

# JOURNAL

OF THE

## BRITISH SOCIETY OF SCIENTIFIC GLASSBLOWERS

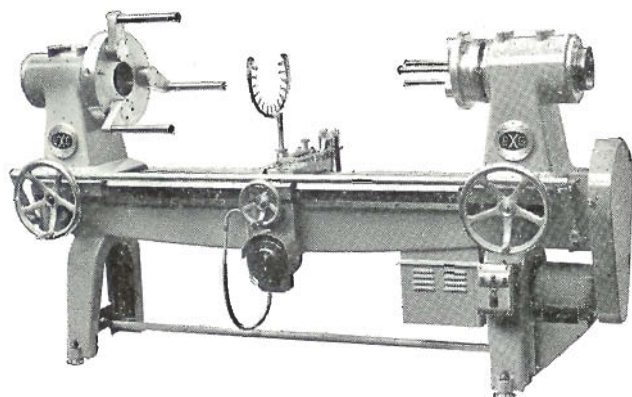
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MARCH, 1966

No. 1

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### CONTENTS

	Page
Editorial . . . . .	1
Vitreous Silica for the Scientific Glassblower . . . . .	2
Abstracts . . . . .	15A
I. C. P. Smith, B.Sc., F.I.C. . . . .	14
Fifth Annual Colloquium . . . . .	15
Air Gauge Method for Exploring Small Tubes . . . . .	16
Section Activities . . . . .	18

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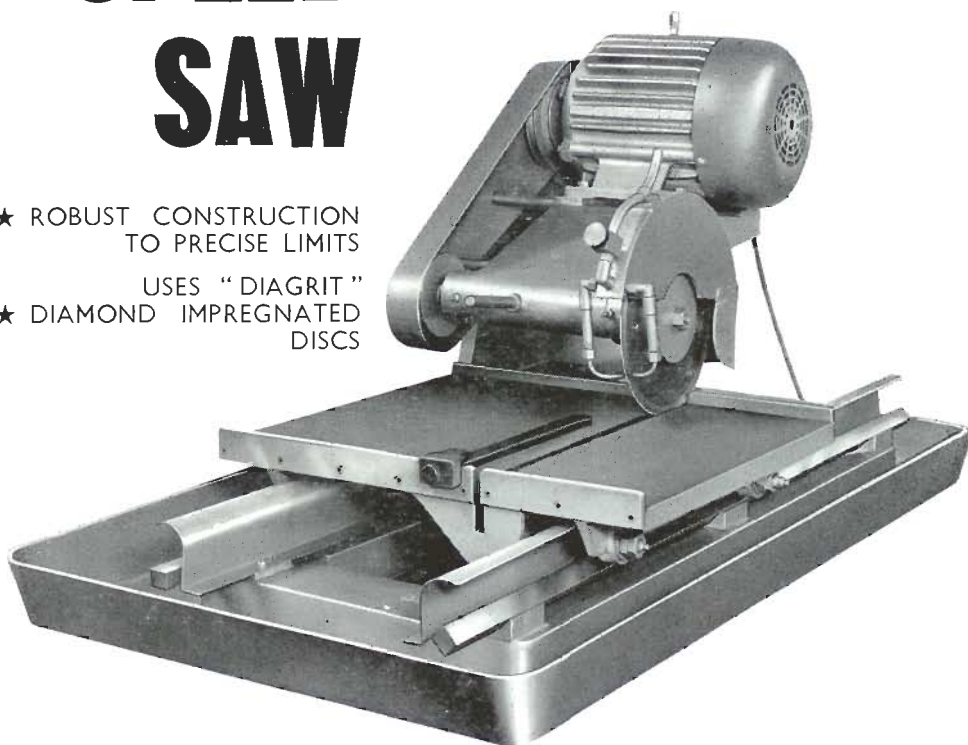
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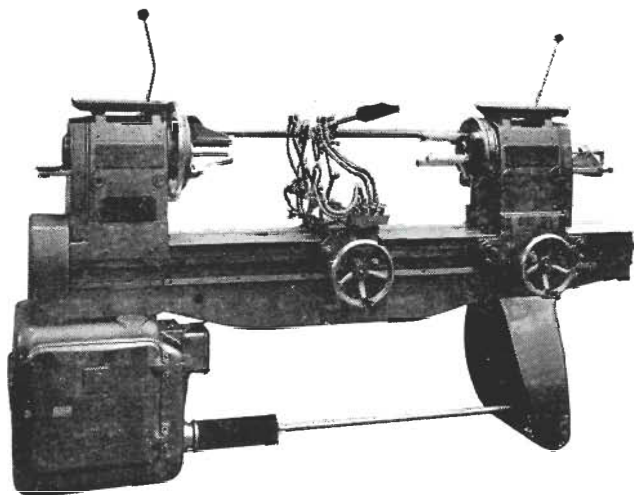
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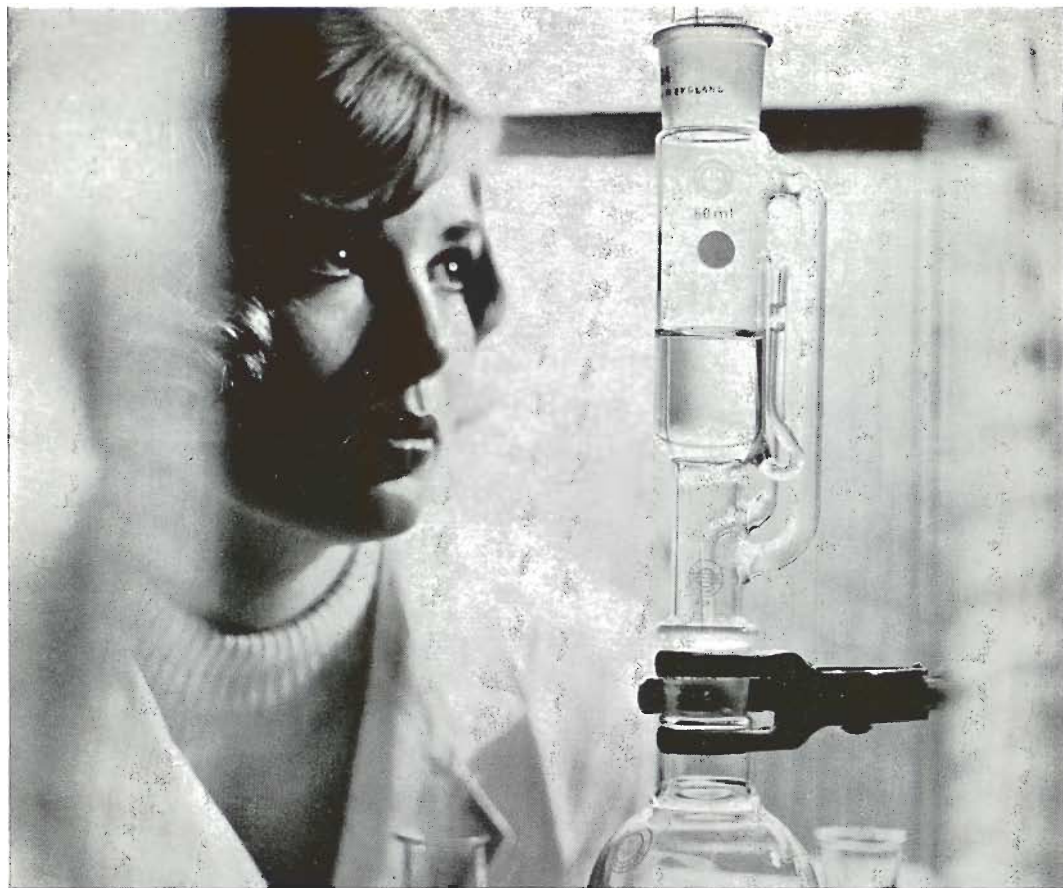
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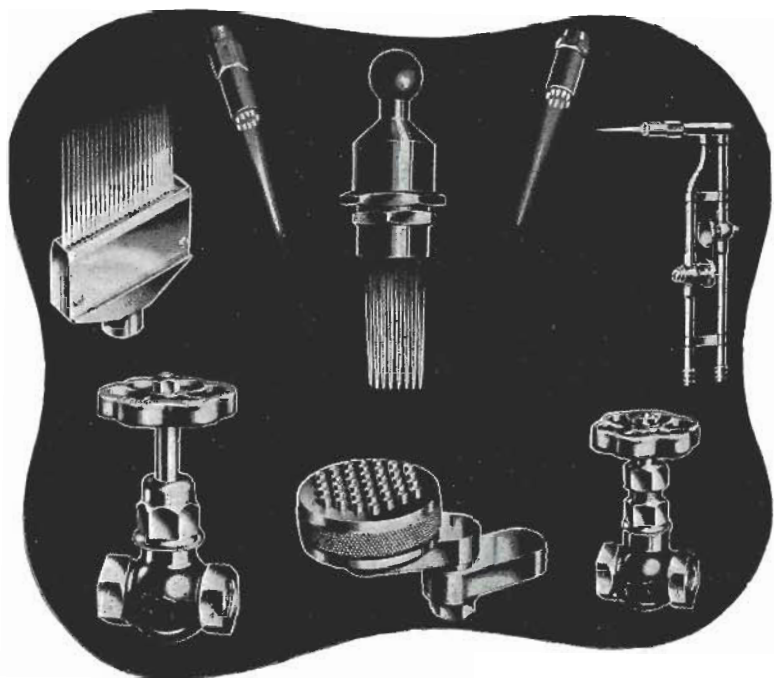
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## EDITORIAL

THE progress of the Examination Board reported below again indicates another step forward towards furthering the objects of the Society and the indications are that the scheme will meet with the same success as other ventures the Society has undertaken.

Some sections are already devoting meetings to discussion and demonstrations of the syllabus with the object of helping their members to qualify.

Together, with other projects such as the publication of this Journal there is no doubt that the Society's promotions fill a need amongst glassblowers and the external support we are now receiving in the form of advertisements for the Journal and increased membership, means that the economics are no longer the main consideration.

Up till now we have flourished under exceptional conditions owing to the work put in by a small group who were keen to see the Society firmly established. This they have done but in the process the work involved in every part of the Society has grown in a way that could not

have been foreseen and the voluntary side has now been stretched to the limit.

There are signs that we are in danger of collapse as one by one these voluntary workers are forced to withdraw and we shall be lucky if their places can be filled in the same way.

Our most recent devastating loss is that of Mr. I. P. C. Smith who has been forced by ill health to give up all his Society activity. We are more than sorry to hear this and wish him a speedy recovery.

Perhaps by the time this editorial is printed a solution to this loss will have been found but it is becoming obvious that the administration of the Society is now too large to be carried out by voluntary effort alone. It is certainly time to examine the work involved in various aspects of the Society's activities and either persuade more people to take part, perhaps on a paid basis, or cut down to a scale which can be more easily managed.

Best of all would be the establishment of a central office with paid services which would give an air of permanence to the Society.

J. H. BURROW

## BOARD OF EXAMINERS MEETING

THE Board of Examiners held a meeting at the College of Advanced Technology, Gosta Green, Birmingham, on the 26th February, 1966, and discussed the following subjects:

### Elementary Scientific Glassblowing Course

The syllabus, examination and form of certificate for the course entitled "Introduction to Elementary Scientific Glassblowing, non professional," as approved by the British Society of

Scientific Glassblowers was discussed and a satisfactory scheme for implementation was considered. A pamphlet was drafted which will be printed and distributed to colleges and instruction centres to introduce the course for general adoption for the Session 1966-67.

The course now being conducted at the Bristol Technical College covers the syllabus as agreed by the B.S.S.G. and the examination for the

*continued on page 20*

# VITREOUS SILICA FOR THE SCIENTIFIC GLASSBLOWER\*

by T. P. BROWELL and G. HETHERINGTON

*Thermal Syndicate Limited, Wallsend, Northumberland*

## The Forms of Silica

**S**ILICA or silicon dioxide,  $\text{SiO}_2$ , is a common substance found in nature, especially as quartz. However, quartz is only one of the modifications of silica which can, in fact, exist in several forms, chemically identical but different structurally. These differences arise from the fact that the silica structure is built-up from tetrahedra with oxygen atoms at each corner equidistant from a silicon atom at the centre, and it is different arrangements of these tetrahedra that give rise to the different structural forms of silica. In the crystalline forms the silica tetrahedra form regular patterns, whereas in the vitreous state they are randomly distributed (this random distribution of one or more inorganic oxides is in fact the main criterion for any glass).

