

# JOURNAL

OF THE

## BRITISH SOCIETY OF SCIENTIFIC GLASSBLOWERS

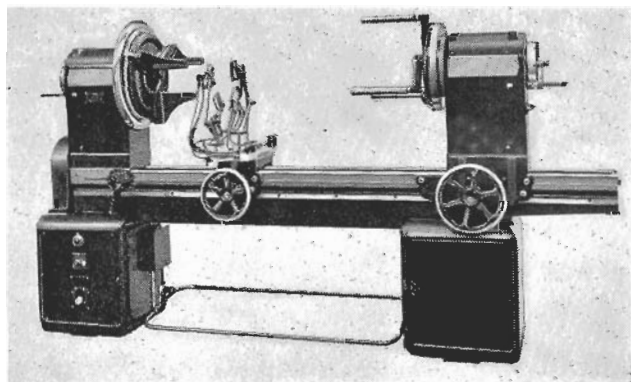
Vol. 1

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No. 4

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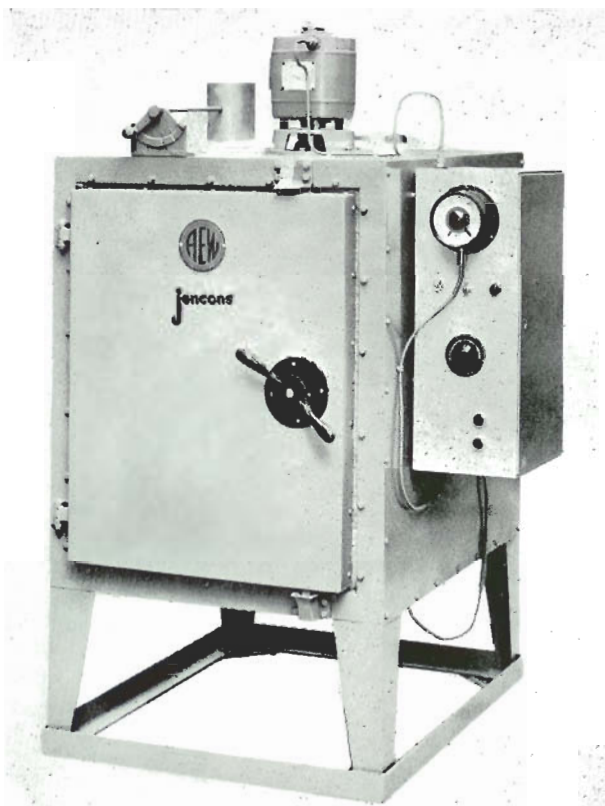
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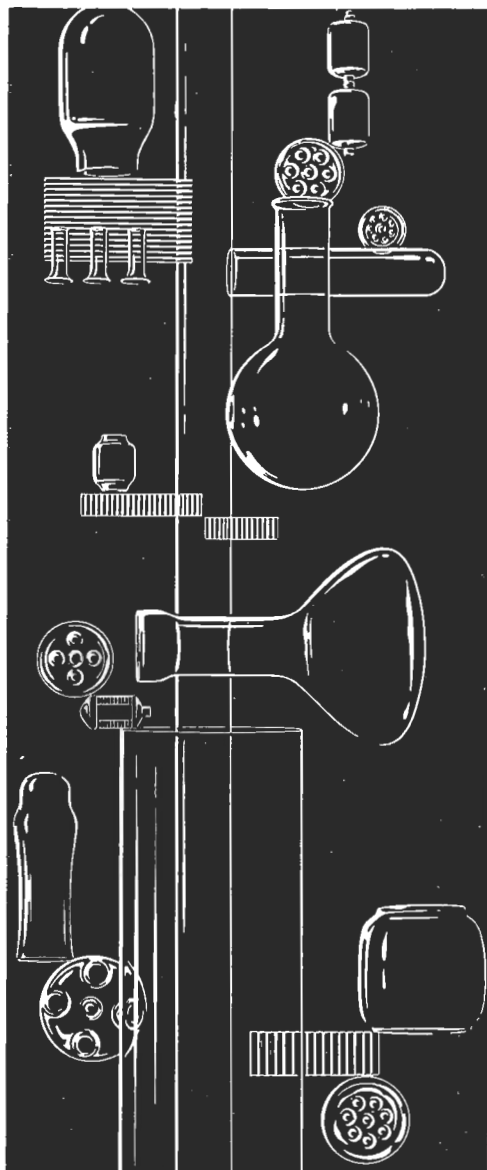


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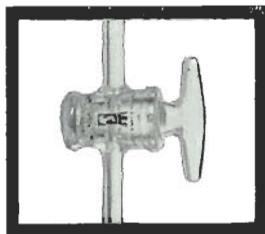
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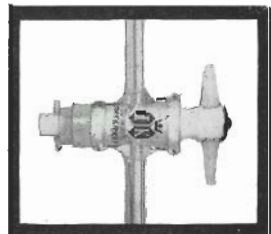
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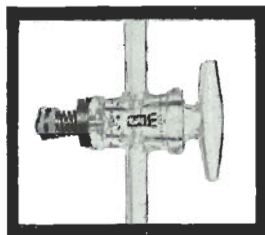
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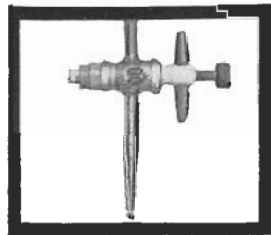
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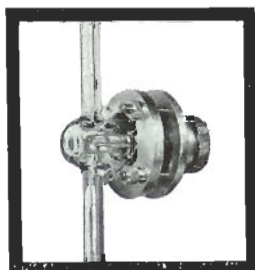
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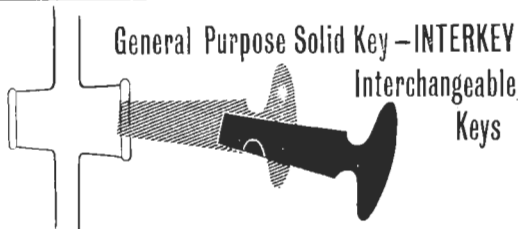


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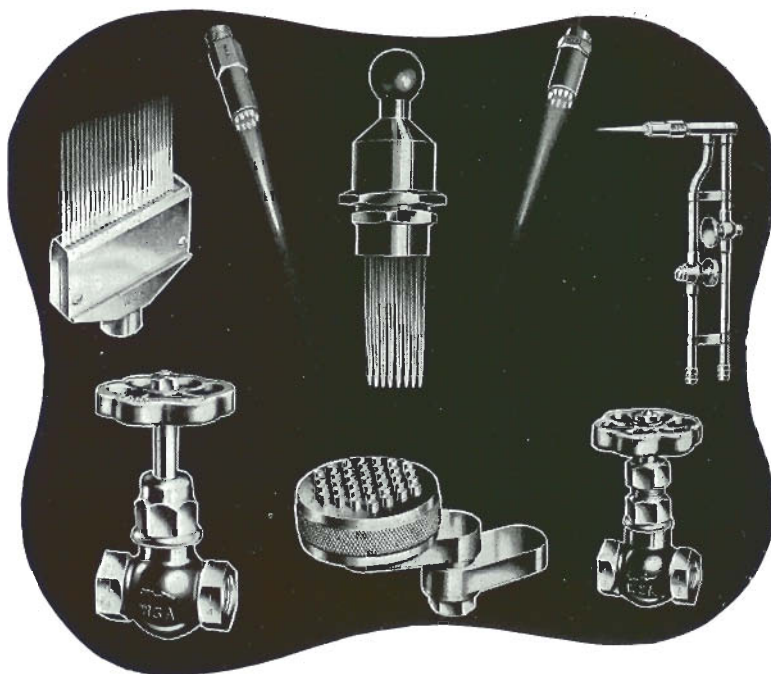
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## EDITORIAL

WITH a wish for success to all members in the coming year the editors hope that there will be renewed energy and enthusiasm in the Society's activities. We are fortunate in having a secretary who is prepared to undertake enormous amounts of work on our behalf and many other officers who also make substantial contributions. But it is possible that changing circumstances could involve us in the loss of one or more of these willing helpers, which could have serious consequences if we do not adopt a policy of persuading more members to take office to ensure future continuity and cover the inevitable increase in Society activity.

