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PREFACE

A need for working glass is frequently encountered in scientific research and teaching, particularly in the field of physical chemistry. In many laboratories this problem is solved by the employment of professional glass-blowers, and the research worker requires little or no skill in the manipulation of glass. Or it may be that a laboratory has no glass-blower and the scientist has to rely on his own resources, combined perhaps with the services of some outside specialist. In other laboratories the scientist may find the available services to be deficient in various respects, or overloaded, so that more satisfactory progress is made when he himself becomes an amateur glass-worker. This possibility was emphasized by W. A. Shenstone in 1889, when he wrote that the amateur with practice can make almost all the apparatus he needs for lecture or other experiments with a considerable saving in expense 'and, which very often is more important, without the delay that occurs when one depends upon the professional glass-worker.' This latter advantage is, he writes, for himself a very weighty one.

We became interested in working glass ourselves when engaged in various researches in the Chemical Laboratories at King's College, London, and over the last few years we have become increasingly convinced that the scientific glass-worker can use methods which differ from those of the professional. The scientist is primarily interested in apparatus which gives him the results he seeks for, and if this apparatus lacks elegance in appearance and is made by methods which are looked upon unfavourably by the professional glass-blower, it by no means follows that the apparatus is defective from a scientific point of view.

We have therefore compiled this work in the hope that it will be useful to scientists faced with problems of glass-working. By avoiding the more difficult manipulations involved in professional glass-working, it seems possible for a scientist to assemble quite complex apparatus, including, for example, his own mercury diffusion pumps, mass spectrometer tubes, molecular beam generators and silica fibres; the preliminary practice required, which depends upon intrinsic ability, may take some hours a week for a period of two or three months. We have also endeavoured to bring together data

scattered in the literature, and to give an account of some of the more difficult methods of manipulating glass.

The researches which led to our interest in glass-working were supported by grants from the Research Fund of the University of London, from the Institute of Petroleum and from the Department of Scientific and Industrial Research (maintenance grants to A. J. C and J. D.).

A. J. B. R.
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December, 1956

Chapter 1

INTRODUCTION

GLASS has been known to mankind for some thousands of years. The manufacture and working of glass became a stable industry in Egypt at the beginning of the 18th dynasty. W. M. Flinders Petrie (1909) has described how, about 1370 B.C. in the time of Amenhotep IV, in the works at Tell-el-Amarna, a lump of glass was patted into a cylinder and then rolled into a rod which was drawn into cane about ¼ in. thick. This cane was wound on a mandrel to produce hollow vases. Later the art of pressing glass into open moulds was discovered. The blowpipe—an iron tube 4–5 feet long with a mouthpiece at one end—was probably discovered about the beginning of the Christian era, and, according to G. W. MOREY (1938), caused an industrial revolution. The use of tongs for manipulating glass was known to the Romans in A.D. 300. By this time, therefore, several of the basic methods which are now useful in constructing complex scientific apparatus had been developed. The glass in those early times was similar in many respects to a modern soda–lime–silica glass. Scientific method does not seem to have influenced glass production very much before the present century, although of important early investigations we may mention those of K. W. Scheele and A. L. Lavoisier on the durability of glass exposed to water and weak acids, and those of W. V. Harcourt and M. Faraday on the production and properties of glass.

The value of glass as a laboratory material is very great. A moderately skilled worker can fabricate complex glass equipment using simple tools; and perhaps of even greater value in research is the ease with which complex glass apparatus can be modified and added to with little or no dismantling. Glass is sufficiently chemically inert for most purposes, and vitreous silica may be used when extraordinary inertness is required. The transparency of glass is often valuable. Glass is a good electrical insulator, and metal electrodes in a glass envelope can be raised to incandescence by eddy-currents induced by a coil, outside the envelope, carrying high-frequency current. In vacuum researches glass is valuable because of the ease with which leaks are found with a Tesla coil, and on account of its

ready outgassing on baking. For nearly all practical purposes glass is vacuum-tight. Glass–to–metal seals of various kinds may be made, and different types of glass can be sealed together with appropriate intermediate glasses.

The manipulation of glass is a craft and has been handed down over the centuries largely by personal example and tradition. Venice was the most important centre of glass-working for the four centuries following its rapid development in the eleventh century to a dominating position; in 1279 a system of apprenticeship needing eight years was introduced there, and the closed and somewhat secretive nature of the craft was reinforced by the employment of assassins to remove glass-blowers who seemed likely to give away valuable secrets to other centres of the industry. Perhaps the aura of mystery which still to some extent surrounds scientific glass-working can be traced back to these times.

The basic techniques developed for scientific glass-working involve the manipulation of glass in the form of tubing or rod, using a flame as a source of heat. The article to be fashioned is held by the operator in both hands, and the part to be worked becomes a semi-molten mass in the flame and is shaped principally by blowing. Generally it is necessary to rotate the article in the flame, and this often requires a very high degree of muscular co-ordination, which can be developed by professional workers to a remarkable extent. This rotation operation is not easy; M. FARADAY (1842) stated the outstanding difficulty in the following words: 'But when the heat has brought the glass into a soft state, it is by no means easy so exactly to turn the tube at both ends alike, and so lightly yet equally to hold them, that the soft part shall retain its cylindrical shape; being neither twisted, nor bent, nor elongated, nor thrust up.' R. E. THRELFALL (1946) considers that one third of the art of glass-blowing consists essentially in being able to move both hands about, rotating a tube with each finger and thumb, and keeping constant both the distance between the hands and the speed of rotation. Considerable practice is necessary to gain great mastery of this technique, which we can call the bench–flame method of work, since the flame is in a fixed position on the bench. The problem facing the scientist who has to engage in his own glass-working is that of simplifying or modifying those operations which depend on extremely good muscular co-ordination. We consider this problem to be soluble when elegance can be sacrificed to utility.

