

RUBBER BASE IMPRESSION MATERIALS

This thesis, based upon an original study by the author, is submitted to the University of Sydney in support of my candidature for the Degree of Master of Dental Surgery.

A handwritten signature in cursive script, appearing to read 'P.W. Kentwell', with a horizontal line underneath.

P.W. KENTWELL B.D.S.

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TABLE OF CONTENTS

Page No.

ACKNOWLEDGEMENTS

INTRODUCTION

REVIEW OF THE LITERATURE

DEVELOPMENT OF ELASTIC IMPRESSIONS MATERIALS 1

CHEMISTRY

- . Thiokol 6
- . Silicone 13
- . Polyether 20

PHYSICAL CHARACTERISTICS

- . Setting 24
- . Elasticity 38
- . Compatability and Surface Reproduction 50
- . Consistency 59
- . Dimensional Stability 64
 - Unrestricted Dimensional Change 66
 - Restricted Dimensional Change 72

THE ORIGINAL INVESTIGATION

SCOPE OF THE INVESTIGATION 88

PART I

- . Materials 90
- . Apparatus 90
- . Test Method 96
- . Results and Discussion 97
- . Summary 105

PART II

. Materials	106
. Apparatus	107
. Statistical Analysis	110
. Method and Results	112
. Discussion and Conclusions	129
. Summary	140

BIBLIOGRAPHY

142

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INTRODUCTION

This thesis is divided into two parts. Part one reviews the literature concerned with the physical properties of the Rubber Base Impression materials. The chemistry of these materials is included so that the physical properties of the materials may be better understood. Those properties which affect the application of the materials to restorative dentistry are discussed, and special reference is made to the variety of test methods used by researchers to study the physical properties of the materials.

The second part of the thesis is an original research project. Study has been made of the linear dimensional changes of several representative rubber base materials. Dimensional changes of the materials in both restricted and unrestricted conditions have been observed.

Finally, the effects of variables common to clinical practice, and which have direct bearing on the dimensional stability of the materials, have been investigated.

THE DEVELOPMENT OF ELASTIC IMPRESSION MATERIALS

The discovery and subsequent development of elastic impression materials has undoubtedly had a profound effect on the current trends in restorative dentistry. The development of these materials has led to the widespread use of indirect techniques for the construction of inlays and crowns as well as many different forms of fixed and removable prosthetic appliances.

In the early years of this century, following the work of W.H. Taggart on a casting process for dental restorations, many different techniques and materials were used to produce models or dies, from which it was hoped that accurate restorations could be made. Limited success was achieved by the construction of dies from modelling compound and copper band impressions. In the case of full coverage this method proved fairly satisfactory, but with intra coronal restorations and partial veneer coverage, accurate dies were seldom if ever obtained (Hollenback 1965). Whenever it was necessary to remove an impression from undercut areas, either fracture or distortion of the impression material resulted.

It was not until the late 1920s that a truly elastic impression material became available to the dental profession. The development of this material is largely attributed to Alphonze Poller (Phillips 1954). The material was a hydrocolloid produced from agar, an extract of seaweed. Originally developed in Switzerland it was later manufactured

in America by L.D. Caulk and Company as "Dentocol". The popularity of this material for operative procedures is generally dated from around 1936-7, when satisfactory techniques for its use in operative dentistry were developed by A.W. Sears. Skinner and Kern (1938) carried out extensive investigations into the dimensional stability of these materials, and their research with that of Paffenbarger (1940) led to proposed standard specifications for hydrocolloid impression material. By this time the material was being widely used with excellent results in all branches of dentistry where a truly elastic impression material was required.

The ideal material however had still not been found. Hydrocolloid was dimensionally unstable, leading to distorted dies unless models were poured within a very short time after removal of the impression from the mouth. Furthermore, the surface of gypsum products used for the construction of casts and dies was sometimes adversely affected by the exudate from the surface of the impression.

During World War II the supply of agar for dental use virtually ceased, and considerable impetus was given to the further development of a new elastic material. This material was also derived from seaweed and had as its basic ingredient a soluble salt of alginic acid. One of the first references to this alginate material in dentistry was by S.W. Wilding in Britain in 1943. Shoonover and Dickson (1943) presented a

paper on the preparation and characteristics of alginate base impression materials, and stated that the new material was not only a substitute for agar type material but may also displace it for some uses, because of certain superior qualities.

Following the war agar was again available, and the dental profession now had at its disposal two excellent elastic impression materials for the accurate reproduction of tooth and soft tissue surfaces. For the next decade emphasis was directed toward the improvement of the physical properties and methods of manipulation of these materials. °

In 1955 reports of a new impression material began to appear in dental literature. This new material was developed from polysulphide liquid polymers, which had been in wide industrial use for many years. The liquid polymers were first produced in the Thiokol Laboratories (U.S.A.) in response to requirements of the synthetic rubber industry. The rubbers evolved proved to have physical properties which made their adaptation to dental use most desirable. They were simple to use and required no special or expensive armamentarium. When cured the material had the strength and resiliency to be removed from undercut areas and to spring back to its original shape and dimensions. The chief advantages of these polysulphide rubbers over the reversible (agar) and irreversible (alginate) hydrocolloids was their remarkable dimensional stability over a wide range of atmospheric conditions and long periods of time. They also showed

excellent compatability with gypsum products used for making casts and dies, and they could be readily electroplated to produce excellent metal dies.

The polysulphide rubbers did however have disadvantages. They showed marked variation in setting time with varying room temperature. They also possessed a characteristically unpleasant odour, and because of the lead peroxide used as the curing agent they were a dark brown disagreeable colour.

About 1956 yet another elastic impression material appeared on the market. This was a silicone polymer which showed great promise as a useful dental material, but initially it had a number of undesirable properties. It was superior to polysulphide in aesthetics, manipulation and cleanliness. However, it had a poor shelf life, produced gas during polymerization which adversely affected the surface of gypsum casts, and it could not be as readily electroplated as polysulphide. Its elastic properties, however, were satisfactory and closely paralleled those of the polysulphides.

The subsequent development of silicone rubbers has largely eliminated their undesirable properties, and excellent products fulfilling all the requirements of specifications for rubber base impression materials are now available.

The most recent addition to the elastic impression materials has been described as a Polyether. This material has been developed and marketed by the Espe Co., of West

Germany. Their manufacturing process was patented in Britain in 1966, and the dental impression material is marketed as "Impregum". Very little investigation of the physical properties of this material has been reported in dental literature, but the manufacturers claim several advantages for their product over both the polysulphide and silicone classes of material.

CHEMISTRY

THIOKOL

Development

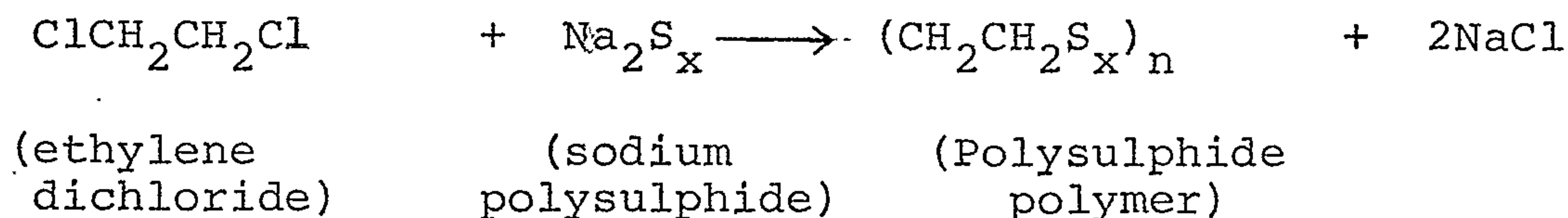
The polysulphide polymers from which the dental Thiokol rubber impression materials have been developed were first introduced commercially in 1929 (Fettes and Jorcyak 1950). Their excellent oil and solvent resistance, as well as good ageing properties made them valuable in a large range of special applications, particularly as sealants, and as liners for the storage of bulk petroleum products. However these polymers were of very high molecular weight, and formed extremely tough crude rubbers which had no application in dentistry.

In 1943 the Thiokol Laboratories in the United States developed liquid polysulphide polymers as a result of research to find a method of reducing the molecular weight of the existing materials which were too tough to process on conventional rubber milling equipment, without being previously chemically plasticized. It is these liquid polymers which can be conveniently cured or vulcanized, at room temperature to form resilient, stable, non irritant rubbers which have a wide use in dental practice.

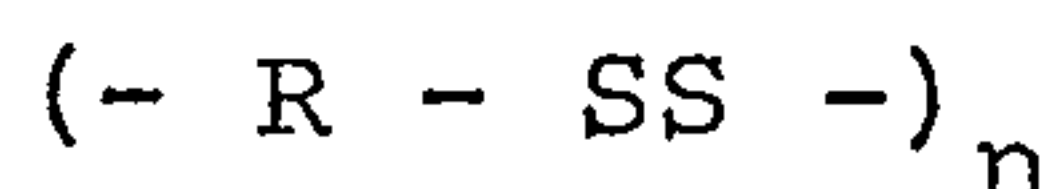
Much of the development of the early polysulphide polymers is attributed to J.C. Patrick of the Thiokol Chemical Corp. (Kinghorn and Allan 1957, Gaylord 1962).

Chemistry

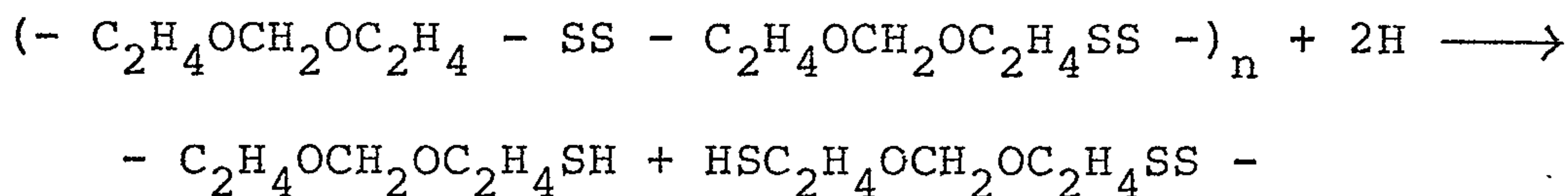
The preparation of polysulphide polymers is an example of condensation polymerization consisting simply of the reaction of an alkaline polysulphide with a suitable organic dihalide, e.g.



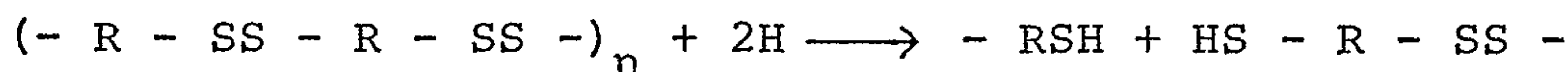
The structure of these polymers can be conveniently represented by the repeating segment



In order to produce the liquid polysulphide polymers of low molecular weight, depolymerization of the long chain polysulphide polymers is brought about by reductive cleavage of the disulphide groups in the chain, to yield short chain products with thiol (or mercaptan) end groups. Jorcyak and Fettes (1951) give the following example of this reduction for a polymer prepared from dichloroethyl formal.

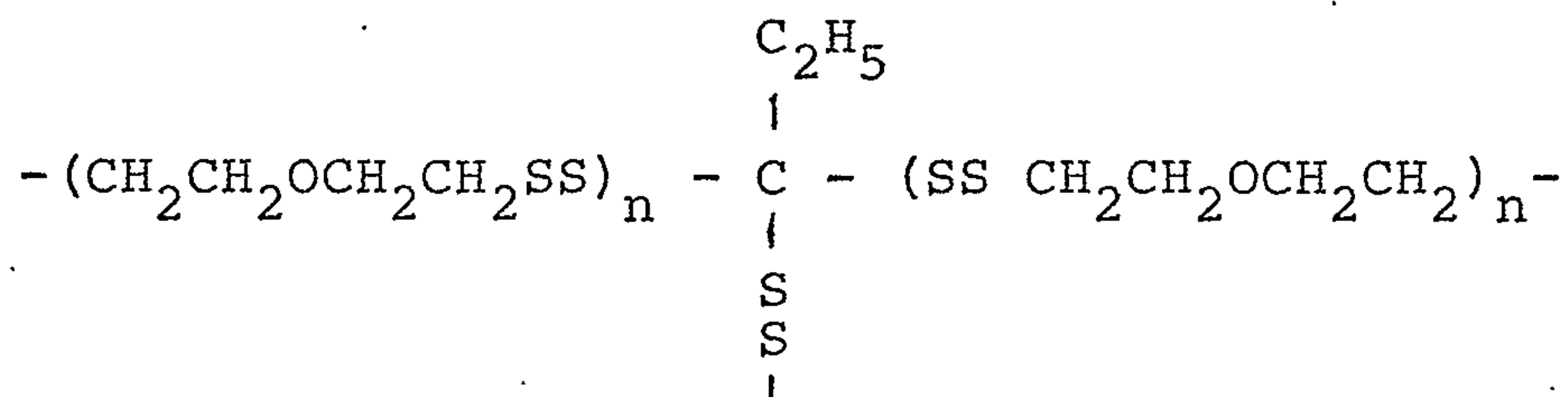
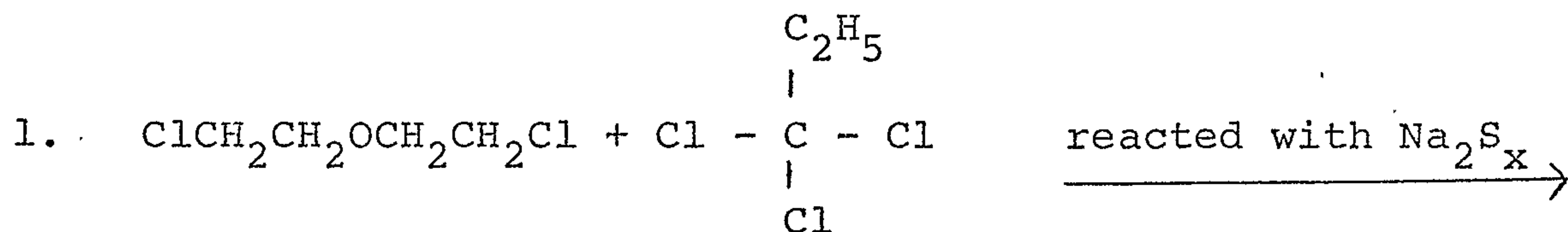


Using the representation above this can be more conveniently expressed as

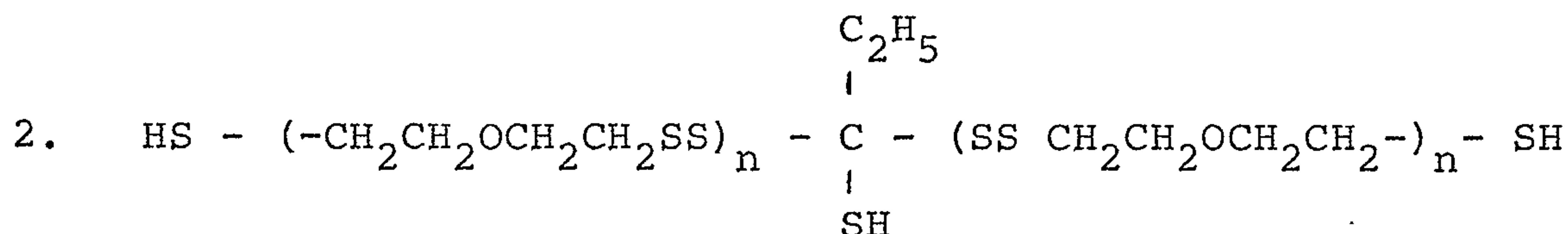


The amount of depolymerization can be readily controlled, and the liquid polymeric product can be readily reconverted to a rubber of high molecular weight by reunion of the thiol end groups.

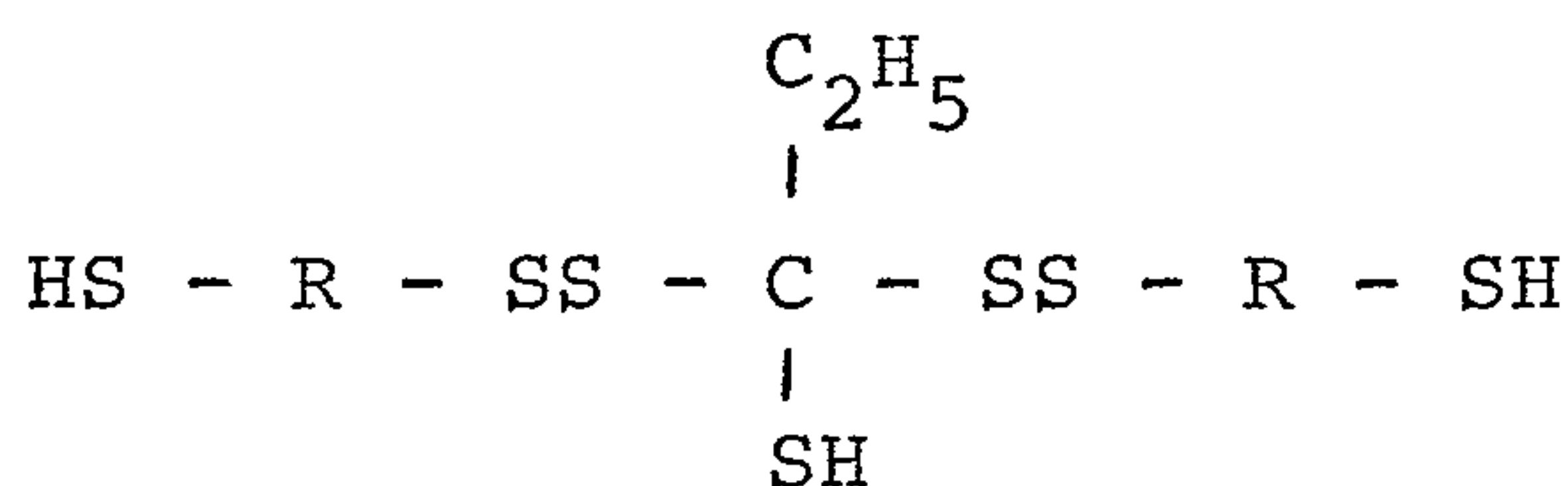
The polymer of particular interest to the dental profession is known commercially as LP-2 and has a molecular weight in the vicinity of 4000. These particular polymers are prepared from dichloroethyl formal with a small amount of trifunctional halide, such as trichloropropane, used to produce a branched polymer. The vulcanized or cured product thus consisting of long linear chains held together by the small amount of trifunctionality placed in the polymer by the trichloropropane. This cross linking is essential to obtain materials with the desired properties for dental impression materials. Braden (1966) points out that textbooks on dental materials generally omit, in their explanation of the curing reaction, this vital feature of cross linking in the polymerization process. Braden presents the stages in the preparation of liquid polysulphide polymers as follows :



This material must be reduced, as described, by cleaving the disulphide groups to leave a material of low molecular weight with both terminal and pendant thiol groups.

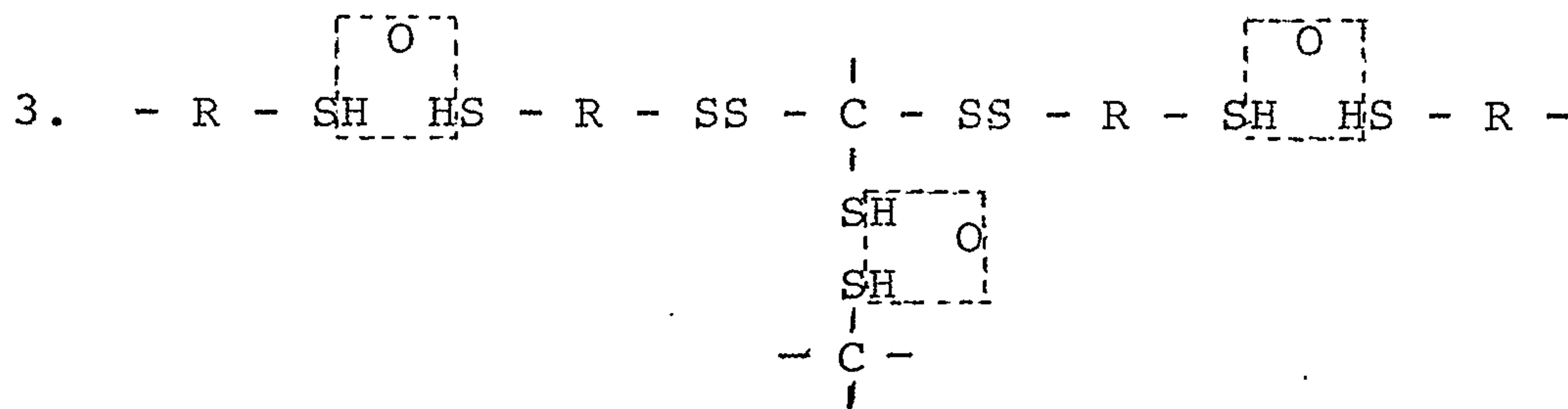


or



The vulcanization or curing of the liquid polymers is a reaction of direct oxidation. This results in the linking of terminal and pendant thiol groups to form the polymeric, cross linked disulphide, with the liberation of water as a by-product.

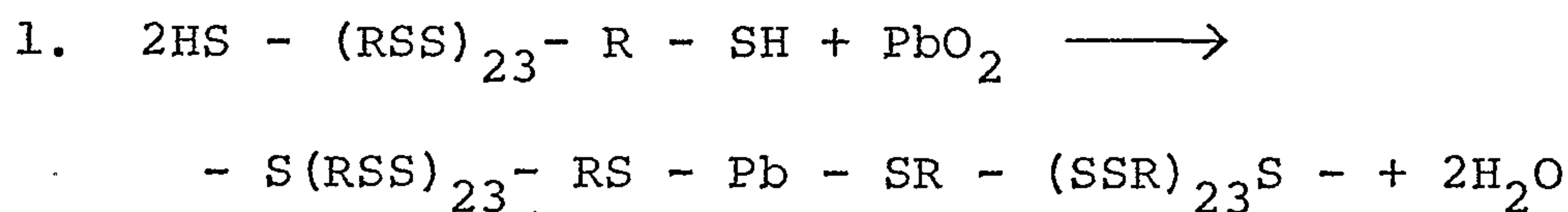
(NOTE Braden has presented a reaction based on dichloroethyl ether not dichloroethyl formal, but the stages of the reaction are valid.).

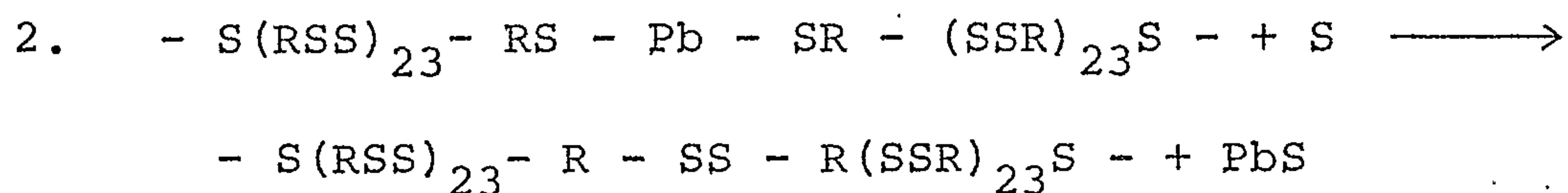


Any reagent which is capable of reacting with two thiol end groups can be potentially useful as a curing agent but lead peroxide has proved one of the most versatile agents and is most suitable in dental preparations, since it will readily vulcanize at room temperature. Heating accelerates the reaction and because the reaction is exothermic, once conversion has started it proceeds at a rapid rate.

In this reaction with lead peroxide the cured product may contain lead mercaptide bonds in the polymer chain, resulting in poor physical properties, especially compression set which is so important in dental rubbers. Additional amounts of sulphur along with the lead peroxide in the curing agent tend to tighten the cure by eliminating the mercaptide as lead sulphide.

In dental literature, Fairhurst et al (1956), Skinner (1958) and Miller et al (1960) represent the complete curing reaction of a dental polysulphide rubber in the following two stages :





where R = (C₂H₄OCH₂OC₂H₄) dichloroethyl formal.

(Note the omission of reference to cross linking in this reaction.)

Because of the specialized application of dental polysulphides, and the well known toxicity of lead compounds, the question was raised very early as to the toxic effect of the lead content in these materials. Tests carried out by Pearson (1955) indicated that there was no evidence of contamination, and showed the material to be quite safe for dental use.

Composition

Most uses of the liquid polysulphide polymers require the addition of fillers as reinforcing and strengthening agents. The industrial polysulphides use a wide variety of these fillers, depending on the requirements of the particular rubber being produced. Several compounds have been mentioned as possible ingredients of dental rubbers. Pearson (1955) and Phillips (1957) suggest zinc oxide and calcium sulphate as possible fillers. Pearson giving the following formula:-

<u>Base</u>		<u>Accelerator</u>	
Polysulphide polymer	- 79.72%	Lead peroxide	- 77.65%
Zinc oxide	- 4.89%	Sulphur	- 3.52%
Calcium sulphate	- 15.39%	Castor Oil	- 16.84%
		Others	- 1.99%

This formula has been queried by Braden (1966), who suggests titanium dioxide as the most likely filler, pointing out that zinc oxide itself may have an effect as a converting agent. Jorcyak and Fettes (1951) when referring to industrial rubbers stated that zinc oxide, while being a good reinforcing filler must be used with special attention because it also acts as a converting agent.

Similar doubt exists concerning the vehicle for the lead peroxide catalyst. It is generally accepted (Jorgensen 1957) that the smaller the particle size of the lead peroxide the more rapid will be the polymerization because of the higher reactivity of the finer catalyst. Because of this reactivity, Braden (1966) suggests that it is most unlikely that such an oxidisable material as castor oil would be used as a vehicle. However although he lists accelerator pastes with a lead peroxide content, ranging from 52% to 82.5% he does not suggest what materials form the remainder of these pastes.

Some control of the rate of cure can be exercised by adjustment of the pH of the reaction. Since the oxidation of thiol groups is accelerated in an alkaline environment, it follows that the addition of acidic retarders will prolong the curing time. To this effect stearic or oleic acid may be included in the catalyst paste.