Our Journal began with good intent but relies on the co-operation of members for a supply of publishable material and of advertisers for financial help. But whereas the latter have given us every encouragement we are somewhat disappointed with the present response from sections and members.

It is true that we did once suggest that reports should be kept short but it was not our intention to discourage submission as they are badly needed at the appropriate time to keep a balance

of material. We intended that the Journal should be a pool of knowledge to which members could both contribute and draw on and we feel that a vast store of information exists which could be made available to members.

It seems that there are still differences in opinion with regard to approach, methods and techniques of glassworking, and through publication and discussion we should endeavour to achieve some uniformity. This will become important when the Society decides on its scheme for issuing certificates and diplomas and among the points at issue will be the "quality to time" factor, it being our opinion that the days of the "time taking" glassworker are over and the best results are achieved quickly without unnecessary manipulation.

To allow full concentration on practical aspects of our Society every effort should be made to complete a permanent set of rules in time for presentation at the next A.G.M. for members' approval. The results could be invaluable in an international sense and we as a Society can have great influence on glassworkers all over the world.

## SILVERING SOLUTIONS

### A Special Precaution

Descriptions are frequently given of the Brashear process for silvering glass, including the making up of stock solutions, without sufficiently emphasising the very serious danger of keeping the mixed ammoniacal silver nitrate solution for any length of time. This solution after standing for a few hours can detonate spontaneously and many serious accidents have been reported due to this; in the last one brought to our notice, which occurred on the 5th January of this year, a young worker lost the sight of an eye. Two

hours should be considered the very limit for keeping this solution and any surplus that has been made up should be destroyed by (1) washing down the drain with a copious supply of water, (2) adding Hydrochloric acid, or (3) adding excess of reducing agent.

The Journal is published quarterly by the B.S.S.G. and is available free to members and at 5s. per copy (or 17s. 6d. per annum) to non-members. Editorial communications should be addressed to the Editors, c/o Departments of Physics and Chemistry, University of Bristol, and enquiries for advertising space to Mr. I. C. P. Smith, 65 Woodberry Way, Chingford, London, E.4. Printed in Great Britain by E. G. Ellis & Sons, Willow Street, London, E.4.

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EDITORIAL NOTE. *Should any case of non-receipt of the Journal or other Society notices become known to members please advise those affected to report the matter to one of the officials.*

# SOME UNUSUAL GLASSES

R. V. Adams, A.E.I. Central Research Laboratory, Rugby

## 1. Introduction

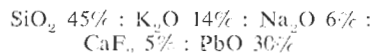
A large variety of glasses are needed in the electrical industry for the many different devices incorporating glass that are manufactured. The industry is always striving to improve its glasses and where possible new glasses are prepared to meet the demand of a new product. Thus research, development and production are a continuous spectrum and consequently there is a close liaison between the departments concerned.

In the context of the present paper the term "unusual" is taken to mean glasses which in general fall outside the sphere of the more or less conventional "soft" soda glasses and "hard" boro-silicate glasses with which most scientific glassblowers are familiar. In view of the large number of glasses which the organisation with which the author is concerned makes, this leaves a fairly wide field from which to pick and choose. Thus a few selected glasses are described, giving where possible some background information relevant to the development of these glasses which may be of particular interest to the scientific glassblower. Finally, two really unusual types of glass prepared and investigated at the author's laboratory over the past two decades are described.

## 2. Development of Iron Sealing Glasses

These glasses were developed as a result of work carried out at Rugby (the British Thomson Houston Co. as it was then) by Dale and Stanworth during the war. The object behind the work was to develop a substitute for a 30% lead glass containing little or no lead oxide as it was feared there might be a shortage of lead oxide. Lead glass is a vital component in the majority of lamps and bulbs because it has a high electrical resistivity. It is also easy to melt, draw into tubing and work on lamp making machinery. It was desirable that any glass developed should have properties similar to those outlined for the original lead glass. Fortunately this anticipated lead shortage did not materialise. Consequently work ceased and no completely satisfactory substitute for the lead glass was forthcoming.

However, it was possible as a result of this work to develop iron sealing glasses of higher softening temperature. Thus a glass of composition:—



described by Hull, Berger and Navias (1), was modified by replacing lead oxide by barium and or zinc oxides. These substitutions were made as the previous work on lead glass had shown that it was possible to increase softening temperatures by these means. It was found that either ZnO or BaO added individually tended to cause devitrification but adding both oxides together considerably reduced this effect. Increasing amounts of ZnO caused the expansion coefficients of the glasses to go down without any appreciable change in softening temperature.

As a result of this effect it was possible to produce glasses matching any particular grade of steel. The compositions of some of these glasses are shown in Table 1 together with the expansion coefficients and viscosity values at 450°C.

A particular use of this type of glass is in a windonut on turbine gearboxes where the oil level has to be checked. In this case the glass has a lower expansion than the steel nut and is consequently in compression. The compositions and some properties of the present commercial glass for compression seals together with the present iron matching glass are shown in Table 2.

TABLE 1

*Composition and properties of lead glasses in which PbO has been replaced by BaO and ZnO*

	1	2	3	4
Wt% SiO <sub>2</sub> ...	45	45	45	45
Wt% K <sub>2</sub> O ...	14	14	14	14
Wt% Na <sub>2</sub> O ...	6	6	6	6
Wt% CaF <sub>2</sub> ...	5	5	5	5
Wt% PbO ...	30	—	—	—
Wt% BaO ...	30	23	19	15
Wt% ZnO ...	—	7	11	15
Thermal expansion coefficient (0-400°C)				
x 10 <sup>7</sup> ...	—	133	127	122
Log viscosity at 450°C	10.2	12.5	12.8	12.8



TABLE 2

Composition and properties of commercial iron  
sealing glasses

				(compression seal)	(matching seal to iron)
				X115	X128
Wt%	SiO <sub>2</sub>	...	...	48.2	45.0
Wt%	K <sub>2</sub> O	...	...	11.8	14.0
Wt%	Na <sub>2</sub> O	...	...	5.0	6.0
Wt%	CaF <sub>2</sub>	...	...	5.0	5.0
Wt%	BaO	...	...	19.0	19.0
Wt%	ZnO	...	...	11.0	11.0
Expansion coefficient (50-300°C) x 10 <sup>-7</sup>				112	120
Annealing Point (°C)				480	450

Figure 1 shows an actual "Windownut" compression seal.

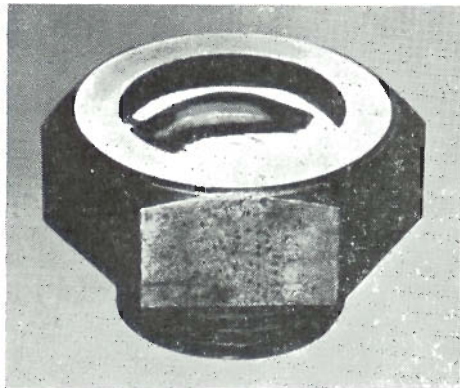


FIG. 1

### 3. Development of Solder Glasses

These glasses were also developed by Dale and Stanworth during the war when it was considered soft glass of good durability would be of value in the construction of miniature valves. The glass would be used to seal an ordinary soda lime glass bulb to a lead glass base. The criteria laid down for this glass were that in addition to good durability under tropical conditions it should be soft enough to seal together the two principal glasses without causing them to deform.