The requirements of a scientist differ from those of a professional glass-worker. The starting point of a new research is new ideas, and when these require subsequent experimental investigation the apparatus used need be only good enough to give results which are

satisfactory from a scientific point of view. A glass apparatus must, in fact, hold together and work, but it may have an unsightly appearance. However, we must also note that experimental skill in itself is of great value to the scientist in pursuing his ideas, for as Faraday (1842) wrote: 'The person who could devise only, without knowing how to perform, would not be able to extend his knowledge far, or make it useful; and where the doubts or questions that arise in the mind are best answered by the results of an experiment, whatever enables the philosopher to perform the experiment in the simplest, quickest, and most correct manner cannot but be esteemed by him as of the utmost value.' In addition to these considerations, experimental skill is of particular value in developing new techniques; and these may lead to many unexpected developments. H. E. ARMSTRONG (1924) tells how the introduction in 1865 of the Sprengel pump—a fairly simple piece of glass-working—revolutionized the English water supply. Again we may note the important practical consequences following on the early work of H. L. CALLENDAR (1892) on vitreous silica tubes, and that of C. V. Boys and R. Threlfall on vitreous silica fibres.

In view of the difference in objective between the scientist and professional glass-worker, we consider that glass-working for the scientist should develop as its own craft, and indeed this has happened to a certain extent. An early systematic account is that of J. J. BERZELIUS (1833), which describes some of the basic operations. Faraday's *Chemical Manipulation* is a masterly treatment, referred to frequently in R. THRELFALL's notable work *On Laboratory Arts* (1898), which devotes 107 pages to glass-blowing and manipulation of glass. Threlfall seems to have been one of the first to describe what is now often called 'in-place' glass-blowing, in which the glass pieces are kept stationary by clamps, and the flame is moved. Joints are made with thin glass rod, now often called welding rod, by a method very similar to that used in 1370 B.C. in the Tell-el-Amarna factory. The difficulty of holding and rotating the glass is avoided, and thus, as Threlfall says, the method is most useful to the experimenter who wants to get on to other things before sufficient skill is acquired for the rotation method. The tradition of the craft of glass-working for scientists is continued, we think, by the works of W. A. SHENSTONE (1889), T. BOLAS (1898), B. D. BOLAS (1921), F. C. FRARY, C. S. TAYLOR and J. D. EDWARDS (1928), R. H. WRIGHT (1943), and J. D. HELDMAN (1946). Amongst eminent scientists who have carried on the glass-working craft with their own hands we may mention R. W. Bunsen, whose skill at the oil-fed blowpipe and whose exemplary patience when one of his pupils rapidly and several times

in succession broke the apparatus glass-blown by the master himself are described by H. E. ROSCOE (1901); J. Dewar, whose vacuum-jacketed glass vessels marked a new era in low temperature research; M. Bodenstein and W. Ramsay, both of whom are described by E. K. RIDEAL (1951) as uncannily skilled in the art of glass-blowing; and F. W. Aston, whose glass bulb discharge tubes and cooled charcoal–in–glass pumping systems first gave those exact mass relations of isotopic chemistry which contributed decisively to the opening of the atomic age. In modern times the old tradition that the masters of science should also be themselves masters of the practical craft of science has failed to persist, and the more eminent scientists are now usually not to be found at the working bench in the research laboratory. There is, we think, a consequent weakening of the craft basis of practical scientific work, and some retreat from the view expressed by I. Langmuir that work in the laboratory can be fun.

The increasing use in scientific research of borosilicate glasses over the last thirty years has not, on the whole, been accompanied by much departure from the traditional methods of glass-working. Generally it is much easier to rotate the flame about a stationary clamped article than to rotate the article in a stationary flame. Quite complex apparatus can be constructed by working with a hand torch—giving a movable flame held in the hand—on clamped apparatus. This method of work is specially suitable for the borosilicate glasses of low thermal expansion, which can be worked into quite knobby apparatus without there being much danger of cracking on cooling—in contrast to soda–lime–silica glass. The joining of two clamped tubes with a movable flame has been described by SHENSTONE (1889), THRELFALL (1898) and TRAVERS (1901). A.L. REIMANN (1952) has described some further uses of this general method, but the great number of complex operations which can be carried out with facility using a hand torch in place of a bench torch does not seem on the whole to have been realized; indeed, HELDMAN writes: 'End-seals with both tubes of approximately the same diameter and T-seals are, with practically no exceptions, the whole repertory of in-place glass blowing.' We are by no means in agreement with this statement. Many operations can be carried out with a hand torch on completely fixed glass, or on a fixed piece of glass to which some other part of glass can be joined by holding it in one hand whilst the other hand manipulates the hand torch. The results are usually not as elegant as those obtained by a skilled worker using rotation in a bench torch, but less skill and practice are required for the hand torch method. It has the further great advantage that the complexity of the apparatus being constructed can be steadily increased without

greatly increasing the glass-working difficulty. Furthermore, the scientist will probably only work glass from time to time, depending upon the requirements of the research or other work, and the hand torch method is far more suitable than the bench torch method for the operator who does not want to spend time on preliminary practice; it is also far more suitable for the scientist working in awkward positions on complex apparatus. We find that very complex apparatus may be assembled with a hand torch.