Figure 1 is a two dimensional comparison of the atomic arrangement for (a) an ordered crystalline structure and (b) the disordered, random structure of vitreous or glassy material. Although there is apparently little difference between these arrangements it will be shown later that these two structures accord the materials entirely different properties.

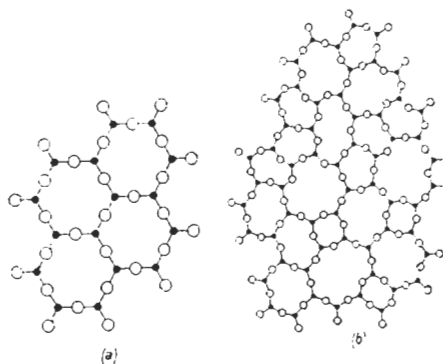


FIGURE 1

*Two-dimensional comparison of (a) an ordered crystalline structure, and (b) the disordered structure of a glass*

Silica occurs naturally in various crystalline forms, the main ones being cristobalite, tridymite and quartz. Quartz is also made synthetically, usually by growth on a seed crystal from a silica-containing solution in an autoclave under high pressure. Natural and synthetic quartz crystals are used as optical polarisers and in crystal oscillators.

It is appropriate at this point to consider the terminology used in describing crystalline and glassy silica. It is important to note that quartz is a crystalline material and that it is incorrect to refer to any form of the glassy material simply as "quartz." In the past vitreous silica has frequently been referred to as "silica glass," "quartz glass," "fused silica," "fused quartz" or simply "quartz." As far back as 1927 Sosman condemned the use of the single word "quartz" and considered that its use had arisen through "carelessness or ignorance and is already causing troublesome confusion." Sosman, who approved the designation "vitreous silica" said "its abbreviated derivation 'Vitreosil'† would have been a useful word to add to the language, had it not been pre-empted by one of the manufacturers as a trade name." The term "vitreous silica" has been adopted now also by the British Standards Institution.‡

The most important form of silica from the point of view of a glassblower is, of course, vitreous silica; therefore the terminology used, and the material itself, will now be considered in more detail.

Opaque or translucent vitreous silica, the form commonly obtained by the fusion of sand and which derives its translucency from the minute gas bubbles disseminated in the structure, is commonly referred to as "fused silica." Transparent vitreous silica, usually made by the fusion of quartz crystal, is referred to as "fused quartz" or sometimes "quartz glass." It is these two terms which have been abbreviated carelessly and wrongly to "quartz," but in any case should only be applied to forms of vitreous silica produced by fusing quartz crystal.

\* Based on part of a symposium on silica, with special reference to items of interest to glassblowers, presented by members of Thermal Syndicate Ltd. to the British Society of Scientific Glassblowers in Bristol, September 1964.

† "Vitreosil" is a registered trade-mark of Thermal Syndicate Ltd. †

‡ British Standards Institution (1962) "Glossary of Terms Used in the Glass Industry," B.S. 3447, 65 pp.

In this article the term "vitreous silica" is used for the glassy or vitreous form of silica generally. The translucent or opaque variety is referred to as "translucent vitreous silica" for convenience, and the transparent variety is similarly referred to as "transparent vitreous silica" or, when made from crystal, as "fused quartz."

Vitreous silica can thus be subdivided into two major groups: *translucent vitreous silica*, which is made by the fusion of quartz sand in a furnace open to the atmosphere, and *transparent vitreous silica*, which, until a few years ago, was always made by the fusion of quartz crystal, but which now can be made also by the hydrolysis or oxidation of a volatile silicon compound and the subsequent fusion of the silica so formed.

These two major groups can be subdivided further as follows:

#### *Translucent Vitreous Silica*

*Sand surface ware* is the quality obtained directly by the electrical fusion of pure silica sand, the unfused sand adhering to the outer surface after fusing and moulding usually being removed by abrasion.

*Glazed ware* is a modification of the sand surface product obtained by remelting the surface, and often reshaping the article, in a flame or electric arc. The material is homogeneous throughout, no fluxes being added.

*Satin tubing or rod* is made by heating externally a fused mass of sand during the drawing operation; the striated bubbles giving the tubing or rod its characteristic sheen.

*Transparent vitreous silica*: It is not intended here to give a detailed discussion on the methods of making transparent vitreous silicas but four fundamentally different types can be recognised with differences based principally on methods of fusing and on "water" and metal impurity concentrations:

*Type I* transparent vitreous silica is usually obtained by electric melting quartz crystal powder in vacuum or in an inert gas at low pressure. It contains negligible "water" but about the same metallic impurities as the unfused raw material (e.g. I. R. Vitreosil).

One type of fused quartz tubing is made by a continuous process, the quartz crystal being melted in a refractory metal pot at atmospheric pressure and the tubing drawn from the bottom

of the pot through a die (Hänlein process). This tubing suffers the disadvantage of long air-lines and the "water" content is somewhat higher than other type I materials, resulting from the protective atmosphere enveloping the furnace.

*Type II* is prepared by fusion of quartz crystal in a flame, and like all vitreous silicas prepared in a water vapour atmosphere it contains varying concentrations of hydroxyl (usually 0.015-0.04 wt. %OH) depending on the fusing conditions and the size of the quartz crystal particles used. Some impurities (e.g. aluminium) may be partially volatilised in the flame giving, generally, lower maximum impurity levels for this type (e.g. Vitreosil 066).

*Type III* "synthetic" vitreous silica is made by the vapour-phase hydrolysis of a pure silicon compound such as silicon tetrachloride and simultaneous fusing of the silica formed in, for example, an oxy-hydrogen flame. It has the highest hydroxyl content (about 0.1 wt. % OH) but is virtually free from metallic impurities (e.g. total metallic contamination in Spectrosil\* is <0.2 p.p.m.). When made from silicon tetrachloride the material contains about 50 p.p.m. chlorine.

*Type IV* is also a "synthetic" vitreous silica but, because it is made by oxidising a silicon compound such as silicon tetrachloride and fusing the silica electrically or in a "water-free" flame, it is free from hydroxyl as well as from metallic impurities. However, in the absence of hydroxyl the chlorine content is increased and may be several hundreds of parts per million. Until recently it has been available only in limited amounts and in small pieces (e.g. Spectrosil\* W. F.).

The situation is further confused because the same commercial name or trade-mark often covers several types (e.g. Vitreosil applies to translucent vitreous silica and Type I and Type II transparent vitreous silicas). Moreover, variations within a type are also possible because special treatments subsequent to manufacture are employed to endow particular properties to a material.

The principle varieties of vitreous silica with their origins are summarised in Table 1, page 4.

It is appropriate at this point to discuss some of the difficulties in manufacturing vitreous silica. Conventional glasses can be melted to fluids with

\* "Spectrosil" is a registered trade-mark of Thermal Syndicate Ltd.

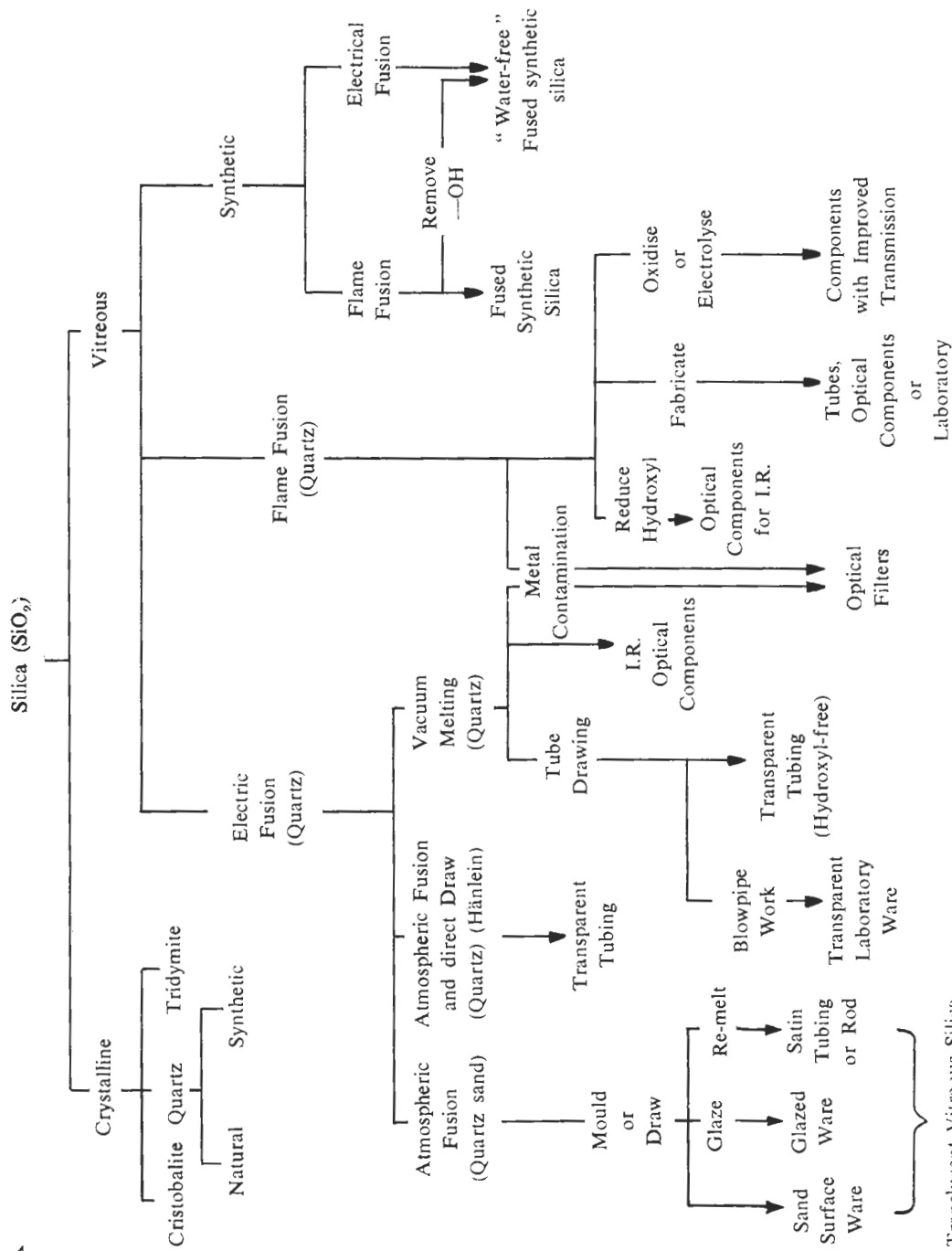


TABLE I Forms of Crystalline and Vitreous Silica

viscosities of about  $10^3$  poises (about the consistency of treacle) and can therefore be poured into moulds—with silica only viscosities of about  $10^6$  poises (something like plasticene) can be achieved at temperatures of the order of  $1,800^\circ$ – $1,900^\circ\text{C}$  because of its tendency to sublime rather than melt. The electrical melting of quartz under vacuum produces transparent material substantially free from bubbles. This is by no means as simple as this brief statement might imply—imagine trying to suck air out of plasticene.

The flame fusion method has some advantages over electrical methods where thick sections are being made in that, since the vitreous material is deposited in thin layers, bubbles are less likely to be trapped in the material and, secondly, it is easier to fuse the material completely to obtain a random structure and hence a material which appears more “glassy.” However the “water” included in the silica structure is linked to the silicon-atoms as Si-OH groups, breaking up the Si-O-Si chains which connect the silicon-oxygen tetrahedra. This has a noticeable effect on such physical properties as the infra-red transmission, viscosity and density. Subsequent flame-working of electrically fused material also introduces “water” to give Si-OH linkages.