From the time of development of the earliest liquid polysulphide polymers, odour has been a problem. This has

been frequently presented as a disadvantage of these materials for use in dentistry. The unpleasant odour was caused by the presence of a small percentage of low molecular weight, volatile dithiols in the liquid polymer. However with the production of polymers from dichloroethyl formal the odour has been greatly reduced. Conversion, by oxidation, to the higher polymer eliminates the volatile dithiols so that the cured material is virtually odourless.

SILICONE

Development

The foundations of organosilicon chemistry were laid in the period 1899-1944 by F.S. Kipping of University College Nottingham, U.K. but nothing in his published work suggests that he foresaw any great technical usefulness for the silicone polymers (Freeman 1962). It was Professor Kipping who coined the term 'Silicone' in silicon chemistry.

Extensive research was carried out by Dr. J.F. Hyde of the Corning Glass Works in the U.S.A. between 1931 and 1940, and this work laid the real foundation for the development of the industrial silicones. In 1940 the General Electric Company became interested in this field, and their work was concentrated on the methyl silicon compounds from which the dental silicone rubbers were eventually developed.

Fordham (1960) states that the early silicone rubbers were extremely weak, had high shrinkage, high compression set,

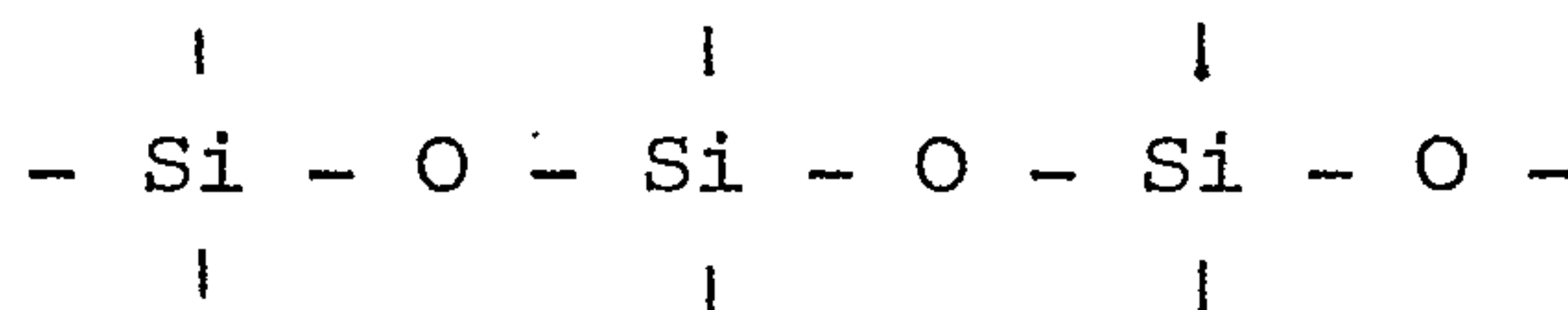
poor solvent resistance, poor processing characteristics, and batch to batch variations. The main application of these rubbers was due to their exceptional resistance to adverse operating conditions, particularly extremes of temperature. Silicone rubbers have been developed with relative constancy of physical properties over a temperature range from -80°C to 250°C .

The years since World War II have seen constant improvement in the physical and mechanical properties of silicone compounds which are now produced in considerable quantity for an ever increasing range of uses. The biggest consumer is the aircraft industry. It has been estimated that a modern passenger aircraft contains about 1000 lbs. of silicone rubber in a wide range of uses involving high and low temperature properties, as well as electrical insulation. In medicine, because of their physiological inertness and lack of toxicity, they are proving excellent for such things as transfusion and drainage tubes etc. They are also used to produce life-like prosthetic replacements in the field of maxillofacial surgery (Kanter 1970).

Chemistry

The simple silicon compound, silane (SiH_4), containing one silicon atom, forms the base from which the very much more complicated silicone compounds are built up. The union of two silicon atoms in a molecule produces a

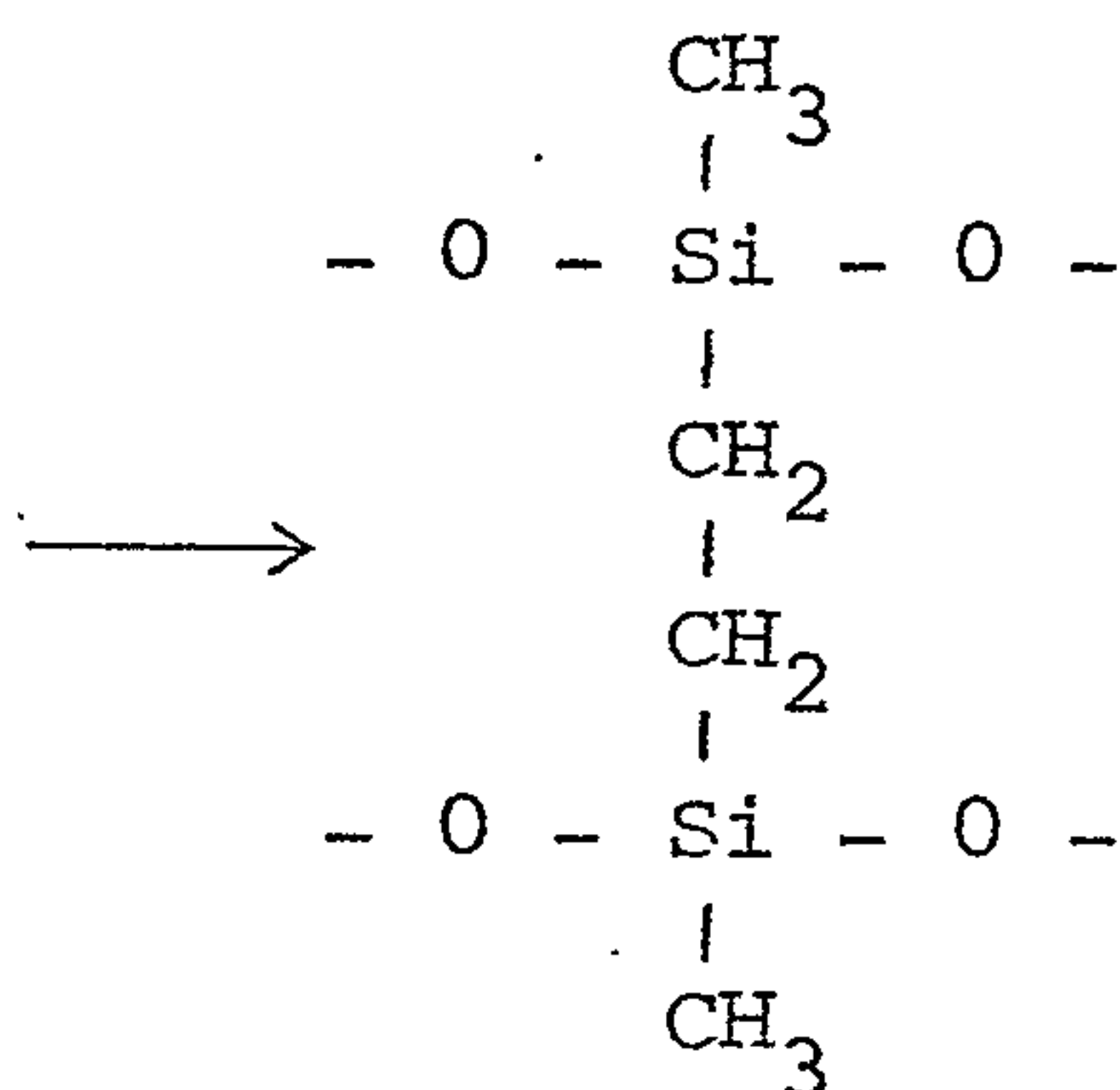
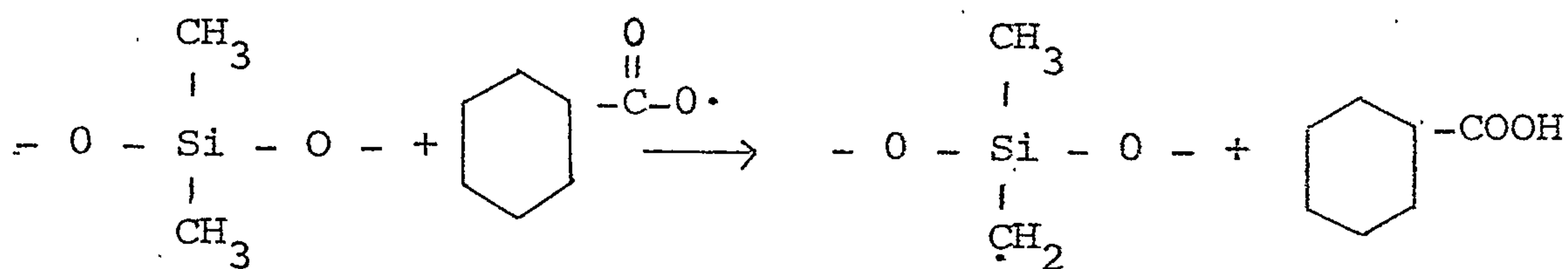
disilane ($\text{H}_3\text{Si-SiH}_3$), three atoms of silicon, a trisilane, and so on to the production of polysilanes. In silicones, the adjacent silicon atoms in the molecule are separated by oxygen atoms, thus :



This is called a siloxane linkage and where a large number of such groupings are present the compound is known as a polysiloxane.

The basis of silicone rubbers is a high molecular weight linear polysiloxane or silicone gum. These gums possess no elastic properties at room temperature but by the addition of curing (cross-linking) agents and reinforcing fillers, at elevated temperatures, silicone rubbers can be produced.

Organic peroxides are widely used as curing agents, the most common being benzoyl peroxide which brings about cross linking of dimethyl polysiloxane by the following mechanism (Fordham 1960):-

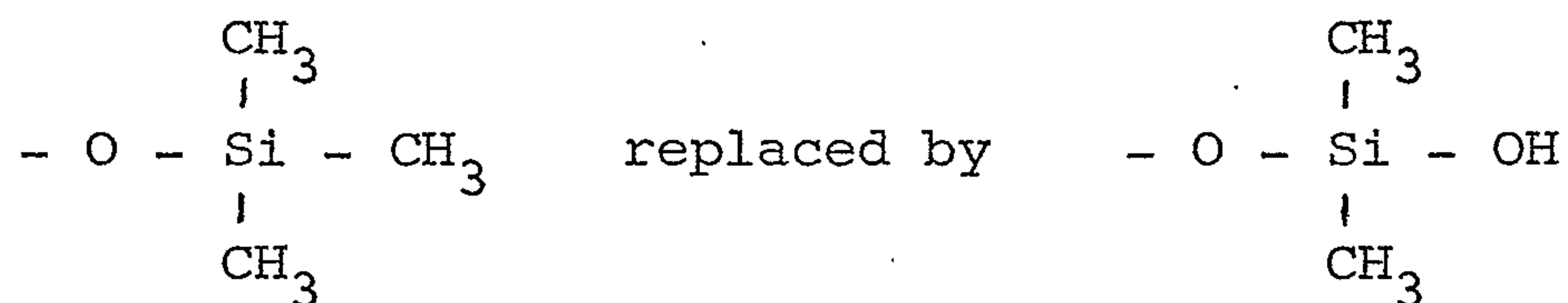


This reaction produces a rubbery gel which is very poor in physical properties and must be heavily reinforced to obtain useful products. To this end a variety of reinforcing fillers are used, the most common being finely divided silica. Coarser grades of silica (diatomaceous earths), calcium carbonate, titanium oxide and iron oxide may also be used as fillers. McLean (1958) lists zinc oxide as the principal filler in dental silicone rubber. The mechanical strength of the rubber is largely imparted by the addition of these inorganic fillers, and the elastic properties are obtained by the inter-molecular cross-linking of the polymer chains.

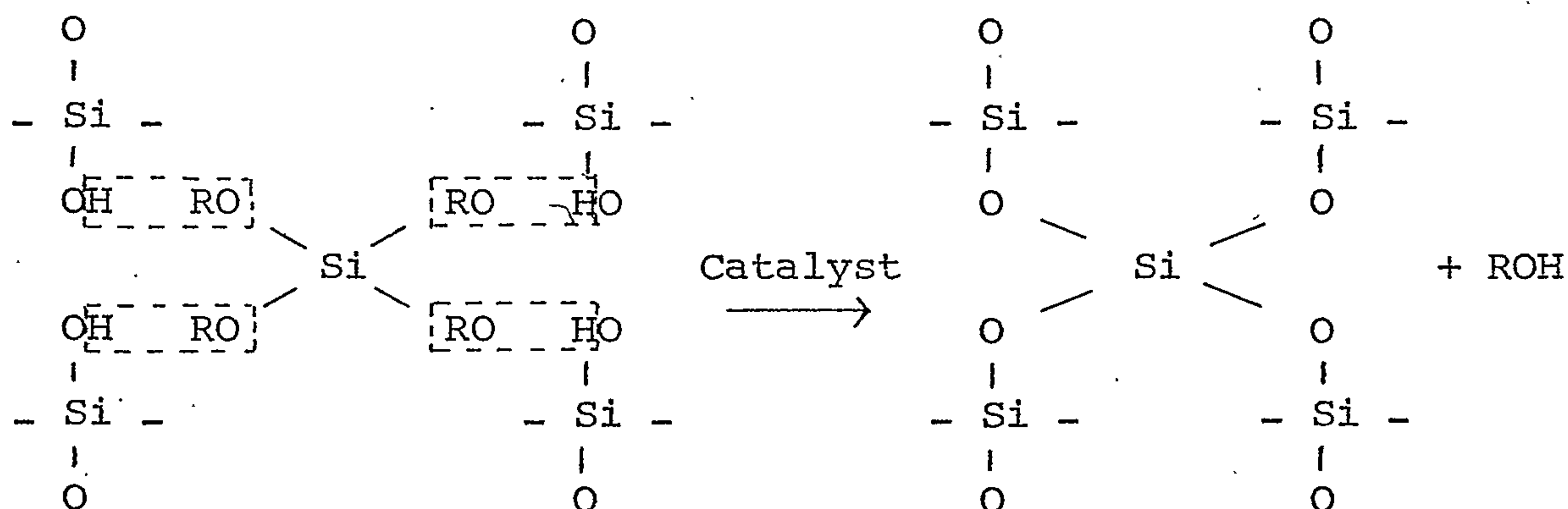
As previously mentioned these industrial elastomers are mainly cured at elevated temperatures, in the order of 115°C-170°C. Further curing may be necessary in temperatures up to 250°C in order to obtain optimal values for mechanical properties.

The rubbers of interest to the dental profession must of course be capable of being cured at body temperature in a relatively short time. Fortunately industry also has use for rubbers of this nature, and the R.T.V. (Room Temperature Vulcanising) rubbers have been developed (Freeman 1962).

By replacing the trimethyl end group in the polysiloxane gum with an hydroxy, or silanol, end group,



the reactivity of the polymer is increased to the point where it can be vulcanised or cured, in the presence of a suitable catalyst, in from a few minutes to a few hours, at room temperature. Organotin compounds such as dibutyltin dilaurate are used to catalyse this reaction. The silanol end-blocked polymers condense with a tetrafunctional silicon ester, in the presence of the catalyst, to produce the necessary cross linked polymer. The reaction taking place as follows :-

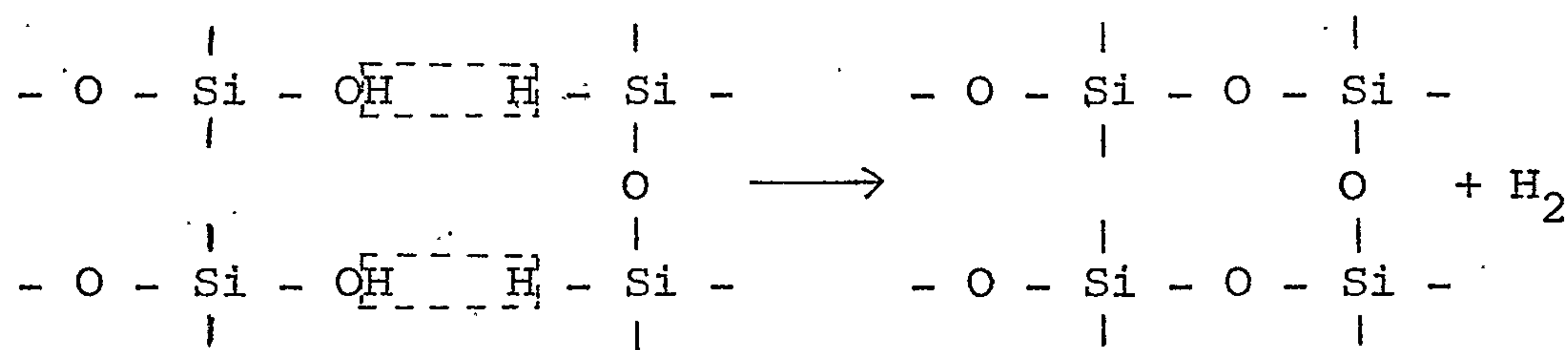


The ROH groups split off in this reaction are volatile alcohols which may be responsible for the gas production reported in the early impression materials (McLean 1958, Phillips 1959). They may also be responsible for a percentage of the shrinkage of these materials on curing, because of evaporation (Braden 1966).

McLean (1958) has given the following probable composition of a silicone R.T.V. paste and accelerator.

<u>Paste</u>		<u>Accelerator</u>	
Dimethylpolysiloxane	50%	Dibutyltin dilaurate	75%
Zinc Oxide	50%	Tetraethyl polysilicate	25%

Another possible mechanism of cold vulcanization involves reaction of silanol groups with a silicon-hydrogen bond (methyl-hydrogen polysiloxane) which may be introduced into the polysiloxane chain. The cross linking in this case produces hydrogen as a reaction product, and this appears to be the more likely source of gas evolution during curing.



The problems associated with hydrogen production during the curing of these rubbers have been minimized by the inclusion of hydrogen acceptors such as aldehydes or chromium oxide (Skinner 1958).

As a result of the curing reaction the long chains of monomer units which make up the linear polymer are cross linked

and anchored together by chemical bonds. These bonds are widely separated in the polymer chains which are randomly kinked in three dimensions, but may be straightened by the application of an external force. The cross linked assembly opposes this force and sets up a reaction which tends to return the assembly to its original state, i.e. the material develops elastic properties.

POLYETHER

Development

In 1966 a new elastomer system was added to the polysulphide and silicone systems already in existence. This system was developed by the German pharmaceutical company ESPE, and is based on polymers containing ethylene imine groups. Polymers suitable for the formation of these synthetic elastomers are substantially linear polyethers with between 1 and 5 ethylene imine groups per molecule and a molecular weight between 5000 and 6000.

The developers of this system claim that the previously known synthetic rubbers have the disadvantage that their preparation processes are comparatively complicated or expensive, and in the case of silicones yield products having unsatisfactory mechanical properties, particularly when made without fillers. They also point to the unpleasant odour of

the polysulphides before, and to a lesser degree after, cross linking and claim the absence of odour of the polyether rubbers as an advantage.

The polyether elastomer has been developed for a wide range of technical applications. The product is used to fabricate seals, and electrical coatings. It has several applications in the automobile and building industries and has also found a place as a dental impression material with some claimed advantages over similar products in this field.

The inventors of the material claim that it is stable and dimensionally accurate, and most importantly that the curing reaction can be so adjusted that cross linking will not become effective, and increase the viscosity, immediately after the addition of the cross linking agent. This allows the base and the catalyst to be mixed to a smooth paste and carried to place in the impression tray, and then for the curing reaction to take place quite quickly. (Further reference will be made to this property in the section of this review dealing with working and setting times of the elastomeric materials).

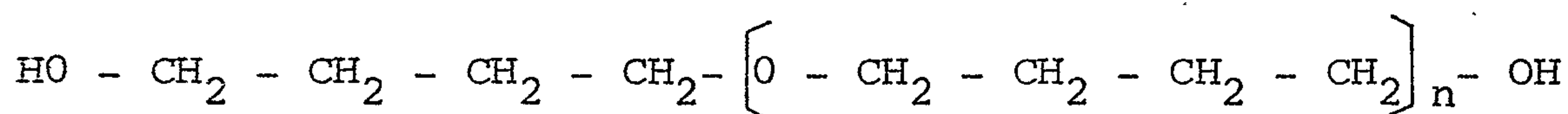
Chemistry

Personal communication with the manufacturer (Purmann 1970) of the only polyether material at present marketed in this country, yielded the information that the basic ingredient of the material is a polyether, mainly of tetramethylene glycol with terminal aziridino groups. The catalyst used to convert this base to a rubber contains a sulphonic acid ester,

which, by reacting with the aziridino groups, causes cross linking and the formation of a stable elastomer.

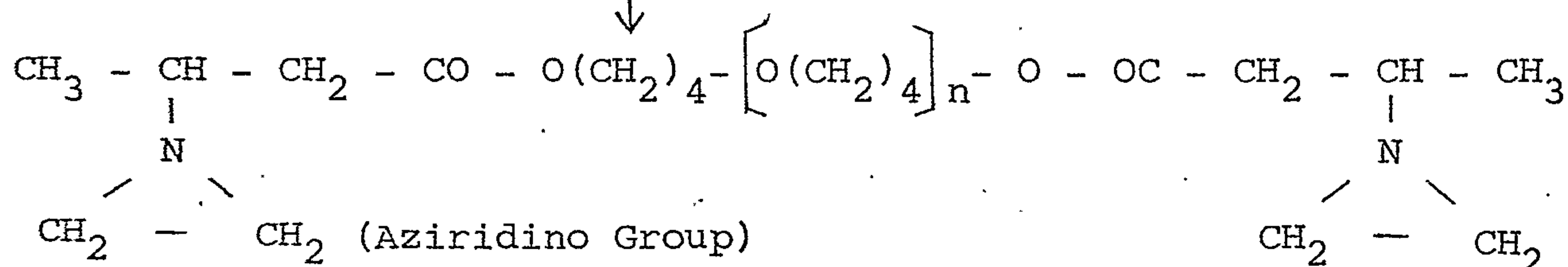
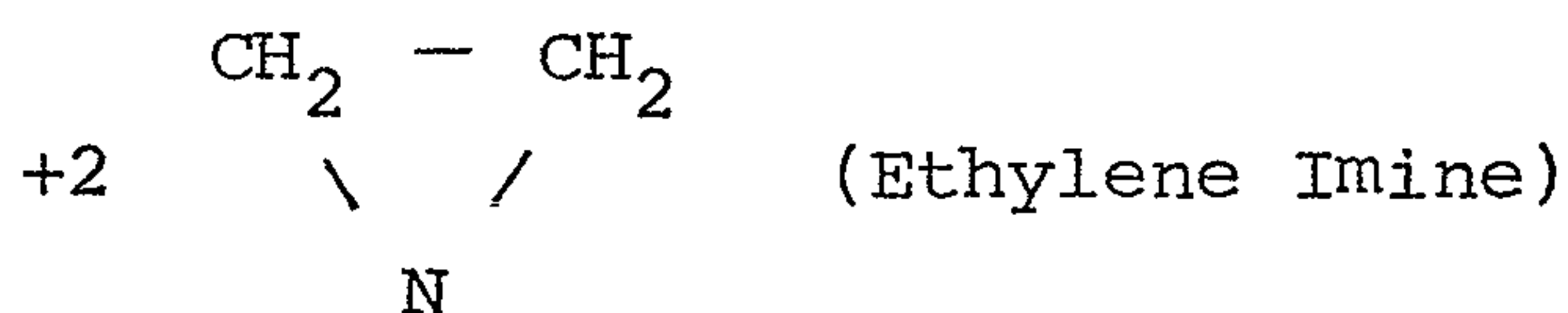
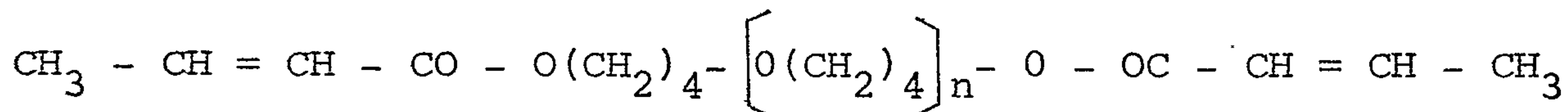
A study of the information supplied in the British Patent No. 1,044,754 leads to the following probable general chemical development of this polyether class of impression material.

Stage 1 - The formation of a linear polymer based mainly on tetramethylene glycol :

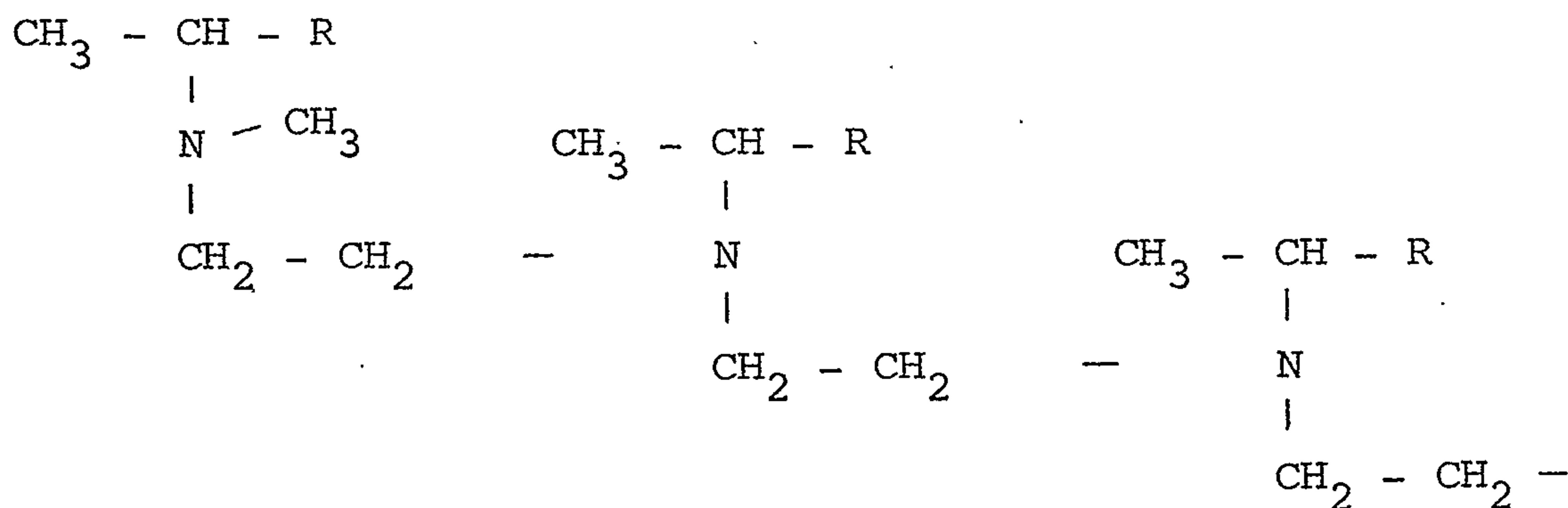


where n would equal approx. 80 to give a molecular weight of ≈ 6000 .