A clear account of professional methods of glass-working has been given by W. E. BARR and V. J. ANHORN (1949). Valuable information on some glass-working problems is also found in the works of J. STRONG (1940), M. C. NOKES (1948), A. J. ANSLEY (1950), A. ELLIOTT and J. HOME DICKSON (1951), and H. J. J. BRADDICK (1954). Accounts of the simpler operations have been given by W. E. PARK-WINDER (1947) and E. H. MORGAN (1953).

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Chapter 2

PHYSICAL AND CHEMICAL PROPERTIES OF GLASS

The Structure of Glass

A GLASS is a product of fusion which has cooled to a rigid condition without crystallizing. This definition includes a large number of organic glasses, and does not restrict the term 'glass' to inorganic substances, which is a frequent practice in the U.S.A. This restriction seems somewhat arbitrary, particularly when we consider how G. Tammann established the general principles of the glass-like state by research on organic glasses, and how the devitrification of technical glasses is paralleled by that of organic glasses. Tammann concluded that a glass could be regarded as a supercooled liquid in which the rotational movements of the molecules had been frozen (see W. E. WARNER, 1952). In fact, as R. Boyle described it about 1660, 'the particles of the glass agitated by the heat, were surpriz'd by the cold before they could make an end of those motions which were requisite to their disposing themselves into the most durable texture.' In modern terminology, a glass is thermodynamically unstable with respect to the corresponding crystal.

Crystalline silica (quartz, tridymite or cristobalite) in its various modifications is built up of SiO_4 tetrahedra linked together in a regular manner so that every oxygen is between two silicons. The tetrahedra therefore share corners. The arrangement in space of the tetrahedra is different in the various crystalline forms, but is always strictly regular. A silica glass, in contrast, again contains SiO_4 tetrahedra with every corner shared; but by slight distortions of the valency angles, as compared with the crystal, a continuous and irregular three-dimensional network is built up. The orientation about the Si–O–Si bond of one SiO_4 tetrahedron with respect to another can be practically random. Thus a two-dimensional picture of a silica glass would show a series of irregular rings, with an average number of about six tetrahedra in each ring, but with the number of tetrahedra in individual rings varying from three to ten or more. The silica glass satisfies the condition for glass formation proposed by W. H.

ZACHARIASEN (1932), namely that the substance can form extended three-dimensional networks lacking periodicity, with an energy content comparable with that of the corresponding crystal network. A glass does not, therefore, produce a regular diffraction pattern with X-rays; but a monochromatic X-ray beam incident on a glass is scattered, and a radial distribution curve may be constructed. The space average of the distribution of atoms round a given one can be deduced (see J. T. RANDALL, 1938). Much work of this kind has been carried out by B. E. Warren and his colleagues.

A soda–silica glass results from the fusion of Na_2O with SiO_2. The number of oxygens is more than twice the number of silicons, and some of the oxygens are bonded to only one silicon. A silicon bonded to one of these oxygens is at the centre of a tetrahedron which shares only three corners with other tetrahedra. With each singly bonded oxygen there is associated one negative charge. The sodium ions are found in the holes in the three-dimensional silicon–oxygen network. On the average, each sodium is surrounded by about six oxygens, and each silicon by four oxygens. In a soda–boric oxide glass of low soda content the extra oxygen is bonded between two borons, and there are no singly bonded oxygens. This can happen because in a boric oxide glass the co-ordination of boron by oxygen is triangular, and in the mixed glass some of the boron atoms become tetrahedrally co-ordinated by oxygen. When there is more than about 13–16 per cent of Na_2O in the glass, the boron atoms cease to change their co-ordination, and some singly bonded oxygens exist (B. E. WARREN, 1942).

Soda–silica glasses are not formed when the soda content exceeds that given by the formula Na_2SiO_3. For this formula, if every silicon atom is surrounded tetrahedrally by four oxygen atoms, then on the average two oxygens round every silicon are singly bonded, and a continuous network is just possible. With still more oxygen it is not possible. In a soda–silica glass with much less soda, there are many SiO_4 tetrahedra sharing every corner, and a number sharing only three corners. The way in which these different tetrahedra are distributed is not yet quite clear. There may be small regions where all the tetrahedra share four corners, and such regions are composed of pure silica; they may alternate with regions of, for example, $Na_2O\ 2SiO_2$. The composition may vary through the glass when sufficiently small regions are considered.

The general picture of a glass as a negatively charged irregular framework containing holes with positive ions in them enables a distinction to be made between the network-forming ions, which comprise the framework, and the network-modifying ions which go

n the holes. Silicon, boron and phosphorus are important network-orming ions. Sodium and potassium are important network-nodifying ions. Other ions can act in both capacities. This is probably true of aluminium, beryllium, zinc, iron and titanium. In a ead–silica glass it seems that lead atoms can take part in the network nd link SiO_4 tetrahedra together. Cobalt ions in network-modifying positions tend on heating to move into the network, and this can ause a colour change from pink to blue.

Most commercial glasses are based on silicates or borosilicates. A ypical hard borosilicate glass for chemical work may contain 80 per ent SiO_2, 12 per cent B_2O_3 and 4 per cent Na_2O. A soft soda–lime–ilica glass (usually referred to as soda glass) may contain 70 per ent SiO_2, 17 per cent Na_2O and 5·4 per cent CaO. Lead glasses, used or lamp and valve stems, may contain 30 per cent PbO, 57 per cent iO_2, 5 per cent Na_2O and 7 per cent K_2O. These glasses have high lectrical resistance. Glasses with exceptionally high softening tem-peratures contain 20–25 per cent of Al_2O_3. Borate glasses, sub-tantially free from silica (8 per cent SiO_2) are used for sodium ischarge lamps.

