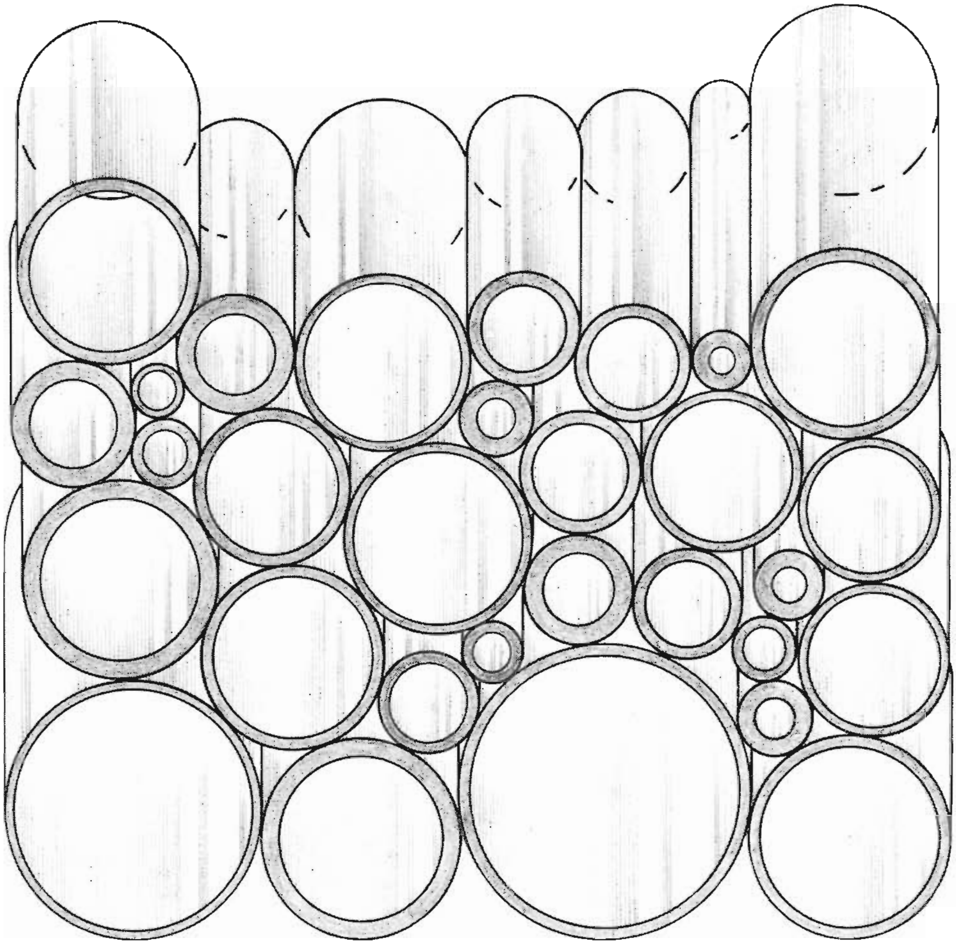
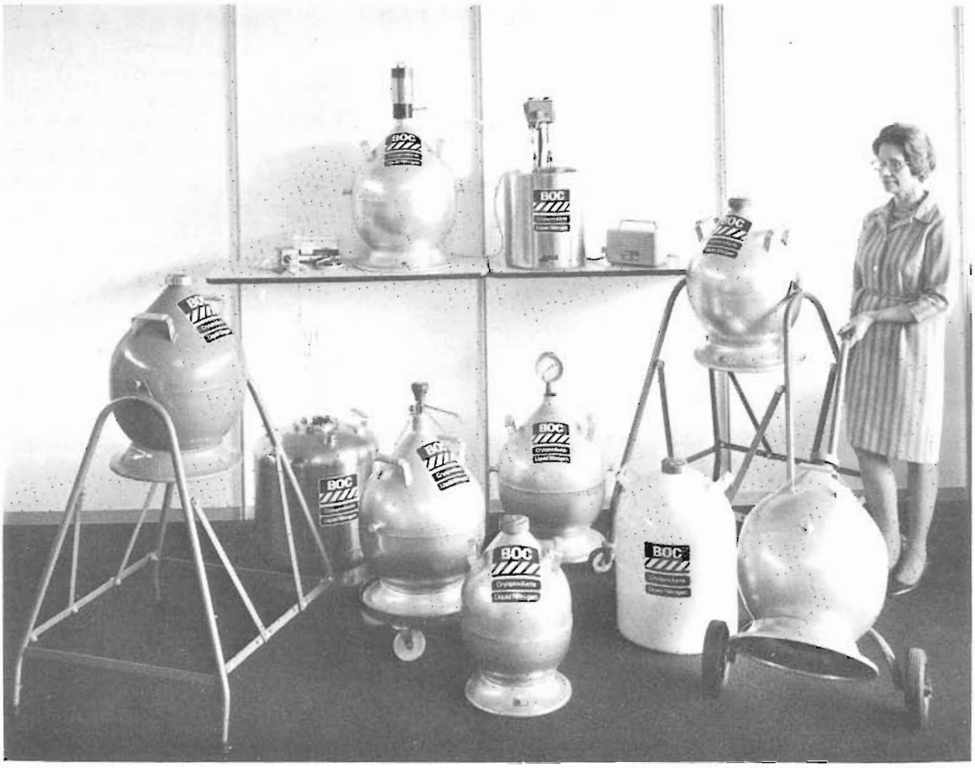
British Society of
Scientific Glassblowers Journal

Vol. 12
JULY 1974
No. 3




BOC

YOU CAN RING US
24 HOURS A DAY
WE'VE INSTALLED A
ROBOPHONE

HON. SEC.
WINKFIELD ROW 3639

OFFICERS OF THE SOCIETY

Chairman R. J. W. Harvey,
73 Long Lane,
Hillingdon,
Middlesex.

Hon. Treasurer G. Robertshaw
83 Beckwith Road,
Harrogate,
Yorkshire.

Hon. Secretary R. Mason,
53A Kennel Ride,
Ascot,
Berkshire.

BOARD OF EXAMINERS

Chairman K. Holden,
School of Molecular and Bio Science,
University, Warwick.

Hon. Secretary N. H. Collins,
8 Holden Terrace,
Waterloo,
Liverpool 22.

Competition Secretary D. Newell,
3 Stratton Gardens,
Southall,
Middlesex.

OVERSEAS MEMBERS AND VISITORS

The British Society of Scientific Glassblowers extend a warm welcome to visitors to the United Kingdom and will be pleased to assist in arranging visits to industrial and other establishments, if this is desired.

It is necessary that early notice is given to the Hon. Secretary . . . . giving,

time and date and place of arrival.
address whilst in the United Kingdom,
length of stay.

Distribution Mr. R. Mason,
53A Kennel Ride,
Ascot,
Berks.

Advertising Manager Mr. C. H. Glover,
"Saraphil", Highfield Lane,
Cox Green,
Maidenhead,
Berks.

British Society of Scientific Glassblowers

Founded 1960

President: Mr. STAFFORD SCHOLES
Hon. Secretary: R. Mason, 53A Kennel Ride, Ascot, Berks.

