. Methods of testing cement. Part 2, Chemical tests. Draft amendment slip no.1, published to BS 4550 : Part 2 : 1970, October 1979.

(86) BRITISH STANDARDS INSTITUTION. Pulverised fuel ash for use in concrete. BS 3892 : 1965 (Now superseded by BS 3892 : Part 1 : 1982)

(87) THE AGREMENT BOARD. Pozzolan - A selected fly ash for use in concrete. Agrement Certificate No. 81/841.

(88) BRITISH STANDARDS INSTITUTION. Methods for sampling and testing of mineral aggregates, sands and fillers. Part 2, Physical properties. BS 812 : Part 2 : 1975.

(89) DIXON, W.J. and BROWN, M.B. Biomedical Computer Programs, P-Series. Developed at the Health Sciences Computing Facility, H.C.L.A. Sponsored by NIH Special Research Resources, Grant RR-3. University of California Press, Berkley, Los Angeles, 1979.

(90) ROBERT, E.C. Alkali-silica reaction, Report on 3^3 Full Factorial Programme. The City University, London. Unpublished progress report, March 1981.

(91) BRITISH STANDARDS INSTITUTION. Specification for sulphate resisting Portland cement. BS 4027 : 1980.

(92) HOBBS, D.W. The alkali-silica reaction - a model for predicting expansion in mortar. Magazine of Concrete Research. Vol.33, No.117, December 1981, pp 208-219.

(93) BRITISH STANDARDS INSTITUTION. Methods of testing mortars, screeds and plasters. BS 4551 : 1980.

(94) BOSWELL, L.F., ROBERT, E.C., BUTTLER, F.G. and MORGAN, S.R. Alkali content and expansion of mortars containing pulverised fuel ash. Proceedings of the 6th International Conference on Alkalis in Concrete-Research and Practice, Copenhagen. Danish Concrete Association, June 1983.

(95) CONCRETE SOCIETY WORKING PARTY. Alkali-silica reaction, new structures - specifying the answer, existing structures - diagnosis and assessment. Papers presented at a one-day conference, London. 11th November 1985.

(96) HOBBS, D.W. Excerpts from paper entitled 'Deleterious expansion of concrete due to alkali-silica reaction : influence of pfa and slag'. Confidential submission to Concrete Society working party. Unpublished, July 1985.