General accounts of the structure of glass have been given by J. E. TANWORTH (1950), B. E. WARREN (1940) and C. J. PHILLIPS (1948).

General Physical Properties of Glass

he physical properties of a given specimen of glass may depend pon the previous history of the specimen. This is particularly the ase for the mechanical strength under tension, when the surface pre-reatment of the specimen is of decisive importance. The thermal xpansion and viscosity of glass also depend to some extent on the istory of the specimen. The importance of this factor has been mphasized by A. E. DALE and J. E. STANWORTH (1945). R. W. OUGLAS (1945) has given a valuable review of the physical pro-erties of glass.

Mechanical Strength

he important property for the practical worker is the strength of lass under tension. The surface of glass very probably contains umerous extremely small cracks extending into the glass, and when tensile stress is applied there is a concentration of stress at the ends f these cracks, which causes them to grow further into the glass, ntil at some crack breakage occurs and is propagated through the pecimen. Glass usually breaks in a direction at right angles to the irection of maximum tensile stress. A newly-drawn glass fibre is ree from these surface cracks and is much stronger than an old

fibre; but touching a new fibre, even with the fingers, greatly weakens it. An old fibre is actually strengthened by removing the surface layer with hydrofluoric acid, even though the cross-section is reduced. The strength of a glass under tension varies from one specimen to another. A further complication is the variation of the tensile strength with the time for which the stress is applied. A tensile stress which does not cause fracture after a short time of application may do so after a long time. There is in fact a delayed fracture of glass.

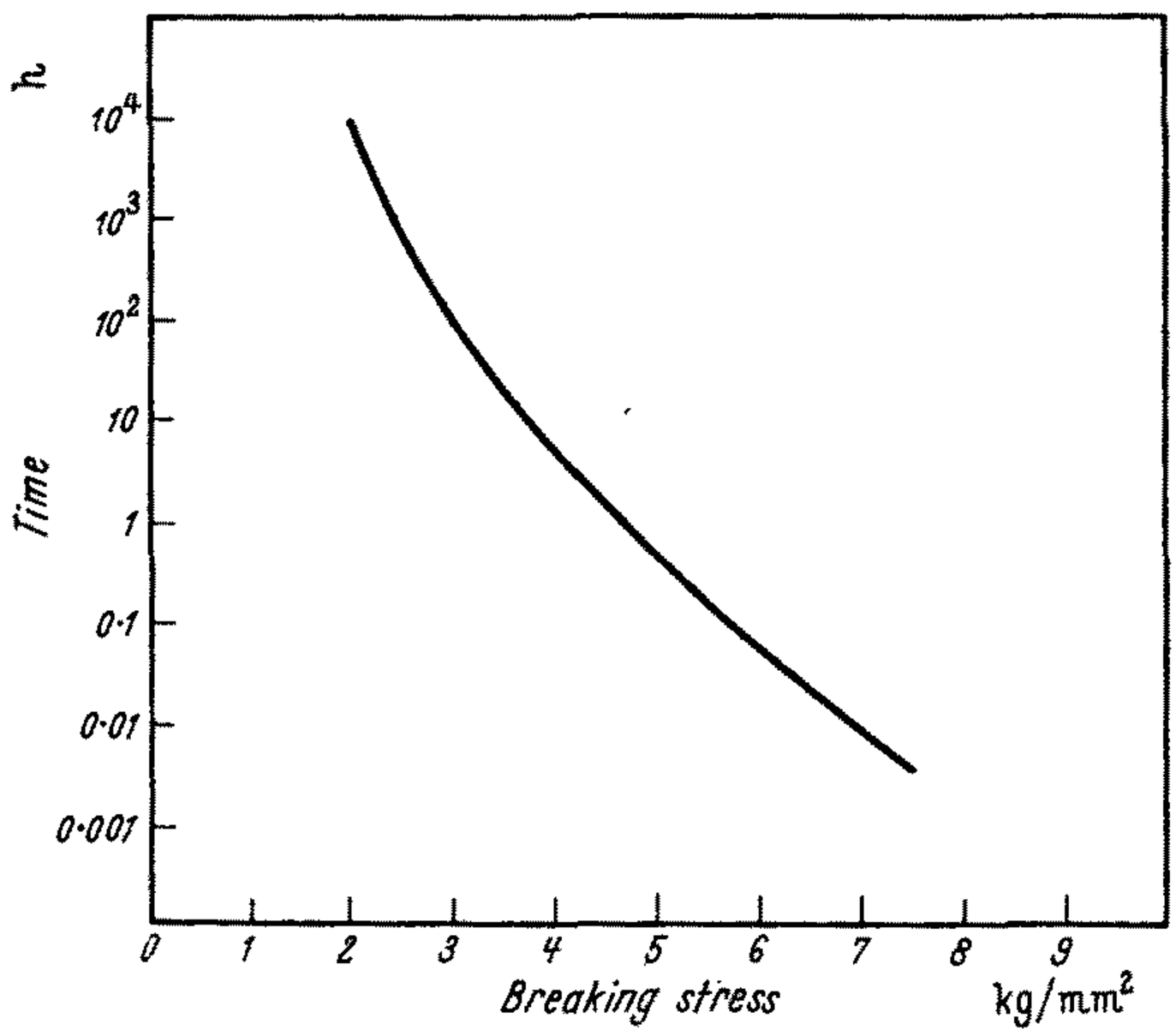

FIGURE 1. Nature of the relation between time of loading and breaking stress for glass. This property is of a statistical nature, and the particular curve shown can only be taken as representative