EDITORIAL STAFF

Vol. 12 July 1974 No. 3

R. E. GARRARD
F. G. PORTER

Journal of the B.S.S.G.
School of Chemistry,
University of Bristol.

CONTENTS

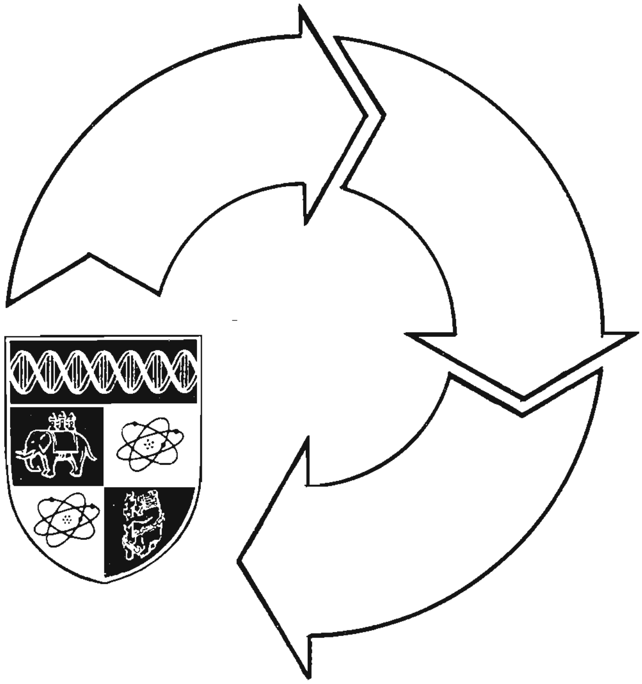
The Journal is published quarterly by the B.S.S.G. and is available free to members and at £1.00 per copy (or £4.00 per annum, airmail extra charge) to non-members. A limited number of back copies are available.

Printed in Gt. Britain by Mergewise Ascot.

SYMPOSIUM 75

SEPTEMBER
11·12·13

UNIVERSITY
OF
WARWICK



PAPERS WANTED
REWARD
AN HOUR OF INTEREST

The committee would be pleased to receive from any member a paper for delivery at the 1975 Symposium, or a proposal of a lecturer who may offer a paper of particular interest to the members.

The Symposium secretary would be pleased to receive a brief synopsis of material for consideration at the next committee meeting to be held toward the end of 1974.

Keith Holden Symposium Secretary
School of Molecular and Bio Science, University of Warwick, Coventry.

METHODS FOR THE ANALYSIS OF NITROGEN OXIDES: THE PRESENT STATE OF THE ART

MISS. L.M. BEDDOWS AND A. WHITE

SUMMARY — This paper describes the present state of the art concerning methods for the determination of nitrogen oxides. Some special emphasis is given to useful techniques and some difficulties encountered during sampling and analysis are reported.

CONTENTS

Introduction

The methods available for the determination of nitrogen oxides can be classified in several ways: as wet chemical and instrumental methods, as batch and continuous methods, or as primary and secondary methods. In practice these describe similar groups, i.e. the wet chemical methods are generally batch methods, and the instrumental methods are in most cases continuous. It must, however, be borne in mind that an instrument needs to be calibrated against a gas accurately analysed by another method.

The threshold limit values [1] (5vpm for nitrogen dioxide, 25vpm for nitric oxide) for the tolerable levels in ambient atmospheres provide an indication of the quantity that may be required to be measured in air. In flue gases values up to 600vpm [2] have been reported whilst concentrations in vehicle exhausts can exceed 5000vpm [3].

1. Wet Chemical Methods

Most of these [4] are based on the Griess?Ilosvay procedure which involves a diazotisation step and coupling reactions of the nitrite in solution. The Saltzman method [5] is preferred by a number of authorities, although there are numerous modifications, an example being that by Stratmann and Buck [6].

Sodium nitrite has been relied upon as a standard because of the difficulty of providing gaseous standards of accurately known composition. Much work has been published [7],[13], [14] concerning the value of the factor which must be used to determine the equivalence of the nitrite in solution in terms of nitrogen dioxide gas and whilst many workers accept a value of 0.72 there is evidence that this figure is not always applicable in practice. Values between 0.5 and 1.0 have been reported. Experiments have indicated that the value may be mass flow dependent; if this is true, different values would be found when making measurements on widely different concentrations of nitrogen oxides. There is general agreement that the value tends to unity at extremely low concentration.

The methods measure only nitrogen dioxide and estimation of nitric oxide is usually achieved by oxidation to nitrogen dioxide. An acid permanganate oxidiser [12] is convenient, although several others have been described [15], [36] but the choice may be determined by other factors such as portability, sample flow rate and the concentration of nitrogen oxides.

The Gas Council analytical method [16] recommended for the analysis of nitrogen oxides is also based on the Saltzman method, and is preferred to the British Standard (BS 1756) which is not suitable for use outside a laboratory. The ASTM method (D1607-60) for atmospheric nitric oxide and nitrogen dioxide is a modified Griess-Ilosvay technique but for total nitrogen oxides in flue gases the ASTM recommends the phenoldisulphonic acid method [17]. It has been reported that the latter is inconvenient and is best suited to higher concentrations [18]. However it has also been said that this is the only true primary method [25]. A method for measurement of nitrous fumes in ambient atmospheres [37], suitable for use in all locations, has been published under authority of the Department of the Environment.

The advantage of wet chemical methods is their simplicity and low cost. Their disadvantage for site work is the time taken in sampling (15 minutes) and to complete the test (a total of 25-30 minutes). It is thus inevitable that an averaged result is obtained. A further disadvantage is that nitrogen dioxide absorption in solution results in the evolution of a small amount of nitric oxide which has to be reoxidised and reabsorbed in a second bubbler. The loss from a similar mechanism in the second bubbler is negligible.

2. Instrumental Methods

The advantage of an instrumental method lies in the speed of analysis and the facility for continuous monitoring, which can seldom be achieved by wet chemistry. Suitable instruments have been reviewed [19], [36] and there are several types, which operate according to different physical principles.

2.1 Electrochemical Transducers

Several commercial instruments are available from the U.S.A. One is marketed by Envirometrics Inc. and another by the Dynasciences Division of the Whittaker Corporation.

Nederlands Gasunie N.V. have tested two types of Whittaker Instrument and their reports [18], [21] indicate that the instruments (Models NR 230, NX 130) are reasonably satisfactory although there is a need for frequent calibration and temperature compensation. For the newer Dynascience model NX 130, Gasunie found that the response of the instrument changed with NO/NO_2 ratio, moreover, there was interference by sulphur dioxide. These two disadvantages seem to limit use to the laboratory.

Attempts by the manufacturers to overcome SO_2 interference by scrubbing and by electronic correction have not proved entirely successful, and these results have been confirmed by U.S. users. However, even if interference effects and mixed response problems can be overcome, the very slow response time of the instrument (minutes) means that its use other than as an averaging device is limited.