It is well known that borate glasses are very soft compared with silicate glasses and initially glasses in the BaO-ZnO-B<sub>2</sub>O<sub>3</sub> systems were prepared. These glasses were too hard to be of any practical use. The system PbO-BaO-B<sub>2</sub>O<sub>3</sub> was therefore tried next. Here again glasses were either too hard or not sufficiently durable. A detailed study was therefore finally made of

the PbO-ZnO-B<sub>2</sub>O<sub>3</sub> system and it was found that suitable glasses containing small additions of silica could be prepared. There are several glasses of this type now in production. One is used for sealing silicate glasses and another is used for sealing mica windows to soda lime glass. These latter seals are used in the construction of Geiger counters.

The compositions of some experimental glasses and one currently in production are shown in Table 3 together with their softening points.

TABLE 3

Composition and properties of experimental high  
expansion solder glasses and also present  
commercial glass

				PZ6	PZS2	X76
Wt%	SiO <sub>2</sub>	...	...	6.3	5.86	5.0
Wt%	B <sub>2</sub> O <sub>3</sub>	...	...	22.5	14.66	17.0
Wt%	PbO	...	...	63.54	63.77	64.0
Wt%	ZnO	...	...	13.96	16.57	14.0
Expansion coefficient (0-200°C) x 10 <sup>-7</sup>				72.5	76.5	76*
Deformation temperature (°C)				415	395	410
* Expansion coefficient over range 50-200°C						

A natural development of this work was to try to develop solder glasses of lower expansion coefficient which could be used in solder seals with for example, tungsten sealing glass or Fernico sealing glass. A satisfactory solution has evolved in the latter case but so far no completely satisfactory solution has been achieved for the former.

With regard to the latter case it was known from research investigations that zinc borate glasses had low expansion coefficients. It was also known that glasses containing high percentages of vanadium pentoxide were very fluid about 800°C. The glass forming region in the ZnO-V<sub>2</sub>O<sub>5</sub>-B<sub>2</sub>O<sub>3</sub> system was therefore determined and expansion measurements were made on selected glasses in this system. The composition and properties of the present commercial glass which resulted from these investigations are shown in Table 4. It was possible to make satisfactory seals at 650°C with this glass.

TABLE 4

Composition and properties of commercial low  
expansion solder glass (X49BK)

Wt%	B <sub>2</sub> O <sub>3</sub>	...	...	...	28.5
Wt%	Al <sub>2</sub> O <sub>3</sub>	...	...	...	5.0
Wt%	ZnO	...	...	...	57.0
Wt%	V <sub>2</sub> O <sub>5</sub>	...	...	...	9.5
Expansion coefficient (50-300°C) x 10 <sup>-7</sup>					48
Deformation Temperature (°C)					530

Figure 2 shows an evacuated butt seal assembly prepared using this solder glass.

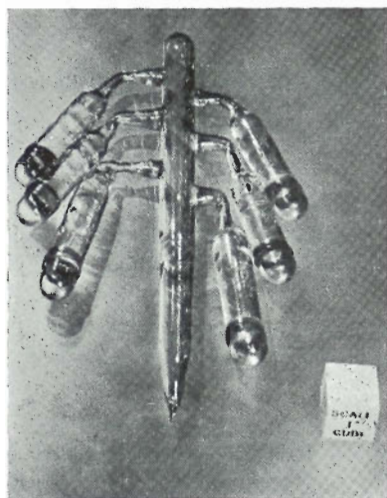


FIG. 2

#### 4. The Development of Commercial Glasses from $B_2O_3$ , $Al_2O_3$ and $P_2O_5$ .

Glasses containing large percentages of  $Al_2O_3$  and  $P_2O_5$  have been known for many years. Until recently, however, the interest in these glasses has been mainly in developing theories of the vitreous state although some glasses containing  $P_2O_5$  have useful UV transmitting and heat absorbing properties. Alumino-phosphate glasses also might find use on account of their resistance to hydrofluoric acid.

Studies were initiated at the Rugby laboratory and were based on work in the system  $Al_2O_3$ - $B_2O_3$ - $P_2O_5$  done by Kreidl and Weyl in America. It was shown that there was only a limited field of glass formation in this system. The introduction of alkaline earth oxides and zinc oxide did however result in alkali free glasses which could be melted rapidly under normal conditions. These glasses had high deformation temperatures and high specific electrical resistances. Their composition and properties are compared with those of "Pyrex" in Table 5.

TABLE 5

Comparison of some experimental glasses in the  $Al_2O_3$ - $P_2O_5$ - $B_2O_3$  system with "Pyrex"

		3a	14	"Pyrex"
Wt%	$P_2O_5$ ...	21.6	33.1	—
Wt%	$B_2O_3$ ...	25.5	14.0	11.9
Wt%	$Al_2O_3$ ...	15.5	15.5	2
Wt%	ZnO ...	9	9	—
Wt%	CaO ...	—	—	CaO
Wt%	MgO ...	16	16	MgO
				$As_2O_3$ } 1.1
Wt%	BaO ...	12.4	12.4	—
Wt%	$Na_2O$ ...	—	—	4.4
Wt%	$SiO_2$ ...	—	—	80.6
Log electrical resistivity				
	at 300°C ...	12.9	12.5	—
Deformation temperature				
(°C) ...		630	635	620
Expansion coefficient				
(50-400°C) x 10 <sup>-7</sup> ...		59	55	32

As a result of this work low expansion borophosphates were developed for use in sodium lamps. Some of the work associated with the development of glasses for sodium discharge lamps is described below.

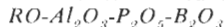
It is well known that normal silicate and borosilicate glasses used in the electrical lamp industry are severely attacked by sodium vapour which causes blackening of the glass. Thus when these glasses are used in the form of sodium discharge tubes a protective layer of sodium resistant glass is needed on the inside of the tube. The glass used should not absorb argon to any appreciable extent as this gas is used together with neon in the sodium lamp to lower the starting voltage.

Considerable skill is needed in making the two ply tubing for sodium lamps and even today sodium discharge lamps are made by hand or by partially mechanised processes. Thus if care is not taken cracking can occur during lamp manufacture or more important still during lamp operation.

Originally work on sodium resistant glasses was done on borate glasses and the expansion of these glasses were matched to a normal soda lime silica glass envelope. It was thought, however, that if sodium resistant glasses could be made to match an outer envelope of lower expansion such as a standard borosilicate glass cracking troubles might be overcome to some extent.

TABLE 6

Some experimental glasses based on the system



	1	2	3	4
Wt% P <sub>2</sub> O <sub>5</sub> ...	21.6	16.0	20.5	15.5
Wt% Al <sub>2</sub> O <sub>3</sub> ...	18.7	23.5	19.8	23.1
Wt% CaO ...	5.0	4.9	4.2	6.0
Wt% MgO ...	13.6	12.0	11.6	12.6
Wt% B <sub>2</sub> O <sub>3</sub> ...	41.1	38.8	40.3	35.6
Wt% SiO <sub>2</sub> ...	—	4.8	2.6	7.1

The preliminary work on aluminoborophosphate glasses looked promising in this respect. Table 6 shows four glass compositions based on the aluminoborophosphate system containing alkaline earths and a small percentage of SiO<sub>2</sub>. Tests on smaller scale melts showed that composition number 1 should be satisfactory and a larger scale melt (10 lb.) was made to produce two ply tubing with C40 borosilicate glass. Unfortunately, however, the glass devitrified, and therefore the glasses from the small and large scale melts were analysed and were of the composition shown in columns 2 and 3. Thus the actual compositions were too far removed from the target composition to be of much use, presumably due to refractory corrosion and volatilisation. Thus corrections were made to the batch and the final glass had the composition shown in column 4. The expansion of this glass was a good match with C40 and satisfactory sodium lamps were made from glass melted on a 50 lb. scale. The composition 1 from which the commercial glass was developed was chosen from the system shown in Figure 3 which gives the glass forming region in the system Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub>-P<sub>2</sub>O<sub>5</sub>-RO.