The nature of the relation between time of loading and breaking stress for a borosilicate or soda–lime–silica glass is shown in FIGURE 1. The curve given must be taken as representative only. A typical figure for the safe tensile strength for prolonged loading times is 0·7 kg/mm² (1000 lb/in.²). Similar results for the relation between bending stress and time to fracture are found when a tensile stress is produced by bending a glass rod into an arc of a circle. From FIGURE 1 we note that an increase of stress by a factor of 4 reduces the time required for fracture by over 10^6 times. If a certain load is supported for one hour by a certain piece of glass, one quarter of the load should be supported for a million hours. This can be made use

of in testing the extent to which stress can be applied to a glass apparatus. Four times the stress the glass must support can be applied for a short time.

The delayed fracture of glass, shown in FIGURE 1, must be borne in mind in assembling apparatus. If, for example, an apparatus is clamped so that bending stress is introduced, the apparatus may break after a long interval. Chemical reactions at the surface of the glass may be partially responsible for delayed fracture: C. GURNEY and S. PEARSON (1952) found a soda–lime–silica glass to be stronger in vacuum, and to be weakened by carbon dioxide and water in the surrounding atmosphere.

Thermal Expansion

The coefficient of linear thermal expansion is almost constant, for most types of glass, for temperatures up to 400–600°C. The actual value depends on the chemical constitution of the glass. It then increases rapidly above a certain temperature, often called the

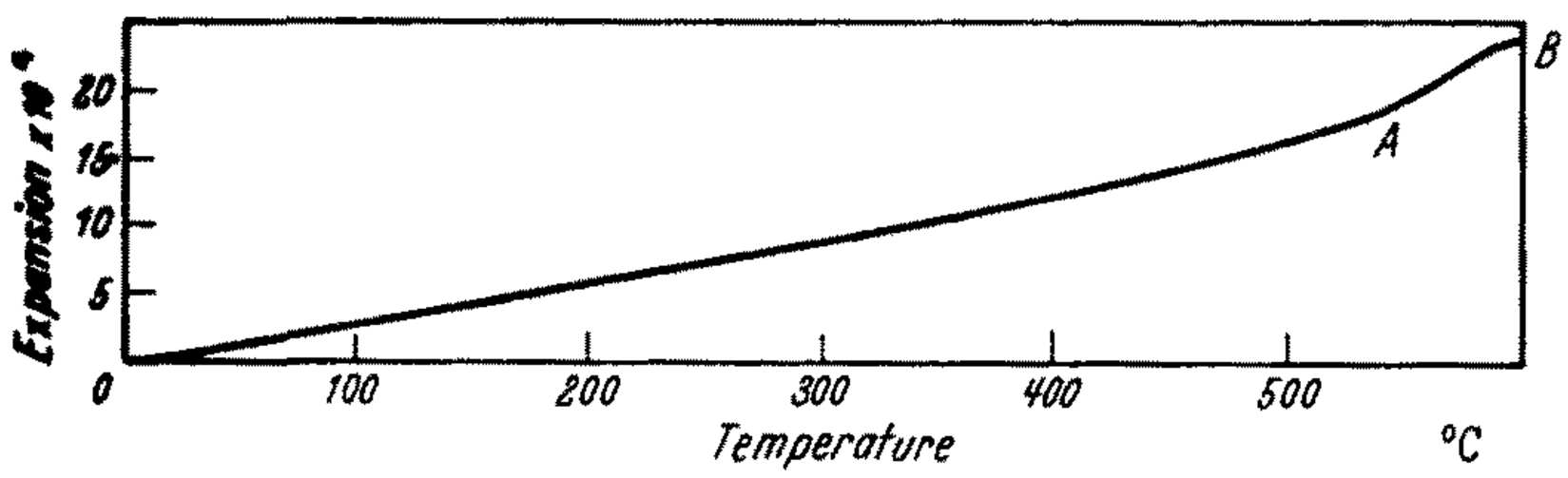

FIGURE 2. A typical expansion curve for a hard borosilicate glass (*Phoenix*)

'transformation point'. This is not, however, a characteristic temperature, since it depends on the thermal history of the specimen and the rate of heating. At a higher temperature the glass softens and ceases to expand. This is sometimes called the 'softening temperature' or the 'Mg point'. Confusion may result from another definition of softening temperature, depending upon the rate of extension of a fibre by viscous flow. This latter softening temperature, which corresponds to a viscosity of $10^{7\cdot6}$ poises, is very much higher than the Mg point. A typical linear expansion curve for a borosilicate glass (*Phoenix*) is shown in FIGURE 2. *A* is the transformation point and *B* the Mg point. The temperature corresponding to *A* is often called the 'lower annealing temperature', and corresponds to a viscosity of about 10^{14} poises; that corresponding to *B* is often called the 'upper annealing temperature', and corresponds to a viscosity of

about 10^{12} poises. Another definition in common use, especially in the U.S.A., is to call the 'annealing temperature' that at which the viscosity is $10^{13.4}$ poises. This is then between *A* and *B* on the thermal expansion curve. It is useful to bear in mind the confused state of terminology and definitions when using tabulated data on the thermal properties of glass. The viscosities mentioned above are, perhaps, not established with certainty. At the lower annealing temperature, annealing is actually extremely slow. This temperature is not used for the practical annealing of laboratory apparatus.