Stage 2 - The addition of reactive groups to the polyether to enable ethylene imine to be added to the molecule. To effect this the starting material may be reacted with crotonic anhydride to establish double bonds at each end of the molecule. The ethylene imine groups can then be added across the double bonds and aziridino groups formed



Stage 3 - The final stage is to cross link the polymer formed by stage 2 and this is done by mixing with a catalyst containing a sulphonic acid ester. This activates the aziridino groups which unite with one another, bringing about a three dimensional cross linking. If a methyl ester were employed, the cross linking could be represented thus :



where R stands for the rest of the molecule from Stage 2.

By the addition of an inhibitor to the mix, the start of the cross linking reaction can be slowed down until most of the inhibitor has been used up. Tertiary amines such as triethylamine are mentioned as possible inhibitors. When this has occurred the cross linking takes place fairly rapidly and this would account for the particular nature of the setting process noted with this material.

As with the other elastomers the physical properties of polyether are influenced by fillers etc., incorporated in the paste. Finely divided silica has been mentioned in the Patent as a probable filler and dibutyl and dioctyl phthalate, as plasticisers.

PHYSICAL CHARACTERISTICS

SETTING

The mechanism which governs the setting process of the rubber base impression materials occurs as a result of linear polymerization, which increases viscosity, and of cross linking which introduces an elastic property to the material. These physical changes progress to the stage where finally an elastic solid is formed.

The measurement of the change in viscosity of the setting material allows us to determine the physical changes taking place which are significant in its correct use as an impression material. The two most important determinations are :

1. The time available for the manipulation of the material.
2. The time at which the physical properties are such that the impression can be removed from the mouth with minimal distortion.

The first of these determinations has been variously referred to as 'working time' or 'initial set' and the second as 'setting time' or 'final set'. (For the purposes of this paper the terms Working Time and Setting Time will be used). Ideally the working time should be long enough to allow the impression procedure to be carried out comfortably and without haste. The setting time should then as short as possible, to reduce patient discomfort and to minimise the possibility

of inducing strains in the setting material due to movement of the impression tray. There must be sufficient working time to permit thorough mixing, unhurried loading of the tray, injection of the impression material into and around the preparations and finally the careful seating of the tray. The physical change should then progress rapidly to prevent any distortion of the material while the tray is in position and to minimize the time required for holding the tray in the mouth. Wilson (1966) suggests that the ideal material should maintain constant viscosity for five minutes at room temperature, and then change immediately to a solid.

Numerous investigators have suggested different methods of assessing the working and setting times of these materials under a variety of changing conditions, which have direct clinical significance upon their use. Before considering the effect of these conditions on the setting reaction it is of interest to review the methods by which these changes have been measured.

Test Methods

Fairhurst et al (1956), studying the elastic properties of Thiokol assessed the setting time as the period from the commencement of mix until the material lost its tackiness to light finger pressure. However, in their conclusions from this investigation, they stated that this was not a satisfactory indicator of setting time for the materials

studied, because the physical properties exhibited at this stage would not allow their use as impression materials. Clearly a more significant and reliable test was needed.

Clark and Phillips (1957) applied a $\frac{1}{4}$ lb Gilmore needle to freshly mixed specimens of Thiokol at 15 second intervals from the commencement of mix. The impression material was readily penetrated at first, and upon removal of the needle would flow into and fill the impression made by the needle. The working time was the time at which the material would no longer flow into and fill this impression. The setting time was the time at which the needle no longer penetrated the surface but made the first of a series of indentations of similar depth. This test was also used by Peyton and Meyers (1959) in relation to both Thiokol and Silicone materials.

For a long time the most common method of measuring working and setting time was by adapting the penetrometer test used in the American Dental Association Specification No. 11 for determining the temperature of gelation of hydrocolloid impression material.

Skinner and Cooper (1955) used this method to examine the effect of temperature on both working and setting time. McLean (1958) carried out similar tests using different commercially available brands of silicone. In describing these tests McLean pointed out that since silicone elastomers (and Thiokol and Polyethers) are polymerizable materials, the term setting time has no real meaning. He demonstrated

that the materials continued to polymerize virtually indefinitely, and that tests carried out on them with the penetrometer were in effect, an indication of the degree of polymerization.

McLean used a penetrometer which permitted the needle holder to move in a guide without appreciable friction, and which was capable of measurements of penetration to 0.1mm. The total moving weight in this investigation was 100 ± 0.25 gm. In contrast Myers and Peyton (1959) and Miller and Myers (1962) used a penetrometer with a total moving weight of 300 gms.

In all these tests the penetrometer needle was placed on the specimen and allowed to penetrate for a given period, when the depth of penetration was noted. As the polymerization of the material progressed, so a decrease in the degree of penetration became evident, until eventually a series of similar indentations was obtained. The time from beginning of mix until the last complete penetration of the specimen was recorded as the working time, and the time from the beginning of the mix until the first of three similar consecutive readings was taken as the setting time.

Both these teams of investigators (Myers and Peyton, and Miller and Myers) also determined working time by means of a syringe test which Myers and Peyton asserted was of more clinical significance than the penetrometer test. In this method, a rubber base syringe with a standard nozzle was used.

The material to be tested was loaded into the syringe and a small quantity was ejected on to a paper pad at 30 second intervals, under normal hand pressure. With the first specimen ejected, the material coalesced and formed a homogeneous globule. Progressively, specimens showed a decreasing tendency to do this, until eventually a specimen failed to coalesce and formed an irregular globule. The time at which this specimen was obtained was taken as the working time.

The use of a more sophisticated machine for measuring the viscosity changes in setting rubbers was mentioned by Ellam & Smith (1966) and further developed by Wilson (1966). Wilson described this machine as a reciprocating rheometer which allows the accurate measurement of viscosity changes with time. With this machine, the change in viscosity of the test material is transmitted through a penarm to a revolving drum, and a characteristic trace is produced. The specimen mix is placed on a grid and a flat plate is drawn over the mix at a given rate. The resistance to draw is recorded as a trace on the revolving drum. The movable plate has a travel of 1 mm at a speed of 10 cycles per minute. Wilson had his machine fitted with water jackets so that constant temperatures up to $90^{\circ}\text{C} \pm 1^{\circ}\text{C}$ could be maintained for given experiments. The characteristic curve of a rubber base material as produced by the rheometer is shown in Fig. 1(a)

In evaluating this trace, working time has been

assessed as the time from beginning of mix until the width of the trace is 95% of the initial width. The setting time is calculated as the time when the trace becomes parallel. The smaller the difference between these two times the closer the setting reaction of the material approaches the ideal. Wilson has represented the ideal impression material as producing the rheometer trace shown in Fig. 1(b). This shows an instant change from a constant working viscosity to a solid which would prevent the material deforming after it had begun to set, thus inducing residual stresses in the set material.

In order to compare tests on materials using the reciprocating rheometer with the more conventional penetrometer tests, Wilson ran tests on the same series of materials, under similar conditions using both types of apparatus. To determine working time with the penetrometer he used a needle of 4 mm diameter and a total load of 30 ± 1 gm. Because of the increased resistance to penetration, with advance in set, a thinner needle (1 mm) and a greater load (100 ± 1 gm) were used to determine setting time. It was found that results obtained by both methods were in general agreement.

Miller et al (1960) used similar variations in penetrometer needle diameter and load to separately determine working time and setting time.

Fig. 1(a) - Characteristic Rheometer tracing produced during the setting of a typical rubber base impression material. (Wilson 1966).

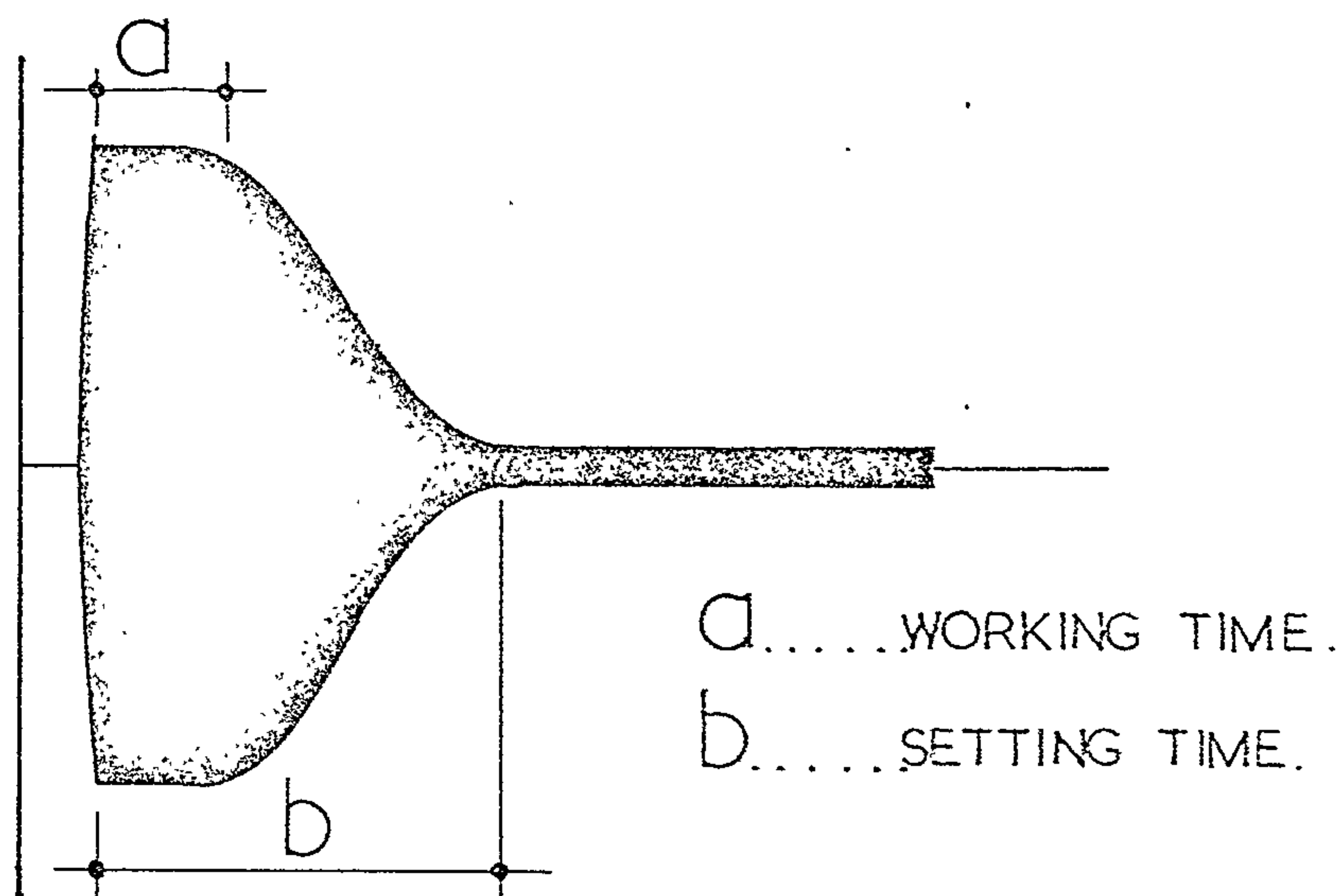
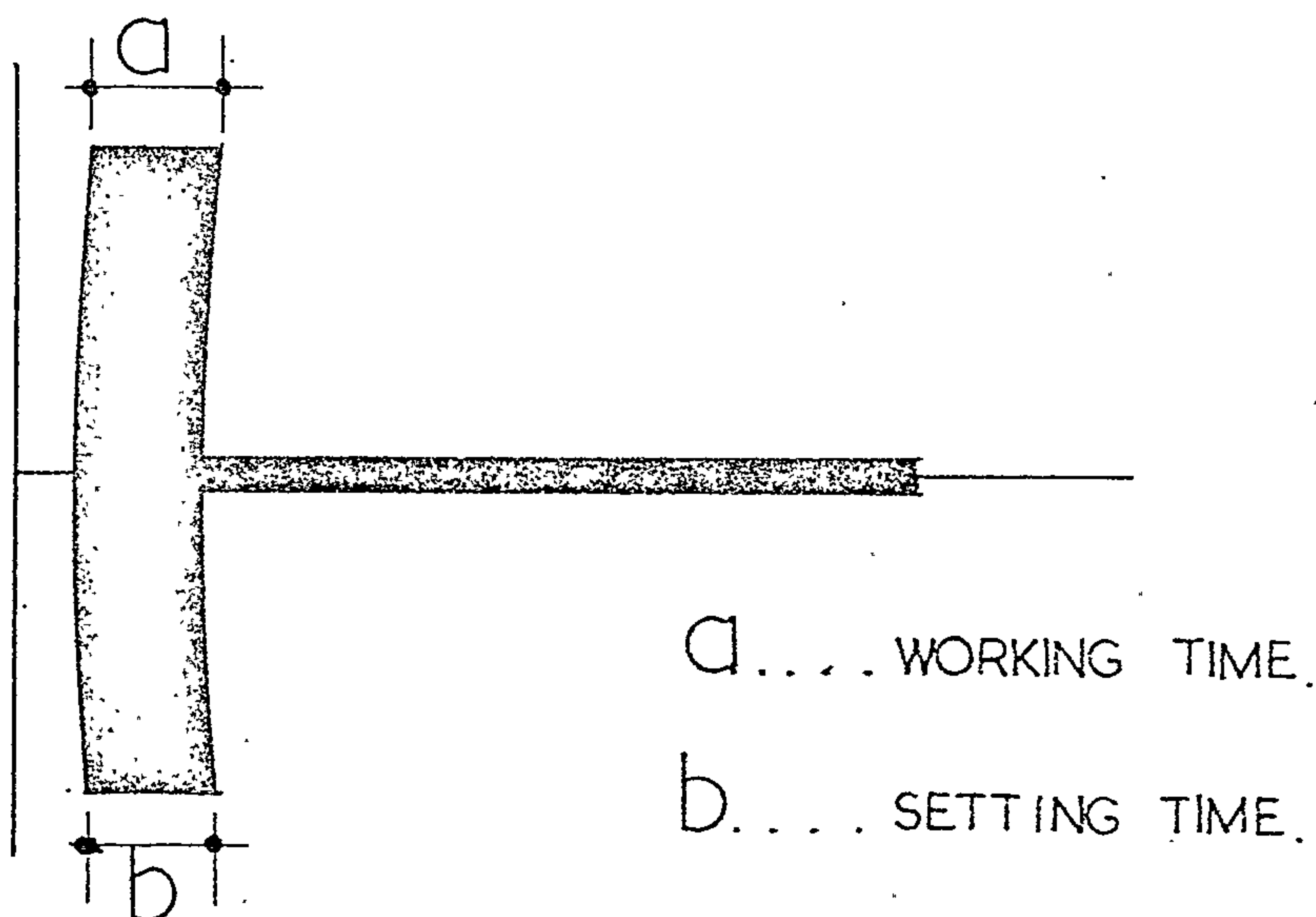


Fig. 1(b) - Rheometer tracing which would be obtained for an ideal impression material. (Wilson 1966)



Braden and Elliott (1966) and Braden (1966) investigated the setting characteristics of rubber base materials using a method of Extrusion Viscometry. This is a measure of the force necessary to extrude a polymer, at a constant rate through a cylindrical capillary, measured as a function of time. It was found in the case of polysulphides that the force necessary increased steadily with time, indicating that for these materials the transition from a viscous to an elastic material is not well defined. This fact is also well illustrated by Wilson's trace. Braden attributes this to the effect of the accelerator reacting with both the terminal and pendant mercaptan groups of the polymer. It is suggested that this contrasts with the setting of silicone materials which are already long chain, high molecular weight polymers and only require cross linking to convert them to elastic materials. The silicones therefore show a more definite transition from the viscous to the elastic state. One could assume, from the chemistry of setting, that polyether materials would also exhibit this more definite transition.

The data amassed by the various methods described has given a clear indication of the effect of significant variables on the rubber base materials. The factors of significance to consider are :

- (a) Temperature and Humidity
- (b) Accelerator-Base ratio.

Temperature and Humidity

The effect of temperature has been shown to have a considerable influence on the rate of set of the polysulphide materials, an increase in temperature decreasing both working and setting time.

Skinner and Cooper (1955) conducted setting experiments at 25°C and 37°C using a penetrometer apparatus. They found that for a representative batch of eight commercial brands of polysulphide material, the working time varied between 3.0 and 9.0 minutes, and the setting times varied between 5.0 and 12.5 minutes when measured at 25°C. At 37°C the two properties varied between 2.0 and 4.0 minutes and 3.0 and 7.5 minutes respectively.

Jorgensen (1957) stated that a rise in temperature of 10°C doubles the reaction rate for polysulphide, and this closely relates to the findings of Skinner and Cooper.

The silicones react similarly to the polysulphides but to a lesser degree (Myers 1958).

Myers and Peyton (1959) and Miller and Myers (1962) carried out working time tests on a number of commercial silicones at 10°F intervals, from 50°F to 100°F, using the syringe method referred to earlier. In general the findings of the two groups were in agreement, and showed that the silicones were less sensitive to temperature change than the polysulphides. Miller and Myers suggested that this reduced sensitivity to temperature variation was an advantage of the silicone materials.

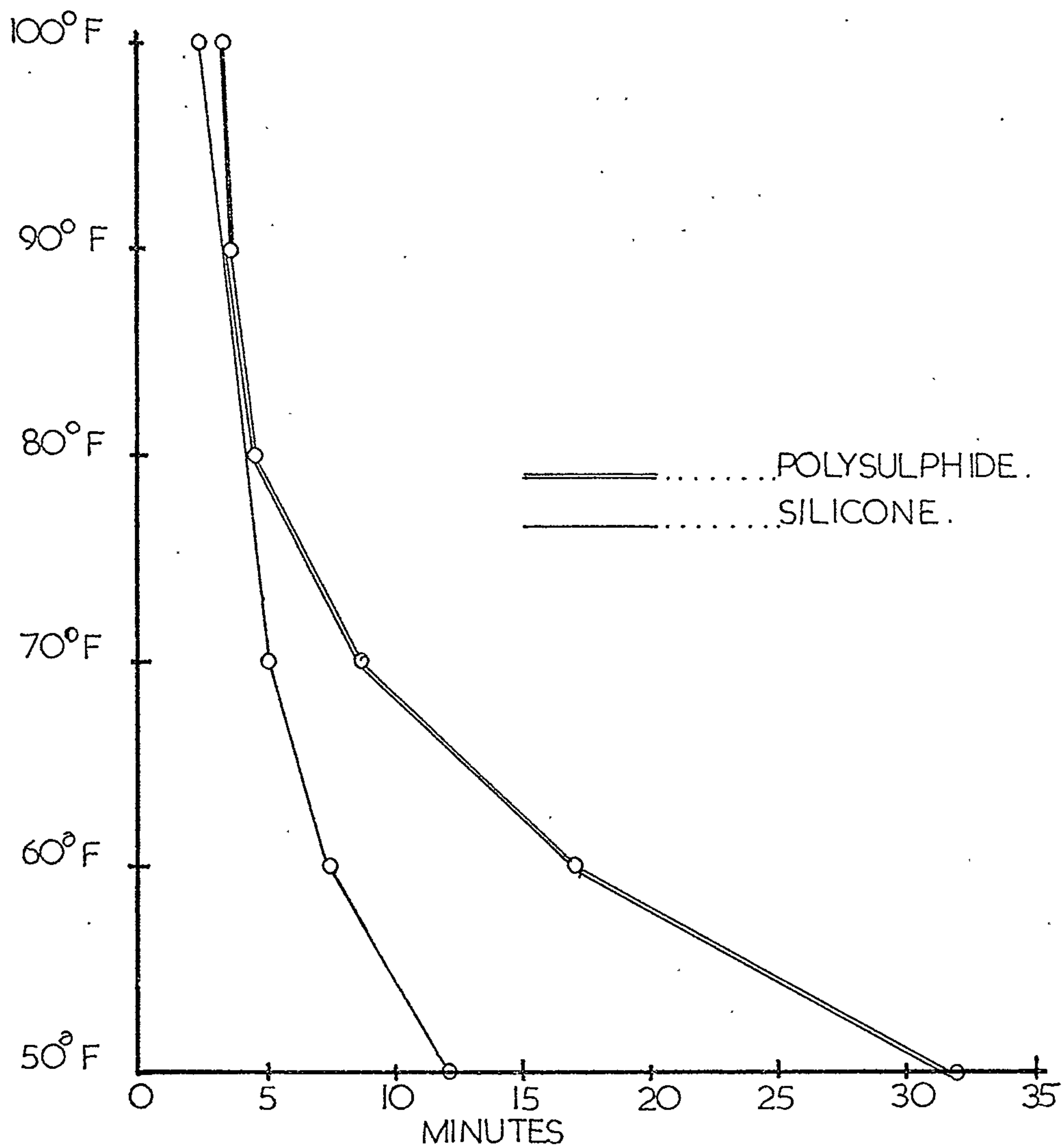
Fig. 2, extracted from Myers and Peyton (1959) shows graphically the relative effect of temperature on working time for representative polysulphide and silicone materials.

Jorczak and Fettes (1951) pointed out that the basic industrial liquid polysulphide LP2 was demonstratively affected in its cure or conversion rate by the relative humidity at which it was mixed. The dental rubbers developed from this LP2 also exhibit this property. Jorgensen (1957) tested Thiokol at 80-85% and 45-50% relative humidity, and stated that working time was reduced by increase in humidity. However, details of the test results were not included in their paper. Phillips (1959) pointed out that this effect was consistent with the fact that the addition of a drop of water to the mix was one way of accelerating the set.

It would appear however, that although increase in humidity does have an effect on cure rate, this may not be of clinical significance over the humidity range likely to be encountered in practice. McLean (1961) found that room humidity experienced in England did not appear to alter the setting time of Thiokol appreciably.

It is difficult to find direct reference to the effect of humidity on the setting rate of silicone materials. It would seem that any accelerating effect within a normal humidity range, is not of clinical significance since silicones appear to be less affected in this regard than the polysulphides.

Fig. 2 - The effect of room temperature on the Initial Set (Working Time) of Polysulphide and Silicone Impression Materials - Myers and Peyton 1959.



Accelerator/Base Ratio

The clinical importance of the effect of variation in proportioning of base and accelerator on the setting reaction has been realized since the introduction of the first rubber base impression material to the dental profession. An early report on Thiokol (Dental Materials 1955) suggested that the setting time of this material could be adjusted to suit the clinician by varying the accelerator base ratio of the material dispensed. Skinner and Cooper (1955) tested two well known brands of polysulphide by determining the setting time (at 25°C) when using, 50% less accelerator, manufacturer's recommended proportions, and 50% excess accelerator, in three separate tests. The results of this research indicated an erratic variation in setting time, and demonstrated that variations in accelerator base ratio did not necessarily affect the setting time proportionately. This led the authors to recommend that some other means should be employed to regulate setting time.

Roydhouse (1958), Phillips (1959), and Feuerstein (1960) all cautioned against variation of proportions as a method of controlling setting time. Phillips stating that noticeable variation from the recommended ratios would adversely affect the elastic properties of the material and lead to distortion.

Myers et al (1958), realising that accurate proportioning was very difficult clinically because of the

methods of dispensing, carried out trials using relatively small variations in proportions. They made the point that while the setting time was affected for all products tested, there was a noticeable difference in effect on different products. Braden (1966) showed that the effect of small variations of concentration of accelerator was relatively slight. He suggested that this may be an advantage of Thiokol, in that the setting time would not be greatly affected by the unavoidable variation in ratio due to dispensing base and accelerator under clinical conditions.

McLean (1958), in two papers, described the effect of increasing the accelerator content of silicone rubbers by 50%. In all cases he demonstrated an increase in setting rate.

Miller and Myers (1962) drew attention to the common recommendation by the manufacturers of silicone materials that the clinical setting time can be varied to suit the operator by altering the accelerator/base ratio. To evaluate this recommendation they experimented with half, and double quantities of accelerator and found that a reduction in accelerator proportion delayed the working time and setting time of all products and that some products responded more than others in this respect. Custer (1964) showed much the same results but contended that in the case of silicones the setting time can be accurately controlled by this method.

Looking more deeply into the chemistry involved in the setting reaction of silicone, Braden and Elliott (1966)

investigated the relative effects of the basic components of the silicone accelerator. They showed that the ortho-silicate, which has been demonstrated to be the primary cross linking agent, has the more definite effect on the commencement of set. Variations in the organo-tin concentration did not show the same marked effect. They therefore suggested that a two reagent accelerator system may be an advantage in adjusting setting time for silicone rubbers.

Summarising the data available, it would appear that slight variation in accelerator base ratio for either thiokol or silicone does not have any great clinical significance. The deliberate adjustment of setting time by large variations in the accelerator base ratio should be discouraged because of unpredictable or detrimental effects upon the physical properties of the set rubber.

ELASTICITY

The property of elasticity possessed by the elastomeric impression materials is that fundamental quality which enables these materials to register undercut areas without fracture or significant distortion of the impression. On removal from the mouth, or area being impressed, the material must be able to spring over undercuts and rebound to its original dimensions and shape. After removal, the impression must have sufficient rigidity or toughness to enable the satisfactory completion of laboratory procedures. The material must be sufficiently rigid to be unaffected by the weight of gypsum, or other product which may be used in making a cast.