It has already been indicated that heating vitreous silica in a water-vapour atmosphere (e.g. a flame) introduces hydroxyl. Conversely, it is possible to reduce the material's hydroxyl content by subjecting the vitreous silica to an appropriate heat treatment in a dry, hydrogen-free atmosphere. The importance of this for infra-red work will be indicated later.

During or subsequent to the fusion process, small amounts of metals (e.g. tin, manganese, titanium) or their oxides can be used to contaminate the vitreous silica and so produce materials which, although behaving as vitreous silica in their mechanical and thermal properties, have modified optical transmission properties which are sometimes useful. One obvious example is that of producing a vitreous silica envelope for mercury vapour lamps which filters out the ozone-producing radiation.

It is now obvious that instead of simply “vitreous silica” there are a number of materials, all essentially vitreous silica, but varying in the randomness of their structure (from fused quartz which, because of the similarity in the structures of the crystalline and vitreous phases and the high viscosity of the melt, must contain some residual crystal structure, to the completely

random synthetic vitreous silicas which have no crystal origin), in their freedom from physical defects such as bubbles, in their chemical purity and in particular, in their hydroxyl contents.

With this in mind it is now possible to examine the effect these differences have on the properties of vitreous silica of most interest to glassblowers.

### Purity

With the increased use of vitreous silica in the manufacture of semi-conductor materials and in radio-chemistry much more attention has been given to the relative purity of different grades of the material and comparisons have been made between the products of one manufacturer with those of another without any real scientific foundation. Almost all commercial grades of vitreous silica are “pure” in the sense that even for the translucent material the total maximum metallic impurity levels are only about 1,000 p.p.m. (0.1 wt. %) and in the transparent grades perhaps 150 p.p.m. (0.015 wt. %). Thus, particularly in the case of transparent vitreous silica, analysis is difficult, expensive and not always sufficiently accurate for someone only interested in a particular impurity. Another important factor to be borne in mind is variability in the impurity levels. Most manufacturers of transparent vitreous silica obtain their quartz crystal from Brazil but there is still a considerable variation in the purity of their raw material. This crystal is graded to some extent at the mines and the manufacturers, themselves, examine the material for surface contamination, “twinning” and “ghosting” (optical defects which can be co-related to some extent with chemical purity), and for inclusions in the crystal. In spite of such examinations, which cannot of course detect impurities present as substitutional ions and interstitial ions in the silica lattice but only macro-defects, there may still be a large variation in the amount present of a particular impurity not only from one batch of crystal to the next, but even within one batch of crystal and conceivably within a single length of tubing made from crystal. It must be emphasised that these variations mentioned are referred to very low impurity levels !

Thus it is difficult for a manufacturer to give a guaranteed or even a typical analysis for any one grade of vitreous silica made from quartz minerals.

One is of course able to give the maximum values of an impurity so far determined, but



since generally these values are much higher than in the majority of specimens, they should not be compared with "typical" values or those resulting from a single analysis.

Figure 2 is an "idealised" curve showing the variation in the occurrence of a particular impurity: although usually insufficient results are available for smooth curves to be drawn, because of the cost of accurate analyses at these levels.

One can only emphasise that it is impossible to compare the purities of the various grades of fused quartz from different manufacturers by analysing only one sample from each grade.

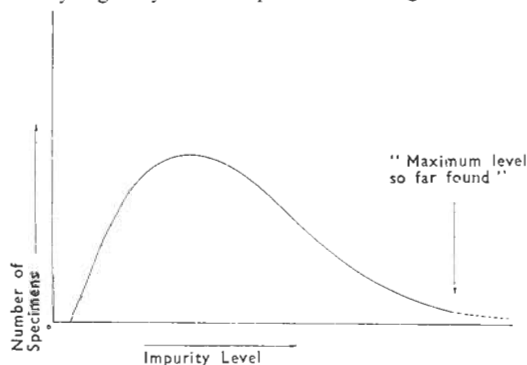


FIGURE 2

*Idealised curve relating impurity content with number of specimens in which this impurity level occurs*

The above remarks apply to material made by fusing quartz crystal. They do not apply to synthetic vitreous silicas where the impurity levels are much lower and are not subject to the same raw material variations as in crystal, being made from a readily purifiable, volatile silicon compound. The impurity levels present in these materials can therefore be given more confidently although it can be seen from Table 2 that, because only very small amounts of impurity are present, they are usually beyond the levels of detection of present routine analytical methods. The methods used for the analyses of synthetic material given in Table 2 are largely either colorimetric or neutron activation methods developed by Dr. C. A. Parker of the Admiralty Materials Laboratory, Holton Heath, Poole, Dorset, who also carried out the original analysis.

Metallic impurities present in vitreous silica are, with the exception of alkali and alkaline earth cations, usually not present as ions but are incorporated in the silica network, generally occupying a position normally occupied by a silicon atom. These impurity atoms are therefore very firmly bound and are not released in high temperature work unless the vitreous silica is dissolved or volatilised. Thus in semi-conductor work, where small impurity levels are obviously important, the presence of impurities in tubes and apparatus used for diffusion work is by no means as critical as the same level of impurity present in a crucible part of whose wall will dissolve in molten silicon during a crystal pulling operation.

TABLE 2  
Chemical Purities of Vitreous Silica  
(Maximum levels so far determined in p.p.m.)

Element	Translucent Vitreous Silica	Transparent Vitreous Silica*		
		Type I	Type II	Type III†
Aluminium . . . .	380	70	60	<0.02
Antimony . . . .	ND	0.3	0.1	<0.0001
Boron . . . . .	7	3	3	<0.01
Calcium . . . . .	10	0.5	0.4	<0.1
Copper . . . . .	ND	1	1	0.002
Iron . . . . .	70	2	1.5	<0.1
Manganese . . . .	ND	0.03	0.02	<0.001
Phosphorous . . .	ND	0.01	0.005	<0.001
Sodium . . . . .	60	4	1	0.04

ND = Not determined.

\* The types referred to are those described in the text.

† Analyses made on ingot material.

(The metallic impurities in Type IV vitreous silica are similar to those in Type III material)



The impurities so far referred to stem largely from the original raw material employed. Manufacturing processes and fabrication are also sources of contamination, and whilst manufacturers take all possible precautions in this respect it is worthwhile mentioning contamination which can occur in the handling of these materials. Copper is a common contaminant since it occurs in the nozzles of normal blowpipes and in many bonded diamond cutting wheels. Moreover, it has a high "diffusion coefficient," which means that if introduced on to a surface it will rapidly diffuse into the material. Thus, whilst surface contamination from blowpipes and cutting wheels might be expected to be removed subsequently by etching the surface with hydrofluoric acid, it may not always be possible to remove contamination such as copper in this way.

It is probably unnecessary to add that vitreous silica should be scrupulously clean before being subjected to high temperatures such as blowpipe flames which will cause reaction with metallic impurities occurring in dust, fingerprints or tap water.

It is not usually possible to judge the chemical purity of vitreous silica by a visual examination, except for obvious inclusion spots which may

have characteristic colours (e.g. iron—black; titanium—blue; copper—red). Some information can however be obtained by observing the colour of the fluorescence emitted when the article is irradiated by ultra-violet light (e.g. from a low pressure mercury vapour lamp). Under these conditions a green colour generally indicates copper although this may be masked sometimes by the blue-violet fluorescence normally associated with fused crystal quartz and caused by metals, such as aluminium, in a reduced state. It is difficult to estimate, even semi-quantitatively, the level of contamination from a visual examination of the emitted fluorescence, as the sensitivity of the human eye varies markedly with the colour observed.

### Optical Properties

Typical ultra-violet and infra-red transmission curves for the different types of transparent vitreous silica are given in Figure 3.

The ultra-violet transmission of vitreous silica is affected by the metallic impurities present in the material. Synthetic vitreous silicas (e.g. Spectrosil) do not exhibit any absorption bands in the ultra-violet on account of their purity and the "cut-off" at about  $160\text{ m}\mu$  is believed to be

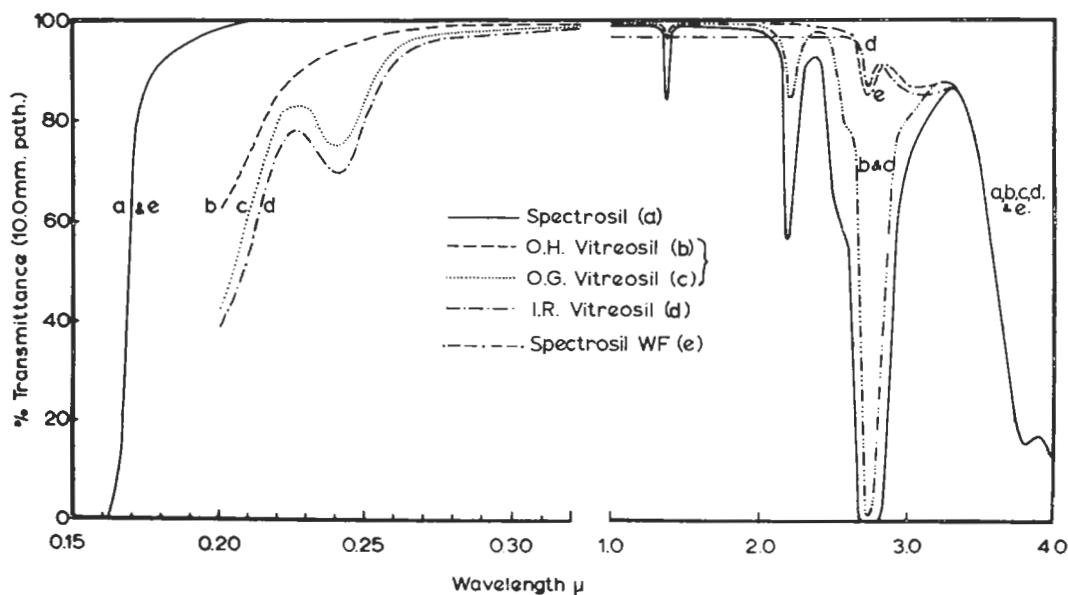


FIGURE 3

*Ultra-violet and infra-red transmittances of vitreous silica (Thermal Syndicate Ltd.)*

the limiting point of transmission for vitreous silica and is not a function of impurities present. The relatively sharp cut-off at this wave length is partly ascribed to the fact that the refractive index of the material increases rapidly in the ultra-violet with a consequent decrease in transmission due to increased surface reflection. Types I and II vitreous silicas exhibit an absorption band at  $240\text{ m}\mu$  due to impurities such as aluminium in association with alkali ions, or to germanium, in a reduced state. The intensity of this absorption band for a given impurity level depends on the "thermal history" of the material and it can be eliminated by an oxidising heat treatment or by migration of ions under high temperature electrolysis. The  $240\text{ m}\mu$  absorption is accompanied by a blue-violet emission and this, of course, is the phenomenon known as fluorescence. It has already been mentioned that other impurities produce characteristic fluorescence emissions, copper giving a green fluorescence.

Certain impurity centres are also responsible for the darkening which occurs under nuclear irradiation. There are two principle defects which, on irradiation, cause a reduction in the ultra-violet transmission at about  $215\text{ m}\mu$  and cause a reduction in the visible transmission at about  $550\text{ m}\mu$ . This latter absorption, which gives the material a purple-brown colouration, is due to aluminium associated with alkali in the silica lattice.

The dose of radiation necessary to produce these effects is still large compared with the dose required to produce similar effects in conventional glasses (except the ceria-stabilised ones). Ordinary glasses may darken under about  $10^4$  Roentgens of gamma or X-radiation and types I and II vitreous silica under about  $10^6$  Roentgens. Synthetic fused silicas (e.g. Spectrosil) have been subjected to doses greater than  $10^{10}$  Roentgens without visible darkening, but with some loss of ultra-violet transmission.

In the infra-red part of the spectrum metallic impurities have almost no effect on transmission and the important factor is the presence or otherwise of hydroxyl in the silica lattice. The transmission curve given in Figure 3 for I.R. Vitreosil is the effective transmission in the infra-red for vitreous silica free from "water." Spectrosil, on the other hand, exhibits not only complete absorption (for a path length of 1 mm. or greater) at  $2.73\mu$ , which is the fundamental

resonant frequency for the Si-OH band, but also associated absorption bands at  $2.22\mu$  and  $1.38\mu$ , which correspond to combination and overtone frequencies of the fundamental. Varying amounts of absorption at these wavelengths are possible between these two limits (I.R. Vitreosil and Spectrosil) depending on the amount of hydroxyl present in the vitreous silica.