Thermal Endurance

This measures the ability of the glass to stand sudden changes of temperature without fracture. When a specimen of glass is suddenly heated uniformly over all its surface, the heat penetrates slowly into the interior. The outside layers are heated first, and being unable to expand fully they become subject to a compressive stress, whilst the inner layers become subject to a tensile stress. When the specimen at a uniform temperature is suddenly cooled over all its surface, the surface layers are subject to tensile stress. Since the mechanism of fracture usually involves surface cracks, glass is more likely to break on sudden cooling than on sudden heating. The magnitude of the stress produced on sudden cooling depends on the modulus of elasticity and the coefficient of linear thermal expansion, and, in a way not important in practice, on Poisson's ratio. Thermal endurance is measured by somewhat empirical methods, and is again a statistical quantity. A heat-resisting glass is one having a high thermal endurance; a hard glass has a high softening temperature. A 1-mm thick beaker of a hard borosilicate glass, such as *Pyrex*, *Phoenix* or *Firmasil*, will require a thermal shock, by sudden cooling, of about 325°C to give appreciable probability of fracture. For a soda–lime–silica beaker the corresponding figure is about 120°C. Beakers of *Monax* glass stand a much greater thermal shock than the soda–lime–silica beaker; the beakers of standard thickness can usually survive a thermal shock of 240–250°C. Thick glass fractures with less thermal shock than thin glass.

The glass-worker subjects tubing to thermal shock by suddenly placing it in a hot flame. The inner surface of the glass tube is then not heated directly, and is very quickly subjected to tensile stress. The hard borosilicate glasses as tubes can usually be placed immediately in an oxy-coal gas flame without fracture, but complex apparatus, especially when internal seals are present, requires more gentle heating. Soda–lime–silica glass tubes need gentle warming at first, particularly when the end of a tube which has not been fire-polished is put in the

flame. The end of a tube is fire-polished by fusing it in a flame, and this process closes up surface cracks. Vitreous silica has very great thermal endurance: small red-hot articles can be quenched in water without cracking.

Thermal Conductivity

For the hard borosilicate glasses and the soda–lime–silica glasses this is around 0·0025 cal $°C^{-1}$ cm^{-1} sec^{-1}. For vitreous silica (*Vitreosil*) in the transparent form it is 0·0025 up to 500°C, and 0·0035 from 500 to 1000°C; for the translucent form it is 0·0033.

Viscosity and Softening Temperatures

These properties have already been mentioned in connection with thermal expansion. The viscosity decreases rapidly with increasing temperature. A linear relation is found between the logarithm of the viscosity and the reciprocal of the absolute temperature. This is convenient for extrapolation. When the viscosity has the value $10^{7·6}$ poises the glass is mobile enough to be drawn into threads, and the temperature is sometimes called the softening temperature (see p. 11). At temperatures between the lower and upper annealing temperatures (*A* and *B* in FIGURE 2) the viscosity can change with time—when the glass is suddenly cooled the viscosity slowly increases to an equilibrium value and when the glass is heated the viscosity slowly decreases to an equilibrium value—in fact time is required for the equilibrium viscosity values to be attained. Glass is often worked when its viscosity is about 10^4 poises; for a hard borosilicate glass this corresponds to a temperature of about 1200°C.

Elastic Properties

Pyrex Chemical Resistance Glass has a Young's modulus of $6·1 \times 10^{11}$ dynes/cm^2, a modulus of rigidity of $2·5 \times 10^{11}$ dynes/cm^2 and a Poisson's ratio of 0·22. Similar values are found for other glasses.

The extension of an amorphous material under a tensile force can be resolved into three parts; first, an immediate elastic extension, which is immediately recoverable on removing the tensile force; secondly, a delayed elastic extension which is recoverable slowly; and thirdly, a plastic extension, viscous flow, or creep, which cannot be recovered. With glass at ordinary temperatures, this plastic extension is practically absent. A very slow delayed elastic extension occurs. This effect can be troublesome in work with torsion fibres. The delayed elastic effect in vitreous silica fibres is 100 times less than in other glass fibres, and viscous flow of silica is negligible below 800°C (N. J. TIGHE, 1956). For exact work vitreous silica torsion fibres are therefore used.

Thermal Capacity

For *Pyrex* glass the thermal capacity (specific heat) between 0 and 250°C is given by C (calgm^{-1}°C) = 0·174 + 0·00036t where t is the temperature in °C.

Electrical Resistance

The resistance of vitreous silica (*Vitreosil*) in the translucent form at room temperature exceeds 2×10^{14} ohm cm. Glasses containing metal ions in network-modifying positions are ionic conductors. In a soda–lime–silica glass, for example, the current is carried by sodium ions and the resistance at 150°C may be around 10^8 ohm cm. *Lemington W.1.*, a hard borosilicate glass, has a resistance of about 10^{10} ohm cm at 200°C. A typical lead glass, *Wembley L.1.*, has a very much greater resistance both at room temperature and normal lamp-operating temperatures than a soda–lime–silica glass, and is therefore valuable for lamp and valve pinches. The resistance of *L.1.* at 150°C is 10^{12} ohm cm. Generally the volume resistance due to ionic conduction decreases rapidly with temperature. The logarithm of the conductivity is a linear function of the reciprocal of the absolute temperature. The surface of most glasses is very hydrophilic, and there is a surface conductivity which depends upon the relative humidity. For *Phoenix* glass, for example, the volume resistance of a centimetre cube at room temperature is about 3×10^{14} ohm, but the surface resistance at 60 per cent relative humidity is 7×10^{11} ohm, and at 81 per cent relative humidity it is $5{\cdot}4 \times 10^9$ ohm. In very humid atmospheres it is possible to have an electrical shock by touching the surface of a soda glass apparatus containing electrodes at high potential. The water layer on the glass becomes slightly alkaline after a time by reaction with sodium from the glass; the apparatus should be wiped from time to time with a cotton cloth. It is best in these cases to use a borosilicate glass. The surface conductivity of glass was discovered by M. FARADAY (1830).