2.2 Infra Red Analysers

(a) Non-dispersive infra red (NDIR) spectroscopy

The method is only applicable to NO but instruments based on the principle are currently widely used throughout the motor industry, since the concentration in exhaust gases is generally higher than from sources not connected with internal combustion. Instruments with ranges up to 4000 ppm or more are available. There is interference by water [18], [20] so that gas drying is essential. Complications are introduced if chemical drying agents are used since, for instance, 'Drierite' can generate NO from NO_2 and 'Aquesorb' may remove NO. Shell Research Limited have used refrigeration, (by means of solid CO_2 traps at $-78^{o}C$) which appears to be an acceptable means to avoid chemical interaction of NO with the drying medium.

(b) Dispersive infra red (DIR) spectroscopy

Long path (40 metre multiple reflection) dispersive infra red absorption has been used for the measurement of low concentrations of NO_2 in the atmosphere [21]. The technique has also been used for the measurement of NO in car exhausts [20]. However, the equipment is liable to be expensive and difficult to use and not therefore suitable for either routine or field work.

The technique of emission infra red spectroscopy has been used for the special application of monitoring NO concentrations at high temperatures in engine cylinders [23].

2.3 Ultra Violet Methods

(a) Non-dispersive ultra violet (NDUV)

The technique only measures NO_2 but an instrument is available commercially (Beckman Instruments Limited) with a range of 0 – 500ppm. Although gas drying problems similar to those experienced with NDIR instruments have been reported it is expected that the instrument will be used under U.S. Federal Regulations for vehicle emissions. However, it is an expensive instrument and not suitable for field use [18].

(b) Dispersive ultra violet (DUV)

By analogy with DIR the dispersive ultra violet techniques are expensive and complicated and suitable only for the laboratory [18], [19].

2.4 Mass Spectrometry

This is clearly an expensive method of analysis and would be of use only if it were a primary standard method. However, it is not an absolute method and no real success has been reported in its use [19], [24], [25] for low levels of nitrogen oxides.

2.5 Gas Chromatography

Gas chromatography is an instrumental method which does not provide a continuous read-out. However, it is relatively cheap, and know-how is widely available. The technique is sufficiently attractive for some time and effort to have been spent in an attempt to develop a suitable method for nitrogen oxides, but with only limited success [26],[30]. Some of the problems encountered are associated with reaction between NO and any O_2 which may be present in traces in the carrier gas; reaction and absorption of the oxides on the chromatograph column walls are possible and there can be interference by water.

2.6 Chemiluminescent Methods

The chemiluminescent method described by Fontijn [31],[32] is based on the oxidation of nitric oxide by ozone to form excited NO_2 at low pressure. The spontaneous (chemiluminescent) decay of the excited NO_2 emits light in the region beyond 600nm, which is a measure of the original concentration of NO:

$$NO + O_3 \rightarrow NO_2^* + O_2$$

$$NO_2^* \rightarrow NO_2 + h\nu \text{ (chemiluminescent)}$$

or $$NO_2^* + M \rightarrow NO_2 + M \text{ (quenching)}$$

The method is virtually free from interference from CO and SO_2, but there is evidence to suggest that whilst the effect of CO_2 and H_2O is not negligible, corrections are easy to apply.

In an alternative procedure, oxygen atoms are used instead of ozone and the chemiluminescent reaction producing NO_2 is observed:

$$O + NO \rightarrow NO_2 + h\nu \text{ (chemiluminescent)}$$

Any NO_2 in the original sample is converted to NO:

$$O + NO_2 \rightarrow NO + O_2$$

Chemiluminescent methods are now sufficiently developed for the procedure to be adopted for Federal Regulation Testing of motor vehicle exhausts in the U.S.A. and some instruments are also being used in the U.K. for this purpose. Numerous models are available at present, generally costing between two and four thousand pounds. Whilst the technique appears to be entirely satisfactory for the determination of NO, there are still minor problems during measurement of NO_2, which requires quantitative conversion to NO before estimation.

2.7 Flame Emission

Flame emission detectors have been developed [33] which are based on similar principles to those already in use for the detection of sulphur, i.e. by the detection of the emission in the infra red region of the spectrum when nitrogen oxides are burnt in an oxygen deficient hydrogen flame. This method probably has as great a potential as the chemiluminescent methods, if development problems can be overcome.

2.8 Ion-selective Electrodes

The application of nitrate ion-selective electrodes for estimation of NO_2, has received limited attention [34]. This method is potentially the cheapest route to the provision of an instrument with a good range, rapid response, ease of calibration, and portability, provided that the sensitivity of the electrode is such that low concentrations can be measured.

2.9 Coulometric Method

The Mast meter [19],[35] is a commercially available monitor working on the coulometric principle. The reaction involves the oxidation of halide ion by NO_2 followed by reaction of the halogen so formed with hydrogen at a polarised electrode, when the current flow is proportional to NO_2 concentration. The meter suffers from interference by NO, water and oxidising agents such as ozone, and its use in flue gas analysis is therefore limited.

2.10 The Hersch Cell

The original cell was further developed by Shaw and was once marketed by B.C.U.R.A. as a nitrogen oxides analyser. In this form it is cheap, simple to operate and portable, but it has a tendency to drift; the response is non-linear over a wide range of nitrogen dioxide concentration and there is interference from sulphur dioxide.

The cell consists of a platinum cathode, kept moist with a neutral buffered electrolyte containing phosphate ion and potassium chloride, which is connected to an anode of active powdered carbon. The cell only responds to nitrogen dioxide, and nitric oxide must be oxidised prior to measurement.

A proposed mechanism [41] involves the reduction of nitrogen dioxide at the cathode according to:

$$NO_2 + H_2O + 2e = NO + 20H$$

while the hydroxyl ions migrate to the anode and are discharged:

$$C^* + 20H = CO^* + H_2O + 2e$$

where $C^* + CO^*$ represents different levels of activity of the carbon.

In view of the insensitivity of the cell to nitric oxide this mechanism may not represent the complete range of reactions in the cell; nevertheless, the output of the cell is proportional to the nitrogen dioxide concentration over a limited range, (0 — 140 vpm), and it can be calibrated for use as a continuous monitor.

3. Discussion

A facility for the measurement of concentrations in the approximate range of 1 to 5000vpm and a continuous recording device is preferred for the determination of nitrogen oxides in air and in combustion products. This requirement means that a suitable primary method must be available to calibrate a continuous recording apparatus. It is clear from the literature that no entirely suitable method is available, although regulations in the U.S.A. concerned with measurements on vehicle exhausts have relied on the wet chemical method with an arbitrary equivalence factor determined by Saltzman of 0.72, which relates the nitrogen dioxide in a gaseous sample to an aqueous solution of sodium nitrite used for calibration. Although it has been shown that results of measurements by wet chemical methods are readily repeatable, there are thus still doubts about the absolute accuracy.

In the near future it should be possible to look forward to a simple wet chemical method which will be accepted on an international basis as a standard method. Collaborating laboratories are now finding a much closer agreement about the relative levels of NO_x to be expected in given situations, and progress is being made towards standardising methods which are continuous. It is clear however that at the present time there is no instrumental technique which satisfies all the requirements for routine determination of nitrogen oxides at all levels of concentration and in any situation.