In considering the physical properties of elastomers, elasticity and rigidity or toughness are generally considered together and like the setting characteristics, have been the subject of numerous different methods of investigations. These fall broadly into two categories :

1. Those methods which measure and evaluate the permanent deformation and percentage strain of specially prepared specimens of material, under given conditions.
2. Those methods which assess the distortion introduced into an impression, by measuring the change in dimensions of dies produced from impressions which have been subjected to a known degree of distortion.

The early assessment of the elastic properties of Thiokol and silicone materials was made by applying the

conditions of the American Dental Association Specification No. 11. This required that permanent deformation (compression set) should not exceed 3% when stresses of 100, 1,000, 0 and 100 gm/cm² were applied for one minute intervals in that order. It was further required that the strain in compression should be not less than 4% or more than 20% between a stress of 100 gm/cm² and 1000 gm/cm². Skinner and Cooper (1955) and Hampson (1956) carried out such tests on a large number of commercial polysulphide materials. When specimens were tested 30 minutes after setting, as stipulated in ADA Specification No. 11, the percentage permanent deformation ranged from 0.5% to 1.9%, and the percentage strain from 4.9% to 9.8%. These results showed the elastic properties of the polysulphide materials to be essentially the same as the hydrocolloids.

However, in comparison with the hydrocolloids, the rubber base materials undergo significant changes in physical properties during the 30 minute interval preceding the test prescribed by the ADA Specification. The cure of the material clinically is at a temperature of 37°C, as opposed to the test temperature of 20-25°C stipulated in the ADA specification. It seemed that a more significant evaluation for both permanent deformation and percentage strain properties for these materials would be obtained under different conditions to those prescribed for the hydrocolloids.

Fairhurst et al (1956) conducted standard ADA tests for a series of polysulphide materials, but they varied the time of application of the initial load. The results for permanent deformation and for strain were obtained for seven representative materials, at times varying from five to ten minutes from the start of mix. At ten minutes, permanent deformation values were between 2.0% and 9.0% and strain values were between 7.0% and 19.0%. The results of these tests, when compared with those of Skinner and Cooper, highlighted the marked improvement in elastic properties of polysulphide with an increase in time. This alteration in physical properties related to time, is of course due to the progress of polymerization of the material. The investigators noted that in most instances the setting time claimed by the manufacturers did not assure the satisfactory development of elastic properties, and they recommended that the definition of setting time should be determined as the time at which optimum elastic properties are obtained, when tested over a practical period of time.

Much more recently Chong and Docking (1969), testing polysulphide, silicone and polyether for permanent deformation, showed that the generally accepted setting times did not allow for the maximum development of elastic properties. These workers suggested that the test for permanent deformation as laid down in the Australian Dental Standard T27 is a better indication of clinical setting time other than tests for setting time.

Another modification of the ADA Specification No. 11 as applied to the rubber base materials was described by Miller et al (1960). Instead of determining the permanent deformation which occurred after the application and release of a load of 1000 gms/cm^2 it was argued that the permanent deformation resulting from a given percentage deformation of the test specimen, would be clinically more significant. This assertion had been previously expounded by Cresson (1949) in relation to hydrocolloid materials. Also the U.S. Federal Specification for Hydrocolloid and Alginate Impression Materials of 1953 incorporated a permanent deformation test based on the release of an applied strain of 12% to the specimen. Wilson (1966) pointed out that the testing of elasticity by the application of a known stress was not suitable, since the results obtained were dependent not only on the elasticity of the material but also on its rigidity. He advocated, as did McLean (1961) and Miller and Myers (1962) that the deformation test is best achieved by the application and release of a given strain so that all materials are compressed by the same amount. This principle is now incorporated in all standard specifications for elastic impression materials.

Wilson (1966) carried out two separate series of tests on both polysulphide and silicone, involving permanent deformation. In the first series each specimen was compressed 10% of its original length, and this strain was

maintained for 1 minute. In the second series each specimen was compressed 30% and the strain was maintained for only 5 seconds. In evaluating the various materials and discussing the merits of his test procedures, Wilson enumerated three factors controlling permanent deformation.

1. The strain applied (clinically this is dependent on the degree of undercut).
2. The time for which the strain is applied.
3. The interval between releasing the strain and measuring the degree of deformation.

He points out that the elastomeric materials are clinically subjected to considerable compression on removal from deep undercuts and he estimates that this may be in the order of 30% strain. In removing the impression from such marked undercuts, this strain should not be exerted for longer than 5 seconds. Both polysulphide and silicone materials showed superior recovery when subjected to the 30% deformation for 5 seconds than when subjected to 10% deformation for 1 minute.

Wilson's tests clearly show that permanent deformation decreases considerably with the time elapsing after the material has been released from the strain. This indicates a progressive recovery of the material towards its original dimensions. He therefore recommended that clinically, impressions should be left at least 10 minutes after removal before the die or model is prepared, in order that the material will recover.

These experiments of Wilson supported the earlier findings and clinical recommendations of Jorgensen (1957). Jorgensen devised a simple method of measuring the elasticity of elastomers by impressing a spherical indenter into the surface of a specimen, and then making a stone die of the indented surface and measuring the height of the resulting projections of stone. He found that by increasing the time for which the steel ball was depressed, from momentarily, to five seconds, the magnitude of the deformation was almost trebled. He also showed that tests carried out at increasing intervals of time after the initial set showed less deformation. Analysis of these two phenomena led Jorgensen to recommend two fundamentals in the clinical use of thiokol :

1. The impression should be removed with a sudden sharp pull;
2. The impression should be left in situ as long as is practical after the apparent set.

In addition to testing permanent deformation following a compressive force, some authors (McLean 1958, Cowell 1959, and Wilson 1966) have employed a tensile force to deform the specimen. It can be argued that a compressive strain is dependent, amongst other things, upon the thickness of material used, whilst a tensile strain is independent of thickness and so is a better guide to the basic behaviour of the material. These tests for determining the permanent deformation following a tensile load (tension set) have been

determined by adapting the British Standard 903 Part A5, 1958 Methods of Testing Vulcanized Rubber. In this test a dumb-bell shaped specimen, with reference points, is assembled in a tensometer and subjected to a given strain for a given period. After release of the strain a second reading is taken of the distance between the reference points. The change in dimension expressed as a percentage of the original measurement is the tension set of the material for a given strain. Both polysulphide and silicone materials have been subjected to this form of testing and in principle the results are in agreement with those of the compression tests.

McLean (1958) tested a popular brand of thiokol under a tensile strain of 0.2 cm/cm, applied for one minute, ten minutes after setting. It was found that the thiokol compared unfavourably with three silicones tested under the same conditions. The results were as follows :

<u>Materials</u>	<u>Deformation</u>
Silicone 1	0
2	0.006 cm/cm
3	0.008 cm/cm
Thiokol	0.028 cm/cm

The following is a summary of the features of elasticity demonstrated by both compressive and tensile methods of testing.

1. The silicones are superior in elastic recovery to the polysulphides.

2. The elastic properties of polysulphide take longer to develop.
3. The recovery of both classes of material improves with time.
4. The amount of recovery is influenced by the rate of deformation as well as its magnitude. A rapid deformation leads to a greater recovery.

It will be appreciated that the tests so far described for permanent deformation are a measure of the behaviour of the material following the application of a simple compressive or tensile force. However in clinical use the materials are subjected to simultaneous compressive, tensile and shearing stresses as a result of the withdrawal of the impression (Viera 1960). Fairhurst (1956), McLean (1958) and Hollenbach (1963) being aware of the significance of the complex nature of the deforming forces in practice, devised methods of evaluating the elastic properties of such materials by subjecting them to all three deforming forces. These tests were designed to simulate the dental impression techniques as closely as possible and to yield numerical values for comparison.

Fairhurst (1956) took polysulphide impressions of an inverted cone shaped die and measured the linear change which occurred between fixed reference points in the impression. He examined the effect of time of removal of the impression, and the effect of storage of the impression. He showed an improvement in elastic recovery the later the die was removed from the impression and also an improvement

on storing the impression for up to 60 minutes after removal of the die. Between 60 minutes and 24 hours there was no further apparent improvement.

Hollenbach (1963) employed yet another method of evaluation which took into account the complex combination of deforming forces met with in practice. He used a comparator to measure the diameter of the pedestals of stone models constructed from impressions of six steel dies. The first of these dies was a straight cylinder 0.500" diameter. The five remaining dies were steel balls 0.500" diameter connected to a base by pedestals varying in diameter from 0.475" to 0.375" in increments of 0.025" or 5%. Removal of an impression from each successive die therefore represented an increase in deformity of the impression material of 5%. The first die presented no deformity and the number six die caused the impression material to be deformed by 25%. Using this test Hollenbach measured the elastic behaviour of several well known brands of polysulphide, silicone and reversible hydrocolloid. His observations led him to assert that the polysulphide and silicone materials could be deformed by at least 25% without showing inaccuracies which would be clinically significant. Both these materials being superior in this respect to hydrocolloid which showed a significant deformity after being subjected to 15% strain.

The results of these more clinically oriented tests of elasticity are of value in that they support the fundamentals of behaviour which have been established by examination of the elastomeric materials in an unsupported condition. However, tests of this nature are influenced by a number of additional factors or variables such as thickness of the impression material, dimensional change of die material, adhesion of impression material to the tray, etc. These factors tend to reduce the validity of clinically oriented tests as a means of determining quantitatively the differences in elastic behaviour of different materials under clinical conditions. More will be said concerning this type of test when considering the dimensional accuracy of elastomers when retained within impression trays.

Rigidity

Rigidity, as measured by strain in compression, has been mentioned earlier in this section but little has been said concerning the practical significance of this property. If a particular material is too rigid when set, it will obviously be impossible to remove it from an undercut area without fracturing or permanently distorting the material. Likewise too flexible a material is likely to be flabby and easily distorted during laboratory procedures.

From the earliest testing of polysulphide it was apparent that this material showed satisfactory strain in

compression values when compared with the existing reversible and irreversible hydrocolloids. The early silicones however were unsatisfactory (Rubinstein et al 1960), showing strain values as high as 30%. This allowed unsupported areas of impressions to be easily distorted by the weight of stone used to make a cast. This situation was quickly overcome by the manufacturers and satisfactory strain values were reported by Miller et al (1960) when these materials were subjected to the test laid down in ADA Specification No. 11.

Peyton (1968) published data for strain values of a representative group of polysulphide and silicone materials, with results for polysulphide varying between 6.1% and 17.4%, and for silicones between 5.5% and 11.4%. These figures show the silicones to be greatly improved, now being marginally more rigid than the polysulphides.

In an attempt to obtain greater clinical significance for strain tests, Wilson (1966) argued that since a stone cast poured into an impression would exert a stress of approximately 10 gms/cm^2 , and that this stress would only be significant for as long as it took the stone to set, then a much more realistic measure of strain characteristics for elastomers would be to test them under stresses between 2 gms/cm^2 and 12 gm/cm^2 applied for a period of five minutes. The results of such tests on four silicones showed a percent strain range from 0.6% to 0.95%, and from 1.25% to 1.55% for four polysulphides. Such results indicate

a very small potential distortion of these materials under clinical conditions, and show the silicones to be slightly more rigid than the polysulphides.

Although figures are not available for strain in compression characteristics of the polyether material, the manufacturers claim low values. It is believed from personal clinical experience that these values would be similar to those of the silicone materials.

COMPATABILITY AND SURFACE REPRODUCTION

The ability of an impression material to reproduce minute detail and a smooth hard surface on artificial stone, or other die materials, is a most important factor in assessing the suitability and relative merit of a particular material for various techniques. This ability is particularly important for the elastic materials used so extensively in indirect inlay and crown techniques. The accuracy of any die while being largely a function of the dimensional accuracy of the impression material is also dependent upon the surface condition of both the impression and the die or cast made from it. The impression material must be capable of reproducing surface detail, and it must be compatible with the die material being used so that the degree of detail obtained in the impression may be reproduced in the die. The hydrocolloid materials, both reversible and irreversible, have always shown limitations in this respect, (Paffenbarger 1940, Skinner & Pomes 1946) due to their hydrophilic nature and to the effect of syneresis and imbibition. Over the years the incorporation into these materials of accelerators for gypsum products, and the use of various fixing solutions has considerably reduced these limitations and satisfactory gypsum dies may now be obtained from hydrocolloid impressions when they are correctly treated. There are however, limitations in the variety of die materials

which can be used because of incompatibility. This has been suggested as a disadvantage of hydrocolloids when compared with the rubber base materials.

Skinner and Cooper (1955) drew attention to the fact that the surface hardness of gypsum products was reduced when these materials were allowed to set in contact with hydrocolloid material. They carried out experiments in which the control was the surface hardness of stone set against glass, and the results showed that the hardness of the surface of stone set against thiokol was of the same general order as that of the control. McLean (1958) demonstrated similar results for silicone set against glass.

Surface Detail

A comparison of the ability to reproduce fine detail between alginate and thiokol materials (Hosoda and Fusayama 1959) showed that although, under ideal conditions, alginate was capable of good surface reproduction thiokol reproduced detail better and was more consistent in its results. The test involved the reproduction of fine lines cut into a brass plate, spots of 8, 10, 20, 30 and 40 μ width on these lines were determined by measuring with an ocular micrometer and reproduction of these spots was compared under a microscope at 40 magnifications. The authors claimed that thiokol would consistently reproduce the 8 μ line in the impression surface and that the 10 μ line was reproduced

consistently in stone models of either Class I or Class II stone.

The silicone materials were initially very disappointing in regard to surface reproduction. The evolution of gas during polymerization produced bubbles at the interface between the impression and the die material which caused pitting on the surface of the die and seriously affected surface reproduction. McLean (1958) recommended the vacuuming of the impression at 28 inches of mercury for ten minutes, and then washing in detergent prior to casting, so that gas formation was retarded sufficiently to allow the stone to set without disturbance. Tomlin and Osborne (1958) suggested that delaying model pouring for up to two hours would have a similar effect. These investigators also related the degree of bubble formation to the thickness of silicone material, showing an increase in pitting of the stone surface poured against a thicker specimen of silicone. They therefore recommended using the minimum practical bulk of silicone material in order to minimise this effect.

The later silicones, by alteration of their formulation, overcame the difficulty of gas production and quite satisfactory surface reproduction with these materials was demonstrated by Ayers et al (1960). This group carried out a comparative study on representative materials of the reversible hydrocolloid, alginate, polysulphide and silicone groups of materials. Their test method was to place a

pattern of Knoop indentations, varying in length from 34 to 422μ and in depth from 2 to 28μ on the polished surface of steel cylinders and to take impressions of these test models. After the impressions were made they were observed under 16 power magnification and the number of indentations reproduced in the impression material was recorded. Within ten minutes of separation gypsum models were poured in these impressions and after one hour the casts were separated and the number of indentations present on these models was recorded. A difference in the ability of various impression materials to produce fine detail was demonstrated. Polysulphide, reversible hydrocolloid and silicone materials gave excellent results, reproducing the 34μ indentations consistently. The alginates were definitely inferior, only showing consistent reproduction of indentations of 100μ or greater. Study of the gypsum models poured in the impressions showed that the ability of all the materials to register detail exceeded the reproduction capability of the gypsum cast materials. It is of interest to compare the results of this investigation with those of Hosoda and Fusayama, mentioned earlier, who claimed much greater reproduction ability for both polysulphide and alginate than was demonstrated in this case.

In evaluating the clinical significance of these tests the authors pointed out that since visual acuity under optimum conditions is approximately 50μ (Nelson et al 1952)

the ability to reproduce the finer indentations was clinically significant because they lay close to the level of visual detection. On the other hand Skinner and Phillips (1967) queried the significance of this type of test, pointing out that the surface interaction between a steel die and the impression material was not necessarily the same as that between the impression material and the oral tissues, and that the detail obtained in the mouth may not be as great as that obtained from a steel block. The results however must be considered as a reasonable basis for comparison between different classes of materials being tested.

Bearing in mind that gypsum die materials generally showed failure to reproduce the finest detail of the impression material Henry and Phillips (1961) reduced the particle size of standard stone by milling and showed that finer particle stone improved detail reproduction in the set die, but still failed to routinely reproduce all detail from the impression.

More recently Toreskog et al (1966) reported the detail reproduction and compatability of a large range of commonly used die materials with polysulphide and silicone. The materials tested included artificial stone, silicophosphate cement, heat fused artificial die material, silica filled epoxy resin, low fusing alloy, and silver and copper plated dies. The test method was essentially the same as that developed by Ayers et al and the findings were similar

regarding the ability of the impression materials themselves to reproduce detail. However their results regarding the ability of the various die materials to reproduce the detail obtained in the impression, and hence the compatability between the impression and die materials, is of interest. Only silver plated dies and those made of silicophosphate reproduced all detail in the tests. The stones and heat fused die materials were incapable of such fine reproduction, and the filled epoxy resins showed such poor compatability that their use with polysulphide or silicone seems unwise.

Detail reproduction on plated dies

One of the significant advantages claimed for rubber base over hydrocolloid materials is their ability to take an electroformed metallic plating. If the rubber base materials permit the production of electroplated metal dies which are accurate and which will reproduce fine surface detail, then they do possess an advantage over the hydrocolloid impression materials. Claims have been made that plating produces the ideal die (Krug and Markley 1969).

The early literature in respect of plating is somewhat contradictory. In Dental Materials (1955) it was suggested that thiokol could be copper plated with ease. Pearson (1955) found little difficulty in copper plating thiokol using colloidal graphite as a conductor over the surface to be plated. Asturias (1957) recommended the use

of copper plated dies from rubber impression materials for the construction of inlays.

On the other hand, Rosenstiel (1955) found difficulty in electroplating with copper, especially when using colloidal graphite as the metalizing agent. Beagrie (1956) also found copper plating using colloidal graphite to be unsatisfactory and did not recommend its use. Campbell Hudson (1958) found copper plated dies to be lacking in fine detail. This was probably because the metalizing agent commonly used occupied space between the impression and the resultant copper die and the fine detail in the impression was lost. He recommended a refinement of a previous method of silver plating (Silver 1956), using finely divided pure silver powder as a metalizing agent. This powder provides a perfect conductor even in the microscopic thickness used, and it has the advantage of becoming part of the die and reproducing intimate surface detail. He found that a comparison between stone and electroformed silver dies favoured the metal type because of detail, accuracy and strength.

The work reported by Phillips and Schnell (1958) seems to have rationalized the conflicting reports concerning the plating of the rubber base materials up to that time. They tested thiokol and silicone materials with both copper and silver plating techniques. Results were assessed for surface detail, distortion and accuracy of fit of castings.

Their conclusions in part were as follows :

1. Both thiokol and silicone impressions can be plated either in basic silver cyanide or in an acid copper sulphate bath.
2. Very finely powdered metals or alloys used as metalizers can produce more accurate surface detail than a brushed on colloidal graphite film.
3. Routinely the most accurate die with the best surface detail was obtained when thiokol was metalized with powdered silver and plated in a basic silver cyanide bath.
4. Because of distortion during the plating process it is doubtful whether electro plating of silicone can be recommended.

Henry and Phillips (1961) carried out detail reproduction Knoop indentation tests, developed by Ayers et al (1960). They used a variety of impression materials including commercially available polysulphides and silicones. Six impressions of each material were plated and six were poured as straight stone dies. Silver plating only was used, and a comparison was made of percentage reproduction of indentations between :

1. The impression surfaces themselves.
2. The unplated stone dies.
3. The silver plated dies.

In the case of both polysulphide and silicone the reproduction of the plated die was superior to that of the unplated stone die. The reproduction of the plated polysulphide was significantly superior to that of the plated silicone.

Stone dies in polysulphide reproduced the smallest indentations of 34μ , 22% of the time. The silver plated polysulphide dies reproduced this smallest indentation 83% of the time and the plated silicone 44% of the time.

Cannon (1964) and Markley and Krug (1969) drew attention to the surface hardness of silver plated dies. When subjected to a Brinell Hardness test silver plate gives a B.H. number of 110-115 compared to 70-85 for silver amalgam or 40-50 for gypsum die materials. This additional surface hardness is a considerable advantage in those laboratory procedures associated with forming wax patterns and finishing restorations.

In summarizing the work on detail reproduction or plated dies it is apparent that superior results are obtained when polysulphide is silver plated using pure silver metalizing powder as the conducting surface. Other considerations involving the dimensional accuracy of the rubber base materials as a result of the plating process will be dealt with in the section of this paper devoted to dimensional change.

CONSISTENCY

It is apparent that in order to be clinically acceptable, a rubber base impression material before it reaches the stage of initial set, must exhibit a consistency which will allow ease of mixing and manipulation. Furthermore the consistency of the material must allow an impression to be taken without undue soft tissue displacement or distortion. To this effect both polysulphides and silicones are marketed in a range of consistencies to facilitate the various clinical techniques advocated. They range from the relatively free flowing syringe material to the much more viscous tray or heavy body materials. The control of this property is largely dependent upon the type and quantity of filler which is used in preparing the pastes.

The American Dental Association Specification No. 19 for Elastomeric Impression Materials, requires the classification of polysulphide and silicone into three classes depending upon their consistency. These classes represent the Heavy, Regular and Light Bodied materials which are available in most commercial brands of rubber base material.

In the case of polyether, the material is marketed with a "thinner" provided in the form of a paste, which when added to the base and accelerator produces a freer flowing material. The proportioning of the thinner relative to the base and accelerator is left to the discretion of the operator,

who adjusts the mix to suit the particular clinical requirement. This thinner also has the effect of slightly increasing the setting time, and also of increasing the strain in compression characteristics of the set material, and therefore must be used with caution.

Early tests of consistency for elastomers were empirical and depended upon a modified slump test which had been developed to evaluate the consistency of zinc phosphate cement (ADA Specification No. 8). A given volume of mixed material was placed between two glass plates and a constant weight was placed on top for a given period of time. Measurement of the mean diameter of the resulting flattened disc of impression material gave a basis for comparison of the consistency of different materials. In this way consistency could be related to such factors as temperature, setting time and accelerator-base ratio (Jorgensen 1957, Clark and Phillips 1957, Myers and Peyton 1959).

It was found that a quantity of 0.1 cc of material placed between two glass plates, two minutes after the commencement of mixing and subjected to a weight of 500 gms for eight minutes at room temperature, gave consistency values for polysulphide of 12-33 mm and for silicone 15-31 mm. Kafalias (1961) suggested that materials showing values from 28-33 mm were suitable for syringe purposes, and those with values between 19-26 mm for use as tray materials. It was however noted that materials with values above 30 mm tended to

be a little thin and to drip or run, particularly from upper cavities.

Miller and Myers (1962) found acceptable values for syringe silicone materials to be higher than with comparable polysulphide material. In fact they suggested that silicone materials with values as high as 40 mm flow well from a syringe, and do not drip from cavities in the upper arch.

The influence of setting time on the consistency values obtained in this standard test is significant. Products with short setting times will generally exhibit low consistency values with this test, which do not necessarily reflect their clinical behaviour.

Because of this influence of setting time on consistency values, Wilson (1966) adapted the standard test described to give more clinically significant consistency ratings to elastomeric materials. He argued that for clinical evaluation, the test should be applied at the time at which the material would normally be inserted in the mouth, under similar load conditions, and for a similar period of time.

The pressure generated in seating a rubber base impression had been calculated as between 2.2 and 4.2 lbs/sq. inch (Douglas et al 1965) and it was estimated that to generate approximately this pressure a weight of 1500 gms should be applied to 0.5 ccs of material. The duration of pressure whilst taking an impression had been shown to be approximately 5 seconds. Therefore Wilson subjected 0.5 ccs of material

under test to a load of 1500 gms for 5 seconds, two minutes after the commencement of mix and the experiments were conducted at room temperature of 20°C.

The very much shorter time of application of the load in this test minimizes the influence of setting time on the consistency value, and the heavier load adds practical significance. Wilson found, using this test, that materials which produced a disc less than 20 mm were too stiff to flow under the normal forces employed in impression taking, and materials producing a disc more than 34 mm were so fluid that they flowed readily under their own weight and were probably not suitable.

In similar tests Braden (1967) applied a load of 1000 gms for periods of 2, 5 and 10 seconds, but he went even further and omitted the setting reagent from the test material so that detailed measurement could be carried out without the occurrence of disturbing effects of the setting reaction. This testing did show definite differences for light, regular, and heavy grades of material, but without the incorporation of the setting reagent its practical significance must be questionable.

Shelf Life

Many references have been made to the shelf life of the rubber base materials (Skinner & Cooper 1955, Gerson 1957, Phillips 1958, 1959, 1962, Miller and Myers 1962) particularly

silicone. For quite some time after their introduction these silicone materials exhibited unsatisfactory shelf life because of autopolymerization which caused a thickening of the base material. In order to test shelf life McLean (1961) conducted parallel plate consistency tests at monthly intervals over a period of one year, with polysulphide and silicone materials stored at either $3.3 \pm 1^{\circ}\text{C}$, $20 \pm 3^{\circ}\text{C}$ and $32 \pm 1^{\circ}\text{C}$. The materials stored at low temperature showed no deterioration measured as a reduction of consistency value over 12 months. At room temperature, $20 \pm 3^{\circ}\text{C}$, there was little or no deterioration, but after storage at the highest temperature some silicone became unworkable after 3 months.

It would appear that under reasonable conditions, the shelf life of rubber base materials as determined by consistency tests are now quite satisfactory, although in areas of constant high temperature the polysulphide may be superior in this respect.

DIMENSIONAL STABILITY

The physical property of the elastomeric impression materials which, more than any other, first brought them to the notice of the dental profession, was their dimensional stability. It was claimed that these materials were dimensionally stable over long periods of time, under normal environmental conditions. Referring to thiokol, Pearson (1955) claimed a good fit for an MOD inlay constructed on a die which was cast in an impression which had been stored in air for 8 days. Skinner and Cooper (1955) constructed a bridge on a die poured 8 weeks after the impression had been taken and they assessed the fit as satisfactory. Stability of this order was of course in marked contrast to the stability of both reversible and irreversible hydrocolloids.

In spite of these early enthusiastic reports it was quite apparent that the new materials did show a definite dimensional change related to time. Dispute concerning the magnitude of this change was in no small manner due to the variety of methods employed to determine their dimensional change quantitatively.