Density

For the soda–lime–silica glasses this is about 2·5 gm/cm^3; for the borosilicate glasses it is very nearly 2·25 gm/cm^3 and hardly changes with slight variations in composition. *Wembley L.1.* lead glass has a density of 3·08. A very dense lead glass has a density of 5·2.

Hardness

Generally glasses with a high silica content are more resistant to abrasion than low silica content glasses. The hardness therefore increases with increase of softening temperature. Lead glasses can be scratched quite easily.

Transmission of Light

A 1-mm thick sheet of *Phoenix* glass will transmit 90 per cent or more of the light incident on it, for wavelengths of 350 millimicrons to almost 2 microns. In the infra-red region a strong absorption occurs at 3 microns and little transmission beyond 4 microns. In the ultra-violet region increasing absorption occurs as the wavelength falls below 350 millimicrons and very little transmission occurs below 270 millimicrons. The transparent variety of vitreous silica (fused quartz) has very superior optical properties, and is widely used in photochemical and optical researches. In the ultra-violet region it transmits at high efficiency down to 1850 Ångström units (185 millimicrons). 'Quality O.H. *Vitreosil*' of The Thermal Syndicate Ltd is a special optical quality in which the absorption band at 2400 Ångströms has been eliminated. A special quality of fused quartz is also available which transmits infra-red up to 3·5 microns approximately ('I.R. quality *Vitreosil*' of The Thermal Syndicate Ltd). In this *Vitreosil* the absorption band at 2·7 microns has been much reduced.

The Stress-Optical Coefficient

It is not usual to take quantitative measurements of the strain in glass apparatus made for research; when a strain-viewer is used (p. 43) qualitative observations are normally made. Quantitative measurements can be made when the stress–optical coefficient is known. The theory involved requires a knowledge of the optical behaviour of doubly refracting materials and depends on the fact that a ray of plane-polarized light entering strained glass is broken into two rays—the 'ordinary ray' and the 'extraordinary ray'—vibrating at right angles to each other. For glass subject to simple axial tension or compression, the extraordinary ray vibrates in the plane which includes the axis of the stress. The birefringence of strained glass is proportional to the strain, and thus to the stress. The stress–optical coefficient is the maximum double refraction or birefringence observed in polarized sodium light for 1 cm path length when there is a uniform stress of 1 kg/cm². It is expressed either in wavelengths of sodium light or in millimicrons. This coefficient varies from one glass to another; it is around 3·5 millimicrons, or 0·006 wavelengths of sodium light. A. JOHANNSEN (1918) has given an account of methods for determining double refraction, and very valuable data for practical work are given by J. H. PARTRIDGE (1949).

General Chemical Properties of Glass

Resistance to Chemical Actions

Vitreous silica is the most chemically inert glass for most purposes.

It is not affected by halogens or acids, except for phosphoric and hydrofluoric acids. Phosphoric acid attacks fused silica at temperatures of 300–400°C, and hydrofluoric acid attacks it at room temperature, forming silicon tetrafluoride and water. At high temperatures silica reacts with caustic alkalis, certain metallic oxides, and some basic salts, and cannot be used for incinerating these materials. Over 1600°C, fused silica is reduced to silicon by carbon. It can also be reduced at high temperature by hydrogen. It is unaffected by water under normal conditions but is attacked by strong solutions of alkalis.

The hard borosilicate glasses are highly resistant to attack by water; but just as the sodium ions in the glass are slightly mobile under the influence of an electric field (p. 14), so also they can be mobile by thermal agitation and escape from the glass into water in contact with it and be replaced by hydrogen ions. This effect is slight: for example, a *Firmasil* beaker in an autoclave containing water at 150°C loses about 0·00015 gm of sodium per dm^2 in four hours. A soda–lime–silica glass loses sodium to water at a much greater rate. The resistance of borosilicate glass to most acids is very good, but strong aqueous alkalis produce visible attack. The network of triangles and tetrahedra is attacked, so the glass tends to dissolve as a whole. Soda–lime–silica glass usually has less chemical resistance than a borosilicate glass. Alkaline attack, however, becomes much greater on glasses with high silica content. Alkalis can also leach out boric oxide from a borosilicate glass. Hydrofluoric acid dissolves glass, and glacial phosphoric acid attacks most kinds of glass.

The Weathering of Glass

A reaction between sodium from the glass and atmospheric water and carbon dioxide can lead to the formation of sodium carbonate, which crystallizes in fine needles. A potash glass forms potassium carbonate, which is too deliquescent to crystallize out. A lead glass can react with hydrogen sulphide, and to a smaller extent with carbon dioxide, sulphur dioxide, and acid vapours.