Acknowledgments
The authors wish to thank British Gas for permission to publish this paper.

References

1. **Threshold limit values for 1969**
Tech. Data Note 2/69 Dept. of Employment and Productivity
Published HMSO

2. **Status of NO_x control from combustion sources**
Crynes B.L., and Maddox R.N.
Chem. Tech. (Aug. 1971) 502-9

3. **Experimental and theoretical study of nitric oxide formation in internal combustion engines**
Lavoie G.A., et al
Combustion Science and Technol. (1970) 1, 313-326

4. **Comparison of fifty-two spectrophotometric methods for the determination of nitrite**
Sawicki E., et al
Talanta, (1961), 10, 641

5. **Colorimetric microdetermination of nitrogen dioxide in the atmosphere**
Saltzman B.E.
Anal. Chem. (Dec. 1954) 26, (12), 1949

6. **The measurement of nitrogen dioxide in the atmosphere**
Stratmann H., Buck M.
Int. J. Air Water Pollution, (1966), 10, 313

7. **Stoichiometric study of the reaction between gaseous nitrogen dioxide and Saltzman reagent**
Hartkamp H.
Staub-Reinhalt-Luft, (Nov. 1969), 29, (11), 6

8. **Kinetic evaluation of the factor used in the Saltzman analysis of oxides of nitrogen**
Freedman R. et al
Amer. Indus. Hygiene Assoc. J., (1970), 31, 76

9. **Reaction of nitrogen dioxide with Greiss-type reagents**
Huyghen C.
Anal. Chem., (March 1970), 42, (3), 407

10. **The measurement of nitrogen dioxide in the air**
Shaw J.T.
Atmos. Environ. (1967), 1, 81

11. **Messung von Stickstoffdioxid in der Atmosphare**
Stratmann H., Buck M.
Air and Water Pollut. J. (1966), 10, 313

12. **The joint and separate determination of nitrogen monoxide and nitrogen dioxide in the atmosphere**
Stratmann H., Buck M.
Staub. Reinhalt Luft, (June 1967), 27, (6), 11

13. **A note on the work on the analysis for NO_2 being carried out at the Watson House Research Station of the Gas Council**
Allen J.D.
11th March, 1971, for presentation at the review meeting, Shell, Egham, 17th March, 1971

14. **Dilution system for low concentrations of nitrogen dioxide and determination of the Saltzman factor**
Nietruch F., Prescher K.E.
Z. Anal. Chem. (1969), 244, 294

15. **An improved form of solid oxidiser for the conversion of nitric oxide to nitrogen dioxide in a flow system**
Bethell K., Shaw J., Thomas A.
Chem. and Industry, (1968), 91

16. **Gas Council analytical methods**
Method No. 3.3.8. Nitric Oxide and Nitrogen Dioxide, June 1970

17. **Oxides of nitrogen in gaseous combustion products (phenol disulphonic acid procedure)**
Method D 1608-60. Amer. Soc. Test Materials, Philadelphia

18. **Third review meeting on sampling and analysis of combustion products for nitrogen oxides**
Minutes by C.J. Halstead,
Shell Centre, London S.E.1, 17.3.71

19. **The measurement of nitrogen oxides from the gasoline engine**
Blackmore D., Crawford K.,
Shell Report MOR 627F, Dec. 1970

20. **Review meeting on sampling and analysis of combustion products for nitrogen oxides**
Minutes by J. Holding,
Shell Research Labs., Egham, 25.3.70

21. **The application of multi-reflection gas cells to infra-red trace analysis**
Thomas L.C.,
Proc. XII Coll. Spectroscopicus Internationali, Exeter 11-17 July 1965

22. **Catalytic reduction of nitrogen oxides in automobile exhaust**
Baker R.A., Doerr R.C.
J. Air Pollution Control Assoc. (1964) 14, (10), 409

23. **Direct spectroscopic determination of nitric oxide in reciprocating engine cylinders**
Newhall H.K., Starkman E.S.,
SAE Paper No. 670122 (1967)

24. **Continuous mass spectrometric determination of nitric oxide in automatic exhaust**
Campau R., Neerman J.,
SAE Paper No. 660116 (1966)

25. **Mass spectrometric analysis of mixtures containing nitrogen dioxide**
Friedel R., et al
Anal. Chem., Sept. (1953), 25, (9), 1314

26. **Gas chromatographic determination of nitric oxide on treated molecular sieve**
Dietz R.,
Anal. Chem., (Aug. 1968), 40, (10), 1576

27. **Analisi di inquinati atmospherici gassosi per via gas cromatografia con rivelatore a elio metastabile**
Castello G., Munari S.,
La Chimica e l'Industria, (Maggio 1969), 51, (5), 469

28. **Optimum conditions and variability in use of pulsed voltage in gas chromatographic determination of parts per million quantities of nitrogen dioxide**
Morrison M., Corcoran W.,
Anal. Chem., (Feb. 1967), 39, (2), 255

29. **Reaction of nitrogen dioxide with Porapak Q**
Trowell J.
J. Chrom. Sci., (April 1971), 9, 253

30. **Chromatographic analysis of some oxides of nitrogen on a Chromasorb 102 column**
Wybrow B.R., Cutting P.A.,
Gas Council London Res. Station Tech. Note No. 150, February 1971

31. **Homogeneous chemiluminescent measurement of nitric oxide with ozone**
Fontijn A., Sabadell A., Ronco R.,
Anal. Chem., (May 1970), 42, (6), 575

32. **Feasibility study for the development of a multifunctional emission detector for air pollutants based on homogeneous chemiluminescent gas phase reactions**
Fontijn A., Sabadell A., Ronco R.,
Report to National Centre for Air Pollution Control, Raleigh, U.S.A., Sept. 1969, Aerochem TP. 217

33. **The detection of NO, NO_2 and other nitrogen compounds by flame emission**
Krost K., Hodgeson J., Stevens R.,
ACS Div. Water, Air and Waste Chem., Preprints, Sept. 1970, 10, (2), 46

34. **Determination of nitrogen dioxide and nitric oxide in the parts per million range in flowing gaseous mixtures by means of the nitrate specific ion electrode**
Di Martini R.,
Anal. Chem., (Aug. 1970), 42, (9), 1102

35. **Instrumental methods for the detection of higher oxides of nitrogen in nitrous oxide**
Shaw J.T.,
Brit. J. Anaesth. (1968), 40, 299

36. **A review of methods of analysis for oxides of nitrogen**
Allen, J.D.
J. Inst. Fuel. (1973) 123

37. **Methods for the Detection of Toxic Substances in Air. Booklet No. 5. Nitrous fumes.**
Pub. H.M.S.O. for H.M. Factory Inspectorate.

We are indebted to Dr R.G. Cockerham of the British Gas Corporation Midlands Research Station who has enabled us to publish this paper in our journal. Dr Cockerham presented a lecture on the subject covered in this paper at the 1972 Symposium held at Scarborough.