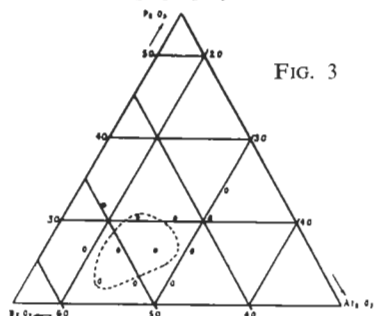


FIG. 3

Fig 1

Theoretical compositions melted in the Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub>-P<sub>2</sub>O<sub>5</sub>-RO system  
 All glasses contain 12.6% MgO and 5% CaO and the (Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub>-P<sub>2</sub>O<sub>5</sub>)  
 content has been calculated to 100. The area enclosed by the dotted line  
 is the region with least tendency to devitrification

It is opportune at this stage to mention the work carried out by Rawson and Elyard at Rugby. These workers made laboratory scale melts of phosphate, borate, aluminoborate, aluminate, and silicate glasses. In brief it was shown that borate and aluminate glasses containing high percentages of modifying oxides were far superior in their resistance to sodium attack than phosphate and silicate glasses. This fact is demonstrated in Table 7 where the compositions and sodium resistance of some of the experimental glasses are shown.

TABLE 7

Compositions and sodium resistance of laboratory prepared glasses compared with commercial glass

	1	2	3	4
Mol% P <sub>2</sub> O <sub>5</sub> ...	65	—	—	—
Mol% SiO <sub>2</sub> ...	—	65	—	—
Mol% B <sub>2</sub> O <sub>3</sub> ...	—	—	65	42
Mol% Al <sub>2</sub> O <sub>3</sub> ...	—	—	—	20
Mol% BaO ...	35	30	35	38
Mol% Na <sub>2</sub> O ...	—	5	—	—
Mol% CaO ...	—	—	—	—
Mol% K <sub>2</sub> O ...	—	—	—	—

Sodium absorption

(mgm/cm<sup>2</sup>/1000 hrs)

at 350°C ... 150 1.9 0.1 0.1

Another result of work in the aluminoborophosphate system was the development of aluminosilicates for certain high temperature applications where conventional borosilicates are likely to deform. Thus a glass of composition and properties shown in Table 8 was developed. This glass has a deformation temperature in excess of 800°C combined with a high electrical resistivity.

TABLE 8

Compositions and properties of A42 glass compared with S96 glass

	A42	S96
Wt% SiO <sub>2</sub> ...	54.5	72.5
Wt% Al <sub>2</sub> O <sub>3</sub> ...	23.5	1.5
Wt% P <sub>2</sub> O <sub>5</sub> ...	3.8	—
Wt% BaO ...	6.3	—
Wt% CaO ...	11.3	6.5
Wt% MgO ...	0.5	3.0
Wt% Na <sub>2</sub> O ...	—	15.8
Wt% K <sub>2</sub> O ...	—	0.5

Expansion coefficient

(50-300°C) x 10<sup>7</sup> ... 43 92

Log. electrical

resistivity at 300°C. 11.9 5.7

Deformation temp. ... 810° 550°

It is not difficult to make glasses of this type having high deformation temperatures but much experimental work is necessary in order that glasses developed have suitable flame working and devitrification properties in addition to high deformation temperatures. The present A42 glass shown in Table 8 has about the highest possible deformation temperature for reasonable ease of melting in the glass works. Its main use is for envelopes of high pressure mercury vapour lamps. Lamps made of this glass can be run horizontally without magnetic control. A42 is also used for chemical combustion tubing which must withstand temperatures in the region of 750°C for long periods without deforming.

### 5. Calcium Aluminate Glass

It is not possible to prepare glasses with alumina as the main glass forming oxide under normal conditions. It was found possible, however, to prepare calcium aluminate glasses under normal conditions on a large scale when a small percentage of  $\text{SiO}_2$  was introduced. Thus we have the glass of composition 1, shown in Table 9. This glass has a very high deformation temperature of 860°C and an expansion coefficient of  $75 \times 10^{-7}$  over the range 50-400°C. Glasses were also prepared containing small amounts of MgO (see for example compositions 2 and 3 of Table 9.) As a result of experiments with compositions such as these the composition of the commercial glass that was finally developed is number 4. This glass has an expansion coefficient of  $84 \times 10^{-7}$  and a deformation temperature of 850°C. It was prepared because there was a need for a glass of high deformation temperature or softening point transmitting infra red radiation in the region of 5 microns (Figure 4). Borate, phosphate and silicate glasses do not transmit in this region. The glass was chiefly developed for windows of infra red detecting equipment. It could not be worked in the form of bulbs or tubes. It was therefore cast in slabs. These slabs could then be sealed to the end of an S96 tube at 550°C using the high expansion solder glass already discussed.

That concludes the description of selected commercial glasses. Other commercial glasses made by the organisation with which the author is associated, that could have been discussed within the scope of this paper, are copper and

molybdenum sealing glasses, various glasses for cathode ray tubes, glasses for germicidal uses, a large number of coloured glasses (22) and also graded seal glasses (12).

Finally, two types of glass that have evolved as a result of pure research at Rugby are described. At present these glasses have not found definite application but it is possible that they will find a use in future. Nevertheless these glasses have been thoroughly examined and their properties listed so that the information is available when and if their use is contemplated. These glasses are described in paragraphs 6 and 7 below.

TABLE 9  
Compositions of experimental and commercial aluminate glasses

	1	2	3	4 (C97)
Wt% $\text{SiO}_2$ ...	6.6	5.8	6.0	6.8
Wt% $\text{Al}_2\text{O}_3$ ...	48.6	39.8	40.8	39.3
Wt% $\text{CaO}$ ...	44.8	49.4	48.2	48.9
Wt% $\text{MgO}$ ...	—	5.0	5.0	5.0
Expansion coefficient (50-400°C) $\times 10^{-7}$	75	—	—	84
Deformation temperature (°C) ...	860	—	—	850

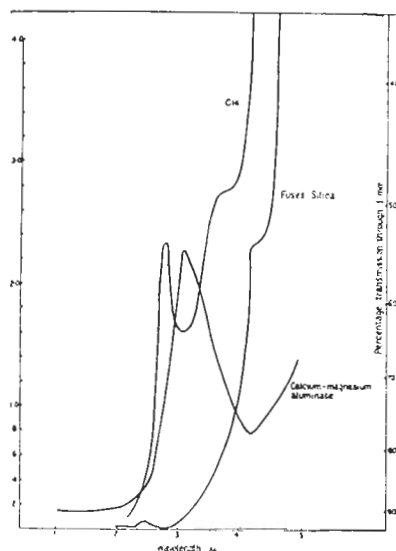


FIG. 4

Continued on page 51



## THE PUREST FUSED SILICA

SPECTROSIL, synthetic fused silica, is readily available in the form of hollow ware, such as crucibles, basins and beakers, and optical components: tubing is supplied in a limited range of sizes. Synthetic fused silica extends the range of commercially available prism spectrophotometers down to 1850Å. Lenses, prisms and windows are offered in SPECTROSIL 'A' and 'B' grades; both are intrinsically the same, in having the highest possible transmissions in the ultra-violet, but the 'A' material is checked against a rigorous optical specification for ultra-violet transmission, fluorescence and uniformity of refractive index. Both grades are free from bubbles and inclusions.