One would expect from a knowledge of their chemistry that these materials would tend to shrink during and after setting. The setting reaction is one of polymerization and a shrinkage always occurs during a polymerization reaction. Additionally, dimensional change would be expected as a result

of volatilization, particularly in the case of silicone materials which, as already mentioned, may produce various gases during the polymerization reaction. The setting reaction of thiokol has been described as a direct oxidation reaction with the production of water as a by-product. The loss of this water may well produce a further shrinkage in this material.

Another source of dimensional change which is clinically significant, is the thermal coefficient of linear expansion. When one considers the temperature range between mouth and room temperature it becomes obvious that if the thermal coefficient is high, then an appreciable dimensional change could be expected.

Still another factor of great clinical importance is the possible distortion which results from the release of stresses introduced when the impression is withdrawn over an undercut area, or subjected to similar deforming forces.

The practical effect of these factors influencing dimensional stability is modified to a marked degree by the restriction of the material within an impression tray or band. In order to obtain a true picture of the dimensional stability of the rubber base materials and its clinical effects, one must consider dimensional change in both an unrestricted or free state, and also in a confined or restricted state.

UNRESTRICTED DIMENSIONAL CHANGE

The measurement of the unrestricted dimensional change of an elastic impression material is a measure of its basic behaviour, and will give an indication of the nature of problems which are likely to arise during clinical usage. With this knowledge the early investigators subjected thiokol and silicone materials to a variety of dimensional change tests.

A number of reports (Jorgensen 1957, Anderson 1958, McLean 1958, Skinner 1958) of dimensional change were based on the behaviour of specimens of material which were floated on a mercury bath. The specimen of material to be tested was attached to one end of the bath, the other end moving freely. Markers were inserted into the specimen and changes in dimension were observed using a comparator microscope, at given time intervals after the commencement of mix. The results published by Skinner are reproduced in Table I.

Table I - Shrinkage of Rubber Impression Materials - Skinner 1958

Material	% Linear Dimensional Change in	
	30 min.	3 days
Polysulphide		
A	-0.05%	-0.13%
B	-0.03%	-0.11%
Silicone		
C	-0.05%	-0.37%
D	-0.06%	-0.87%

These results show a shrinkage in all materials tested which continued over a considerable period of time. The shrinkage for the silicone materials was appreciably greater than for thiokol materials. Ostlund (1958) had earlier drawn attention to the fact that thiokol was more dimensionally stable than silicone. It is interesting to note that Skinner suggested that no appreciable change in dimension could be observed before 30 minutes after mixing had begun. This observation will be referred to later when considering the work of Chong and Docking (1969).

Miller et al (1960) described a method of testing dimensional stability in which specimens measuring 57 x 19 x 3 mm were employed. Metal reference points were embedded in the specimen's surface. The specimens were placed on a glass surface which had been thoroughly dusted with talc, as had the contacting surface of the specimen, permitting the specimen to change dimension freely on the glass. The distance between the reference points was measured at various times from 15 minutes to 24 hours from the beginning of mix. The results of this investigation supported the knowledge to that date in that all the materials tested showed a shrinkage over a 24 hour period, with the greatest change being evidenced by the silicones.

Rubinstein et al (1960) using the same test method found that no silicone tested satisfied the 0.2% dimensional change limit after 6 hours, which had been proposed as a

standard specification for dimensional stability of rubber base materials.

McLean (1961) developed yet another test method which, in principle, had earlier been described by Molnar (1958). In this test, cylinders of rubber material were constructed and transferred to the stage of a measuring instrument (a Microkator). The microkator was adjusted to receive a specimen of the exact height of the cylinders. Contact was made, under a load of not more than 2 grammes, with the top of the specimen in such a way that any change in height could be registered to 0.0005 mm. McLean showed the same relative shrinkage of thiokol and silicone, but the materials tested gave higher shrinkage values than previously recorded studies. It was suggested that this might be attributable to a more positive degree of change over a given length due to the stability of both ends of the specimen, as opposed to the chance of distortion or movement of the rubber, as might be the case when measuring a long specimen on a mercury bath.

More recently a test method was been devised by Chong and Docking (1969) which suggests that the unrestrained dimensional change of the rubber base materials is considerably greater than was previously demonstrated. They pointed out that factors in both the design of apparatus, and the technique, had previously contributed to incorrect assumptions concerning the magnitude of the dimensional change. It was stated that all effective movement in the material was not recorded by

earlier test methods. Several factors of significance in determining this movement were tested, the most important being the time at which the first reading was taken. By developing a method whereby the fiducial reading was registered before the material had set, they observed that significant contraction took place within the first 30 minutes after mixing. This observation is completely contrary to the findings of Skinner, referred to earlier, and would account for an appreciable proportion of the additional contraction demonstrated by Chong and Docking. In previous tests, measurement was not started for varying times after the commencement of mix, usually about 15 minutes.

In spite of an increase in magnitude, the relative dimensional stability of thiokol and silicone remained the same in this as in earlier tests, i.e. the silicones showed appreciably greater shrinkage than the thiokols. Also the newer polyether material was demonstrated to have a dimensional behaviour very similar to thiokol.

These tests showed dimensional change of a magnitude that led the authors to recommend that the proposed limit of 0.2% maximum dimensional change after 6 hours should be extended, to 0.4% at 6 hours for polysulphide and polyether materials, and 0.8% at 24 hours for silicones, provided that this test, which reproduced very consistent results, is used.

The mechanical details of this test method of Chong and Docking will be dealt with more fully in the second part

of this paper, which describes the writer's original research.

THERMAL DIMENSIONAL CHANGE

An impression material mixed at room temperature will warm to mouth temperature during the period it is setting in the mouth. This will take place quite rapidly where the material is used in relatively thin sections, as is recommended in most clinical techniques. After removal from the mouth and cooling to room temperature again, a linear contraction can be expected to take place. The magnitude of this contraction will be dependent upon the thermal coefficient of linear expansion of the material being used.

The thermal coefficient of polysulphide was first reported, in dental literature, by Jorgensen (1957) who indicated an average figure of 150×10^{-6} per $^{\circ}\text{C}$. Anderson (1958) determined the figure for silicone to be approximately 200×10^{-6} per $^{\circ}\text{C}$. These figures indicate potentially significant linear dimensional changes due to thermal expansion or contraction and point to the importance of :

- a) temperature control when carrying out tests to determine the nature and degree of dimensional change (Phillips 1959),
- b) an understanding of the effect of thermal change on the clinical behaviour of any particular material (Cowell 1959).

McLean (1961) confirmed the above values for the thermal coefficients of a representative group of both polysulphide and silicone materials. He measured the expansion of

cylindrical specimens of rubber base material when subjected to a temperature rise from room temperature of 22°C to body temperature of 37°C . The specimens had been aged to ensure that a state of near equilibrium had been reached so far as polymerization shrinkage was concerned. From McLean's figures it was calculated that, over a normal operating temperature range, polysulphide could be expected to contract by approximately 0.2% and silicone by 0.35%. This contraction in addition to polymerization shrinkage indicates a considerable potential percentage dimensional change. Skinner and Phillips (1967) have stated that such a dimensional change would be beyond the limits of clinical tolerance. They state that in practice thermal shrinkage is greatly inhibited by the restraining effect of the impression tray. McLean suggested that in order to compensate for whatever contraction may be attributable to thermal change, impressions should be poured in stone mixed with water at 37°C . He also stated that because of the high coefficient of linear expansion of these materials, copper and silver plating techniques were contraindicated at room temperature.

Hosoda and Fusayama (1961) conducted clinically oriented tests on polysulphide, which indicated the extent of possible effects of temperature change on dimensions. They took a series of impressions of a die at 37°C and poured stone casts at 17°C . They compared the dimensions of these casts with a second series of casts which were prepared from impressions

taken and poured at 17°C. It was shown that far less distortion took place when the impression was taken, and the cast poured, at the same temperature thus eliminating as far as possible the influence of thermal shrinkage. This finding supports the suggestion that the cast should be poured at mouth temperature to help minimize distortion.

RESTRICTED DIMENSIONAL CHANGE

The most common means of determining the dimensional accuracy of rubber base impression materials and certainly the one with the most direct clinical application, is by comparing the fit of castings directly prepared on master dies, with the fit of the same castings on dies prepared from impressions of the master dies (Schnell and Phillips 1958, Gilmore et al 1959, Myers and Stockman 1960). The accuracy with which these castings fit the replica dies is a measure of the dimensional stability of the impression material. In some tests, machined parts which accurately fit master dies have been used instead of castings (McLean 1958, Eberle 1959, Schnell and Phillips 1962, Hollenback 1963, Marcroft 1964). Also dies made from impressions have been measured directly, and the dimensions compared with those of the master die (Hollenback 1957, Tomlin and Osborne 1958, Stackhouse, 1970).

Tests made by these methods give results which can be equated clinically, since the materials can be subjected to conditions under which they are commonly used. Important

variations in technique can also be evaluated for their effect on dimensional accuracy.

Trays

Since the introduction of the rubber base elastomers, a considerable variety of impression trays has been presented as being suitable for use with these materials.

Understandably the materials were first used in the perforated stock trays, used for the existing hydrocolloid elastomers. This type of tray however did not allow control of either the bulk of material, or its even distribution. It was also uneconomical and because of the tacky nature of the material, untidy. Myers and Stockman (1960) showed an appreciably lower percentage of satisfactory castings constructed on dies from impressions taken in stock trays compared with dies from impressions taken in special trays.

Beagrie (1956) recommended the construction of an individual tray, using a snap compound impression, which was relieved and provided with mechanical retention for a lining of rubber base material. Morrison (1956) incorporated gauze into the surface of the compound to provide retention, and he claimed that the bond between the rubber and the compound was maintained for as much as 18 hours. Cowell (1960) suggested that the use of rubber adhesive with a trimmed compound impression eliminated the need for mechanical retention.

This type of tray did not prove satisfactory because of the instability of impression compound which was a potential source of distortion. Also the rubber cement used as an adhesive tended to soften the surface of the compound, leading to unsatisfactory results (Kingham and Allan 1957).

Pinkerton (1956) and Gruenwald (1957) described trays made from relieved plaster impressions. These provided a stable base for the rubber material, but the technique was awkward and it was not used extensively.

Sectional custom made trays of expanded or perforated sheet metal have been used with satisfactory results (Skinner and Cooper 1955, Joseph 1961, Duxbury 1963).

Mobbs (1964) recommended an inexpensive and easily constructed tray made from a double layer of shellac base plate, adapted over a relieved study model. Retention for the rubber being provided by perforations and/or adhesive.

The metal shell or band, as a tray for individual tooth impressions has been recommended (Grassl 1957, Eastman 1959, Brinsden 1964, Cranin 1965, Hirshberg 1965). This is an adaptation of the compound and copper band technique which has been used for many years in indirect restorative work, and is capable of providing excellent results. However unless rigid bands are used (Hailey 1957, Sturdevant 1957) distortion is likely to occur during removal of the impression from the tooth. Kinghorn and Allan (1957) suggested that this type of impression tray had the disadvantage of not providing

sufficient bulk of rubber material to spring over undercuts. This could lead to distortion by straining the material beyond its elastic limit.

Undoubtedly the most commonly used and satisfactory impression tray is the custom built tray made of acrylic resin. Bailey (1957), Davis (1958) and Rosen (1963) have described the construction of such trays and pointed to their following advantages :

1. They are simple and inexpensive to construct.
2. They are rigid and stable.
3. They allow for the optimum thickness and even distribution of impression material.

The self curing resins are recommended for this type of tray construction because of the speed and simplicity of manufacture, and the fact that they are more stable than heat processed resins (Anthony and Peyton 1962, Woelfel & Paffenbarger 1961).

The ideal tray has been described by Yoder and Thayer (1962) as a custom built, self curing acrylic tray, including the whole arch and soft tissues 3-4 mm gingivally to the teeth. Extreme undercuts should be avoided. A spacer should be made of wax or asbestos to provide an even 2 mm thickness of impression material. There should be three widely separated stops provided outside the area of the restorations to facilitate definite seating of the tray.

Bulk

The determination of the bulk of material allowed within the tray has a marked influence on the accuracy of subsequent dies constructed from such impressions. It has been shown by transferring master castings on to dies made from impressions using varying thickness of impression material, that a reduction in the bulk of material increases the accuracy of the impression (Schnell and Phillips 1958, Gilmore et al 1959). This is of course providing that sufficient bulk is allowed to prevent the material being strained beyond its elastic limit when being removed from undercuts. Gerson (1957) recommended an optimum thickness of 3-4 mm for Thiokol. Phillips (1959) stated that wherever possible the bulk should not exceed 2 mm for either thiokol or silicone. Also, the bulk should be evenly distributed around cavity preparations, since non-uniform masses of material will produce distortion (Gilmore et al 1959).

Storage

From a knowledge of the unrestricted dimensional change of the rubber base materials it can be anticipated that the longer an impression is left before the construction of a die, the less accurate the impression will be. In spite of exaggerated claims to the contrary (Blank 1957, Sturdevant 1958, Darrow 1958, McGregor 1958) this has been proved to be the case.

Schnell and Phillips (1958) tested five polysulphide and three silicone materials for accuracy using a variety of master models to represent different clinical situations. They tried master castings, or machined parts back on stone dies poured in impressions stored for 15 minutes, and 6 and 24 hours. They found that storage produced marked distortion with all materials tested.

Hollenback (1963) using a tapered bar and block test, noted that polysulphide and silicone materials were similar from the standpoint of accuracy when impressions were poured within 30 minutes. After one hour storage the polysulphide showed slightly better results than the silicone. In a 24 hour test the results obtained with polysulphide were greatly superior to those of silicone.

Consecutive Pours

Because of the practice of pouring more than one die in a rubber base impression, several investigators have examined the relative accuracy of dies made from first and second pours. The most significant factor in any variation in the dimensions of such dies is the continued polymerization shrinkage associated with the storage time of the impression. If undercuts are present in the impression, the straining of the material on removal of the first die will also play a part in distortion evident in the second die.

Myers and Stockman (1960), using polysulphide showed a slight reduction in the number of master castings which accurately fitted second pour dies, as opposed to the number which could be fitted to the dies poured immediately. This reduction in accuracy was more apparent in the case of intra-coronal, than extra-coronal restorations. This may be explained by the presence of undercuts in the master models of the intra-coronal preparations. The investigators in this study were of the opinion that, for clinical purposes, dies made from second pours are acceptable in most cases.

Silicone materials also show a loss of accuracy with successive pours. Gilmore et al (1959), using master castings, showed that the distortion of silicone increased with each die. This distortion was considerably greater than that shown for polysulphide materials under the same conditions.

A possible improvement in the stability of silicone may be indicated from the work of Stackhouse (1970). This investigator measured the dimensional change, in length and diameter, of stone dies made from impressions of steel crown master dies. Successive pours of stone were obtained from impressions at intervals of $\frac{1}{2}$, $1\frac{1}{2}$ and $2\frac{1}{2}$ hours from the start of impression mix. All materials showed increasing distortion with successive pours, but the polysulphide was not significantly superior to the silicone material in this study.

Plating

The effect of storage time on the accuracy of an impression becomes significant when metal plating is contemplated. Cannon (1964) stated that the accuracy of electroformed models was limited by the physical and chemical stability of the impression material during the plating process.

As mentioned in a previous section of this Review dealing with detail reproduction, the metal plating of both polysulphide and silicone materials has been recommended. The manufacturers of polyether material suggest that this also can be either copper or silver plated. The plating of these materials has been shown to produce harder, and more detailed dies than other methods of die construction, but the effect of plating on the accuracy of impressions and therefore on the resultant dies has led to some controversy.

Silver (1956), Campbell Hudson (1958), Shipee (1960 and Markley and Krug (1969), presented papers indicating the production of highly accurate silver plated dies from polysulphide impression material. Azaria (1962) and Rosenstiel (1965) claimed accuracy with copper dies made from polysulphide.

McLean (1958) suggested that because of the lack of dimensional stability, the plating of silicone was not recommended. This view was endorsed by Phillips (1959).

Phillips and Schnell (1958) used master castings transferred to copper, and silver plated dies, to assess the accuracy of dies from plated impressions. They reported that

silver plating of polysulphide gave acceptable clinically accurate metal dies. Copper plating of polysulphide gave accurate results but the actual plating technique proved difficult and unreliable. They found that copper dies from silicone impressions were more accurate than silver dies from silicone impressions, but neither compared favourably with the silver dies from polysulphide impressions.

It is generally accepted that the rubber base materials are chemically unaffected by either the acid copper sulphate or alkaline silver cyanide electrolytes used in the copper or silver plating process. Therefore the inaccuracy evident in plated dies should be able to be predicted by a knowledge of the restricted dimensional change of the impression material, relative to the time taken to complete the plating process. This hypothesis is in fact substantiated by the findings of Phillips and Schnell (1958). The greater distortion of silicone, either copper or silver plated, is attributable to the higher rate of polymerization shrinkage, and also to the higher thermal dimensional change of that material, compared to polysulphide. The fact that copper plating of silicone proved more accurate than silver plating, in spite of the longer time required for the copper plating process, may be explained by the phenomenon noted by Toreskog et al (1966). They observed that silver deposited on silicone tended to pull loose from the silicone impression surface allowing greater shrinkage of the silver deposit and therefore more distortion in the die.

Adhesion

The requirement for retention of rubber base impression materials to the walls of impression trays is twofold :

1. There must be retention to prevent gross distortion of the impression, due to sections of it being stripped from the walls of the tray when the impression is removed over undercut areas, and against the forces of vacuum caused by removal of the impression.
2. Adhesion must be provided to control the direction of the forces of contraction demonstrated to take place in the setting impression material.

Skinner and Cooper (1955) stated that "without adequate retention in the tray the impression material is useless". Hailey (1957) was equally adamant regarding the requirement for retention and suggested that, "regardless of the type of tray employed, the use of a rubber cement is imperative in order to obtain maximum retention of the material to the tray".

The retention of these materials to the impression tray is generally provided either by the use of perforated trays, or by the application of an adhesive layer to the areas of the tray which will come in contact with the impression material. It is felt that while the use of perforated trays will adequately provide for prevention of distortion due to (1) above, it will not have the same degree of control on the direction of dimensional change of the impression material.

Shipee (1960) compared the clinical accuracy of polysulphide and silicone materials when controlling or altering a number of clinical variables. He found that more accurate results were obtained when using acrylic trays coated with adhesive, than when using perforated trays.

A recent paper by Mitchell and Damele (1970) has also cast doubt on the effectiveness of perforations in controlling the dimensional change of rubber base materials. They took impressions in undercut, rimlock, and perforated trays, none of which were coated with adhesive, and they found the greatest distortion in impressions taken in perforated trays. These produced both oversized and undersized dies and the investigators concluded that the shrinkage of impression material towards the attachment to the tray was a major contributor to distortion.

Ellam and Smith (1966) have studied the effectiveness of rubber adhesive on the bond between polysulphide materials and various tray materials. They indicated excellent bonding to roughened acrylic resin provided that the adhesive was allowed to dry thoroughly. They also related the bond strength to the rate of application of tensile and shear forces and they found these to be directly related. Clinically this indicates that the snap removal of an impression is less likely to fracture the bond between the impression material and the tray wall than a slow dragging action on removal.

Direction of Dimensional Change

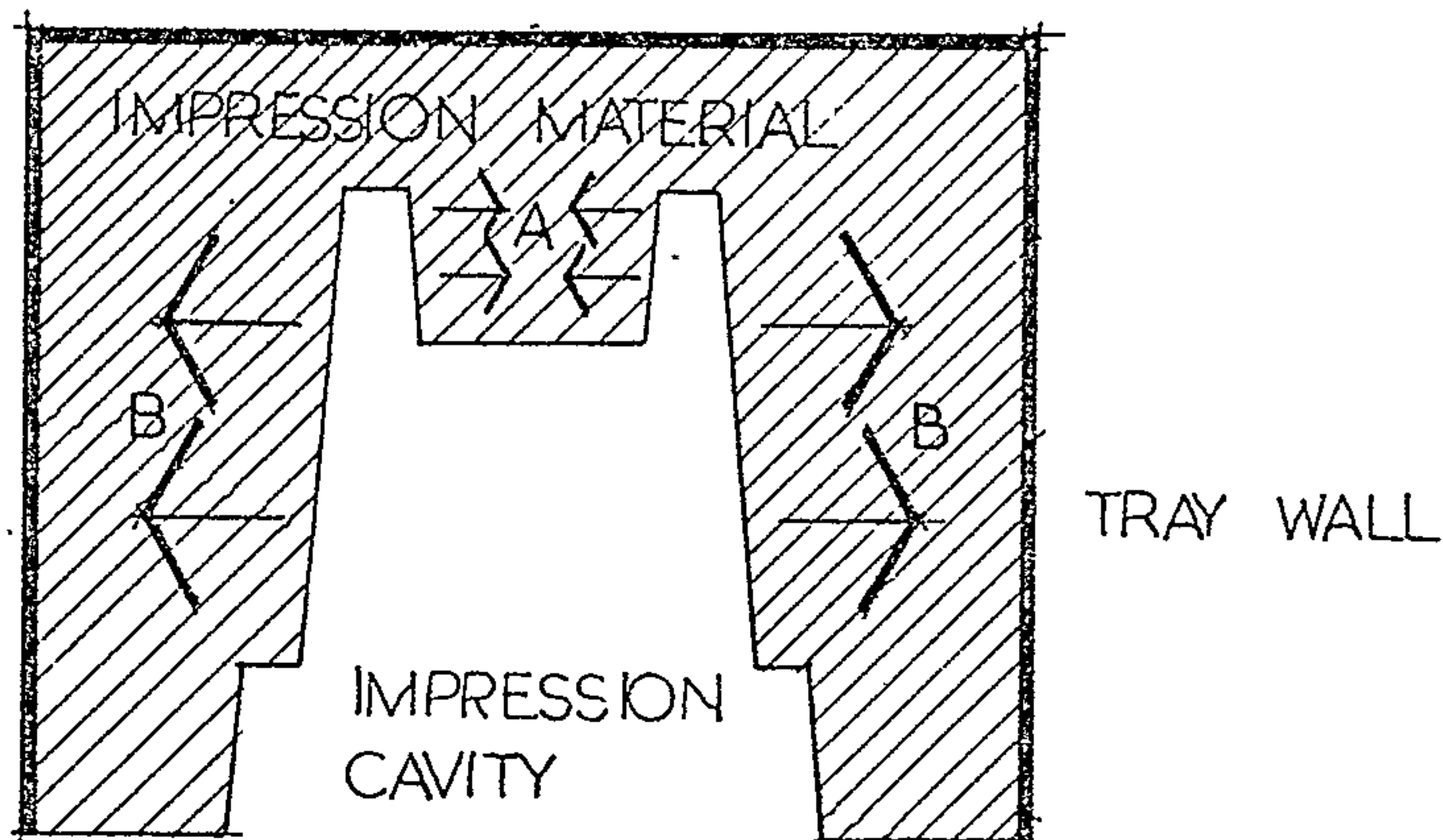
By creating a firm adhesive bond between the impression material and the walls of the impression tray it may be expected that any shrinkage which took place in the material would result in a contraction of the impression material towards the walls of the tray. In the simplest instance this is in fact the case, as is illustrated by the consistent construction of oversized dies of full crown preparations. However the complex design of preparations in dental restorative work are such that one cannot simply assume that all sections of a rubber base impression will contract towards the walls of the impression tray, thereby producing a die which is oversize in all dimensions.

Hosoda and Fusayama (1961) developed a die by which it was possible to compare both the outside (extra-coronal) and the inside (intra-coronal) dimensional changes in an impression. This work showed quite conclusively that dies from rubber base impressions were not oversize in all dimensions. An intra-coronal cavity was cut in the top of a steel full crown die, and impressions of this die were taken in a tube tray. When the impression material, which was bonded to the walls of the tube tray contracted, it was shown that the outside dimensions of the subsequent dies increased because of contraction of the impression material towards the walls of the tray. The inside dimensions decreased since the inside impression core contracted towards the centre.

Fig. 3 shows diagrammatically the relative effect of this shrinkage. These investigators also showed that the later the impressions were poured, the larger were the outside dimensions and the smaller were the inside dimensions. This phenomenon being due to the continued polymerization shrinkage of the impression material.

Stackhouse (1970) further illustrated the complexity of dimensional change when comparing the relative accuracy of different, commonly used impression techniques using polysulphide and silicone. He took impressions of steel, full crown master dies and showed that generally there was an increase in diameter of resultant stone dies and a decrease in length. This decrease in length is difficult to explain but may be partially due to the result of release of back pressure, built up in the occlusal area, after removal of the impression. The one exception to this general pattern was exhibited when a wash impression was taken over an unrelieved heavy base primary impression which had been vented to allow the escape of excess material used for the wash. In this case the diameters of all stone dies were undersize. Stackhouse suggested that this was probably due to distortion caused by insufficient relief provided for the wash, creating a back pressure which was released after the impression had been removed.

Fig. 3



Showing a cross-sectioned impression of the die used by Hosoda and Fusayama (1961) to illustrate the directional effect of contraction of rubber base impression material confined within an impression tray.

NOTE : A - Contraction inwards towards the centre of the core
B - Contraction outwards towards the walls of the tray.

Relining of Rubber Base Impression

The question of accuracy of relined rubber base impressions has caused controversy since their introduction. It was originally argued that the relining would create compressive forces in the original impression which upon removal would be released, and cause distortion resulting in undersized dies. Skinner and Cooper (1955), Myers and Stockman (1960) and La Forgia (1967), all suggested that this back pressure could be overcome by venting the original impression.

Podshadley et al (1970) carried out a detailed study of this problem. They made heavy bodied impressions of metal dies representing MOD preparations. These impressions were trimmed to remove the rubber material which would interfere with reseating on the master die. The impressions were then completely relined with a thin layer of light bodied material and reseated. After separation, casts were poured and changes in dimension in both vertical and horizontal planes were measured. In all cases the dies from relined impressions showed less distortion than was exhibited in dies from conventional double mix (light and heavy body) techniques. They also showed that the direction of dimensional change varied with the geometric configuration of the impression, as has already been mentioned above. In their conclusions they stated that, "casts produced from relined impressions more accurately duplicated the master die".