We gratefully acknowledge the British Gas Corporation for allowing this publication.

TOM PARSELL

Most members of the British Society of Scientific Glassblowers will know Tom Parsell, many will have cause to be grateful to him for his help and guidance in their glassblowing careers, many others will recall he has been the Chairman of the Southern section for a number of years.

Tom is retiring this year, after having been with the Training Service Agency (formerly the Ministry of Labour or Department of Employment) for 28 years.

The young Tom Parsell was trained by the same establishment that he is now leaving and he remembers from those days such names as Tom Wingent, Bob Bartle and Jack Tandy.

After leaving the training establishment he joined G.E.C. at the Research labs working under R.L. Breadner, he calls to mind Mr Lewis, Dr Dudding, Dr Partridge and Dr Bennett ..... during his 15 years with the General Electric Co. in research labs and the factory.

Now very experienced in all aspects of glass-working and allied techniques Tom returned as instructor to the G.T.C. Waddon, this in 1946. Many names will be associated with him in those times, such as Ted Evans, Jim MacDonald, Bob Beer, Lou Morrison and Jimmy Gardner with R.B. Radley at a somewhat later date ..... many others are in different parts of the world and more recent names such as Hepburn and Le Pinnett are to be found engraved upon the A.D. Woods Cup, these also being graduates of the Government Training Centre.

Tom has sent his best wishes to the society with his hope that it will grow from strength to strength. He hopes to get around to see many of his old colleagues in the future, once he has got some of his "around the house" chores completed to his satisfaction.

The British Society of Scientific Glassblowers we are sure, would want to thank Tom for his great service to glassblowing and to the Society ..... wishing him a very pleasant retirement, good health and well being for many years to come.

Editor

GLASS-SHOP DEPARTMENT OF ORGANIC CHEMISTRY, UNIVERSITY OF LIVERPOOL

P. HALLIWELL

Peter Halliwell started glassblowing in 1958 at the age of 15 as apprentice to Mrs. Yvonne Brooks with Messrs. Ferranti Ltd., Oldham, Nr. Manchester. After 5 years in the valve production department he aspired to take charge of the glassblowing unit of the Electronics Display Division, formerly the Cathode Ray Tube laboratory, within the same plant. Things went smoothly for him for 3 years or so, but then after two troubled years his service with Ferranti Limited ended.

In late November 1968, Peter took up the position of assistant glassblower to Mr. J. Eisenberg in the University of Liverpool's Department of Organic Chemistry, but tragically in early January 1969, John Eisenberg died. Peter carried on alone as Senior glassblower for Organic Chemistry until August 1969 when Mr. E.C. Hegarty was engaged as Peter's apprentice.

The organics glass-shop is compact at 440 sq.ft. and consists basically of two glassblowing benches, two work planning benches and a fume cupboard, a certain amount of glass tube and sundries are stocked in the glass-shop, the majority though being kept in a separate store room.

Equipment available, a Heathway SIEV 4½" bore lathe, an annealing furnace, a 15" flat grinding mill, a Tyslyde 5" diamond saw, plus both the usual and unusual gadgets that make a glass-shop.

Whilst Mr. Hegarty (Eddie) fulfills the needs of the four teaching laboratories, Peter concerns himself mainly with the requirements of the research chemist, their needs as we all know fluctuate from the usual chemists requests for sinter funnels, fractionating columns, distillation apparatus, extraction apparatus etc., to the, we had better say, chemists unusual requests, rather than unusual chemists requests, like helium ionization guns for use in conjunction with a mass spectrometer U.V. irradiation equipment using a special silica glass imported from Germany, filters fabricated in 'Corex' glass, only to find after numerous telephone calls and letters to Belgium, Switzerland and New York, that 'Corex' was a chemically toughened glass so if they could obtain it, they could not work it, multi limbed liquid mixing kinetic cells where space was at such a premium in the spectrophotometer that the cells had to be twisted and bent into some quite odd shapes.

In conclusion, Peter has been a member of the North Western section since 1968 and at present holds the positions of Section Representative to Council and Board of Examiners Section Representative, and Eddie is at present a student member of the Society.

SAFETY

INSERTION OF GLASS TUBING OR ROD INTO RUBBER BUNGS

F. WAINWRIGHT

Drilling of bungs

a) Where work allows, drill the rubber bung first and select the glass to fit afterwards. Although this is not always convenient, it must be understood that a wider variety of glass than drill diameters is obtainable, and consequently a better match can be made.

b) This method should, therefore, be used wherever possible. In some instances, rubber bungs can now be obtained already drilled. This alleviates any dangers from actual drilling.

c) If manual drilling is necessary a 'moleskin type' glove or mit should be worn on the hand gripping the bung. These gloves as used by us have a double thickness of material and yet still not thick and clumsy. The drill should be lubricated with glycerine. Both drill and bung should be rotated in opposite directions, with rubber bung firmly held against a bench top or similar steady base. When drilling is completed the drill and bung should once again be rotated to release. If a drilling machine is used, **both hands** should be gloved. Glycerine should once again be used as a lubricant.

Selection of glass

All glass tubing (or rod) due to be inserted into a rubber bung, should have both ends fire polished. Annealing should also follow wherever possible. Bends, straight tubing or rod, should be shaped or fused by glassblowers only. Prior to inserting the glass (which should now be cool), the drilled holes and the glass itself should both be lubricated with glycerine or even water, if the former is not readily available. Meths is a useful lubricant which evaporates leaving the job dry.

Protection

Before inserting glass, ensure both hands are adequately protected by either 'moleskin type' gloves or mits. If, due to the surface property of the glove a 'slip-feeling' is encountered between the glove and the glass, it is felt that this is a good indication that the glass is too tight, therefore, a new piece of tubing or rod should be selected.

Method of insertion

a) Grip the glass tubing (or rod) no more than three tubing diameters distance from the end to be inserted, and place into entrance of hole.

b) As glass enters, rotate both bung and glass in opposite directions until hand reaches bung.

c) Move hand back along tubing for a similar distance of three tubing diameters.

d) Continue with rotation until hand once again reaches bung.

e) Continue this method in small steps until required position of glass is obtained.

Problems

If any tightness is suddenly encountered, do not proceed any further. More accidents can be caused at the 'half way over tight' stage than at any other time. Seek advice of glassblowers if this happens.

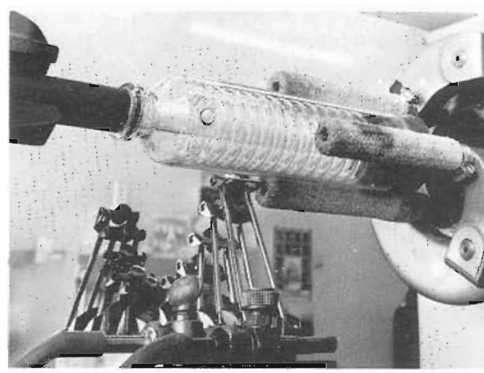
Accidents

If an accident should take place, any cuts should receive medical attention however slight they may be.