It is convenient at this stage to mention another feature of synthetic fused silicas, in addition to their ultra-violet transmissions, which make them suitable for optical work. Fused quartz crystal generally has a granular structure due to refractive index changes at the surface of the original crystal grains, where "melting" has not been quite complete. A convenient method of examining optical material for "granularity" is to use a very small, bright source of light (as can be obtained by focusing a light on to a pin-hole in a sheet of metal) to project an image of the specimen on to a screen. Figure 4 shows such "pin-hole" photographs (shadowgraphs) of different varieties of vitreous silica. A typical synthetic fused silica, Spectrosil, exhibits none of the granular structure exhibited by the materials made from quartz crystal.

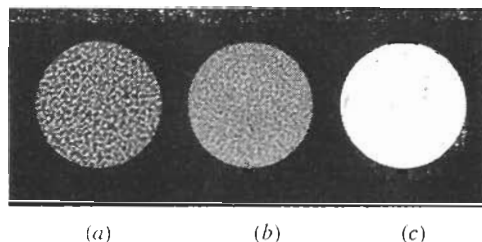


FIGURE 4

*Pinhole photographs of optical vitreous silicas, showing granularity*

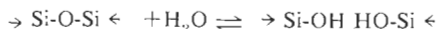
- (a) OG Vitreosil (type II)
- (b) OH Vitreosil (modified type II)
- (c) Spectrosil (type III)

#### "Water" in Vitreous Silica

The effect of the hydroxyl content on the infra-red transmission of vitreous silica is referred to under the heading "Optical properties." Many other physical properties are affected by the hydroxyl content (e.g. density, viscosity, thermal expansion) and this is referred to later but, at

this stage, a few words of explanation on the role of "water" in vitreous silica may be useful.

The reaction of water with silica is a reversible equilibrium process and may be represented by the equation:



indicating that the hydroxyl content of vitreous silica is not a fixed amount but can vary continuously (although usually slowly) with changing conditions. Given time to establish equilibrium, the hydroxyl content depends on the amount of water in the atmosphere surrounding the piece of vitreous silica and is, in fact, proportional to the square root of the partial pressure of water vapour in the atmosphere at a particular temperature. The reversibility of the process is demonstrated in Figure 5 which shows

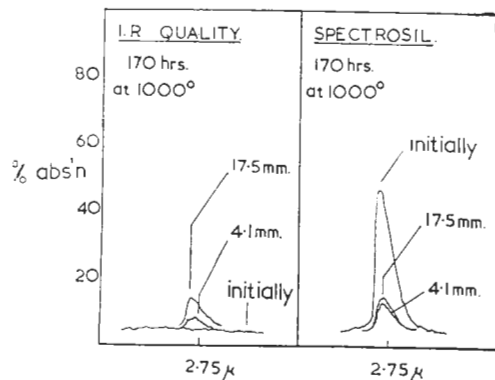


FIGURE 5

*Infra-red absorptions of vitreous silicas after heat-treatment in water vapour atmospheres*

how Spectrosil, with a high hydroxyl content, and I.R. Vitreosil, with a very low hydroxyl content, reach the same final hydroxyl concentrations, as measured by the  $2.7\mu$  absorption peak, when heated in the same water vapour atmosphere. (Specimens 0.2 mm. thick heated for 170 hours at  $1,000^\circ\text{C}$  under water vapour pressures of 4.1 mm. and 17.5 mm. respectively.)

From the glassblower's point of view, no vitreous silica can be regarded as completely "water-free" if it has had any blowpipe treatment since blowpipe flames contain large amounts of water vapour, as a product of combustion. In this respect oxy-hydrogen flames introduce

hydroxyl more quickly into vitreous silica than do oxy-propane flames. Figure 6 shows some comparative rates of entry of hydroxyl into vitreous silica from different blowpipe flames (the hydroxyl content being represented as the optical density per mm. at  $2.73\mu$ ,  $d/t$   $2.73\mu$ ).

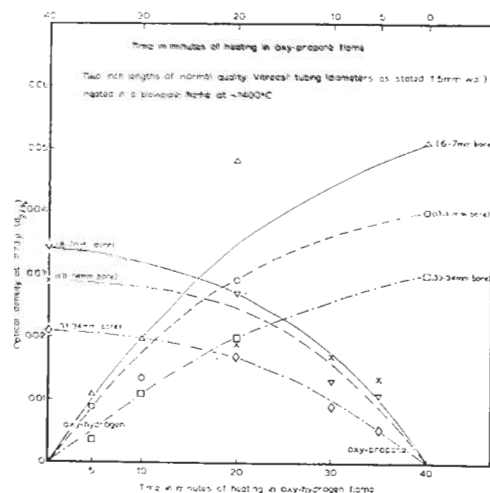


FIGURE 6

*A comparison of the effects of oxy-hydrogen and oxy-propane flames on the hydroxyl content (represented by the optical density at  $2.73\mu$ ) of transparent Vitreosil tubing (type 1)*

Although it is seen from the above discussion that it is impossible to give precise figures for the actual hydroxyl content of a particular grade of material, and batch-to-batch or sample variation must be allowed for, some typical figures are given in Table 3.

TABLE 3

### Hydroxyl Contents of the Various Forms of Vitreous Silica

	Wt. % OH
I.R. Vitreosil (Type I)	0.0003
Blowpipe worked fused quartz tubing (modified Type I)	0.006
O.G. Vitreosil and Vitreosil 066 (Type II)	0.04
Spectrosil (Type III)	0.12
Spectrosil W.F. (Type IV)	0.0005

## Structure and Viscosity of Vitreous Silica

Vitreous silica has a relatively high viscosity compared with other glassy materials at the same temperature and this influences some of its other properties.

When a liquid such as a metal cools a temperature is reached at which crystallisation occurs rapidly, the temperature remaining constant during solidification. When liquid glassy materials cool there is no fixed temperature at which crystallisation occurs rapidly; instead they pass through a temperature where crystallisation is possible. However, at this temperature the viscosity of the "liquid glass" is much higher than that for ordinary liquids (e.g. metals) and this corresponds to a much lower mobility of the atoms in the glass structure. As the temperature falls the atoms become progressively less mobile resulting in a decreased ability for them to crystallise into a regular pattern. This process is summarised in Figure 7.

Thus there is a temperature at which a glass will crystallise (devitrify) most rapidly but this process is extremely slow compared with the crystallisation of metals and other materials. Again, because vitreous silica has a viscosity much higher than other glasses its tendency to crystallise is less and it is a stable material at high temperatures. The curve in Figure 7 assumes that the cooling rate has been sufficiently slow for the glass to follow this curve. Usually a glass may be cooled sufficiently slowly to follow the first part of this curve but, as there is a rapid decrease in mobility of the atoms on cooling, the glass has a "frozen in" structure corresponding to the point where it could no longer follow this curve. Thus a glass has a different degree of "order" corresponding to each temperature on the curve (Figure 7), that is a different "frozen

in" structure. The temperature corresponding to this "frozen in" structure is known as the "fictive" temperature.

At first sight this may appear confusing but, from a practical point of view, it is important to realise that the structure and hence many of the

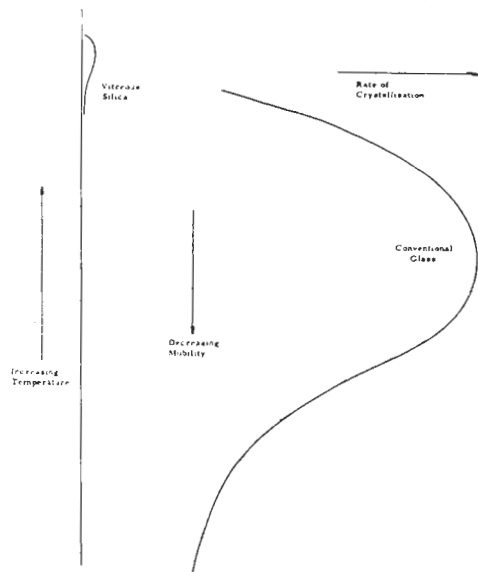


FIGURE 7

Diagrammatic representation of the rates of crystal growth in vitreous silica and a conventional soda-lime-silicate glass versus temperature

physical properties of glasses, including vitreous silica, depends on the previous thermal history. It is worth noting, for example, that for a "fictive" temperature change of 400°C the viscosity of I.R. Vitreosil at 1,100°C changes by

*continued on page 11*

TABLE 4

### Softening, Annealing and Strain Points of Vitreous Silicas and Borosilicate Glass

	Type I I.R. Vitreosil (°C)	Type II O.G. Vitreosil (°C)	Type III Spectrosil (°C)	Borosilicate glass (°C)
Softening point ( $10^{7.6}$ poises) . . .	1,583	1,596	1,594	790
Annealing point ( $10^{13.0}$ poises) . . .	1,190	1,108	1,082	560
Strain point ( $10^{14.5}$ poises) . . .	1,108	1,015	987	520

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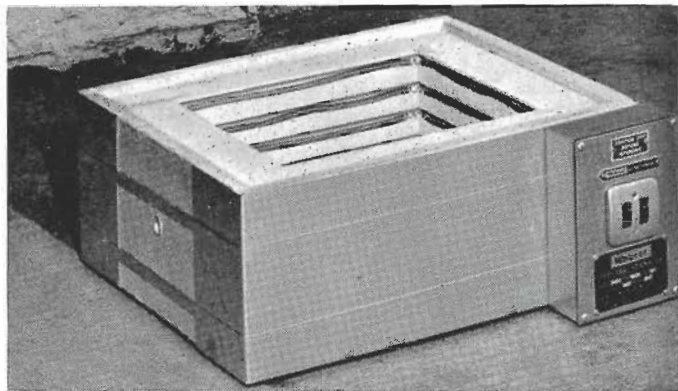
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## ABSTRACTS

### BRAZING

(223) **Brazing in Hydrogen Atmosphere in Laboratories.**

Karkera, B. A., *Fusion*, Vol. 12, No. 3, 1965, 9-11. Short account of a laboratory rig in which the brazing is carried out, sketch of unit. D.W.S.

### COLORIMETERS

(224) **An Isothermal Microcalorimeter Operated in a Water Triple Point Cell and Designed for Radioactivity Measurements.**

Lowenthal, G. C., and Hicks, R. M., *J. Sci. Instrum.*, Vol. 43, No. 1, 1966, 36-8.

By means of a triple point cell the isothermal microcalorimeter has a temperature constant envelope  $\pm 0.0001^\circ\text{C}$  is claimed. Temperature change in the calorimeter is measured by a thermistor, the smallest detectable change is  $0.00012^\circ\text{C}$ . Details of the apparatus are given. D.W.S.

### CHROMATOGRAPHY

(225) **A Glass Centrifugal Machine for Chromatographic Investigation.**

Vertebnyi, P. Ya., *Industrial Laboratory*, Vol. 30, No. 12, translation published May 1965, 1895.

The device consists of a glass chamber, a removable metal holder and motor with autotransformer control. Can be run at several hundred r.p.m. D.A.H. See also 229.

### EVAPORATORS

(226) **A Laboratory Evaporator for Corrosion Tests.**

Babkina, V. Ya., Chub, E. G., Gapunina, O. U., and Skudar, I. K., *Industrial Laboratory*, Vol. 30, No. 10, translation published March 1965, 1586. Schematic diagram of the apparatus. D.A.H.

### GAS-ANALYSIS

(227) **Use of the Chizhevskii Method for Determining Nitrogen in Slags.**

Chuchmarev, S. K., and Kamyshov, V. M., *Industrial Laboratory*, Vol. 30, No. 9, translation published March 1965, 1317.

Nitrogen measured at low pressures, calibrated volume divided into three parts. Complete diagram of apparatus. D.A.H.