In direct contrast Shelvin et al (1970) found, when using methods of relining both relieved and unrelieved primary impressions, that the results regarding accuracy were unpredictable and that relining techniques should not be regarded as substitutes for the proficient use of standard impression procedures.

THE ORIGINAL INVESTIGATION

SCOPE OF THE INVESTIGATION

The purpose of this research project is to examine the dimensional stability occurring in the various types of rubber base impression materials currently available, and in common use in dentistry. The paper is presented in two parts.

PART I is an investigation into the unrestricted dimensional change which occurs in representative polysulphide, silicone and polyether type elastomeric impression materials. This part of the project is concerned with the measurement of the linear change occurring in these materials over a period of twenty four hours from the commencement of mix. The materials were tested when the curing process was carried out at a simulated oral temperature of 37°C , and also when curing was carried out at room temperature. In all cases, after curing, the specimens were maintained under constant conditions of temperature and humidity.

PART II concerns the effect of dimensional change taking place in impressions under simulated clinical conditions. This was measured by comparing the dimensions of dies constructed from impressions taken of a master model representing a dental arch. The influence of several variables of practical significance was examined and related to the accuracy of the resulting dies.

The effects of the following aspects of clinical usage, on the accuracy of impressions were investigated :

1. The restraining effect of adhesion of the impression material to the walls of the impression tray.
2. The effect of the bulk of impression material used.
3. The construction of more than one die from a single impression.
4. The time lapse, after removal of the impression, before construction of the die.
5. Electroplating of the impression surface to produce metallic dies.
6. The use of perforations, as opposed to contact adhesive, for the retention of the impression material in the impression tray.
7. The effect on the impression material of the dimensional change taking place in the selected die material.

PART I

The determination of the unrestricted dimensional change of representative polysulphide, silicone, and polyether type rubber base impression materials.

MATERIALS

Because of the large number of brands available to the profession, and the impracticability of testing all these materials, it was decided that one of each of the three types of rubber base materials should be selected for testing. It is believed that the polysulphide and silicone materials chosen are representative of their types, and the polyether material is the only brand of its type known to be available.

Manufacturers' recommendations regarding proportioning of the base and accelerator, of the various materials tested, was strictly adhered to. So also were the mixing times recommended.

The same brands and types of materials (Table 2) were used in Parts I and II of this project.

APPARATUS

The Mould. Figure 4 shows the apparatus used to form the specimens of rubber base material to be tested. This apparatus is an adaptation of that used by Chong and Docking (1969) for a similar study. It consists of a surface ground stainless steel plate approximately 4 x 2 x $\frac{1}{4}$ inches.

Table 2 Materials Tested

Material	Trade Name	Batch No.	Manufacturer
Polysulphide	Coe Flex Regular	03019 03049 02129	Coe Laboratories Inc., U.S.A.
Silicone	Elasticon Heavy Bodied	1074-M-438	Kerr Mfg. Co., U.S.A.
Polyether	Impregum	033577a 033510a 032772a	Espe Co., West Germany

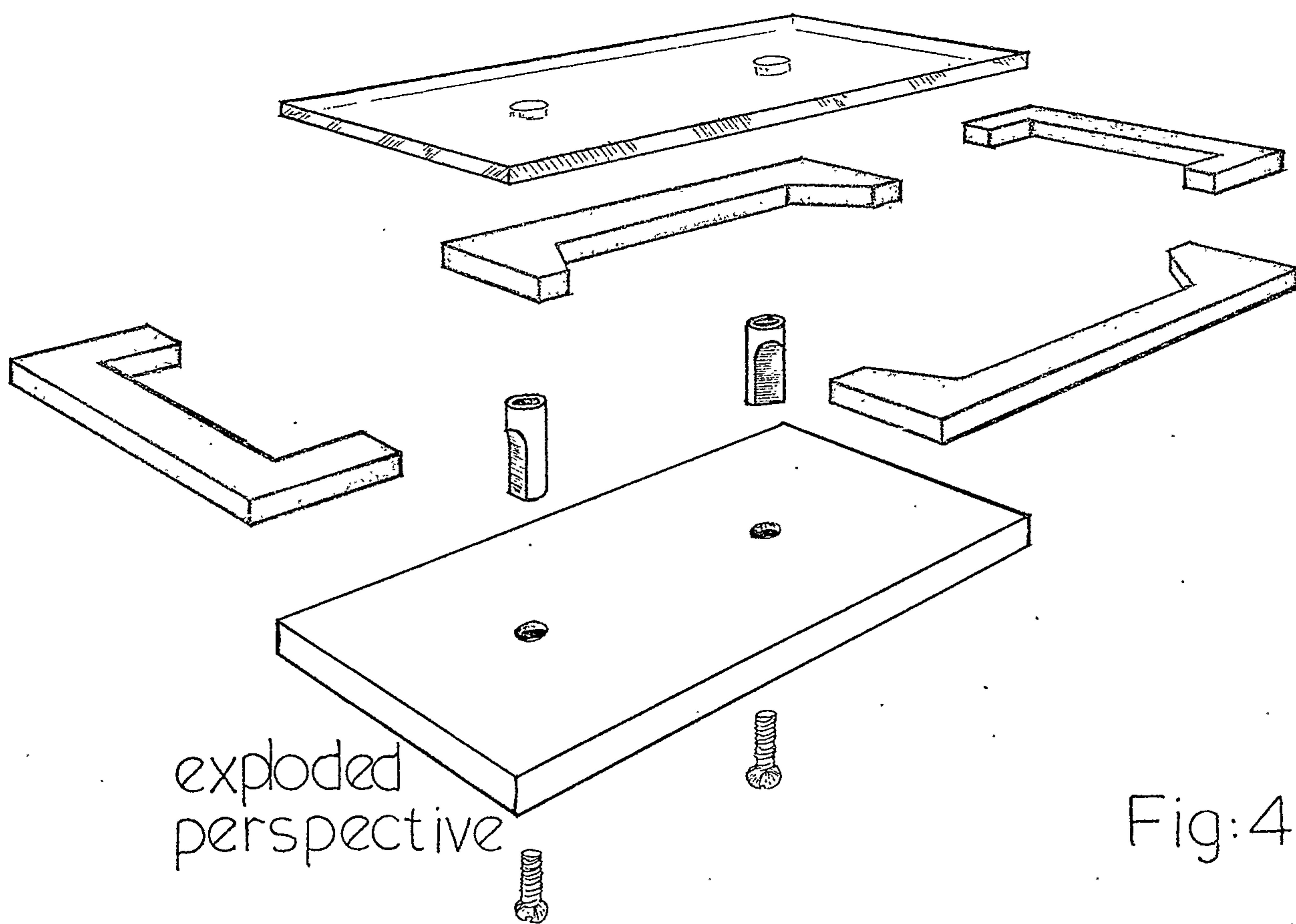
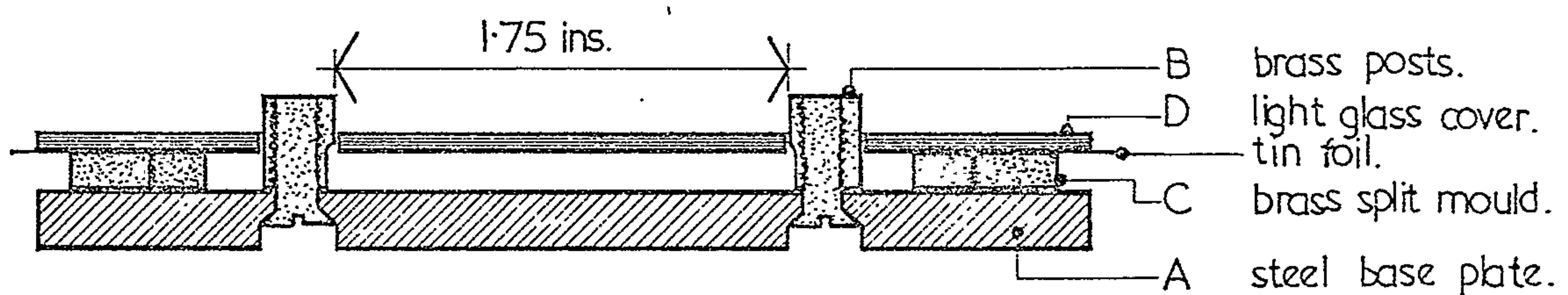
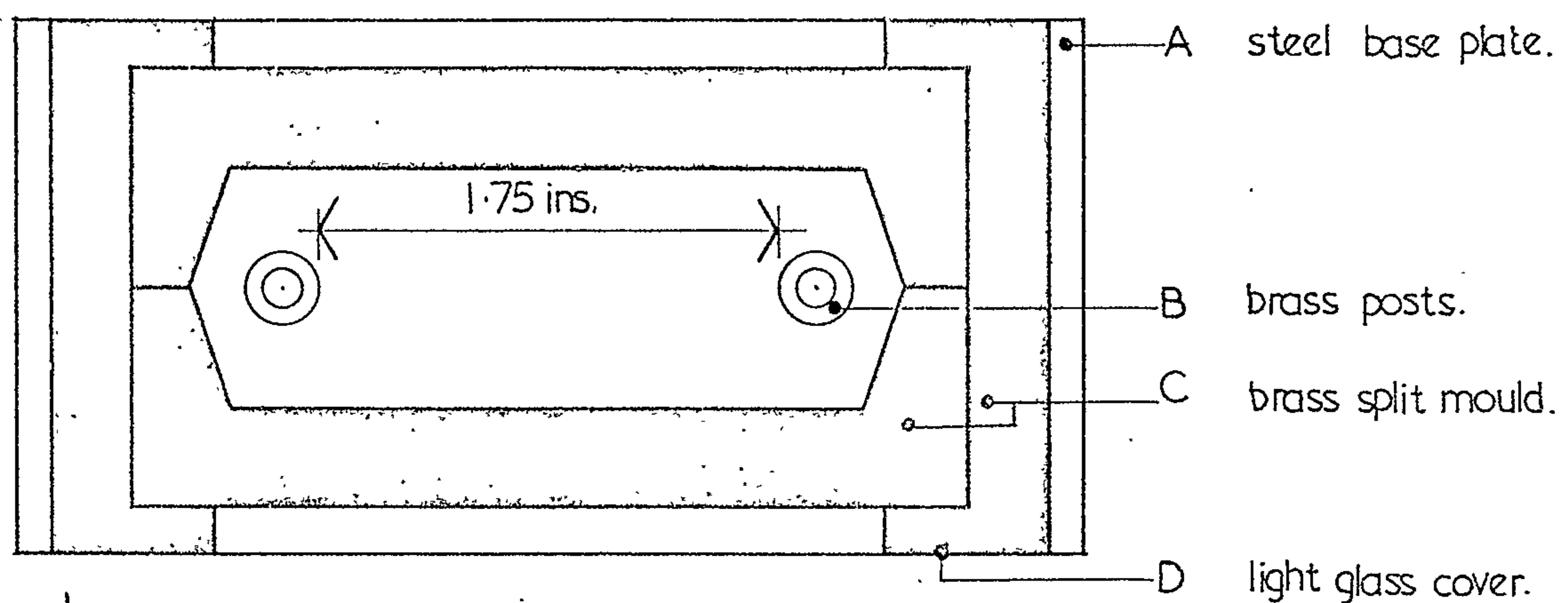


Fig:4

THE MOULD

Two counter-sunk holes were drilled, from underneath this base plate, to take screws which attached accurately turned cylindrical brass marker posts. The holes were positioned to allow a distance of approximately 1.75 inches between the inside tangents of the marker posts when the apparatus was assembled. The brass posts were bored and threaded, as illustrated, so that they could be screwed down flush with the base plate, thus preventing any possibility of movement when the mould was being loaded with impression material.

Both ends of the brass posts were very carefully paralleled and surface ground so that they were exactly the same height when screwed firmly to the ground surface of the base plate. The careful turning, and surface grinding of these posts presented a very sharp and definite circumference from which a tangential reading could be taken, using the measuring device to be described.

Square facets were cut on the ends of the marker posts designed to approximate the base plate. This reduced any tendency of the posts to turn in the cured rubber, when the screws were being released to free the specimen with the markers embedded in it.

When the marker posts were attached to the base as described, a four piece brass split mould was set symmetrically around the markers on the base.

A light glass cover, with holes bored to accept the ends of the marker posts was made to fit over the whole assembly.

The under surface of this glass plate was covered with a layer of thin tin foil during the construction of each specimen.

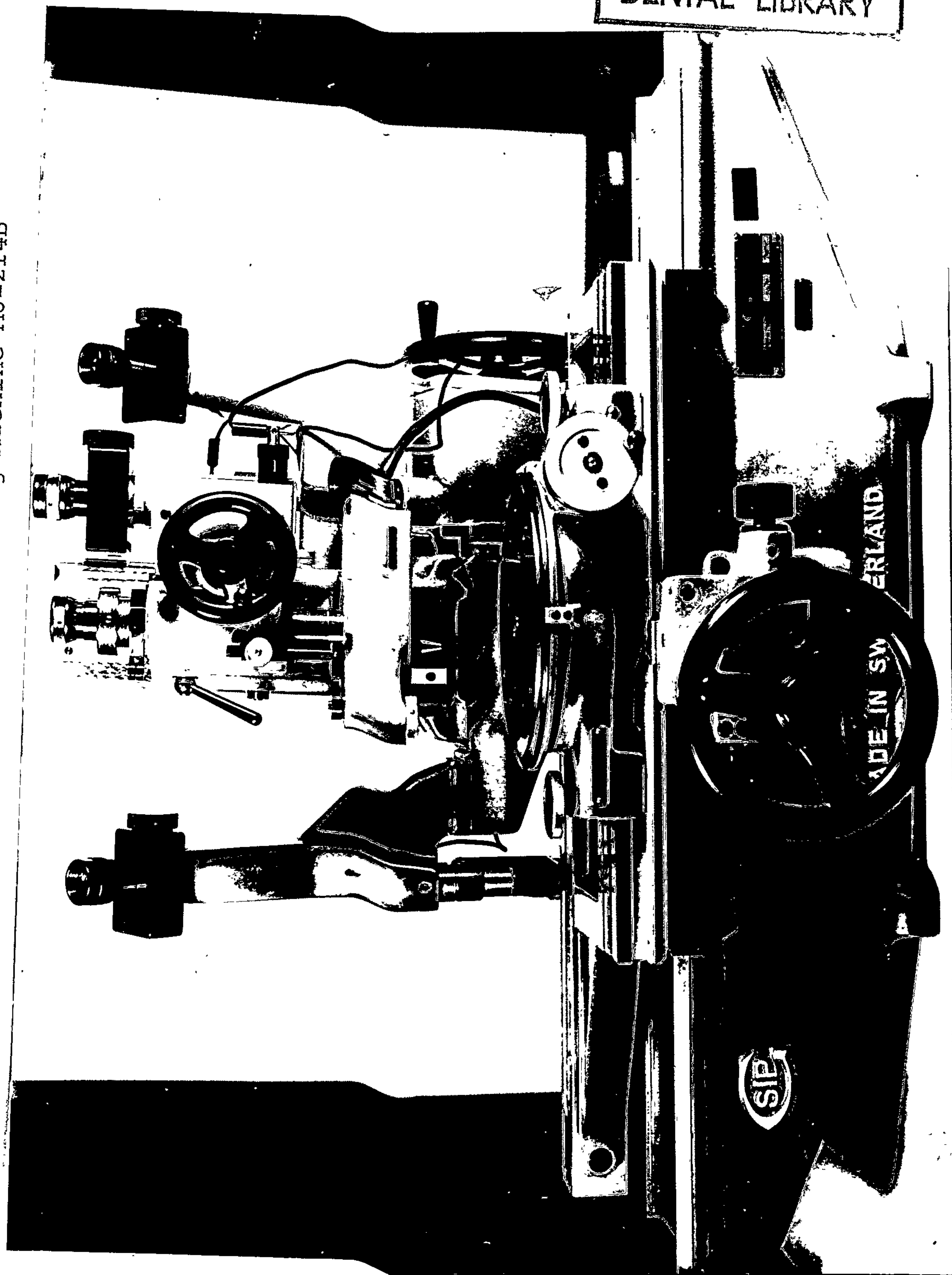
The Measuring Device. An S.I.P. Universal Measuring Apparatus Type MU 214B was used. This machine (Fig. 5) consists of a locating microscope with a x185 magnification, supported on a transverse carriage and mounted above a work table on a longitudinal carriage. Readings of linear measurement are made using a micrometer microscope graduated in one hundred thousands of an inch. This machine is capable of measurements accurate to ± 0.00005 inches.

The object being measured is normally illuminated by a fluorescent ring light, mounted around the object lens of the locating microscope. It was found in this study that the heat generated by this illuminating device had a measurable effect on the dimensions of the specimens being measured. It was therefore disconnected from the machine and all observations were taken without direct illumination from the fluorescent light.

The Curing Apparatus. This consisted of a water bath to which a B.T.L. 'Circon' heating and circulating unit was attached. This unit was set to maintain a constant circulation of water in the bath at a temperature of 37°C .

Temperature and Humidity. A room temperature of $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$, and a relative humidity of 50% were maintained during the entire conduct of Part I of this study.

Fig. 5- S.I.P. Universal Measuring Machine MU-214B



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TEST METHOD

The brass marker posts were firmly attached, by means of screws, to the steel base plate of the mould apparatus and this was placed on the work table of the measuring machine. The work was aligned so that the distance between the inside tangents to the two marker posts could be measured. This measurement was recorded as the original length of the specimen, and all subsequent readings were related to this fiducial reading.

The split mould was then set around the markers (Fig.4) and a quantity of rubber base material was mixed, and spatulated around the posts so as to slightly overfill the mould. The glass cover plate, lined with tin foil, was settled gently over the whole assembly.

In the case of those specimens which were cured at 37°C , the assembly was then transferred to the curing bath two minutes after the commencement of mix, and allowed to remain there for a further eight minutes. The specimens cured at room temperature were left to bench cure for twenty five minutes in the case of polysulphides, and fifteen minutes in the case of silicone and polyether materials.

After curing, the specimens were carefully removed by unscrewing the marker posts, lifting the glass cover plate, stripping off the tin foil and then sliding the specimen from the base plate. The specimen was then separated from the sections of the split mould and any flash of material was trimmed with scissors.

Although the use of a mould release agent was not found necessary with silicone and polyether specimens, it was noted that polysulphide adhered very firmly to the clean surface of the brass split mould. A release agent proved essential if specimens of this material were to be separated from the mould without distortion. The most effective separation was afforded by a light smear of liquid soap on all brass surfaces likely to come into contact with the polysulphide.

After separation, the base of the specimen was lightly dusted with talcum powder, and the specimen was placed on a Grade "A" Engineers Steel Parallel, the surface of which had also been lightly dusted. The specimen, on its steel parallel, was then aligned on the measuring instrument and a second reading taken of the distance between the posts. This second reading was taken twenty minutes after the commencement of mix for those specimens cured at 37°C , and ten minutes after the curing time allowed, for specimens cured at room temperature. Further readings were taken at hourly intervals up to five hours, and then a final reading at twenty four hours. Once a specimen had been placed on a steel parallel it could be transferred to and from the work table of the measuring instrument without being disturbed.

RESULTS AND DISCUSSION

The results, Table 3, show the effect of time on the dimensional change of unrestricted rubber base impression.

materials cured at 37°C . Each result is the mean of observations of five specimens, and is expressed as a percentage of the fiducial reading for each specimen.

It will be noted that in all cases a progressive contraction occurred during the twenty four hour period. The contraction demonstrated to take place in the first twenty minutes after mixing is of considerable magnitude in all materials tested. This finding is in agreement with that of Chong and Docking (1969), and contrary to that of Skinner (1958), who asserted that there was no demonstrable change during the first thirty minutes, with either polysulphide or silicone materials.

Figure 6 illustrates the relative contraction which occurs between the different materials under the conditions of this experiment. The polysulphide and polyether materials have both been demonstrated to be considerably more stable than the silicone material. All the results closely support the contention that the actual dimensional change of the rubber base materials is considerably greater than was previously realized. This is due to the fact that previous test methods did not measure movement in the material during and shortly after setting. Using the apparatus and technique in this test the fiducial reading for each specimen is established before any movement has taken place. As a consequence all effective movement is recorded by this method.

Table 3 - The Effect of Time on the Dimensional Change of
Unrestricted Impression Materials Cured at 37°C

Material	Percent Dimensional Change at Given Time Intervals from Commencement of Mix						
	20 min	1 hr 20'	2 hrs 20'	3 hrs 20'	4 hrs 20'	5 hrs 20'	24 hrs
Polysulphide	-0.26(.02) [*]	-0.29(.03)	-0.30(.04)	-0.30(.04)	-0.31(.05)	-0.32(.05)	-0.40(.05)
Silicone	-0.46(.03)	-0.59(.04)	-0.64(.04)	-0.68(.05)	-0.71(.05)	-0.73(.05)	-0.92(.04)
Polyether	-0.34(.04)	-0.36(.04)	-0.37(.04)	-0.37(.04)	-0.37(.04)	-0.37(.04)	-0.43(.07)

* standard deviations in brackets

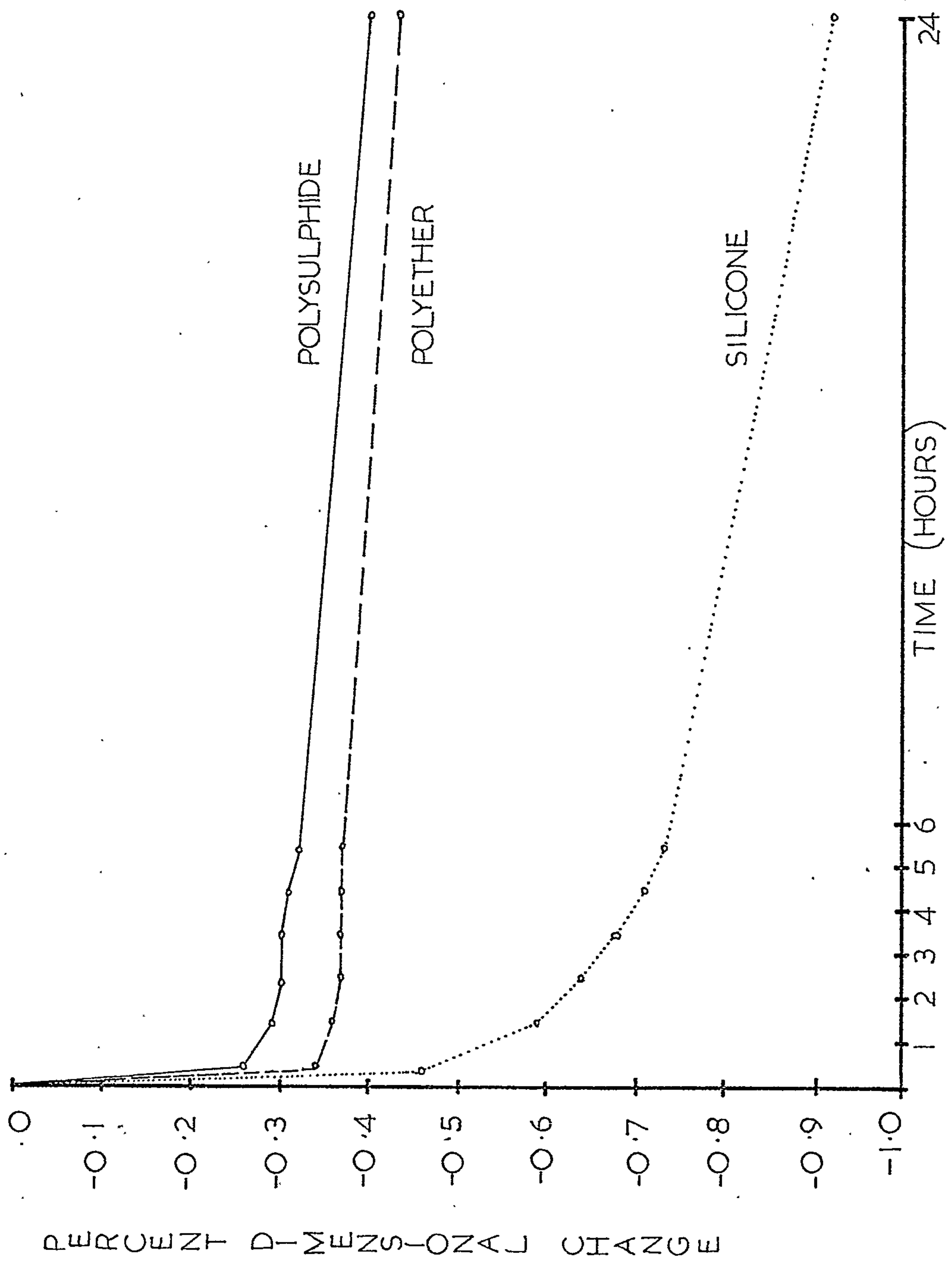


Fig. 6 - The dimensional stability curves of unrestricted impression material cured at 37°C.

The reproducibility of results using this test method can be shown to be similar to that of the tests by Chong and Docking (1969). They stated that for earlier test methods (Draft Australian Standard T.27) the average of the standard deviations expressed as percentages of contractions at six hours was 37.4%. For their test method the average was 10.6%. Similar calculations made for this series of tests show an average of 11% for the readings taken at five hours and twenty minutes after the start of mixing.

Table 4 shows the effect of time on the dimensional change of specimens cured at room temperature. The results are the observations of only one specimen of each material. Because of restriction on the use of laboratory facilities it was not possible to conduct more tests in this series. However, having established the reproducibility of results for this test method, it was considered that observations of one specimen could still contribute useful information.

All materials showed a continual contraction over the twenty four hour test period. Polysulphide and polyether showing less than one third of the contraction of the silicone material over this period. Figure 7 shows graphically the relative changes between the different materials tested.

By examination of Figures 6 and 7 it is immediately apparent that the magnitude of contraction is substantially less in the case of room temperature cured specimens than in those specimens cured at body temperature. It is also

Table 4 - The Effect of Time on the Dimensional Change of Unrestricted Impression Materials Cured at 20°C.