Total metallic impurities are less than 0.2 parts per million.



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## ABSTRACTS

### BURETTES

(87) Free Piston Pipettes and Burettes—Recent Developments.

Smith, I. C. P., *J. Brit. Soc. Sci. Glassblowers*, Vol. 1, No. 3, 1964, 41-2.

Short account of lecture given to Southern Section of B.S.S.G., deals with accuracy and tolerances. D.W.S.

### CEMENTS

(88) C.G.W. has Solder Glass Kit.

Anon., *Nat. Glass Budg.*, Vol. 79, No. 15, 1963, 12-3. The kit consists of a powder of solder glass in the expansion range of 8–10 x 10<sup>-6</sup>/°C and a nitrocellulose solution in amyl acetate. The components are mixed to give a thick paste which is applied to the glass tube over the area to be joined. After firing, the paste forms a permanent vacuum-tight seal. S.G.Y.

### CERAMICS

(89) Some Recent Developments in High Strength Glasses and Ceramics.

Shaver, W. W., *Proc. Roy. Soc. A.*, Vol. 282, No. 1388, 1964. A.J.L.-M.

### CHROMATOGRAPHY

(90) Extraction Apparatus for Gas Chromatographic Micro-syringes.

Ott, D. E., Gaston, L. K., and Gunther, F. A., *J. Gas Chrom.*, Vol. 2, No. 7, 1964, 223.

A microsyringe washer has been designed to operate safely and continuously in the laboratory yet permitting easy introduction or removal of a single syringe. Based on the Soxhlet extraction principle, drawing. D.A.H.

(91) Micro Determination of Dithiocarbamates by Gas Chromatography.

Corrado Bichi, *Chrom. J.*, Vol. 14, No. 3, 1964, 353. Photograph of the manifold for the introduction of the samples. Apparatus for the distillation of CS<sub>2</sub>—drawing, also trap to collect liquid CS<sub>2</sub> in liquid air. E.G.E.

(92) Photoionization Detector for Gas Chromatography.

Mikaya Yamane, *J. Gas. Chrom.*, Vol. 14, No. 3, 1964, 355.

Photograph and good descriptive drawing of construction of photoionization detector. E.G.E.

### COMBUSTION

(93) A Wet-Combustion Method for Carbon-14 Analysis.

Kuyper, A. C., Cortese, T. A., and Aghdashi, M., *Anal. Biochem.*, Vol. 8, No. 2, 1964, 272-5.

This method reduces the time needed for the analysis of a sample and simplifies the apparatus. A drawing and explanation is given. D.W.S.

### CRYOSTATS

(94) A Cryostat for the range -60°C to 20°C.

Mucel, M. A., and Singer, N., *J. Sci. Instrum.*, Vol. 41, No. 8, 1964, 506.

Description with a drawing of a small laboratory cryostat which can be left unattended for up to 9 hrs. Control of temperature to 0.05°C between -55°C to 20°C is achieved. D.W.S.

### ELECTRODES

(95) New Electrodes for Chromopotentiometry in thin layers of solutions.

Hubbard, A. T., and Anson, F. C., *Anal. Chem.*, Vol. 36, No. 4, 1964, 723.

Five figure drawings in detail incorporating Glass to Metal Seals and sintered ware. E.G.E.

(96) Capillary Electrometer-Sippman.

Mulher, O. H., *J. Chem. Education*, Vol. 41, No. 6, 1964, 321.

For Polarographic examination of Hg drops, illustration and description. F.G.P.

### EVAPORATION

(97) Analytical Applications and Construction of Long Tube Vaporizers.

Ash, B. O., *Anal. Chem.*, Vol. 36, No. 7, 1964, 1363. Detailed drawings of Cyclone Separator and long tube vaporizing system. E.G.E.

(98) Forced Volatilization Clean-up of Butterfat for Gas Chromatographic Evaluation of Organochlorine Insecticide Residues.

Ott, D. A., and Gunther, F. A., *Ag. and Food Chem.*, Vol. 12, No. 3, 1964, 240.

Shows photographs of three pieces of apparatus: 1. The basic all-glass unit for forced volatilization, 2. A taped Heater for unit for 1.

3. Design of Scrubber for trapping emergent volatiles. E.G.E.

### FLOWMETERS

(99) The Accuracy of the Bubble Meter Method for Gas Flow Measurements.

Levy, A., *J. Sci. Instrum.*, Vol. 41, No. 7, 1964, 449-53.

This discusses the standards needed for this type of flowmeter and the size and shape of the tube. Errors can be kept below ±0.25%. D.W.S.

### FURNACES

(100) 250 KW Electron Beam Furnace.

Anon., *Elect. Rev.*, 14th August, 1964.

A.J.L.-M.

### GLASS ANALYSIS

(101) The Development of a New Form of Hempel Pipette.

Layzell, B., *J. Brit. Soc. Sci. Glassblowers*, Vol. 1, No. 3, 1964, 44.

Gives drawing and reasons. D.W.S.

### GLASS MANUFACTURE

See 89.

### GLASS—PHYSICS

(102) Sources of Weakness in Glass.

Gurney, C., *Proc. Roy. Soc. A.*, Vol. 282, No. 1388, 1964.

A.J.L.-M.

(103) Properties of Porous Sintered Glass.

Schönborn, H., *Silikat Tech.*, Vol. 13, No. 12, 1962, 419-24.

Difficulties encountered in sealing porous sintered plates to ordinary glass tubes were investigated by measuring the thermal expansion of cubes made from Rasotherm glass powder. The Fizeau-Pulfrich type of dilatometer was used. The thermal expansion of the sintered bodies was up to six times that of the bulk glass and showed a sudden rise at 200°C, indicating the presence of crystallite. The results lead to the conclusion that the increase in expansion is caused by the growing crystallite crystals. S.G.Y.

See also 89.

### GLASSWORKING—MACHINES

(104) Manufacture of Lamp Bulbs from Tubing.

Mickley, E., and Thomas, M., *Glas. Tech. Ber.*, Vol. 36, No. 7, 1963, 273-7.

Economical mechanical manufacture of small glass bulbs from glass tube. The machines and forming processes and difficulties encountered are discussed. A machine is mentioned having 36 heads. The curvature of the tubing caused difficulties but a method of supporting the tubing corrected the fault. S.G.Y.

(105) Chucking with Planetary Bar Chucks—An Application Note.

Litton, C. V., *Fusion*, Vol. 11, No. 3, 1964, 9. Short article on how to chuck a long and heavy glass cylinder. D.W.S.

## GLASSWORKING—METHODS

### (106) How to make $\frac{1}{8}$ " to $\frac{1}{4}$ " Solid Glass Balls.

Lorimer, R., *Fusion*, Vol. 11, No. 3, 1964, 3.  
Gather the end of a glass rod in a flame to form a ball, tack on a piece of rod, flame cut off from original rod, crack off, grind. D.W.S.

### (107) Copper Moulds.

Anon., *Glass Age*, Vol. 6, No. 3, 1963, 56.  
Copper alloys containing Ni, Al, Fe and Co have good oxidation and thermal shock resistance, high hardness and tensile strength with good machinability and surface finish. Advantages of the alloy over cast iron moulds are claimed for casting glass. S.G.Y.