A METHOD OF REPAIRING BUCHI ROTOVAPOUR CONDENSERS

P. HALLIWELL

In a department which uses amongst other types, some 25 Buchi* rotavapours throughout its laboratories each day, it's no surprise when a number land in the glass-shop for repair, the results of a survey over the past 18 months showed that 95% of the 12 condensers for repair had been broken in much the same manner i.e. due to careless insertion of the vapour duct through the rubber seal, the first turns of both internal and external coils had been broken, the odd couple with coils intact had split moulded flanges so bad in fact, they were treated as the others in that they had to be re-built.



The method adopted for repairing these condensers was, that for the ones with broken moulded flanges a carbon former was made in our workshops to facilitate their manufacture. Once made they followed the same procedure as the others, in that a very simple tool was made out of 36mm dia. carbon rod at one end its diameter was reduced to 34mm along a length of 1cm, the same end was bored out to a 24.5mm dia. for a depth of 1cm.

This holder was placed into a lathe's headstock chuck, the tailstock chuck holding a broken condenser, 2 or 3 turns of damp asbestos paper was then wrapped around the machined end of the holder and the tailstock moved along to enable the good moulded flange of the broken condenser to be pressed tightly over the asbestos paper, the moulded flange was then flame parted from the body of the condenser from which only the coils were worth re-claiming.

The next stage was to rejoin the parted moulded flange to a new body, i.e. a pre-formed length of 85mm dia. tube, if the asbestos paper had been correctly tensioned and positioned it would be then possible to open the tailstock chuck, and place a re-claimed coil into the supported body (the support used here is a flexible headed ring burner), at this point a rubber bung drilled centrally to take a piece of 24mm dia. tube was fitted to the open end of the body, the centralising tube passing through the middle of the coils, and locating in the bored out section of the holder (Fig. 1). Coils correctly positioned and blowing connections made, the tailstock chuck was tightened and the support taken away.

The remaining operations are quite straightforward providing one has remembered to pre-fabricate one's side arms etc., and should result after annealing in a rewarding, sparkling re-built condenser (Fig. 2).

Department of Organic Chemistry,
University of Liverpool.

* W.Buchi Scientific Apparatus Flawil, Switzerland.

PRESS RELEASE

JOBLING CUP PRESENTED TO BORN-DEAF GLASS TECHNICIAN

Every year since 1966 the handsome Silver Cup given by Jobling Laboratory Division at Stone to the British Society of Scientific Glassblowers is presented to the most outstanding trainee member of that Society. This year the successful candidate merits particular laurels because he has won the trophy despite the severe handicap of being totally deaf and unable to communicate other than visually.

The winner, 21 year old Peter Brindley of Warwick University was born deaf and thus has never learned to speak except by hand signs. On Friday last he came to Jobling's Stone factory with his Tutor (and self-taught interpreter) from the University Mr. K. Holden, to receive the trophy and replica from Mr. A.R. Dygert, Managing Director of Jobling Laboratory Division. He was given the V.I.P. treatment by Jobling, with a conducted tour of the factory to see the firm's famous QUICKFIT and E-MIL laboratory glassware being manufactured, followed by the presentation of the trophy and then lunch as personal guest of Mr. Dygert and his senior Jobling colleagues.

Our picture shows Mr. Brindley receiving the trophy replica from Mr. Dygert (the trophy itself can be seen on the table). Also at the presentation were (left to right) Mr. L. Morrell, Divisional Manufacturing Manager, Mr. F.E. Hankey, Managing Director of the Jobling Group, Mr. K. Holden of Warwick University and Mr. P. Jaram, Managing Director of Jobling's Process Plant Division.

SOCIETY DIARY

CHAIRMANS REPORT

The first Council meeting after the 1973 A.G.M. was devoted to the financial position of our society, with particular regard to the running costs of the journal and the providing for the members the means to enable them to achieve qualifications within the society through the Board of Examiners. Not that acquiring these qualifications would be compulsory but rather to provide opportunity and assistance to members, if so desired.

By Christmas, because of the rail dispute and petrol situation we did not know if Council members would be able to get to Birmingham for the meetings and when the three day week hit the country, some of us certainly could not do so. Even locally held meetings were not always possible owing to the different area working times. Despite these difficulties and the contingent hardships that attended them, the Board of Examiners have put in a remarkable amount of work and have laid the foundations of the Master glassblower as the immediate grade between the Full members and the Fellows. The Master glassblower grade to be achieved by qualifying on technical ability as directed by the Board of Examiners, unlike the Fellows which it will be the prerogative of the Council to award.

All sections of the society welcome any help given by members, but the East Anglian section would particularly appreciate interested members accepting office, any members who feel they lack the experience would be welcome and would be assured of assistance and guidance from the Officers of the Society.

It has not been an easy year but we have come through it considerably better than we might have done, owing to the efforts of the Officers of the Society and the Council members to whom I would like to give my, and I am sure your, thanks.

R. Harvey, Chairman, BSSG.

BOARD OF EXAMINERS' REPORT

Arising from the A.G.M. of 1st September, 1973, a referendum, on the subject of the relationship between the proposed "Institute of Glass Engineers" and the B.S.S.G., was called for.

The Council meeting 14/1 held on 6th October, 1973 recorded the result of the referendum as follows.

> "That the B.S.S.G. has no part in the formation of the Institute of Glass Engineers but instead develops through its Board of Examiners, a stronger academic role by the inclusion of technical or educationally qualified persons on the Board of Examiners, so that they might assist in the promotion of courses, lectures and examinations."

The B.o.E. meeting of 27th October, 1973 received a directive from Council to consider the result of the referendum and forward proposals to Council.

Council, on 26th January, 1974 (meeting 14/2), accepted the recommendations forwarded by the B.o.E. as follows.

The B.o.E. propose to use the following structure in building up an examination system in both academic and practical fields. Both practical and theory will be linked together, though on a variable bias to one or the other, but never completely divorced. It is envisaged that the credit will be obtainable singly and accumulated.

Fellow B.S.S.G.
Credits in theory and practical.
(Subjects to be listed.)

Master Glassblower
Credits in theory and practical.
(Subjects to be listed.)

Full Member

Five years experience. Vetted.	or	Certificate of competence.

Considerable progress has been made on subject headings and content up to the Master Glassblower level, with a view to completion during 1974.

Overseas Scientific Glassblowing Lectures

From time to time the Society is approached regarding the availability of B.S.S.G. members to act as Scientific Glassblowing Course Tutors. A recent example of such an approach was an invitation to the B.S.S.G. to run an Elementary Course terminating in exams at the University of Ibadan, Nigeria. The invitation was forwarded to the B.S.S.G. through the Inter Universities Council.

Mr. R. Mason was elected to represent the B.o.E. and the B.S.S.G. in the University of Ibadan during the period from 24th June, 1974 to 24th August, 1974.

In the anticipation of future invitations, members of the B.S.S.G. willing to act as course tutors should write to the Secretary, B.o.E.