### GLASSWORKING—MACHINES

(228) **A Machine for Winding Quartz Springs.**

Bryushkov, B. S., Bychkov, V. P., and Mamonov, A. F., *Industrial Laboratory U.S.S.R.*, Vol. 30, No. 11, translation published April 1965, 1769.

Automatically produces springs and provides for automatic compensation of consumption of glass. Schematic diagram. D.A.H. See also 229.

### GLASSWORKING—METHODS

(229) **Cutting Chromatography Troughs.**

Porter, F. G., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 3, 1965, 43.

Describes the cutting of chromatography troughs by means of slitting tube with a diamond wheel in a milling machine. D.W.S.

(230) **A Method of Making Glass Micro Leaks.**

Fisher, I. P., and Herrick, D. A. R., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 4, 1965, 49-50.

Describes the technique and the equipment used to produce micro leaks (30 and 90 micron diameter). D.W.S.

(231) **Small Diameter Pyrex Sinters (for Mercury Sealed Gas Cut-Offs).**

Maple, T. J., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 4, 1965, 54.

The Pyrex sinter 1½-2 mm. diameter is placed in a sleeve of B37 glass, this being sealed in turn to a pyrex capillary tube. Vacuum is applied and the B37 sleeve is shrunk on to the pyrex sinter in a small furnace. D.W.S.

(232) **Hot Plate Glassworking.**

Pike, K. G., and Huckfield, J., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 4, 1965, 55-6.

Suggests that a hot plate can be a very useful tool. D.W.S.

### GLASSWORKING—TOOLS

(233) **Fast Release Jig for Glass.**

Hawkins, A. J., *Fusion*, Vol. 12, No. 3, 1965, 19-20. This jig is constructed from chain clamps mounted on a Heathway T/V Tube faceplate. Its use is for tee and angle pieces. D.W.S.

### LAMPS

(234) **D.C. Mercury Vapour Lamp.**

Hussain, J., *Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 4, 1965, 55.

Gives details of a low pressure D.C. mercury vapour lamp for use with interferometers and spectrometers. D.W.S.

### LASERS

See 238.

### OVENS

(235) **An Efficient and Moderately-Priced Annealing Oven.**

Stevens, G., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 4, 1965, 53-4.

Describes the construction of an annealing oven from kiln extension units. D.W.S.

### PUMPS—CIRCULATING

(236) **A Circulating System for Differential Spectrophotometry.**

Schertel, M. E., *Fusion*, Vol. 12, No. 3, 1965, 16-7. Apparatus in which titration, mixing and introduction to the optical cell is carried out, this is an accessory to a spectrophotometer. The pump is operated by a vacuum powered windscreen wiper. D.W.S.

### SAFETY

(237) **Hazards in the Glass Industry.**

Grounds, P. A., *J. Brit. Soc. of Sci. Glassblowers*, Vol. 2, No. 3, 1965, 40-3.

Discusses the complaints of the 19th century glass-makers, exhaustion, silicosis and lung diseases, optical ailments, consumption, etc. Health of the glassblower no longer a problem but still has accidents, burns, cuts and eye injuries. Goes on to talk about first-aid treatment. Lists safety spectacle lenses and filters. D.W.S.

(238) **Treat Lasers with Respect.**

Christie, H. L., *Fusion*, Vol. 12, No. 3, 1965, 21. Short article on some harmful effects of the Laser beam. D.W.S.

### SEALS—GLASS TO METAL

(239) **Behaviour of Interlayers of Glass to Tungsten Seals.**

Takamori, T., and Tomozawa, M., *J. Am. Ceramic Soc.*, Vol. 48, No. 8, 1965, 405.

The relation of the stability of the glass-to-tungsten seals to glass composition was examined. Differences in thermal expansion were considered with respect to seal strength. D.A.H.

## STOPCOCKS

(240) **Fundamentals in the Design of Stopcocks.**  
Smith, I. C. P., *J. Brit. Soc. of Sci. Glassblowers*,  
Vol. 2, No. 4, 1965, 45-8.  
Discusses the relationship of dimensions, deals with  
angle of taper. Various types of stopcock are dealt  
with. D.W.S.

## THIN FILMS

(241) **The Technology of Cathode Sputtering.**  
Pleshivtsey, N. V., *Instruments and Experimental  
Techniques*, No. 5, translation published April 1965,  
929.  
Comprehensive survey of the field (11 pages). Lists  
361 journals abstracted. D.A.H.

## ULTRA-HIGH VACUUM

(242) **Recent Developments in Ultra-high Vacuum.**  
Klemperer, D. K., *J. Brit. Soc. of Sci. Glassblowers*,  
Vol. 2, No. 3, 1965, 28-38.  
This paper given by a leader in the field discusses  
systems, valves, pumps and gauges. D.W.S.

## VACUUM—APPARATUS

(243) **Synthesis of the Higher Sianes and Germanes.**  
Gokhale, S. D., Drake, J. E., and Jolly, W. L., *J. of  
Inorganic and Nuclear Chem.*, Vol. 27, No. 9, 1965,  
1911.  
Vacuum line apparatus shown. D.A.H.

## MISCELLANEOUS

(244) **Synthesis of Uranium Monophosphide by the  
Phosphine Reactions.**  
Baskin, Y., and Shalek, P. D., *J. of Inorganic and  
Nuclear Chem.*, Vol. 26, No. 10, 1965, 1679.  
Glass apparatus used for synthesising Uranium Mono-  
phosphide by reacting finely divided uranium with  
phosphine ( $\text{PH}_3$ ), is shown. D.A.H.

## (245) Atomising Systems for the Spectral Analysis of Solutions.

Tarasevich, N. I., and Mokhamed, M., *Industrial  
Laboratory U.S.S.R.*, Vol. 30, No. 11, translation  
published April 1965, 1757.  
Systems gave stable highly reproducible results and were  
designed so that 1 to 4 jets could be used so as to  
charge sensitivity required with solutions of varying  
concentrations. D.A.H.

## (246) Cell for Measuring the Activity of $\beta$ — Emitters in Volatile Media.

Kachanov, J. A., *Industrial Laboratory U.S.S.R.*,  
Vol. 30, No. 9, translation published March 1965,  
1420.

Two vessels joined by stopcocks and sinter. Emitter  
measured via a Teflon window. D.A.H.

## (247) Gallium Phosphide Light Sources and Photo- cells.

Grimmeiss, H. G., Kischio, W., and Scholz, H.,  
*Philips Tech. Review*, Vol. 26, No. 4/5/6, 1965, 136.  
Apparatus for preparation of Gallium Phosphide is  
shown. D.A.H.

## (248) Automatic Column for Chemical Purification of Mercury.

Artamonov, V. G., *Industrial Laboratory U.S.S.R.*,  
Vol. 31, No. 2, translation published July 1965, 312.  
The column is shown schematically. In normal operation  
speed of circulation is approximately 120 ml/min. In  
one hour mercury passes about 100 times through  
purification cycle. Caustic soda, nitric acid and distilled  
water are used in three stages. D.W.S.

## (249) An Adsorber for Removing Surface-Active and Colloidal Impurities from Electrolytes during Determination of pH values.

Efimov, I. A., and Bacashova, N. N., *Industrial  
Laboratory U.S.S.R.*, Vol. 31, No. 3, translation  
published August 1965, 462. D.A.H.

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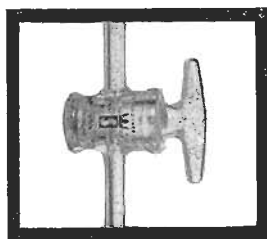
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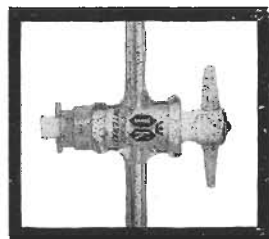
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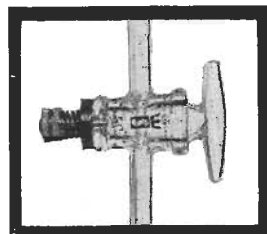
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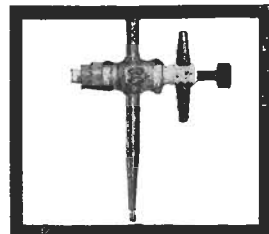
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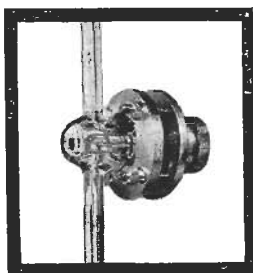
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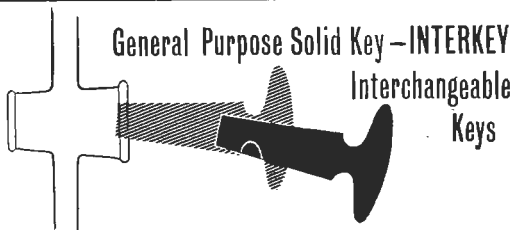


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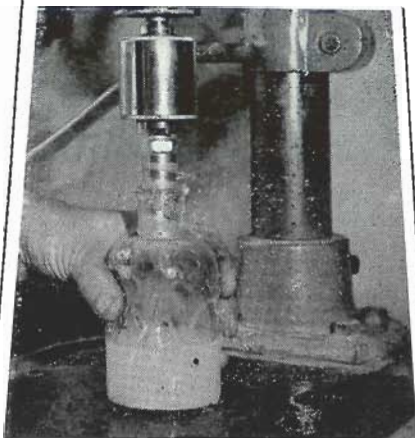
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continued from page 10

a factor of approximately 2,500 (Figure 8). Hydroxyl in vitreous silica also lowers the viscosity of the material and therefore it is worth remembering that a material with a high hydroxyl content (e.g. Spectrosil) given the same heat treatment as a hydroxyl-free vitreous silica (e.g. I.R. Vitreosil) will have a different "fictive" temperature and hence slightly different physical properties. This is demonstrated in Table 4 which compares the softening points, annealing points and strain points of the different vitreous silicas (value for a borosilicate glass are given for comparison purposes).

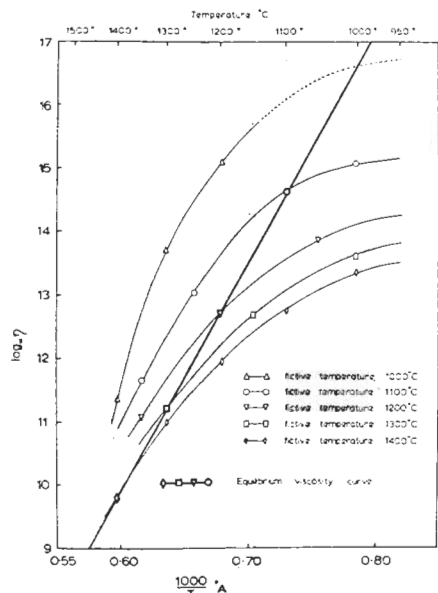


FIGURE 8

Variation of viscosity with temperature for I.R. Vitreosil of different fictive temperature

### Devitrification

Factors influencing the rate of crystallisation of vitreous silica have been discussed above. Vitreous silica also crystallises because, being a super-cooled liquid, it is "unstable" and tends to revert to a more stable phase of silica, usually cristobalite. A complete phase diagram for silica is given in Figure 9. So far as vitreous silica is concerned the right-hand part of this diagram is the most important. It shows that vitreous silica is an unstable form but at temperatures below

1,050°C it will remain in this form regardless of temperature changes. Above 1,050°C there is a slow and irreversible change to  $\beta$ -cristobalite. Under clean conditions this process occurs slowly and is a surface effect. This white devitrification will often have been observed on vitreous silica subjected to temperatures above 1,050°C, but it should not be confused with the white deposits of evaporated silica left after blowpiping.