### (108) Transfers or Lithographs. A New Material for the Decoration of Glass or Ceramic Materials.

Baumer, H., *Sprechsaal*, Vol. 96, No. 8, 1963, 165-6.  
The advantages of lithographs compared with transfers are discussed. The former can be applied automatically and the quality of the pattern need not be impaired. S.G.Y.

## GLASSWORKING—TOOLS

### (109) Some Effects of the Shape and Structure of Diamond Particles on Grinding Efficiency.

Seal, M., *Engelhard Tech. Bull.*, Vol. 4, No. 1, 1963, 9-19.

Natural diamond, metal or resin-bonded wheels can wear in two ways. The diamonds themselves may wear, so that flat areas are developed on the wheels, or the wheels may wear through loss of diamonds. Remedies for these occurrences are discussed, also the behaviour of synthetic diamonds in a wheel. S.G.Y.

## GRADUATION

### (110) Graduation Lines on Borosilicate Glass.

Smith, I. C. P., *J. Brit. Soc. Sci. Glassblowers*, Vol. 1, No. 3, 1964, 43.

Short account of lecture to Southern Section of B.S.S.G. Discusses use of HF, HF/H<sub>2</sub>SO<sub>4</sub>, HF/Ammonium bifluoride. D.W.S.

## MANOMETERS

### (111) A Differential Mercury Manometer.

Hanley, H. J. M., *J. Sci. Instrum.*, Vol. 41, No. 7, 1964, 486.

This consists of a U-tube with wide limbs but the flat-bottomed U-bend of capillary bore. An air bubble is trapped in the bend, the mercury is lubricated with 100% H<sub>2</sub>SO<sub>4</sub> for sensitivity. D.W.S.

## PIPETES

See 87, 101.

## POLISHING

### (112) Etching Polished Depressions in Glass Plate.

Kendall, P. W., *J. Sci. Instrum.*, Vol. 41, No. 7, 1964, 485.

Uniform polished depressions up to 25  $\mu$ m deep in polished "Pyrex," soda-glass and Quartz plates can be produced by HF etching. D.W.S.

## PUMPS—CIRCULATING

### (113) A Circulating Pump for Vacuum Systems.

Eden, C., *J. Sci. Instrum.*, Vol. 41, No. 9, 1964, 572.  
This article with a sketch describes a modified Sprengel Pump. D.W.S.

## PUMPS—ION

### (114) A Compact Titanium Vacuum Pump.

Ponomarev, U. P., *Instrum. and Experimental Techniques* (translation June, 1964) No. 6, 1963, p. 1147.  
This article describes a Titanium ionic-sorption pump for removing air with a pressure of 10<sup>-6</sup> to 10<sup>-7</sup> Torr at 40 L/sec. D.A.H.

## SEALS—GLASS TO METAL

(115) Kovar Glass Sealing Alloy from Metal Powder. Thornbury, D. R., *Glass Industry*, Vol. 44, No. 7, 1963, 384-5 and 410.

The use of powder metallurgy techniques leads to finer grain size, better composition control, fewer inclusions from trace elements and less scrap. Offers a tool for overcoming certain material problems. S.G.Y.

### (116) Glass-Sealing Alloys.

Eberly, W. S., *Glass Industry*, Vol. 48, No. 8, 435-8 and 466-7, 1963.

The author lists the standard glass to metal sealing alloys and discusses the factors to be considered in design to form perfect gas-tight glass seals to metal. See also 95. S.G.Y.

## SILICA

### (117) Production of Silica Glass by Vacuum Sintering of Quartz Powder.

Moriya, Y., *Vacuum*, Vol. 13, No. 2, 1963, 47-52.  
Investigation into the production of a transparent silica glass ingot from 60 mesh quartz powder in a vacuum furnace at 1720°C. S.G.Y.

### (118) The Preparation of Pure Silica.

Nikitina, O. N., *Soviet Res. Glass Ceram. Consultants Bur. Transl.* 1957-58, Part 2, 263-4.

Research into preparation of a sample of pure silica for use as a spectroscopic standard. Prepared by the hydrolysis of purified Si Cl<sub>4</sub> in ice cooled platinum vessels, the sample is then heated to 1100°C for two hours. S.G.Y.

## MISCELLANEOUS

### (119) Constant Level Oil Bath and Melting Point Apparatus.

Powell, H., and Burow, D., *Chem. Education*, Vol. 41, No. 6, 1964, 345.

Modification of Thiele and Deris M/P apparatus. Illustration and description of use. F.G.P.

### (120) History of the Warbourg Apparatus.

Oesper, P., *Chem. Education*, Vol. 41, No. 6, 1964, 295.

Development and use of Warbourg apparatus from 1897 to date. F.G.P.

### (121) Vitreous Calcium Metaphosphate — Some Properties of its Aqueous Systems.

Brown, E. H., and Lehr, J. R., *Ag. and Food Chem.*, Vol. 12, No. 3, 1964, 202.  
Details given of apparatus. E.G.E.

### (122) High Dilution Apparatus.

Eglinton, G., and McCrae, W., *Advances in Organic Chem.*, Vol. 4, 1964, 313.

For coupling of acetylenic compounds. Illustration and description of use. F.G.P.

### (123) Fifty Years of the Gas Filled Lamp.

Lokker, J. C., *Philips Tech. Rev.*, Vol. 25, No. 1, 1963-4, 2-15.

After a brief history of the development of the incandescent lamp, a short account is given of the theory underlying Langmuir's invention. Problems of manufacture are discussed. Review of subsequent developments in use of inert gas and coiled-coil filament. D.A.H.

### (124) An Internal Circulating Method for Growing Water Soluble Crystals.

Oliver, C. B., and White, E. A. D., *J. Sci. Instrum.*, Vol. 40, No. 12, 1963, 598-9.

Details of apparatus and techniques used for growing crystals from small volumes of liquid. D.W.S.

### (125) New Tools for Sub-Atomic Searches.

Anon., *Engineering*, Vol. 197, 1st May, 1964, 606-8.  
Briefly describe "Nimrod" and its work. D.W.S.

### (126) "Nimrod" 7GeV Proton Synchrotron.

Anon., *The Engineer*, Vol. 212, 1st May, 1964, 776-7.  
A short account of "Nimrod" is given, with a brief description of its method of working. D.W.S.

### (127) Releasing Siezed Stopcocks and Ground Glass Joints.

Stockton, J., *J. Brit. Soc. Sci. Glassblowers*, Vol. 1, No. 3, 1964, 40.

The article emphasises the need to cool the inner member and heat the outer, suggestions of ways are given. D.W.S.

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## 6. Tellurite Glasses

The first group of glasses are glasses based on  $\text{TeO}_2$  as the glass former. These glasses (tellurites) were first prepared and investigated by Stanworth and his colleagues. Initially binary systems of  $\text{TeO}_2$  with  $\text{BaO}$ ,  $\text{PbO}$ ,  $\text{WO}_3$  and  $\text{MoO}_3$  were examined. A number of ternary systems were then examined which included in addition to the oxides already mentioned  $\text{SiO}_2$ ,  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{P}_2\text{O}_5$ ,  $\text{V}_2\text{O}_5$ ,  $\text{As}_2\text{O}_5$  and  $\text{ZnF}_2$ . Small amounts of  $\text{MnO}_2$ ,  $\text{CaO}$ ,  $\text{TiO}_2$ ,  $\text{GeO}_2$ ,  $\text{La}_2\text{O}_3$ ,  $\text{CeO}_2$  and  $\text{ZrO}_2$  were added to some glasses.