Chairman
Board of Examiners
5th June, 1974

BRITISH SOCIETY OF SCIENTIFIC GLASSBLOWERS 1973 SUBSCRIPTIONS

Members are reminded that subscriptions are now due for renewal. Renewal forms will shortly be circulated and should be completed and returned to the Hon Treasurer enclosing the appropriate amount, by not later than 31st March 1973.

Three years subscription may now be obtained at the cost of £10.

MATERIAL FOR PUBLICATION

All contributions to this journal must be in the hands of the Editor by the following dates for each issue...

November 21st...	for the January issue.
February 21st...	for the April issue.
May 21st...	for the July issue.
August 21st...	for the October issue.

REFERENCE/PAGE CHARGES

Full page	£20.00
Half page	£12.50
Inside front cover	£25.00
Back page outer	£35.00
Plus colour	£12.50

G.T.M.

ABSTRACTS 'Glastechnische Mededelingen' 1974

No. 1, March 1974

- **Behaviour of glass under stress** *(A.L. Zijlstra, Eindhoven, 8 p., 11 fig.).*
  Text of a paper read at the Symposium 1973 at Nijmegen. After an introduction a treatise is given on tensile strength, influences of scratches and flaws on strength, load v. deformation, fatigue, various tensions in glasses (mechanical and thermal), introduction of preliminary tension in glass (prestress) and annealing. The paper deals furthermore with measuring the C. of E. of glasses and the selection of suitable materials in glassworking.
- **Velocity of fragments of Dewarvessels on exploding** *(C.D.F. Eerbeek and A.C. Sterk, Amsterdam, 4 p., 1 fig., 11 form.).*
  After a review of various formulae of mechanics an approximation is made on determining the speed of flying fragments of an exploding Dewarvessel.
- **Safety blowing valve for glass instrument working** *(R. Brehm and J. van Wershoven, Eindhoven, 4 p., 1 fig., 11 form.).*
  The article deals with a safety valve to protect the worker from inhaling noxious fumes (nitric fumes etc.), throws a light on some risks and gives a description of the construction of such a valve.
- **Production and application of packed capillaries for scientific research** *(J.C. Hendriks, Eindhoven, 3 p., 7 fig.).*
  To reach theoretical conclusions on the behaviour of various porous media models are wanted in which the number and dimensions of 'chanels' are exactly known. To realise this object use is made of 'packed capillaries'. Simple capillaries are drawn with the aid of a drawing machine, subsequently packed together and drawn out again, and so on. Weight is laid on the relation capillary holes v. intermediate solid material as this determines the quality of the model. Results are:– bundles with an outer diameter of about 1,5mm with up to 9,660 holes of .6 micrometre each. With the aid of mathematical models practical experiments can be carried out with less imponderable data, as size of pores, pore distribution, porosity and labyrinth factor, as occur in concrete, brick and other building materials.
- **Agenda of events** *(8 in no.).*
- **Index 1973.**
- **Information** *(4 p.).*
  Safety in laboratories: publication of Dutch Labour Inspection No. P 130.
  Summary of Proceedings Werkgroep "Zuid" voor Glastechniek.
  Notice on examination 1974.
  Wertheimer Glastage 1974.
  Etching of patterns on ultraviolet sensitive glass (Corning).
  Risks on the use of laser rays.

Van der Burgh.

NEWS FROM

JAPAN

The Japanese Society of Scientific Glassblowing held its first session in 1974 on 22nd February at Kameido Kinro Fukushi Kaikan in Tokyo. Following president's address, guest speakers gave lectures:–

"Economic policy in the overseas"
Mr Saburo Harada, Koto Electric Co. Ltd.

"Chemical solutions and hazard"
Specialist, Tokyo Fire Agency

"Filter Glasses"
Dr Hachiro Shimada, Shimada Glass Laboratory, Inc.

The lectures were received very well by the attendants and the session adjourned at 5.00 p.m.

On that day, some committee meetings were held at the same place. Election committee, for instance, experienced a candid discussion of a delicate and irritating question concerning the election of directors for the 1975-1976 terms.

On 9th March, the Sendai section of the society held a session at Tohoku University. Mr Saburo Sasaki, chairman of the section, gave an opening address at 1.00 p.m. The first program was showing films on glass and glassblowing. Some technical discussion was also carried out by the attendants. The session was adjourned at 5.00 p.m.

Coe Gotoh

SOUTHERN

Meeting was held on 13th February, at Queen Elizabeth College. It was a lecture "Carbon in the Glass Industry" by J. Brewer of Morganite Speical Carbons Ltd. This thoroughly worthwhile lecture commenced at 7.40 p.m.

Mr. Brewer launched himself into his subject quickly, by giving a quick description of what Carbon and Graphite/Carbons are. What is Carbon? He said it is a composition of pitch, tar, soot oil residues and graphite substances mixed together and formed into various shapes. How is it made? The various forms required are heat treated up to 1250°C and for up to 9 days in muffle furnaces. Carbon itself is very hard, and graphite is very soft, so most carbon tools used by glassblowers are a mixture of the two. This allows resiliance to both wear and oxidation. The blends of carbon/graphite vary according to low temp. or high temp. requirements e.g. CY9 Carbon is suitable for low temperature work – soda, lead glass forming etc. EY9 Carbon is more suitable for high temperature work – pyrex glass, silica etc., although for silica work Carbon EY110 is recommended.

He stated that one of the main advantages of Carbon/Graphite tools is that they may form the glass without crazing or oxidising the surface, whereas metal or other substances can and do contaminate the glass.

Carbon and graphite are used extensively in the valve and special glass component industries, as well as by large bottle making and sheet glass industries. It can be used in all forms of glass moulding or forming, according to any particular requirement. The lecture finished at 9.00 p.m. but not before many questions had been placed. Judging by the amount of questions, Mr. Brewer had tapped a real spring of interest.

March Meeting

"Ballotini and the Manufacture of Glass Balls" by Mr. T. Lawson of English Glass Co. Ltd., Leicester on 20th March 1974.

The meeting began at 7.40 p.m. After a brief introduction by the Chairman, Mr. Lawson quickly pursued his subject with descriptions, slides and examples. He described the rod making process by which quite large diameter glass rod has the ends heated and passed into a mould. The heating requires careful judgement and skill, in order to avoid any faulty moulding or damage to surfaces. Although some of the processes are secret, we were given much helpful information concerning processing. The temperatures in melting and moulding were highly critical, the examples shown were a good example of judgement and skill.

The glass rod used in the processes are optical glass, coloured glass rods, pyrex, soda and lead glass rods.

Ballotini balls come in steps of 0.005, in the range of 0.5mm to 10.0mm O.D. Precision ground glass balls from 1.0mm to 52.0mm. Tolerances are quite high, in the range 1.0 to 5.0mm O.D. + or – 0.0005 in the range 5.0 to 50.0mm + or – 0.001.

Questions flowed and many showed interest in the examples displayed. A worthwhile lecture, delivered in a pleasant and efficient manner. The meeting closed at 8.50 p.m.

Mr. Harvey gave the following report of the lecture by Dr. Torrant, held at the University of Surrey.

We went into the lecture theatre where there was a battery of projectors, lighting equipment, food of differing types and packaging, coloured lights, U.V. lamp and oh yes, an attractive young girl student model.