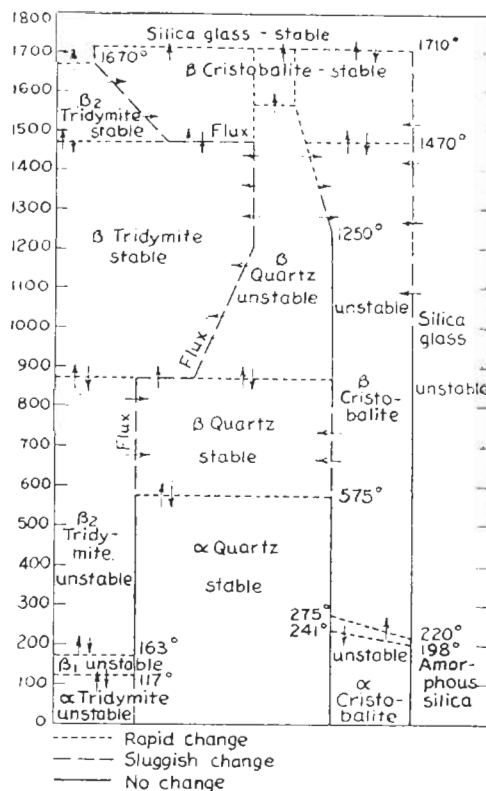


FIGURE 9

Stability relations of the silica minerals

This  $\beta$ -cristobalite does not initially harm a vitreous silica component as its coefficient of thermal expansion is quite close to that of the vitreous material (Figure 10). However, as the temperature falls the  $\beta$ -cristobalite changes to  $\alpha$ -quartz via  $\beta$ -quartz at 570°C or to  $\alpha$ -cristobalite at about 270°C (see Figure 9). These phase changes are accompanied by large volume

changes and consequently the materials are not compatible with the low expansion vitreous silica substrate. If only a small amount of cristobalite has been formed the crystallised material will be weaker than the vitreous silica substrate and will flake off. If, on the other hand, a considerable part of the vitreous silica has been devitrified the strength of the crystalline material will be greater than the remaining vitreous silica and the component will shatter.

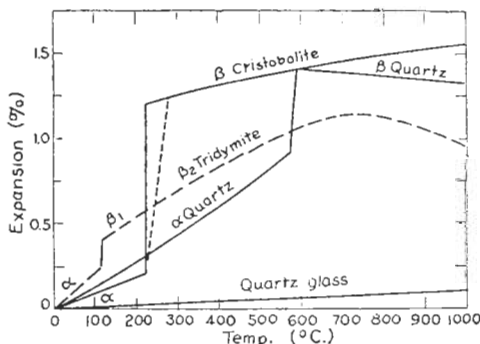


FIGURE 10

*Thermal expansion of silica minerals*

Thus in using vitreous silica above 1,050°C there is a choice between leaving the component above this temperature for as long as is necessary knowing that, due to phase changes, it will shatter on cooling, or to remove components periodically after short times above 1,050°C and dissolve away the small amount of cristobalite formed with hydrofluoric acid.

Devitrification is a "nucleation" process, that is it spreads from centres of impurity or mechanical surface imperfections. For this reason it is usually worthwhile blowpipe working a surface which has been treated in hydrofluoric acid so as to restore a "glassy" surface.

Although components may be heated for hundreds of hours at temperatures above 1,050°C under very clean conditions, very small amounts of contamination by alkali ions (sodium, potassium, lithium, calcium, etc.) will accelerate devitrification. Even fingermarks or tap-water stains will produce the effect very quickly and scrupulous cleanliness is vital in preparing components for blowpiping or high temperature work.

## Thermal Properties

Probably the most significant property which makes vitreous silica such a useful high temperature material is its extremely low coefficient of expansion. Figure 11 gives comparative expansions for some common laboratory materials. It should be noted how small is the expansion of silica compared with even conventional heat resisting glasses of the borosilicate type.

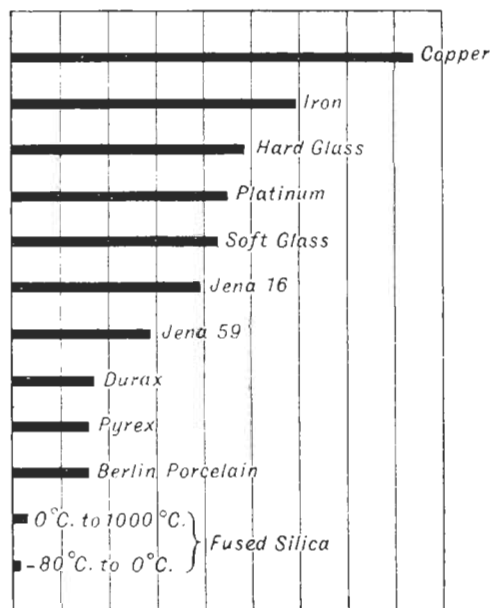


FIGURE 11

*Chart comparing the coefficients of expansion of various common laboratory materials (drawn to proportional scale)*

This low expansion gives vitreous silica its almost unique position among high temperature materials in its resistance to thermal shock. This statement has been qualified by the word "almost" because two kinds of thermal shock need to be considered. Where one surface of a material is suddenly subjected to a temperature very different from the opposite side the important factors are that the material should have the lowest possible expansion ( $\alpha$ ), the highest possible strength ( $S$ ), and a low elasticity ( $E$ ) so that deformation can occur. For this type of application vitreous silica is much better than

other high temperature materials. There are, however, other applications in which steady differences in temperature are maintained and here the conductivity ( $k$ ) is obviously important. The translucent forms of vitreous silica have low effective thermal conductivities which tend to counteract the beneficial facts of their low thermal expansions. Table 5 shows some typical high temperature materials with comparative thermal shock values for the two types of conditions.

### Electrical Properties

All glasses have good electrical insulation and dielectric properties at ordinary temperatures. This is because they are largely "polar" in structure, that is different from the ionic structure of, say, a metal containing free electrons. Ordinary glasses, however, usually contain some ionic structure, or contain ionic impurities, and as the temperature rises these ions become more mobile and the electrical resistivity falls off rapidly.

There are virtually no free ions or electrons in vitreous silica and therefore not only is the electrical resistivity higher at ordinary temperature but the fall off with rise in temperature is very much less than for conventional glasses.

TABLE 5  
Relative Thermal Shock Indices for High Temperature Materials

Material	$\frac{S}{E\alpha}$ ( $^{\circ}\text{C}$ )	$\frac{KS(\text{cal. sec.}^{-1})}{E\alpha}$ $\text{cm.}^{-1}$
Vitreous Silica . . .	3,000	13.0
Graphite . . . .	740	310
Zircon . . . . .	95	1.1
Zirconia (stabilised)	78	0.38
Beryllia . . . . .	53	11.7
Alumina . . . . .	47	1.5
Magnesia . . . . .	34	1.3
Thoria . . . . .	33	0.46

### Summary

In choosing vitreous silica for a particular purpose considerations of purity, optical transmission structure, devitrification and water content may all be important with the knowledge that no one form of silica is the best in all respects. It is hoped that this brief account will be of help to glassblowers in their choice of vitreous silica for a particular purpose and at the same time explain the differences in manipulation between vitreous silica and other glasses which are likely to be encountered during working.

### Preliminary Notice

## 1966 COLLOQUIUM

to be held at

**THE METROPOLE HOTEL, LEEDS, on SATURDAY, 24th SEPTEMBER**

Cost to members: 28s. 6d., which includes tea, coffee and full lunch, but not drinks. There will be a full programme of lectures on interesting topics and it is hoped an account of the Chairman's experiences in the U.S.A. will be given.

Trade exhibits are being arranged and student members are invited to submit scientific apparatus and items of equipment which they have constructed. These will be divided into classes and judged for the A. D. Wood Trophy, which will be presented at the meeting.

The North-Eastern Section are making every effort to provide facilities of the highest standard and the journey to Leeds will be well worth while.

Full programme in the June issue

## I. C. P. SMITH, B.Sc., F.I.C.

### Chairman's appreciation of his services for the Society

As many of you will already have heard, Mr. I. C. P. Smith has had to resign his post as Secretary to the Society. The decision to retire from active service has been forced upon him purely for health reasons and on orders from medical authorities.

Let me on your behalf, and as your Chairman, offer sincere wishes for a speedy recovery and return to good health. We hope that we may have the pleasure of his company at many meetings in the future.

We cannot under-estimate the enormous value and amount of extremely hard work that he has put into the Society. Mr. Smith was in at the beginning amongst the founder-members, and has held the post of Secretary since the resignation of Mr. D. Ivens in the early days of the Society. He was the Chairman of the Southern Section and he also negotiated with the City and Guilds Committee on behalf of the Society.

From all these angles he has watched, helped and guided the Society to the excellent body that it is today. His technical knowledge and con-

tacts in the world of glass have on many occasions assisted us to survive.

He was Advertising Manager of the Journal, in fact the present pattern of the Journal is largely due to his early guidance. His active interest in this field is beyond our praise, for the Journal is our livelihood and is recognised as such throughout the Society.

We deeply appreciate everything that he has done to fulfil the aims of the Society. We owe our heartfelt thanks to him. E. G. EVANS

### Editor's appreciation

Having worked in collaboration with Mr. I. C. P. Smith since the early days of this Journal, I can endorse the foregoing remarks and add that my association with him during this period has resulted in a growing respect and admiration for the way he has applied his energy and experience to the production of this Journal. It was a great help to know that he was on hand to ease production difficulties, and on many occasions his advice has been invaluable. I have learnt much from him and although we shall try to maintain his high standards he will be difficult to replace. J. H. BURROW

## SPECIAL COUNCIL MEETING

*Held on 19th March, 1966 in Birmingham*

**I**N consequence of the enforced retirement of Mr. I. C. P. Smith, the chairman called this special council meeting at which he acquainted the council with the reason for his resignation, the events which followed and the resulting situation which had to be considered.

Mr. I. C. P. Smith's resignation was accepted with regret by the Council but with full and sympathetic understanding, and a vote of thanks to him was placed on record in appreciation of the enormous amount of work he had carried out for the Society. A wish for a speedy return to health was expressed.

The chairman stressed that the work of administering the Society should be dispersed and no one person should be expected or allowed to carry a similar load of responsibility to that which had built up on Mr. I. C. P. Smith. The appointment of an assistant secretary was essential and the main officers of the Society should not have other responsibilities within the Society. Mr. D. W. Smith of the Western Section was unanimously elected Society Secretary and he

agreed to investigate possible assistants, forwarding to the council the name of the member able and willing to help him.

Mr. D. A. Henson, having previously expressed a wish to retire from the office of Society Treasurer in order to concentrate on Journal finances, introduced Mr. F. C. Branfield of the Southern Section as capable and willing to take his place. Mr. Branfield was duly nominated and elected treasurer to the Society.

Mr. Burrow outlined the current position of the Journal and the part played by Mr. I. C. P. Smith. After discussion it was agreed to invite Mr. J. A. Frost, of Reading University, to become business manager with responsibility of arranging advertising and printing.

Mr. Butler of the North-Eastern Section gave a brief account of the arrangements so far made for the 1966 Colloquium. These are reported on page 13.

It was also decided that the Council should, at an early date, consider the conditions needed to bring Rule 7 into operation. D. W. SMITH

## GLASS IN CHEMISTRY

### Fifth Annual Colloquium of the British Society of Scientific Glassblowers

GLASS IN CHEMISTRY was the subject of the Colloquium which took place on the 24th September, 1965, in the Haworth Building, University of Birmingham, under the auspices of the British Society of Scientific Glassblowers

#### Design in Glass for Vacuum and Pressure Application

IN his first talk Mr. G. P. Helliwell of Quickfit & Quartz Ltd. outlined the essential design factors for glass where a state of tension might be built up. Glass is very strong in compression but weak in tension, and this is more marked when the state of tension is prolonged. Although glass will generally withstand a tensile stress of 6,000 p.s.i. and even as high as 10,000 p.s.i. for short periods, the presence of small flaws, micro (Griffiths) cracks, or effects of moisture and chemical action, may initiate major cracks if subjected to stress over a long period. This has led to the adoption of a design tensile stress 1,000 p.s.i. for general purposes, and of 500 p.s.i. for prolonged stress or more hazardous applications. For spherical and cylindrical vessels under internal pressure the simple formulae are respectively

$$f = \frac{Dp}{4t} \text{ and } f = \frac{Dp}{2t}$$

where  $f$  is the design stress,  $D$  the inside diameter,  $p$  the working pressure and  $t$  the thickness: for

a flat base the formula is  $f = .18 \frac{D^2}{t} p$ . For a

glass vessel under vacuum  $\frac{D}{t} = 19$  is a good working rule for minimum thickness. These design factors have already been adopted in several B.S. specifications connected with glassware.