The compositions and properties of some binary lead tellurite glasses are shown for example in Table 10. It can be seen that in general the refractive indices, densities and expansion coefficients are high and deformation temperatures low. Tables 11 and 12 show the compositions and properties of some three component glasses based on lead tellurite melts. It can be seen that most of the remarks made about the properties of the ternary lead tellurite glasses apply to the ternary glasses also. The glasses generally had very good infra red transmission properties (Figure 5).

TABLE 10  
*Properties of PbO-TeO<sub>2</sub> glasses*

Glass No.	PbO	TeO <sub>2</sub>	Percentage SiO <sub>2</sub> by analysis	Crucible	Refractive index (approx.)	Density	Thermal expansion coefficient x 10 <sup>7</sup>
6	80	20	4.39	S	2.22	7.24	171
7	80	20	4% added	A	2.25	—	—
15	25	75	0.1	S	2.21	6.22	182
17	22	78	—	S	2.25	6.15	177
18	22	78	—	A	2.25	—	—
22	18	82	—	Z	2.20	6.05	185

TABLE 11  
*Composition and properties of three component glasses based on lead tellurite*

Weight per cent TeO <sub>2</sub>	...	85.9	73.25	72.3	74.7
Weight per cent PbO	...	12.0	22.7	22.4	21.4
Weight per cent third component	...	2.1 MgO	4.05 TiO <sub>2</sub>	5.3 GeO <sub>2</sub>	3.9 La <sub>2</sub> O <sub>3</sub>
Expansion coefficient (50-200°C) x 10 <sup>7</sup>	...	170	166	169	177
Deformation temperature (°C)	...	315	322	315	309
Density (g/cc)	...	5.773	6.0295	6.071	
Refractive index for 5780Å		2.16	2.24	2.17	

TABLE 12  
*Composition and properties of three component glasses based on lead tellurite*

Weight per cent TeO <sub>2</sub>	...	80.4	88.2	86.4	42.5	44.1	63.5	33.2	66.3
Weight per cent PbO	...	13.5	9.8	9.6	29.6	42.5	22.2	18.5	23.2
Weight per cent third component	...	BaO	Li <sub>2</sub> O	Na <sub>2</sub> O	B <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>3</sub>	MoO <sub>3</sub>	WO <sub>3</sub>	ZnF <sub>2</sub>
	...	6.1	2.0	4.0	27.9	13.4	14.3	48.3	10.5
Expansion coefficient (50-200°C)	...	175	197	215	102	154	179	127	173
Deformation temperature (°C)	...	305	271	276	404	335	316	399	282
Density (g/cc)	...	5.927	5.564	5.465	4.413	5.994	5.933	6.758	6.077
Refractive index for 5780Å		2.17	2.14	—	1.82	2.04	2.04	2.21	2.10

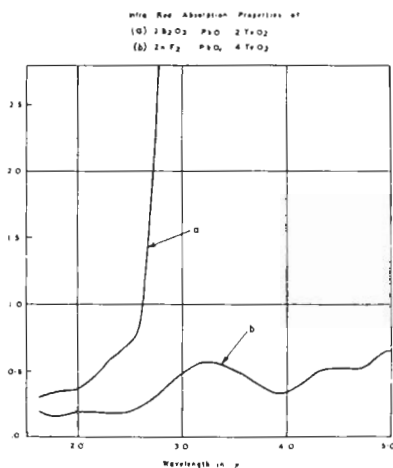


FIG. 5

One particular thing against the use of tellurite glasses is the fact that  $\text{TeO}_2$  sublimes when melted emitting an extremely noxious vapour, which, unless precautions are taken, is absorbed by the worker and still emitted by him after several weeks. Indeed, early workers with crystalline  $\text{TeO}_2$  were complete social outcasts as a result of this smell.

## 7. Vanadate Glasses

Here again a general investigation into the glass forming behaviour of  $\text{V}_2\text{O}_5$  with various secondary components was inaugurated at Rugby by Stanworth and his co-workers.

It was found possible to make glasses containing large proportions of  $\text{V}_2\text{O}_5$  with one other oxide. The other oxide could be a glass former such as  $\text{P}_2\text{O}_5$ ,  $\text{GeO}_2$ ,  $\text{TeO}_2$  or  $\text{As}_2\text{O}_3$ . Non-glass forming secondary oxides included  $\text{BaO}$  and  $\text{PbO}$ .

The glasses prepared were all black and hence opaque to visible radiation and they had low softening points. The most interesting property of these glasses is the low electrical resistivity that they possess. Thus a glass of composition 90%  $\text{V}_2\text{O}_5$  10%  $\text{P}_2\text{O}_5$  had a resistivity of  $10^{4.3}$  at room temperature which is much lower than

that of any other known glass. It is considered that these glasses are semiconductors as no polarisation effects are observed when direct current is passed through the glasses and a marked thermoelectric effect is also observed. It is very likely that if any good use is made of vanadate glasses it will be on account of their semiconducting properties. The variation of log. resistivity with  $\text{V}_2\text{O}_5$  content in the binary  $\text{P}_2\text{O}_5$ - $\text{V}_2\text{O}_5$  system is shown in Figure 6.

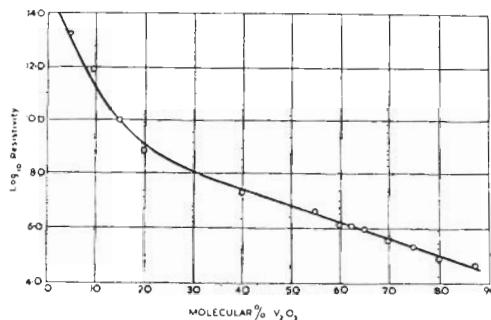


FIG. 6

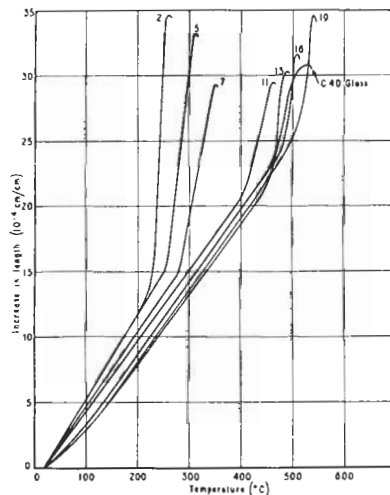


FIG. 7

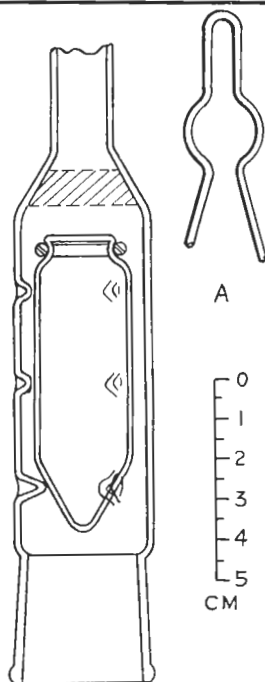


The expansion coefficients of the vanadate glasses containing major proportions of  $V_2O_5$  are somewhat higher than those of conventional soda lime silicate glasses, usually being greater than  $100 \times 10^{-7}$ . One of the most remarkable things about the expansion curves of these glasses is the very steep neck between the transformation point and the deformation point. This effect is demonstrated in Figure 7.

This section on vanadate glasses completes a necessarily brief description of a selected number of glasses. One point that emerges from this description is the large number of glasses of diverse properties that the electrical industry need in order to keep abreast of new developments and new devices in which glass is a vital component. As previously mentioned, the industry is always striving to improve existing glasses; but whether developing new glasses or improving existing ones, a close liaison between research, development and production personnel is always necessary if suitable products are going to be forthcoming.