"Keep your eyes on the black dot in the centre of the green elephant (green yes, not pink) and observe what change takes place." You know the sort of thing where one looks at a black image on a white screen and then at a black area and the retina retains an image in reverse. Well, this was slightly different. The green soon started becoming lighter round the edges and the elephant floated gently upwards to the top of the screen. There we were, in the dark and I felt we were being mesmerised. This was one of the demonstrations used by Dr. Tarrant, lecturer in the Dept. of Physics at Surrey University to show the effects of coloured light on coloured objects.

First of all he described just what colour was and by means of three projectors showing red, green and blue respectively, how white light was formed and how it was possible to match a colour given by a fourth projector by adding and subtracting light colours superimposed one upon another and varying the intensity.

Proof was given that colour was not just a physical effect but a psychophysical phenomenon, by inducing colour to appear from a spinning disc which had only black lines on white but at the correct speed appeared yellow, red and blue. We all saw the same colours here but it was explained that different people normally saw variations in colour tones so that colour T.V. sets up and down the country varied in their colour balance to the point where one person could quite happily be watching actors with mauve tinted faces and green shaded hair without being aware of anything unusual. This was not to say they were colour blind, but merely had a different colour bias. Advertising agencies are well aware of these colour light effects and make good use of warm tinted lighting which is emitting strongly in the red end of the spectrum giving meat and other goods that fresher than fresh look and UV reflecting powders giving the washed whites that popular whiter than white appearance.

One of the confusing demonstrations was food which was of a completely different colour to norm. Even after I had been told that I was tasting custard, the black colour ruined my taste buds, I tasted only the texture, it was flavourless. The strawberry jelly was yellow, and I could not guess the flavour. The bananas looked good on display, but in ordinary light they turned out to be powdery blue-grey, revolting.

A full lecture, with excellent demonstrations, what a difference a professional lecturer makes.

R. Newman

WESTERN

The February meeting was held at the Medical School, Bristol University, where members heard an interesting talk by Mr. Thomas, the Research and Development Manager of I.D.P. Gloucester.

Mr. Thomas started his talk by giving a brief resume of the natural diamond as mined by someone like DeBeers. DeBeers grade diamonds as mined into gem stones, larger industrials and industrials, he noted here that the value of a gem stone goes up by a 4th power of its weight. Larger industrials differ to a gem stone only in colour and there would be about 120 to the carat (1 carat = 0.2gm). The smaller industrials are crushed from perhaps the size of white sugar grains down to flour grain size.

As far as glassworkers are concerned the glass cutting diamond is one of the few uses of a single point diamond.

Mr. Thomas then went on to explain the process for preparing sintered diamond cutting tools, diamonds are set in a carbon mould with glue which is then packed with inert metal powder, e.g. Tungsten, Brass is laid on top of the tungsten and the whole mould is heated to 1080-1150° when the brass melts and sucks into the tungsten compact. He stressed that it was important with impregnated tools that the wear rate of the diamond was the same as that of the matrix bearing in mind the proportion of matrix to diamond. Mr. Thomas then warned of how machine vibration can badly reduced tool life.

A lot of the diamonds in impregnated tools are man made by high pressure technology, basically

$$\text{Carbon} \quad \begin{matrix} 1600^{o}C \\ 400 \text{ tons/sq. inch} \end{matrix} \quad \text{Diamond}$$

DeBeers make diamonds at Shannon, Eire. These crystals being stronger than crushed natural diamond are therefore slightly more expensive. Mr. Thomas pointed out that diamond begins to "graphetise" above 1000°C and recommended a white bauxite stick for cleaning clogged slitting discs etc.

The evening ended with everyone being extremely interested in the exhibits that Mr. Thomas had brought with him especially the sealed tubes of natural diamonds.

The March meeting was held at Rex Garrards workshop, School of Chemistry, Bristol University when Mr. R. Harvey, Society Chairman gave an extremely interesting talk to one of the largest audiences that we have had. Ron's talk on Enamelling and Small Jewellery started by giving a brief history of enamelling and then moved through the technical details to the practical methods used. Ron then demonstrated this fascinating art to an enthralled audience. The meeting then closed with everyone agreeing that it had been an extremely entertaining and informative evening.

The April meeting of the section was a business meeting when future programmes were discussed. Due to the very few members present the venue of the Annual dinner was left in abeyance as was nominees for the Thames valley award. This year's works visit is to the factory of Messrs. T. Wingent Ltd., Southampton.

P. Houlden

MIDLANDS

The Midland Section held it's A.G.M. on 9th April 1974 at the New Victoria Hotel, Birmingham, commencing at 6 p.m. The election of officers was as follows:–

Chairman	Mr Brian Cutforth
Vice Chairman	Mr P. Sala
Secretary	Mr Keith Holden
Treasurer	Mr Peter Brindley
Councillor	Mr Keith Holden
Section Council Rep.	Mr Frank Daysh
B.o.E. Members	Mr Keith Holden
Re-confirmed	Mr Jim Huckfield

The A.G.M. was followed at 7 p.m. by a Social Evening sponsored by W.G. FLAIG & SONS LIMITED manufacturers of EXCELO INTERCHANGEABLE GLASSWARE.

"Excelo" Sponsored Evening

Mr Walter Zuber of W.G. Flaig & Sons Ltd., accompanied by Mr Jim Clayton, Midlands Area Representative, and Mr Doug Ireland were introduced by Section Chairman, Mr Bryan Cutforth, to the members and wives present.

Mr Zuber responded by indicating his pleasure in being able to meet the glassblowers within the Midlands Area as it coincided with the recent expansion of W.G. Flaig & Sons Ltd. to within the Midlands with the opening of a supply depot at Haybrook, Halesfield, Telford, Shropshire.

Mr Zuber introduced Jim Clayton to those few members who had not already had the pleasure of Jim's company and also Doug Ireland as the Telford branch warehouse manager.

Mr Zuber further expanded his "chat" to include some very interesting facts concerning the history of "Excelo" interchangeable glassware which extends to some sixty years in the laboratory glassware field.

Mr Zuber further outlined his company's association with JENA GLASWERK SCHOTT & GEN, MAINZ of West Germany and their position as supplies of the well known SCHOTT borosilicate glass DURAN 50.

Mr Zuber invited those present to inspect the range of DURAN 50 laboratory and Microbiological Glassware on display, along with the many fine examples of "Excelo's" own products.

Bryan Cutforth thanked Mr Zuber for a most interesting talk and his company's generosity in laying on a most delightful evening and an excellent buffet and asked those present to show their appreciation in the usual way.

K. Holden

NORTH WEST

The A.G.M. of the Section was held at the Slip Inn, Warrington on 16th May 1974. The reports of the Chairman, Secretary and the statement of accounts was approved. The following were elected into office:—

Chairman	Mr S. Hill
Secretary	Mr Paul Le Pinet
Treasurer	Mr G. Blackburn
Reporter	Mr H. Chappell

The meeting was followed by a Symposium committee meeting.

B. Chappell