Material	Percent Dimensional Change at Given Time Intervals From Commencement of Mix						
	35 min	1 hr 35'	2 hrs 35'	3 hrs 35'	4 hrs 35'	5 hrs 35'	24 hrs
Polysulphide	-0.03	-0.05	-0.05	-0.05	-0.05	-0.06	-0.13
	25 min	1 hr 25'	2 hrs 25'	3 hrs 25'	4 hrs 25'	5 hrs 25'	24 hrs
Silicone	-0.10	-0.26	-0.29	-0.33	-0.37	-0.39	-0.60
	25 min	1 hr 25'	2 hrs 25'	3 hrs 25'	4 hrs 25'	5 hrs 25'	24 hrs
Polyether	-0.07	-0.08	-0.08	-0.08	-0.08	-0.09	-0.14
	25 min	1 hr 25'	2 hrs 25'	3 hrs 25'	4 hrs 25'	5 hrs 25'	24 hrs

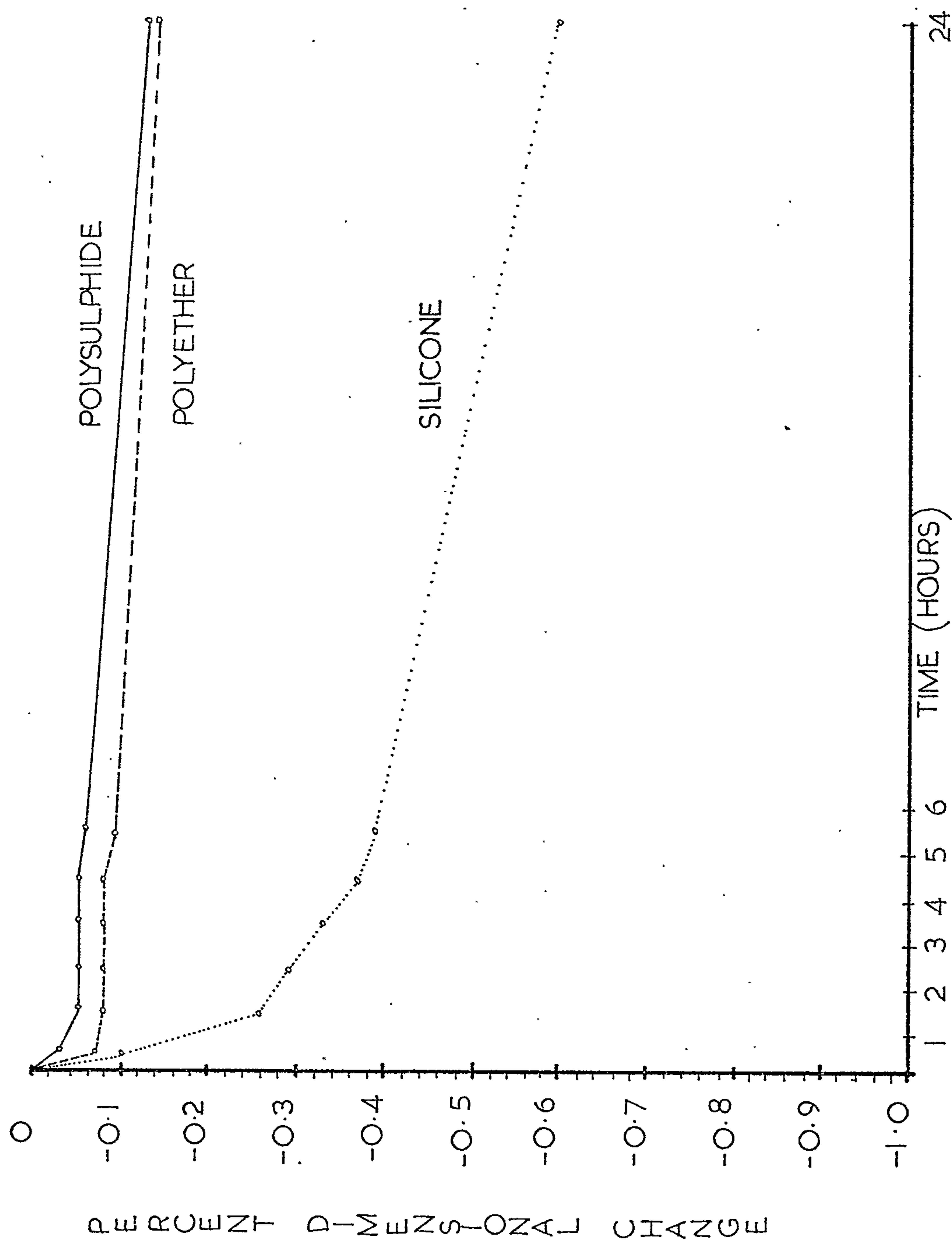


Fig. 7 - The dimensional stability curves of unrestricted impression material cured at 200C.

apparent that the bulk of this difference in percentage contraction takes place during the first hour after mixing. It is suggested that this difference in contraction is attributable almost entirely to the thermal shrinkage of the materials which were cured at 37°C. Working from this assumption it should be possible to calculate the thermal coefficient of linear expansion of a material from the difference in percent contraction between specimens cured at 37°C and those cured at 20°C.

At one hour from the commencement of mix it can reasonably be assumed that a specimen cured at 37°C has reached a state of equilibrium with the room temperature i.e. the temperature of the specimen has dropped 17°C. By reference to the one hour contraction values, Figs 6 and 7, it will be noted that the difference in percent contraction between specimens cured at 37°C and bench cured specimens is ; polysulphide 0.24%, silicone 0.37% and polyether 0.27%. Dividing these figures by seventeen gives the percent change per °C. From this, an assessment of the thermal coefficient of linear expansion of each material tested can be calculated. The results of such calculations are :

Polysulphide	-	140 x 10 ⁻⁶	in/in°C
Silicone	-	220 x 10 ⁻⁶	" . "
Polyether	-	160 x 10 ⁻⁶	" "

These figures for the thermal coefficient of polysulphide and silicone compare favourably with those presented by McLean (1961)

The coefficient for the polyether material is shown to be similar to that of polysulphide.

SUMMARY

The dimensional stability of unrestricted rubber base materials has been tested. With the method used, all materials showed a continual contraction over a twenty four hour period. The relative magnitude of this contraction, for the various materials tested, has been illustrated.

When materials were cured at mouth temperature and tested at room temperature they showed a contraction of greater magnitude than those both cured and tested at room temperature. The difference in this degree of contraction has been shown to be attributable to the thermal coefficient of linear expansion of the materials being tested. These coefficients have been calculated from the data of the experiments.

The contention of Chong and Docking (1969) that previous test methods, which established a fiducial reading at varying times after a specimen had been cured, did not record all effective movement in the specimen, has been substantiated.

PART II

In this section nine separate experiments were conducted in order to determine the effects of certain aspects of clinical usage on the dimensional change of polysulphide, silicone and polyether type rubber base impression materials.

MATERIALS

The same selection of brands and types of impression materials was used in Part II of this study as in Part I (Table 2).

With the exception of Experiments 5 and 9, all dies were constructed of Vel Mix Stone (Kerr Manufacturing Co. U.S.A.), which has a one hour setting expansion of 0.05%. This was carefully weighed to produce a water powder ratio of 22/100. The water used to mix the stone was maintained as closely as possible to 22°C. Each mix was hand spatulated for 40-45 seconds, and then vibrated into the impression using an electrical vibrator.

In the case of Experiment 5, the impression surface was first silver plated and then filled with Vel Mix to form the die.

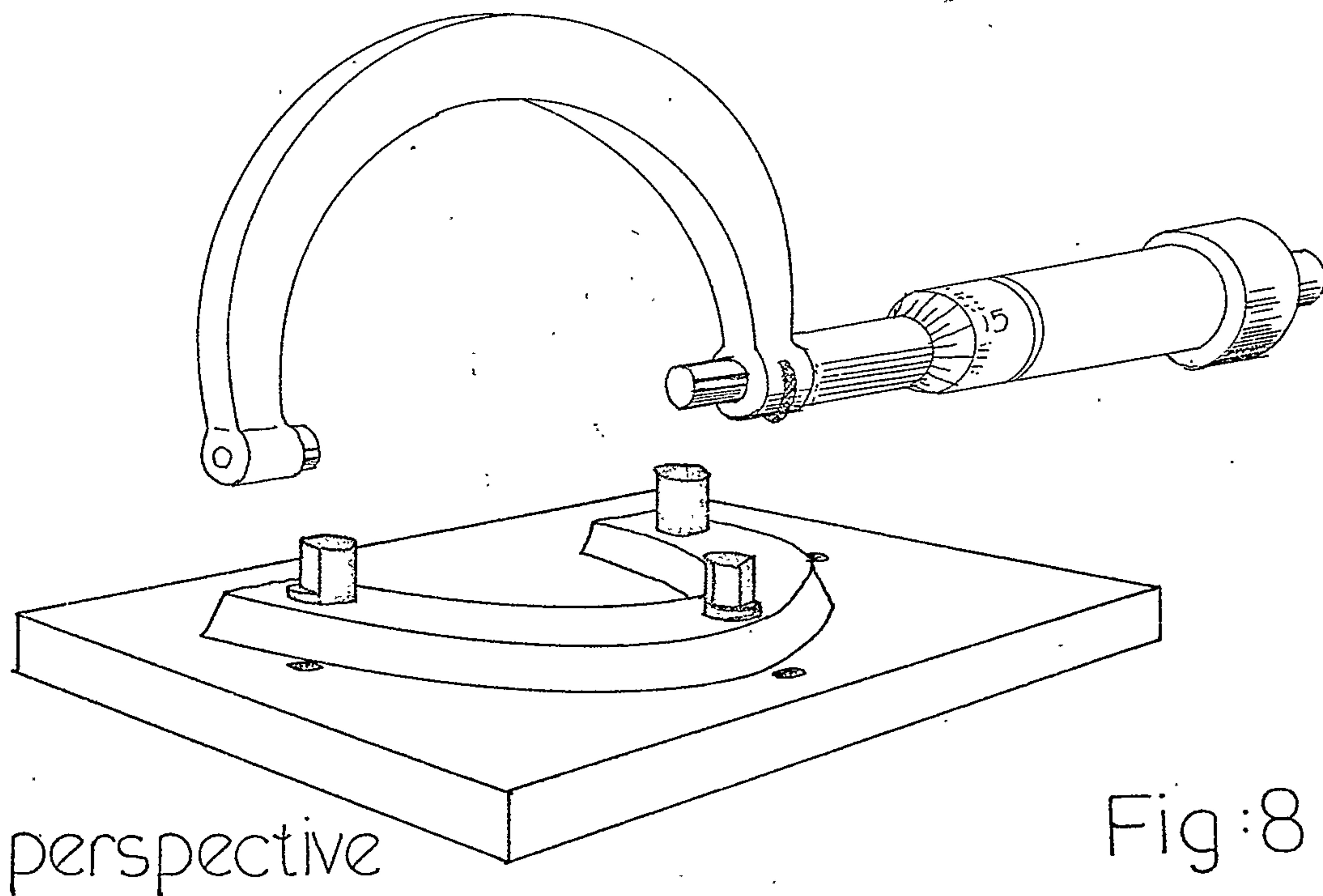
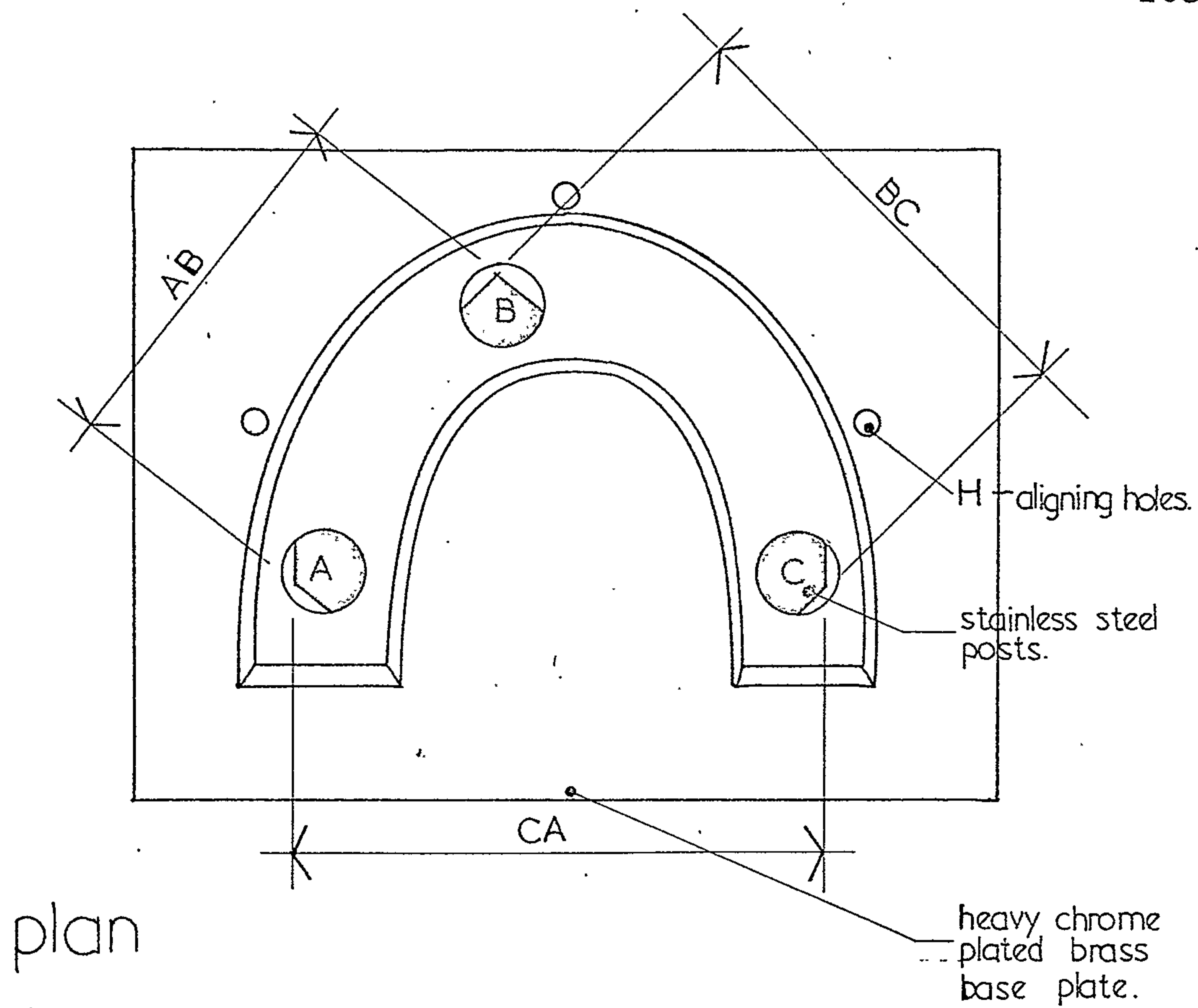
Experiment 9, employed a Class I stone with a two hour expansion rate of 0.35%. This was Investo Yellowstone (Investo Manufacturing Co. Aust) mixed with a water powder ratio of 33/100.

All stone dies were allowed one hour to set before removal from the impression. They were then left on the bench for a further twenty four hours before being measured.

APPARATUS

The Master Model. Fig. 8 shows the master model used in these experiments. This consisted of a heavy chrome plated brass base-plate, on which was mounted a horse-shoe shaped simulated alveolar ridge of the same material. Three stainless steel posts, A, B, and C projected from this ridge. These posts were ground to a minimal taper of 2° towards the occlusal, to reduce drag on removal of the impression. Parallel facets were ground on the posts to enable measurement of the dimensions A-B, B-C, and C-A to be made with a micrometer caliper. Positioning of the micrometer was facilitated by the presence of small shoulders on each measuring facet, at equal heights above the ridge. The actual magnitude of these dimensions was not critical. The model was designed to allow measurement of medium (A-B) and long (B-C) spans, and also measurement of the distance across the heels of the model (C-A). The actual dimensions were determined after the model had been conditioned in a water bath at 37°C for thirty minutes. These dimensions were :

A-B	=	1.6705"
B-C	=	1.9793"
C-A	=	2.3611"



THE MASTER MODEL

Three holes, H, were drilled in the base plate to receive the pins incorporated in the impression tray. These were for the alignment of the trays when seating the impression, thus ensuring an even distribution of impression material within the tray.

Trays. All trays were constructed of self curing acrylic resin. Three separate series of trays were made :

1. Full arch trays including all three posts and allowing for an average thickness of 2 mm of impression material.
2. Sectional trays including posts A and B only, and allowing the same 2 mm thickness of impression material.
3. Sectional trays including posts A and B only, and allowing an average thickness of 5 mm of impression material.

All trays, with the exception of those which were perforated, were coated internally with adhesive. The adhesive appropriate to the brand and type of material was used according to the manufacturer's instructions. The perforated trays were prepared with perforations 2 mm in diameter placed 2 mm apart for maximum retention (Fusayama and Nakazato 1969).

Measuring Device. All measurements of length were made using a hand held 'Starret' Micrometer Caliper which was graduated in ten thousandths of an inch.

Curing Apparatus. The same arrangement of water bath and temperature control unit was used as in Part I.

Electroplating Equipment. All electroplating was carried out in a 'Galloni' electroplating apparatus, capable of supplying a regulated current of from 0-500 MA. Kerr Silver Metalizing Powder was used to provide a conducting surface. The electrolyte used was made according to the formula recommended by Skinner and Phillips (1967) for silver plating, and a pure silver anode was used.

Temperature and Humidity. Room temperature, during the conduct of all experiments, was kept within a range of 65-75°F. The humidity was neither controlled or recorded.

STATISTICAL ANALYSIS

When measuring dies, each measurement was repeated three times. The mean of these three readings was recorded as the actual measurement of the die.

Five dies were produced from each impression material, in each experiment, and the mean dimension was calculated together with the standard deviation.

The difference between the mean dimension of the die, and the dimension of the master model was expressed as the percent dimensional change of the die, corrected to two decimal places.

For the purposes of this research the difference between two values was termed statistically significant when the "t" ratio indicated a probability of less than $P = 0.05$ (95% confidence level). The result was considered to be

statistically highly significant when the probability was less than $P = 0.01$ (99% confidence level).

The "t" distribution when comparing the dimensions of dies with the dimensions of the master model, were calculated for $n-1$ degrees of freedom, in accordance with the following formula (Hoel 1966) :

$$t_{(n-1)} = \frac{\bar{x} - \mu}{s} \sqrt{n}$$

where n = the number of dies measured

μ = the dimension of the master model

$\bar{x}$ = the mean dimension of the dies

s = the standard deviation of $\bar{x}$

When comparing the dimensions of dies made using different materials, or different techniques, the statistical significance was calculated for $n_1 + n_2 - 2$ degrees of freedom using the following formula : (Hoel 1966)

$$t_{(n_1+n_2-2)} = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{\frac{(n_1-1)s_1^2 + (n_2-1)s_2^2}{n_1+n_2-2} \times \frac{n_1+n_2}{n_1n_2}}}$$

where n_1 and n_2 = the number of dies measured in samples 1 and 2 respectively

$\bar{x}_1$ and $\bar{x}_2$ = the mean dimension of the dies from sample 1 and 2 respectively

s_1 and s_2 = the standard deviations of $\bar{x}_1$ and $\bar{x}_2$ respectively.

All actual measurements were transferred to computer punch cards, and a Fortran computer programme was written to provide all statistical information required. This programme was run on a Honeywell D.D.P. 516 Computer.

METHOD AND RESULTS

Experiment No. 1

Five impressions of the master model were taken, using each of the rubber base materials being tested. Full arch trays, allowing an even distribution of impression material 2 mm thick, were used. Each tray was coated with a layer of bonding adhesive appropriate to the impression material used, and this was allowed ten minutes to dry.

The master model was conditioned in the curing bath at a temperature of 37°C for at least thirty minutes prior to impression taking. The impression material was mixed and loaded into the tray. The master model was removed from the water bath and the loaded impression tray was immediately settled firmly into position, aided by the guide pins which had been incorporated in the impression tray. In each case two minutes were allowed for the mixing of the impression material, and the loading and positioning of the tray. The whole assembly was then returned to the water bath and allowed to remain until the impression material had reached its final set (eight minutes in the case of polysulphide and five minutes in the case of silicone or polyether materials.)

After removal from the curing bath the impression was separated from the master model. The impression was washed and dried and a Vel Mix stone die was poured. This die was allowed one hour to harden before being separated from the impression. The die was then left on the bench for a further twenty four hours, before measurements were made of the distances between posts A, B and C.

It will be seen from the results presented in Table 5 that the dimensions of the dies produced in this experiment were generally undersize. The dies produced from polysulphide showed the smallest percent dimensional change. In all cases the percent change in dimension is extremely small and is of no clinical significance.

Statistically, dimensions A-B and B-C in dies produced from polysulphide impressions are not significantly different to those of the master model.

When comparing the results of dies made using one particular impression material, against dies produced from either of the other materials, it was established from "t" tests, that there was no statistical difference between the various dies.

It was noted during the progress of this experiment that some polysulphide impressions exhibited a number of sub-surface bubbles, which were reproduced as raised areas on the stone dies. The existence of these sub-surface bubbles has been previously reported (Schnell & Phillips 1958, Miller et al 1960). Since the bubbles were not evident in silicone

Table 5 - Comparison of Die to Master Model employing Impression Material for Optimum Usage*

Material	Dimension of Die	Mean Dimension of Die	Standard Deviation	% Dimensional Change	Significance	
					t_4	P
Poly-sulphide	A-B	1.6704"	0.0003"	-0.01	0.88	0.1<P<0.05
	B-C	1.9793"	0.0005"	0.0	0.17	0.5<P
	C-A	2.3604"	0.0003"	-0.03	5.87	P<0.01
Silicone	A-B	1.6702"	0.0002"	-0.02	3.83	0.01<P<0.02
	B-C	1.9790"	0.0002"	-0.02	2.87	0.02<P<0.05
	C-A	2.3602"	0.0004"	-0.04	5.37	P<0.01
Polyether	A-B	1.6698"	0.0005"	-0.04	3.38	0.02<P<0.05
	B-C	1.9784	0.0007"	-0.06	3.02	0.02<P<0.05
	C-A	2.3603"	0.0007"	-0.04	2.04	0.05<P<0.10

*Employing A) Manufacturers recommended adhesive

B) 2 mm thickness of material

C) Immediate pour

or polyether impressions it was concluded that they were due to air bubbles which were incorporated in the paste as supplied by the manufacturer, and which could not be entirely eliminated during mixing of the material.

Experiment No. 2

Because of the common clinical practice of pouring duplicate models in rubber base impressions, it was decided to test the effect of this practice on linear accuracy.

In this experiment the same impressions made in Ex. 1 were used to produce duplicate dies. Following the removal of the first die, one hour after pouring, a second Vel Mix Stone die was poured in the same impression. This die was allowed one hour to harden. It was then removed and left on the bench for twenty four hours before being measured.

The results, Table 6, show dimensions A-B and B-C to be oversize for dies from polysulphide and silicone materials. All dimensions of dies from polyether impressions are undersize. It is interesting to note that dimension C-A is undersize in dies from all three impression materials.

As in Ex. 1 dimensions A-B and B-C for dies from polysulphide show no statistically significant difference to the master model.

The percent dimensional change in all dimensions, for polysulphide and silicone, is still extremely small and of no clinical significance. The percent change exhibited by the

Table 6 - Comparison of Die to Master Model employing a Second Pour immediately following separation of First Model

Material	Dimension of Die	Mean Dimension of Die	Standard Deviation	% Dimensional Change	Significance	
					t_4	P
Poly-sulphide	A-B	1.6706"	0.0003"	+0.01	0.57	0.5 < P
	B-C	1.9794"	0.0007"	+0.01	0.26	0.5 < P
	C-A	2.3603"	0.0003"	-0.03	6.76	P < 0.01
Silicone	A-B	1.6711"	0.0002"	+0.04	5.81	P < 0.01
	B-C	1.9799"	0.0002"	+0.03	6.04	P < 0.01
	C-A	2.3609"	0.0008"	-0.01	1.05	0.1 < P < 0.5
Polyether	A-B	1.6690"	0.0005"	-0.09	7.10	P < 0.01
	B-C	1.9775"	0.0005"	-0.09	7.60	P < 0.01
	C-A	2.3593"	0.0005"	-0.08	5.06	P < 0.01

dies from polyether impressions is considerably larger than in Ex. 1, but it is still not clinically significant.

In contrast to the results of Ex. 1 there is a significant statistical difference in the results between dies made from different impression materials. In most cases this difference is highly significant, e.g. comparing dimensions A-B for dies made from polysulphide, and polyether impressions, $t_8 = 6.2$. The probability therefore is $P < 0.01$ and the difference between the two mean measurements is highly statistically significant.

The results of the collapse of sub-surface bubbles in polysulphide impressions was more apparent in this experiment. Several experiments had to be repeated because of deformation of the measuring facets of the dies.

Experiment No. 3

In practice many rubber base impressions are not poured for several hours after removal from the mouth. At times impressions may be mailed from country areas, or overseas, and dies are not poured for days. Experiment 3 was designed to determine the effect of delay in pouring on the accuracy of impressions. The method was identical with that in Ex. 1, to the stage where the impression was removed from the master model. In this case each impression was left on the bench for twenty four hours before the Vel Mix die was poured.

The results, Table 7, show very little change compared with those of Experiment 2. The dimensional change in dies from polysulphide impression material was again demonstrated to be less than in dies from either of the other materials tested. There was no statistically significant change in dimensions A-B and B-C, for dies produced from polysulphide, compared to the master model. As in previous experiments the dies from polyether impressions were undersize in all dimensions.

Generally there was statistical significance demonstrated between the dies produced from different materials under the conditions of this experiment.

The presence of sub-surface voids was even more apparent after 24 hours, and again caused the rejection of several dies.

Experiment No. 4

Looking for an explanation for the consistent production of undersize dies from polyether material, it was assumed that the adhesive used with this material was ineffective. The polymerization and thermal shrinkage, demonstrated to take place in the unrestricted material, was causing it to contract away from the walls of the tray, thus producing an undersized die. It was decided therefore to examine the dimensions of dies produced from rubber base material used in perforated trays, rather than in trays coated with adhesive.