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## WORKSHOP NOTES

### A NON-RETURN FLOAT VALVE FOR VACUUM PUMPS

I. C. P. Smith, E.R.D.E., Waltham Abbey

THOSE who use the old type oil-sealed rotary vacuum pumps should take precautions to see that oil is not sucked back into the vacuum system when the pump is turned off, but nevertheless this does happen occasionally and glassblowers have to spend valuable time opening up, cleaning out and resealing the apparatus. The figure shows a simple non-return float valve which can be placed immediately above the pump and will provide against most accidents. The construction is simple, and differs from previous practice in such valves in that an "O" ring is used to make the seal, instead of the two ground glass surfaces, which may fail through faulty manufacture, causing incorrect bedding, or the presence of foreign matter.

The construction of the valve will be fairly clear from the figure, which is to scale, and is based on a B.29 socket, but any preferred finish may be applied to either the upper or the lower end. The following points should, however, be noted. The upper shoulder should be made with about  $60^\circ$  taper, and ground and smooth ground inside. A "tool" for this can be made by shaping the end of a piece of 24 or 25 mm. tubing similar to the lower end of the float as drawn. The body is given two rows of three dimples to locate the float easily, and finally three more deep ones when the float has been inserted. These should allow about 10 mm. of vertical movement for the float.

24 mm. light wall tubing is suitable for the float and the "O" ring size No. 115 (O.S. 13) made from neoprene or silicone rubber. The channel for the "O" ring should be quite shallow, at its upper side not more than half the depth of the ring section and the glass shaped here so that it falls away rapidly and cannot touch the body seating instead of the "O" ring. Circularity of the channel relative to the axis is important and it should be lapped using a thick copper wire bent as shown (A) and finished with fine carbo. The root diameter should be about 18 mm. Centrifugal forming would probably be adequate. Naturally the completed valve cannot be furnace annealed, but flame annealing at each stage is sufficient.

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## SECTION ACTIVITIES

### *Southern Section*

**T**HE Southern Section held the first meeting of the 1964-1965 season in the main lecture theatre, Chemistry Department, Queen Elizabeth College, Campden Hill Road, London, W.8, on Wednesday, 14th October, 1964. Mr. G. Isaacs of The Royal Radar Establishment, Malvern, was guest speaker. The Section was very pleased to have a founder member of the Society give the first lecture in the 1964-1965 programme.

Mr. Isaacs gave a brief outline of the various types of glass to metal seals as an introduction to his lecture on "Micro-Wave-Tube Techniques." The speaker went on to describe in great detail the many intricate techniques used in the fabrication of micro-wave tubes.

On Wednesday, 11th November, 1964, in the main lecture theatre, Queen Elizabeth College, Campden Hill Road, London, W.8. Mr. J. Frost of the University of Reading entertained the Southern Section with a lecture on "Strain in Glass." Mr. Frost can always be relied upon to provide an interesting lecture and this occasion was no exception.

With the aid of several demonstrations, some rather spectacular, the speaker explained the complex subject of the strain in glass. Question time contained many interesting questions, and a lively discussion followed.

The next meeting of the Southern Section will be held on Wednesday, 9th December, 1964, in the main lecture theatre, Chemistry Department, Queen Elizabeth College, Campden Hill Road, London, W.8. The lecture will be on "Basic Glassblowing Principles" by J. Burrow, Esq., Bristol University.

### *Thames Valley Section*

Our Secretary Mr. A. G. Thompson has taken up a new appointment in the North and the section wishes him good luck in his new position. Mr. M. Priem will be acting Secretary.

A meeting took place on 23rd October when a lecture on "Ultrasonics" was given by Mr. B. F. Green of Kerry Ultrasonics.

A film show has been arranged for the December meeting and a series of lectures for 1965 is being planned.

### *Western Section*

The main activity of the section has been to organise the Annual Symposium on behalf of the Society. Lectures and demonstrations on Fused Silica were by Thermal Syndicate Ltd. and the recorded attendance was 151. Thanks are expressed to T.S.L. for the excellent content of their lectures and exhibits.

Officers of the section are now: Chairman, Mr. R. A. Redford; secretary, Mr. D. W. Smith; treasurer, Mr. D. A. Jones.

### **1964 FUTURE PROGRAMME**

#### **22nd December**

Dinner at the Hawthornes Hotel, Bristol. Members and their ladies.

#### **1965**

#### **25th January**

The Use of a Glassblowing Lathe, by J. H. Burrow (University of Bristol), Glassblowing Workshop, Department of Physics, University of Bristol, 7.30 p.m.

#### **22nd February**

Brazing and Soldering, by B. W. D. Harris (Research Department, Imperial Tobacco Co. Ltd.), Glassblowing Workshop, Department of Chemistry, University of Bristol, 7.30 p.m.

### *North-Western Section*

On Wednesday, 6th November, a general meeting was held in the Warrington Technical College. Rules and education formed most of its discussion time, and also some provisional arrangements for a social evening for members and their wives at a night club were made.

A definite date has now been fixed for the North Western Annual General Meeting which will be held on Friday, 20th November, in the Committee rooms of the White Hart Hotel, Warrington.

### **SUBSCRIPTION REMINDER**

Members will appreciate that in a small Society such as ours every member's subscription is needed and prompt payment avoids extra work for those who run the Society and in addition assists in a more accurate assessment of the funds which will be available in the next financial year.

We therefore wish to point out that 1965 subscriptions are now due and should be forwarded to the Treasurer, Mr. D. A. Henson, cheques and postal orders being made payable to BRITISH SOCIETY OF SCIENTIFIC GLASSBLOWERS.

## ADDITIONS TO LIST OF MEMBERS

### *Full Members*

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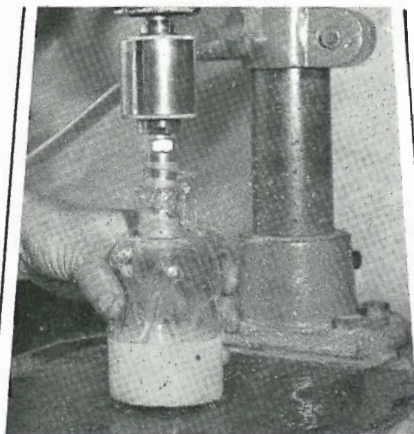
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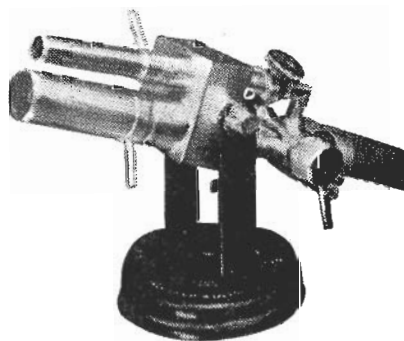
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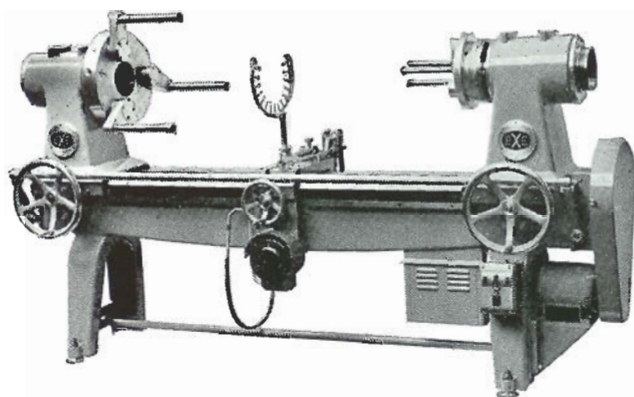
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