#### The Function of a Glassblower in Industrial Research

This was the subject of an encouraging talk by Dr. H. Coates of the Research Department of Albright & Wilson (Manufacturing) Ltd., that is to the research glassblowers present—the majority of the audience.

Himself an able glassblower he had a real appreciation of the value of a versatile man in a research department. He stressed the need of the glassblower working in close co-operation with all the research staff, and his practical experience could be of great value in the use of glass in the development of projects. His role is sometimes

difficult when pressure of work makes it necessary for him to assess the real needs of various categories of research workers so that effort is not wasted. To be able to use his skill fully the glassblower must be supported by a well equipped glass-shop with annealing oven, lathe and other machinery in the same way that a mechanical workshop would be equipped, and it seems that research managers do not always appreciate this. In the latter respect he added that if the research manager failed to see the need for these expensive items and expected his glassblower to work at a small bench in the corner he should be confronted with the need to work on a 20-litre flask.

#### Chemistry and Light

Professor J. P. Cobb, Chemistry Department, University of Birmingham, gave a very interesting talk and demonstration on the sources of light through the ages, and beginning with the simple bunsen burner and gas mantle a wide variety of chemical and physical methods of producing luminosity were demonstrated. These included phosphorescence, chemiluminescence, ultra-violet light and electrical discharges. The demonstrations had been well prepared and were extremely effective and the address was received with acclamation.

#### Recent Advances in Laboratory and Industrial Glassware

In his second talk, Mr. Helliwell outlined some of the more recent innovations in this sphere: one is the series of screwed plastic fittings made to go with glass tubes having a screw formed on one end. Another was a set of very small reaction apparatus, and at the other end of the scale the developments in 24 in. pipe-line, of considerable interest to chemical engineers who are already calling for larger sizes.

Considerable applause was given to each speaker at the end of his address and as previously reported Mr. E. G. Evans, the Chairman of the Society concluded the meeting, acknowledging the Society's indebtedness to the speakers and to the Midland Section for their part in organising the colloquium.

Compiled from reports submitted by Mr. L. C. Haynes and Mr. I. C. P. Smith, the latter having appeared in "Chemistry and Industry."

# AN AIR-GAUGE METHOD FOR EXPLORING THE BORES OF SMALL TUBES

by I. C. P. SMITH

*E.R.D.E. Waltham Abbey, Essex*

AIR gauges for exploring the bores of tubes are commercially available, but not generally for sizes less than about 4 mm. The gauge head of these is a cylindrical member, having two jets at opposite ends of a diameter, connected to the governed airflow, control jet and pressure gauge, which give the dimensional reading.\* The gauge heads are comparatively expensive to make, and one is needed for each nominal size under investigation. The method to be described uses a similar air flow control, but is otherwise inexpensive, and gives comparative results, i.e. of the order of .001 mm.

The system is illustrated in Fig. 1, in this case the calibrated air flow is attached to the tube under examination, and a ball of selected size,

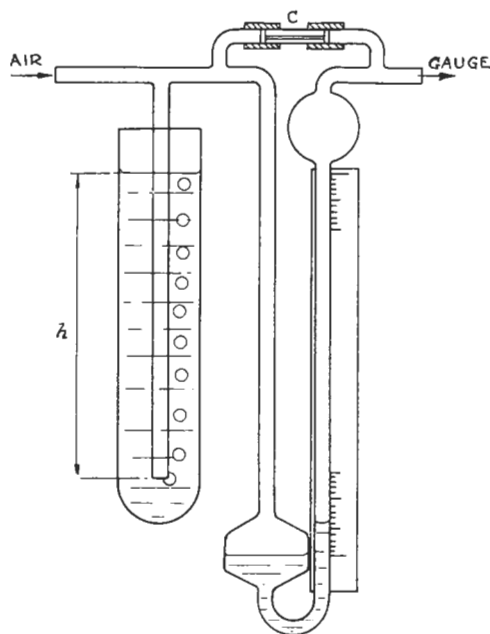


FIGURE 1

attached to a thin rod, is inserted at points in the tube, when readings of the gauge are obtained, to be converted into differences in diameter along the tube. The principle of operation is simple, and as illustrated in Figure 2, there is a crescent shaped space between the ball and the tube through which air may pass.

The area of opening in Figure 2 is  $\frac{\pi}{4} (d_1^2 - d_2^2)$ ;

the term in brackets may be factorised into  $(d_1 + d_2)(d_1 - d_2)$ . When the difference is small  $d_1 + d_2$  practically equals  $2d_1$ , and the area may be expressed as  $\frac{\pi}{4} 2d_1 (d_1 - d_2)$ , this shows that

over small differences this area is proportional to the difference in diameters.



FIGURE 2

If the capillary control C has been chosen to be of the same order as this opening a reading of flow rate is obtained on the manometer. If a slightly larger ball is inserted the airflow rate will be smaller, and a smaller manometer reading obtained; likewise for a smaller ball larger manometer readings. Similarly, if the original ball is placed in a section of tubing of slightly smaller diameter a smaller gap is produced and a smaller gauge reading, and vice-versa. The purpose of this article is to show how these gauge readings may be interpreted as differences in diameter from a norm.

To set up and calibrate an air-gauge, so that a batch of tubing of one nominal size may be explored the procedure is as follows:—

The air flow part of the apparatus illustrated in simple form in Figure 1, consists of the usual pressure stabilising tube, connected to a flowmeter. The height  $h$  is conveniently 50 cm., the manometer greater than this, and a number

\* The jet system is described in "Pneumatic Gauging Applied to Standard Ground Glass Joints," I. C. P. Smith, Laboratory Practice, March 1958, Vol. 7, No. 3.



of 20 or 30 mm. lengths of different sizes of capillary tube are available to connect across it at C. The capillary forms the control jet of the commercial types of set-up. The manometer scale is better in inches and tenths than in millimetres.

It is necessary next to obtain balls of good quality of sizes slightly smaller than the nominal sizes to be explored, e.g. for 2.0 mm. sizes about 1.88, 1.91 and 1.95 mm. These may be obtainable from a ball-bearing manufacturer, or made by patient grinding, and stuck with a trace of Araldite to a straight wire or length of hypodermic needle tubing; or alternatively, a glassblower may proceed as follows. A long strand of glass rod is drawn out, not more than 0.6 mm. diameter, and on lengths of this a small bead is melted up at one end. With practice, and with prior careful selection of the starting rod, beads of good sphericity may be made and selected for size, approximating to the values given above. The rods attached should be at least half the length of the tubing to be examined, and so thin that they do not cause errors in reading owing to back-pressure at deep insertions. Steel balls may alternatively be held in a desired position in the tube by means of a powerful magnet or a solenoid, when the wire is unnecessary. The balls are carefully measured with the best micrometer available, and the stems labelled.

The air flow, using in this case a 0.8 mm.  $\times$  20 mm. control capillary, is attached to one end of the 2 mm. tube, a mark made say 10 mm. from the other end, the balls inserted in turn to this mark, and readings of the gauge taken in inches w.g. The readings were as follows:

Ball 1.95 mm.	Reading 3.0 in.
1.915 mm.	8.3 in.
1.89 mm.	11.0 in.

From these a graph is drawn, line 2 of Figure 3. The major part is seen to be approximately a straight line, the slope of which gives the sensitivity of the instrument in fractions of a mm. per inch of reading. In this example the spread of diameter is  $1.95-1.89 \text{ mm.} = 0.06 \text{ mm.}$  and the spread of manometer readings  $11.0-3.0 = 8.0 \text{ in.}$ , so that  $1.33 \text{ in. equals } 0.01 \text{ mm.}$  As the instrument can be read to 0.1 in., this gives a sensitivity of 0.0006 mm.

One of the balls, e.g. the 1.915 mm., is now run down the tube, stopping at intervals to take a reading; if this differs markedly from the original reading of 8.3 in., for that ball, it should

be noted and the difference in diameter calculated using the above factor, noting that a smaller gauge reading means a smaller difference between ball and bore, and a smaller bore diameter.

The graph contains also readings taken on 1 mm., 5 mm. and 11 mm. bore tubes, using as control capillaries respectively 40 mm. of 0.61

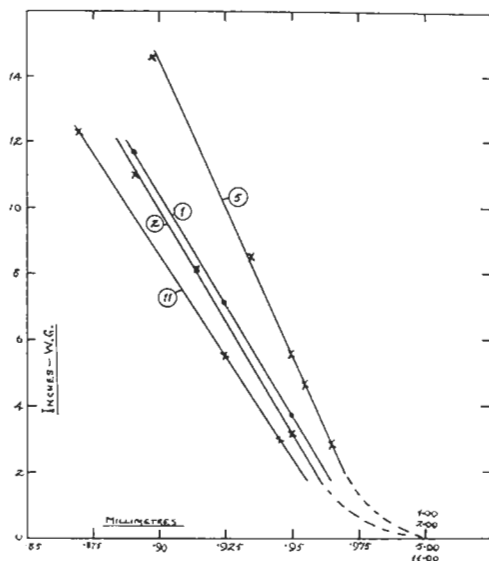


FIGURE 3

mm., 30 mm. of 0.88 mm. and 20 mm. of 1.25 mm. It is seen that the straight lines of the graphs do not meet the X axis at the nominal diameter, but that each curves round to this position. This part of the curve has been confirmed by making balls of close dimensions in this region, and is characteristic of most air gauge measurements, approximating to the first .001 in. in the usual jet methods. All of these readings were made using melted up beads on glass stems, except for the 1 mm., which used small beads melted on .005 in. Pt wire.

All the tubing sizes referred to, both under test and the control capillaries were precision bore. The ball sizes chosen are generally in steps between 0.05 and 0.20 mm. less than the size under test, and a control capillary used which will give manometer readings between about 3.0 in. for the largest and 12 to 14 in. for the smallest ball. Absolute diameter measurements using the balls can only be derived from an

absolute measurement of the tubing at one place. It is for this reason that the first set of readings is taken just inside the tubing, where the diameter can be determined by any usual means.

The gauging method described has been used successfully for testing the capillary tubing to be used for reference McLeod gauges and for

viscometers. It has found small irregularities caused by inclusions in the glass tubing wall, where the tubing did not sink onto its mandrel in the expected manner. In this it has an advantage over the mercury thread, as also in examining metal tubes in which mercury would be invisible.

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## SECTION ACTIVITIES

### *Southern Section*

The 1965-66 programme of the Southern Section opened on Wednesday, 13th October, 1965, in the main Chemistry Lecture Theatre, Queen Elizabeth's College. The section were very fortunate to have Mr. C. K. McEwan of Q.V.F. as guest speaker for this meeting. The speaker had the misfortune to have his car break down during the long journey to London and as a consequence arrived only just in time for the meeting. During his talk "The Application of Glass Equipment in the Chemical Industry," Mr. McEwan described the special techniques involved in fabricating the huge glass apparatus manufactured by Q.V.F. Ltd.

On Wednesday, 17th November, 1965, Mr. J. Burrow of the University of Bristol presented a lecture on "Glass Vacuum Systems" in the main Chemistry Lecture Theatre, Queen Elizabeth College. A lecture by Mr. Burrow is an annual event in the Southern Section programme and as is usual when Mr. Burrow visits the Southern Section, an eager audience awaited him. The speaker traced the use of glass for vacuum systems from the days when the research glass-blower had to make his own stopcocks, standard joints and diffusion pumps in the laboratory in order to produce apparatus to achieve low pressures. Glass, the speaker felt, was still the cheapest and cleanest material from which to construct small scale vacuum systems. The talk included pump design, sorption pumps and getters, special mention was made of the care needed in cleaning glass to be used for vacuum apparatus.

A meeting was held on Wednesday, 8th December, 1965, in the main Chemistry Lecture Theatre, Queen Elizabeth College, to hear Mr. L. D. Cole of E.R.D.E., Waltham Abbey, talk about "Safety Aspects." This is a very important subject for all glassblowers and we are grateful

to Mr. Cole for a most interesting lecture. This talk will be fully reported in a later issue.