Table 7 - Comparison of Die to Master Model, Casting Delayed twenty four hours

Material	Dimension of Die	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
					t_4	P
Polysulphide	A-B	1.6706"	0.0003"	+0.01	1.04	0.1<P<0.5
	B-C	1.9792"	0.0005"	-0.01	0.30	0.5<P
	C-A	2.3610"	0.0002"	0.0	1.30	0.1<P<0.5
Silicone	A-B	1.6712"	0.0004"	+0.04	4.11	0.01<P<0.02
	B-C	1.9797"	0.0003"	+0.02	3.07	0.02<P<0.05
	C-A	2.3607"	0.0005"	-0.02	1.97	0.1<P<0.5
Polyether	A-B	1.6688"	0.0005"	-0.10	6.84	P<0.01
	B-C	1.9773"	0.0008"	-0.10	5.73	P<0.01
	C-A	2.3585"	0.0012"	-0.11	4.73	P<0.01

The general method of Experiment 3 was repeated, the only difference being that perforated trays without adhesive were used. Tests were conducted using only silicone and polyether impression materials.

Table 8 shows the results of this experiment. The dimensional change in the dies from silicone is very small and shows no statistically significant difference to the master model. However it should be noted that the standard deviations of the means are considerably higher than is the case in Experiments 1, 2 and 3. This indicates a generally unpredictable result for dies produced from silicone material using this method.

The dies from polyether impressions are still under-size in all dimensions, and the magnitude of the change can be seen to be greater than in any of the previous experiments. The difference in the mean dimensions of these dies, compared to the master model, is highly statistically significant.

Experiment No. 5

The purpose of this experiment was to assess the effect of metal plating on the linear dimensional change of rubber base impressions.

Attempts to copper plate the materials, using copper metalizing powder as the conducting agent, were disappointing. It was found that the copper plate could not be separated from the polysulphide. The metalizing powder tended to float off the surface of the polyether material, and only silicone

Table 8 - Comparison of Die to Master Model using Perforated Trays*

Material	Dimension of Model	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
Silicone	A-B B-C C-A	1.6706" 1.9799" 2.3612"	0.0012" 0.0007" 0.0003"	+0.01 +0.03 0.0	t_4	P
					0.18	0.5<P
					1.91	0.1<P<0.5
					0.07	0.5<P
Polyether	A-B B-C C-A	1.6680" 1.9763" 2.3574"	0.0003" 0.0002" 0.0009"	-0.15 -0.15 -0.16	20.67	P<0.01
					27.91	P<0.01
					8.90	P<0.01

*Casting delayed twenty four hours

produced consistently well plated copper dies. For these reasons only the results of silver plating experiments are recorded here.

For this and subsequent experiments, sectional trays, which included the medium length span A-B only, were used. The appropriate adhesive was used, and all aspects of impression taking were as described for Experiment 1.

Immediately after separation from the master model the impression was coated with a layer of silver metalizing powder. The impression was then connected with the cathode terminal of the electroplating apparatus, which had been set up for silver plating operation. An initial current of 15-20 MA/in² was employed for one hour. The current was then raised to 50 MA/in² for a further seven hours. The electrolyte, during the entire plating operation, was maintained at a temperature between 65-75°F.

When the plating operation was completed Vel Mix Stone was vibrated into the mould and allowed an hour to set.

The results of the dimensional change recorded in the plated dies are listed in Table 9. Unexpectedly, the dies from polysulphide impressions are seen to be slightly undersize. The plated dies from silicone and polyether appear to follow the same dimensional change pattern as the unplated stone dies from these materials. Those from silicone being slightly oversize and those from polyether slightly undersize. In all cases the percent change is very small and of no clinical significance.

-Table 9 - Comparison of Die to Master Model Silver-Plated
Impression *
(Dimension A-B Only)

Material	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
				t_4	P
Polysulphide	1.6696"	0.0007"	-0.05	2.78	P=0.05
Silicone	1.6711"	0.0002"	+0.04	6.39	P<0.01
Polyether	1.6699"	0.0006"	-0.04	2.27	0.05<P<0.10

* 2 mm Thickness of Impression Material

Experiment No. 6

Experiment 6 was carried out using sectional trays constructed to allow a bulk of 5 mm of impressions material. Only the posts A, B were included in these trays.

The method of mixing, impression taking, and curing was identical with Experiment 1. After separation of the impression from the master model a stone die was immediately poured, and when set, was allowed twenty four hours to age. Measurement of these dies produced the results set out in Table 10-A.

All dimensional change was of very small magnitude, with only dies from silicone impressions indicating any increase in size. Comparing the results for each of the materials in this experiment with results for the same material in Experiment 1, it can be shown that there is a statistically highly significant difference between the dies produced from silicone by the different methods. The dies from polysulphide and polyether are not significantly different from those in Experiment 1.

Experiment No. 7

The same impressions made in Experiment 6 were used to produce duplicate dies. Following the removal of the first die from each impression a second die was poured. The dimensions of these dies are shown in Table 10-B.

The dies from polysulphide are slightly oversize indicating contraction of the impression material back towards the walls of the tray. As would be expected, dies from silicone material show a more marked contraction towards the walls of the tray than in the previous experiment. The polyether impressions continue to exhibit a shrinkage inwards, resulting in smaller dies.

In all cases the difference between the mean dimensions of dies and those of the master model is shown to be statistically highly significant. Again, the dies from silicone impressions are statistically highly significantly different from those produced under similar conditions but with less bulk of impression material, in Experiment 2.

Experiment No. 8

The method in this experiment was identical to that of experiment 6, to the stage where the impression was removed from the master model. The impression was then left on the bench for twenty four hours before the die was poured. The measurements of these dies appear in Table 10-C.

Dies from polysulphide and polyether show relatively the same dimensional change as in Experiment 7. Dies from silicone are considerably oversize, showing the greater contraction of the silicone material over the twenty four hour period. It is considered that a percent dimensional change of this magnitude would be clinically significant.

Table 10 - Comparison of Die to Master Model using
5 mm thickness of Impression Material,
Dimension A-B only

A. Immediate Pour

Material	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
				t_4	P
Poly- sulphide	1.6704"	0.0001"	-0.01	3.16	$0.02 < P < 0.05$
Silicone	1.6709"	0.0003"	+0.02	3.07	$0.02 < P < 0.05$
Polyether	1.6700"	0.0002"	-0.03	7.96	$P < 0.01$

B. Second Pour

Material	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
				t_4	P
Poly- sulphide	1.6711"	0.0002"	+0.04	6.39	$P < 0.01$
Silicone	1.6722"	0.0004"	+0.10	17.33	$P < 0.01$
Polyether	1.6695"	0.0004"	-0.06	5.20	$P < 0.01$

C. Casting Delayed 24 hrs.

Material	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
				t_4	P
Poly- sulphide	1.6712"	0.0002"	+0.04	9.90	$P < 0.01$
Silicone	1.6741"	0.0004"	+0.22	18.76	$P < 0.01$
Polyether	1.6696"	0.0006"	-0.05	3.42	$0.01 < P < 0.02$

Experiment No. 9

During all the previous experiments in this project it was assumed that the setting expansion of the die material used, had little or no influence on the dimensional change of the die, relative to the master model. This final experiment was conducted to determine what effect, if any, the setting expansion of the die material may have on the dimensions of a die from a rubber base impression.

The method was identical with that of Experiment 6, with the exception that a Class I stone, with a setting expansion of 0.35%, was used to construct the die. Only polysulphide impression material was used in this experiment.

The results, Table 11, show a definite influence being exerted by the expansion of the stone, when compared with the results for polysulphide in Experiment 6. In this case a highly statistically significantly oversized die resulted, presumably due to the effect of expansion of the die material used.

Table 11 - Comparison of Die to Master Model using High Expansion Class I Stone* (Dimension A-B Only)

Material	Mean Dimension of Model	Standard Deviation	% Dimensional Change	Significance	
				t_4	P
Polysulphide	1.6740"	0.0004"	+0.21	19.88	P < 0.01

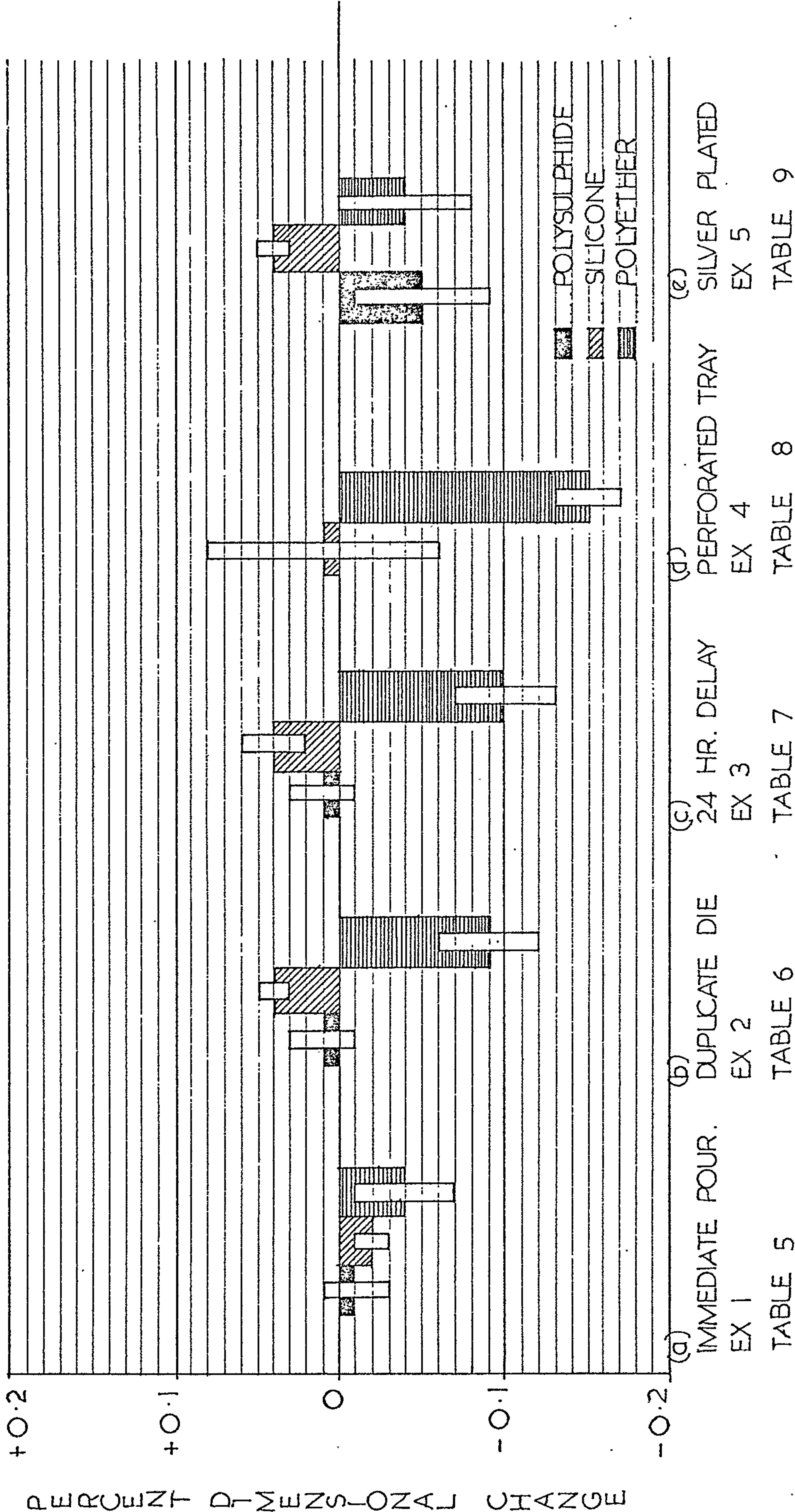
* 5 mm Thickness of Impression Material.

DISCUSSION AND CONCLUSIONS

In discussing the findings of PART II of this project, it was decided to confine comment on the results of linear dimensional change to dimension A-B only. In those experiments where full arch trays were used (Tables 5, 6, 7 and 8) it can be seen that under the same experimental conditions, the percent dimensional change of A-B and B-C is very similar in all cases. This is to be expected since B-C simply represents a relatively longer span than A-B. For clarity, the results (related to dimension A-B) of all experiments are depicted in Figs. 9 and 10. These histograms show at a glance the relative change in dimension of the dies from different impression materials, under the conditions of each experiment.

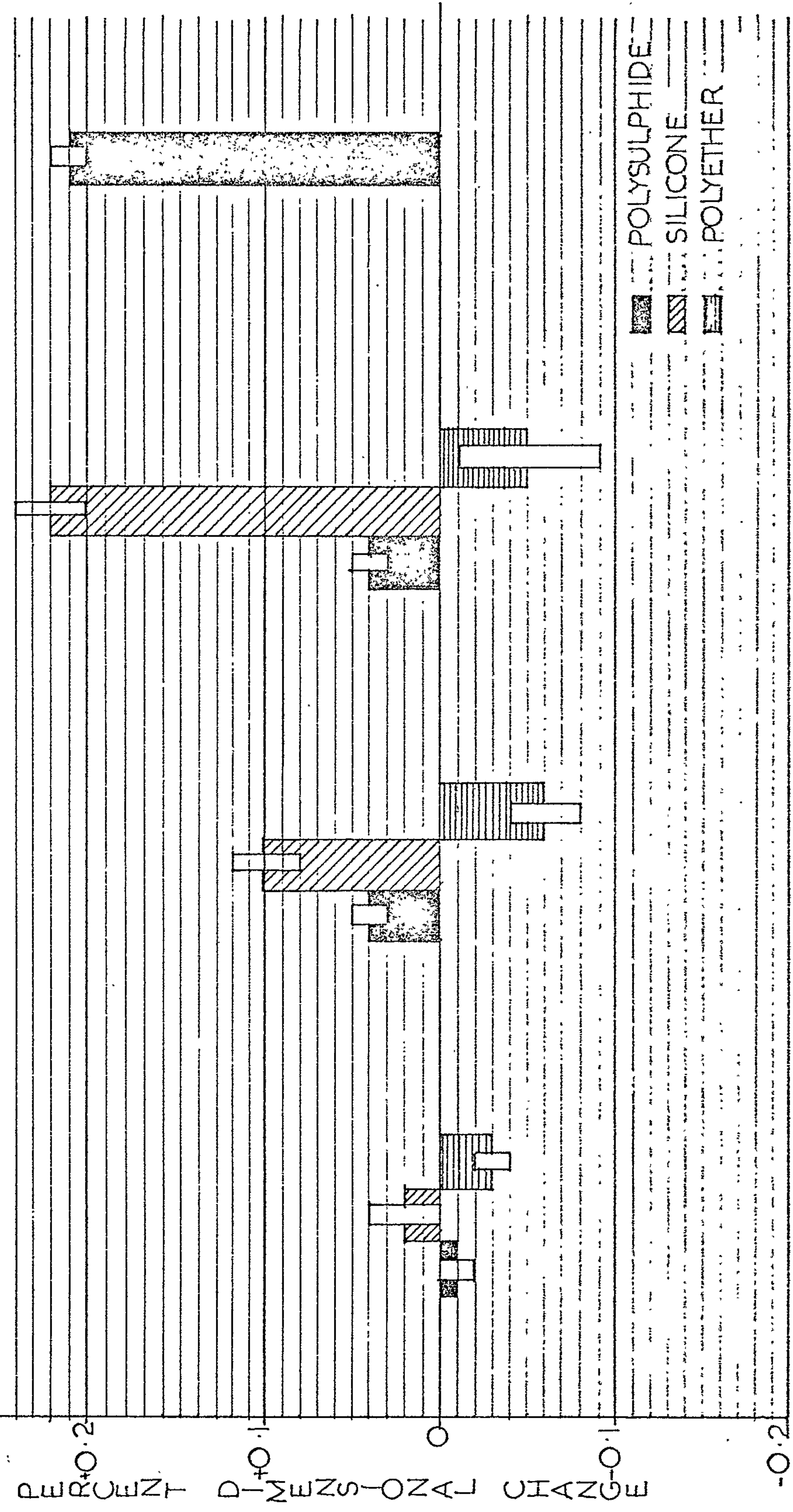
It has also been decided to exclude dimension C-A from detailed discussion, beyond drawing attention to the fact that, in virtually all cases, the percent dimensional change of C-A gives a negative result. This indicates the existence of some factor which does not affect the other dimensions measured. It is suggested that, because of the arched configuration of the trays, this additional factor may be associated with a flexing across the heels of the impression due to thermal dimensional changes involved under the conditions of the experiment. The results of this investigation suggest that this aspect of dimensional accuracy of impressions could well

Fig. 9 - The percent dimensional change in dies from rubber base impression material - using 2 mm thickness of material (Dimension A-B).



Note - One standard deviation, plus and minus, is depicted in the centre of each column.

Fig. 10 - The percent dimensional change in dies from rubber base
impression material - using 5 mm thickness of material.
(Dimension A-B)



Note - One standard deviation, plus and minus, is depicted in the centre of each column.

be further investigated, particularly as related to full, and partial denture construction.

The results of PART II of this investigation will be discussed in the order indicated in The Scope of the Investigation, page 88.

It has been shown in PART I that all impression materials tested contract during setting, and continue to contract after they are clinically set. The relative magnitude of this contraction for the different types of material has also been illustrated. It is generally accepted that when these materials are firmly bound by adhesive to the walls of the impression tray, then the direction of the contraction will be back towards the walls of the tray. This should result in the production of a die of greater dimensions than the master model. Clinically, because of the complex configuration of dental impressions, resultant dies are not oversize in all dimensions. However, with a relatively uncomplicated master model such as that used in this investigation, oversized dies would be expected, particularly in the horizontal plane in which measurements have been taken.

From Fig. 9 part (a) it can be seen that dies from all materials were in fact slightly undersized when 2 mm thickness of impression material was used, and dies cast immediately. This may be explained by an assumption that there are two different directional components of the contraction of these materials, under these conditions. One component pulling the

material back towards the walls of the tray, and the other pulling the material in towards the preparation. When dies are poured immediately, and the magnitude of the contraction of the impression material is not great, the resultant contraction would seem to be inwards. With the passage of time the percent contraction of the impression material increases, altering the direction of the resultant contraction back towards the walls of the tray, producing a larger impression cavity. This is well illustrated for polysulphide and silicone in Fig. 9 part (c), where die construction was delayed 24 hours. The acknowledged greater percent contraction of silicone has caused a relatively greater dimensional change than has occurred with polysulphide material.

The behaviour of the polyether material, Fig. 9 part (a) and Fig. 9 part (c), is not consistent with this hypothesis. From a knowledge of its unrestricted dimensional change it would appear reasonable to expect polyether to behave in a similar manner to polysulphide. This similarity has not been demonstrated. The behaviour of polyether material in all experiments is consistent with the assumption that the adhesive used to bind the material to the impression tray is not entirely effective. The impression material is in effect, contracting much more as it would in an unrestricted state, and so is contracting inwards and producing undersized dies. This assumption is supported by the ease with which polyether material can be stripped from the impression tray after use.

The actual magnitude of the contraction of rubber base materials can be seen to be considerably inhibited by adhesion to the impression tray. For example, Fig. 9 part (c) shows the contraction of a silicone impression which has been left on the bench for 24 hours before construction of a die. Measurement of this die indicates a contraction of 0.04% in the impression. When this is compared with a 24 hour contraction figure of 0.92% for unrestricted specimens of silicone (Fig. 6) it is readily seen how effective this inhibiting effect is.

Clinically, if fractionally oversized dies are required, it is suggested that when using polysulphide or silicone, the pouring of the die should be delayed $\frac{1}{2}$ -1 hour. This practice will also assist the development of elastic recovery, after the impression has been deformed by being removed over undercuts (Wilson 1966).

In the case of polyether, it would appear that a more effective adhesive must be found before it will follow the same dimensional change pattern as the other rubber base materials.

The effect of bulk of material on the dimensional change of rubber base impression materials is shown to vary considerably with the time at which the die is constructed, and with the type of impression material used.

By comparison of Fig. 9 part (a) and Fig. 10 part (a), it can be seen that increasing the bulk of material from 2 mm

to 5 mm has very little significant effect, for any of the materials tested, when the die was poured immediately. It is suggested that the production of slightly oversized dies from silicone materials, in this case, is consistent with the hypothesis already advanced concerning the direction of the resultant contraction. In all cases the magnitude of the dimensional change is extremely small and is of no clinical significance.

When die construction is delayed, and a greater bulk of impression material is used, the effect of contraction of the impression material becomes much more evident (Fig. 9 part (c) and Fig. 10 part (c)). The behaviour of polysulphide and silicone under these conditions is predictable from knowledge of their unrestricted dimensional change.

The behaviour of polyether in thick section is confusing. Accepting a lack of adhesion to the walls of the tray, one would expect a greater contraction inwards with this material when an increase in bulk was employed. This has not been demonstrated. However, contraction inwards is evident in all experiments employing this material in thick section.

Clinically, provided that dies are constructed soon after removal of the impression, all types of rubber base materials tested will produce clinically accurate dies from thick impressions. Where very marked undercuts are evident an increase in bulk may be an advantage because of the relative

reduction in percent elastic deformation involved in removing such impressions.

Duplicate dies constructed immediately after removal of the first die, from polysulphide and silicone impressions, have been shown to be slightly less accurate than the first dies constructed (Fig. 9 part (a) and Fig. 9 part (b)). For polysulphide and silicone this tendency is shown to be more definite when a greater bulk of impression material has been used, Fig. 10 part (a) and Fig. 10 part (b). For polyether there is no statistically significant difference demonstrated between the dimensions of the first die and the duplicate die under such conditions.

Since little or no elastic deformation is involved in these experiments, it seems reasonable to suppose that any loss of accuracy, under these conditions, is attributable to the continuing contraction of the impression material with time. The magnitude of the contraction is not considered to be of clinical significance.

Provided that no great elastic deformation is involved in a particular impression it is suggested that the practice of constructing duplicate dies from rubber base impressions is clinically acceptable. Particularly when a relatively thin bulk of material (2 mm) is used.

Generally it has been shown from this investigation that the most accurate dies are obtained from rubber base

impressions when dies are constructed as soon as possible after removal of the impression. Any delay in die construction will allow progressive contraction of the impression material and consequent loss of accuracy in the die.

It is interesting to compare the magnitude of this progressive contraction for the different types of material tested. From Fig. 9 part (a) and Fig. 9 part (c), it can be seen that when polysulphide is used in thin section (2 mm), the effect of continuing contraction is negligible. Dies from impressions of this material have been poured up to 24 hours later without any statistically significant change of dimension. The same material in thick section (5 mm) shows a definite increase in dimensional change over a 24 hour period (Fig. 10 part (a) and Fig. 10 part (c)), but this change is not clinically significant.

Silicone material shows a relatively greater change. In thick section, over a lapse of 24 hours, this change is probably of a magnitude which is clinically significant. Therefore delay in die construction after using silicone impression materials is not recommended.

Progressive increase in dimensional change of polyether material has been shown to occur, but again the direction of this change is complicated in these experiments by lack of adhesion of the material to the impression tray.

The incidence of imperfections on the surface of dies constructed from the polysulphide material used, has been shown to increase when die construction is delayed. These imperfections are due to the collapse of sub-surface voids, thought to be due to air bubbles incorporated in the material at the time of manufacture. Imperfections of this type were not encountered in dies from silicone or polyether material.

All three types of impression material used in this investigation can be readily silver plated, with the consequent production of clinically accurate dies, Fig. 9 (part e). The dimensional change noted in plated dies from silicone and polyether impressions is consistent with the normal thermal and polymerization contraction effective during the period of the plating operation. The plated dies from polysulphide impressions are, unexpectedly, slightly undersize. This may be partly associated with the absence of any effective expansion in the die material, such as might be expected when stone dies are employed.

For the production of dies with a very hard surface and excellent detail reproduction, the silver plating of any of the rubber base materials is highly recommended.

The results of this investigation suggest that the use of perforations rather than contact adhesive, to retain rubber base materials within the impression tray and to restrain the effect of contraction of these materials is contra-indicated. By comparing Fig. 9 part (c) and Fig.

9 part (d), it can be seen that for both silicone and polyether materials the dimensions of dies are less satisfactory when perforated trays were used. In the case of silicone the large standard deviation indicates an unusually wide range of results, when compared with the other experiments conducted. Further research into this aspect of the use of rubber base materials seems to be indicated.

In all experiments in the investigation where Vel Mix stone dies have been used, no allowance has been made for the possible effect of the setting expansion of the stone on the eventual dimensions of the die. (This is because the unrestricted expansion rate of Vel Mix, 0.05% after 1 hour, is considered to be of no clinical significance). It is interesting therefore to note that by employing a Class I stone with a high expansion rate of 0.35% after 2 hours, a very definite influence can be demonstrated, Fig. 10 Part (d). The expansion of the die in this case is seen to be +0.21%, as opposed to -0.01% when Vel Mix stone dies are cast under identical conditions, Fig. 10 part (a).

It is apparent that the impression material is partially effective in restraining the expansion of the stone dies, but if a die material with too great a setting expansion is used the resulting dies may not be clinically acceptable. For the attainment of the highest degree of accuracy, a die material with minimal setting expansion is recommended.

SUMMARY

The effects of a number of variables on the dimensional accuracy of representative polysulphide, silicone and polyether impression materials, employed under simulated clinical conditions, has been investigated.

All the materials tested have been demonstrated to be capable of producing highly accurate stone dies. The polysulphide material has proved the most accurate and stable material.

Delay in construction has been shown to increase the inaccuracy of dies, due to the continuing contraction of the impression material. The greater the bulk of impression material used the more this inaccuracy is accentuated.

Duplicate dies, constructed immediately after removal of the first die, have been shown to be clinically acceptable when no great elastic deformation of the impression is involved.

The adhesive recommended by the manufacturer of the Polyether material used, proved to be ineffective and undersized dies always resulted from impressions of that material.

Impressions taken using perforated trays have proved to be less accurate, or of less predictable accuracy, than those taken in unperforated trays coated with contact adhesive.

All three types of impression materials tested have been readily silver plated to produce clinically accurate metallic dies.

The setting expansion of stone die material has been shown to have a definite effect on the accuracy of dies constructed in rubber base impressions. For optimum accuracy a low expansion die stone is recommended.

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