The Annual General Meeting of the Southern Section was held on Wednesday, 12th January, 1966, in the main Chemistry Lecture Theatre, Queen Elizabeth College. At this meeting the following officers and committee were elected: *Treasurer*: F. Branfield; *Auditor*: W. Brench; *Secretary*: E. White; *Committee*: L. Bengé, E. Evans, F. Luadaka, T. Parsell, R. Reader, D. Smith, I. C. P. Smith, I. Zamit, Councillor D. Smith.

At the end of the Annual General Meeting Mr. I. C. P. Smith presented a most interesting lecture entitled "Millions of Gas Capillaries." During his talk Mr. Smith explained the method used to make small glass tubes filled with liquid which were to be used to detect the presence of poison gas in war time. The methods used included the redrawing process and filling the tubes with liquid using vacuum and centrifugal force.

The next meeting of the Southern Section is a Stag Dinner to be held on Friday, 18th February, 1966, at the Horse Shoe Hotel, Tottenham Court Road. Members of all sections of the Society are welcome to attend the Southern Section Stag Dinner.

E. WHITE

### *North-Western Section*

A meeting of this section was held on the 28th January, 1966, at the Training Centre of Messrs. Joseph Crossfield and Sons Ltd.

Before the business commenced members observed a one minute's silence in memory of Mr. W. Taylor, a member of the section.

Various subjects were then discussed and some decisions agreed upon which will, it is hoped, result in increased activity and smoother running

of the section. A visit to Messrs. Joblings has been fixed for the 4th May and other visits and lectures are being negotiated.

The future pattern of section meetings will be first to deal with Society matters and follow with discussions on glass working topics. On the 4th March the agenda for the Society A.G.M. will be examined and Mr. Elson will then lead a discussion on the making, silvering and pumping of Dewars.

Two proposals were made to be brought before the Council meeting on the 5th February:

- (1) That a more detailed agenda for these Council meetings be circulated to give more information to attending Councilors.
- (2) That an item be included in the agenda of the Society A.G.M. to consider either honorary membership or reduced fees for retired members.

Mr. Edkins and Mr. Collins were nominated to attend Council meetings.

Among other subjects discussed was the difficulty experienced because of long distances of some members.

It was concluded that the Training Centre was the most convenient meeting place and with a vote of thanks to Messrs. Crossfield for the use of the Centre the meeting ended.

J. STOCKTON

## Western Section

THE Annual Dinner for members and their ladies was once again a very pleasant evening. More members than last year attended but next year we must try and get most of the Section there.

The Annual General Meeting in October, as is usual, was rather sparsely attended. In fact there were only just enough full members present to make the proposed changes to the Section rules. The number of members in the section, it was reported, had increased but the retiring Committee had been constantly concerned with the low attendance at monthly lectures. During a discussion after the main business, it was decided that the section should continue to meet monthly, not quarterly, as had been suggested. The retiring chairman, Mr. R. A. Redford, thanked members for their support during his term of office, and said that his main concern had been the failure of many members to attend meetings which had a high technical value.

## Elections

*Chairman:* Mr. R. E. Garrard; *Secretary:* Mr. D. W. Smith; *Treasurer:* Mr. D. A. Jones; *Committee:* Messrs. Lyons, Leeson-Magry Porter and Spear.

It was agreed that the publication of *Revue* (the Western Section News Letter) should continue.

At the November meeting we were honoured to have as visiting lecturer, Mr. W. V. Baker of A.E.I. Research Laboratories, Rugby. His subject was copper-glass tubular seals and as usual Mr. Baker spoke with great authority interspersed with humour.

Mr. J. A. Frost from Reading University and a member of the Thames Valley Section, gave us an insight into "Strain in Glass" during the December lecture. This masterly lecture was accompanied by demonstration and slides. Mr. Frost, who is known to most members of the section, cleared up many false impressions that some members had, and made a very difficult subject seem an easy one.

During the January lecture on "Ultrasonic Machining of Glass" by Mr. B. F. Green of Kerry's Ultrasonics Ltd., the subject was explained in very simple terms. Mr. Green explained in great detail the workings and dimensional features of an Ultrasonic Generator. After discussing shapes, sizes and materials of construction of the tools we were treated to a demonstration. The evening was well rounded off by the showing of a film on Ultrasonics in general application.

D. W. SMITH

## OBITUARIES

WE regret the passing of Mr. D. Hugh, glassblower to the Physics Department, University of Birmingham, who died in November 1965.

A former member of the Midland Section, he had been seriously ill for some time and our sympathy goes to his relatives in their sad loss.

Mr. W. Taylor, a member of the North-Western Section collapsed and died at his home on the 22nd December, 1965.

He worked at I.C.I. Pharmaceuticals Ltd. until his retirement shortly before death, and Mr. Elson, chairman of the section, who had known him for over 30 years held him in high esteem.

*continued from page 1*

award of the certificate will be held at the end of the current session.

This elementary glassblowing course has attracted the interest of principals and lecturers at several colleges who have requested information regarding the syllabus and it is anticipated that the course will be adopted at these colleges as the elementary glassblowing course. The Society recommends that the instructors should be full members and if necessary, the Board of Examiners will advise on full members in the area convenient to the college.

Although glassblowing instruction has been included in scientific and laboratory technician syllabuses as ancillary subjects for many years, there is evidence that laboratory staffs, research workers, and science teachers will find the B.S.S.G. course advantageous to their careers.

#### **Professional Qualification for Glassblowing**

The Board have agreed on a scheme for the implementation of the examination for the awarding of the certificate of competence which will be the professional qualification required for the status of full membership of the Society. The Board will present the scheme to the Annual General Meeting in April, and advise that it will take effect from August 1966.

The theoretical examination is being compiled, and will include questions on burner design, use and hazards of gasses, properties and applications of various glasses, also materials and devices with which the scientific glassblowers should be conversant.

#### **Presentation Awards**

The Board has pleasure in announcing that the British Society of Scientific Glassblowers has received two generous offers of presentations for glassblowing ability or the knowledge of glass application.

Mr. A. D. Wood, a notable stopcock and glass apparatus manufacturer, has offered a Challenge Cup available to student members and to be awarded for a meritorious piece of scientific glassware constructed entirely by a student member. The Board proposes to arrange with Mr. Wood to have the entries judged and to have the Cup presented during the Colloquium programme.

The Management of Thermal Syndicate Ltd., Wallsend, Northumberland, have offered to

present an award to the value of £50, to a member of the B.S.S.G. for either a written article defining a unique technique in manipulation or application of silica, or for a meritorious designed and constructed apparatus in silica. Arrangements are to be considered for the implementing of this award.

#### **Glass Manufacturing and Processing Industries—Training**

The Exploratory Committee arranged by the City and Guilds of London Institute for the Glass Manufacturing and Processing Industries on which the B.S.S.G. is represented is finalising the arrangements for establishing a Glass Operatives Course to be offered to colleges. It is anticipated that the training will be a "full day" release course and will be advantageous for glassblowers requiring technologist knowledge.

S. G. YORKE

Chairman to the Board of Examiners

### **MEMBERS QUERIES**

*From Mr. T. P. Young, Glassblowing Department, Department of Applied Science, Loughborough College of Technology, Ashby Road, Loughborough, Leics.*

Mr. Young asks if any member can furnish him with information with regard to working B.S.39.B Glass. He wishes to seal two polished windows of B.S.39.B Glass, 40 mm. diameter + 1 mm. thick, to the end of Pyrex tubing, to form a gas-tight cell, with I.R. transmitting windows.

He would appreciate any help with this technique.

*From Henry Keating, Biochemistry Department, University College, Cork, Eire.*

I would be grateful to anybody who could let me have any information or references on building and using a high frequency heating apparatus for making of glass to metal seals up to 12 mm. diameter.

### **NEWS ITEM**

Our Chairman 'Ted' Evans has been invited by the American Scientific Glassblowers Society to give a talk and demonstration on "Glass Centrifuging" at their Symposium in Boston. We wish him every success.—Ed.

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We are a young and expanding company engaged in the fabrication of all types of laboratory apparatus in quartz glass, and we have immediate vacancies for qualified glassblowers and lathe workers.

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# **The University of Warwick**

Applications are invited for a post of

## **Technical Assistant**

in the School of Physics to install and operate facilities for glassblowing. Applicants should have previous experience of glassblowing and other laboratory techniques

Applications, giving details of age, qualifications and experience should be sent to the Registrar, the University of Warwick, Coventry, Warwickshire, quoting Ref. No. 42/65



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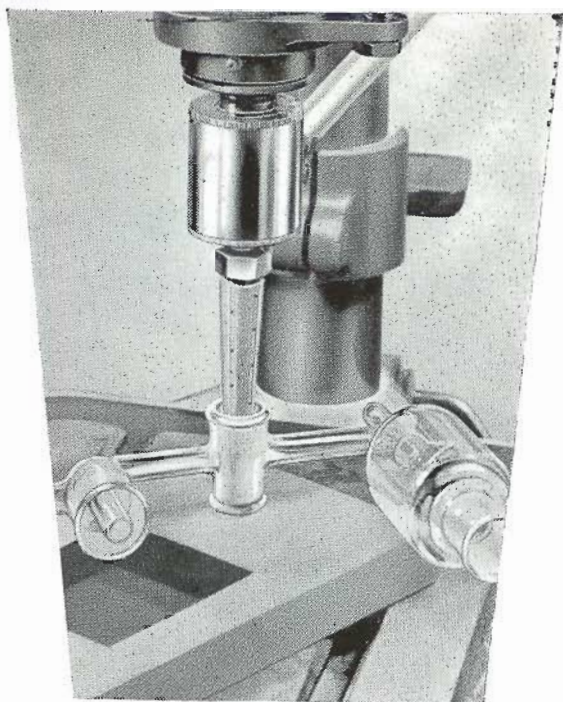
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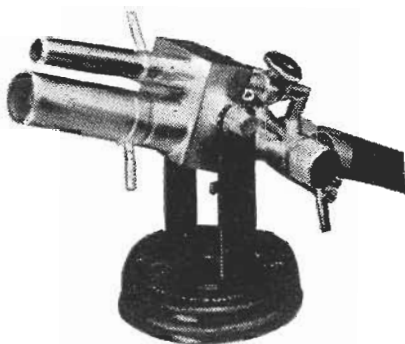
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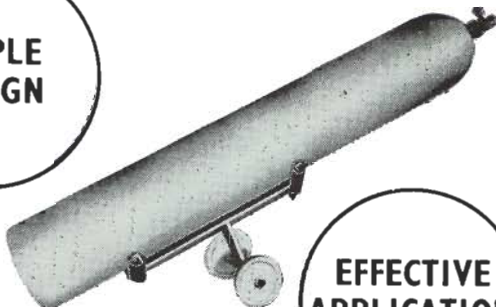
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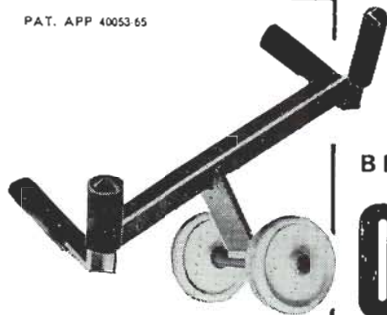
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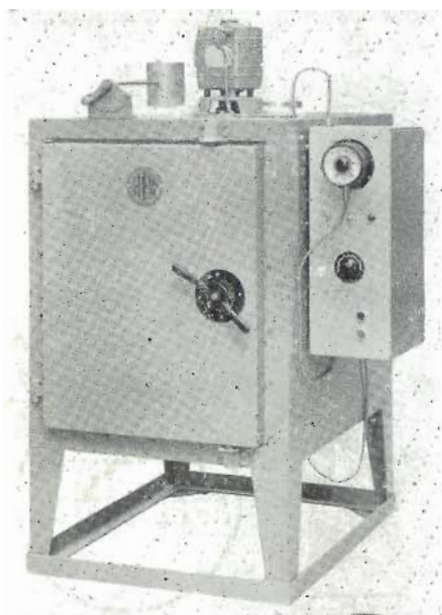
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