Phenomena Arising from the Heating of Glass

A rapid evolution of adsorbed water first occurs on heating glass; this is followed by a persistent evolution, due to gas (mostly water) diffusing from the interior. Above 300°C the two processes are fairly clearly separated. The adsorbed water is rapidly and completely removed, and the quantity of gas evolved by the persistent evolution

is proportional to the square root of time. The process has an activation energy. For a soda–lime–silica glass over 98 per cent of the evolved gas is water. B. J. TODD (1955) has studied these effects in detail. The adsorbed water on glass can be troublesome in gaseous manipulation, as R. W. Bunsen first appreciated.

At high temperatures glass loses its more volatile components. The loss of silica, lime, magnesia and alumina is negligible, but boric oxide, lead oxide, sodium oxide and potassium oxide can also be lost. When the glass is heated in a flame, reaction may occur with some of the flame gases; sulphur dioxide can react with soda glass and lead glass to form sodium sulphate and lead sulphate respectively, and of these only the former can be washed off. An account of these effects is given by W. E. S. TURNER (1945). The loss of weight of vitreous silica on ignition is negligible; crucibles can be heated to 1050°C, and precipitates can be ignited at 1000°C in crucibles with a porous base of vitreous silica.

Diffusion through Glass

The mobility of the sodium ions in a soda–lime–silica glass at elevated temperatures is fairly high; if an evacuated bulb of such a glass is dipped into molten sodium nitrate and electrolysis is brought about by bombarding the inside of the bulb with electrons, the circuit being completed with an electrode in the sodium nitrate, then metallic sodium appears in the bulb. By immersing the bulb in other molten salts the sodium ions can be replaced by ions of silver, copper, thallium and vanadium. These ions also diffuse into glass from their molten salts in the absence of an electric field. When potassium is distilled in a borosilicate glass vessel it becomes slightly contaminated with sodium which diffuses from the glass and is replaced by potassium (D. K. C. MACDONALD and J. E. STANWORTH, 1950). Vitreous silica allows helium, hydrogen, neon, nitrogen, oxygen and argon to diffuse through it, with the permeability decreasing in the order given. The permeability of silica becomes greater if the glass devitrifies. The permeability to helium of soda–lime–silica glass is 10^5 (or more) times less than that of vitreous silica. For practical vacuum purposes soda and borosilicate glasses can be regarded as impermeable to gases at ordinary temperatures, except in work at extremely low pressures when the diffusion of atmospheric helium through the glass may become significant.

The permeability of glass at high temperatures seems to have been discovered by R. Boyle. In his collected works published in 1744 there is a paper in Volume III 'A discovery of the perviousness of glass to ponderable parts of flame' in which he writes '. . . it is plain

that igneous particles were trajected through the glass, which agrees with the Epicureans . . .'

Some Surface Properties

The chemical properties of glass are largely determined by its surface, because the metal ions present in most types of glass are in fact reactive enough, but only those at the surface can react. The very slow rates of diffusion in glass at ordinary temperatures limit its reactivity, so that, as M. FARADAY (1830) wrote 'Glass may be considered rather as a solution of different substances one in another, than as a strong chemical compound; and it owes its power of resisting agents generally to its perfectly compact state, and the existence of an insoluble and unchangeable film of silica or highly silicated matter upon its surface.' The surface composition of glass may be very different from the bulk composition, for volatilization occurs during the forming process, and weathering occurs subsequently; both processes produce a surface resembling vitreous silica. It is possible that the SiO_4 tetrahedra on the surface terminate in OH groups to which adsorbed water is normally bound. The glass surface can be made hydrophobic instead of hydrophilic by allowing adsorbed water and surface hydroxyls to react with various monoalkyldichlorosilanes ($RHSiCl_2$), when hydrogen chloride is formed by elimination.

The surface properties of glass are of great importance in many reaction kinetic studies, particularly those involving the termination of reaction chains on the walls of the vessel. When a glass reaction vessel is used in such cases, it is usually found that it must be matured, by carrying out a number of reactions in it, before reproducible results can be obtained. In many cases, reproducible results are only obtained when the vessel is kept continuously at the reaction temperature, and exposed only to the reaction mixtures; if the apparatus is cooled, and air let in to make an alteration or repair, different results may be obtained subsequently. This is particularly the case in oxidation reactions.

Devitrification

This is the process of the crystallization of one or more of the constituents of glass. Generally a glass is thermodynamically unstable with respect to these crystals, but at ordinary temperatures the crystallization rate is quite negligible. Crystallization may occur when the glass is worked at high temperature. The crystals which appear in a supercooled melt are not necessarily those of the stable solid phase at the temperature concerned: for example, cristobalite can appear at temperatures for which tridymite is the stable crystalline

form of silica. The crystals which are most likely to separate from soda–lime–silica glass are those of calcium silicate, tridymite and cristobalite. Calcium silicate occurs usually as the monoclinic form wollastonite, but sometimes in a hexagonal form. The monoclinic form tends to appear as very long, thin crystals bunched together to give a brush-like appearance. The hexagonal form is not acicular. Tridymite is hexagonal, and forms hexagonal stars, like snow, by twinning at 60°. The cristobalite forms twins at 90°. Devitrification on the surface of glass seems to depend upon loss of alkali-oxides, and may be assisted by dust particles.

Transparent vitreous silica (transparent *Vitreosil*) is liable to devitrify if potassium or sodium compounds are present. The surface of the material must be thoroughly cleaned, and the part to be worked should then not be touched with the fingers.

When devitrification is observed in the soda and borosilicate types of glass, the crystals may sometimes be removed by fusion of the glass in the flame; alternatively the semi-molten devitrified portion may be removed with tongs, and replaced by fresh glass added as thin rod. If there is a large extent of devitrification the portion of glass should be completely cut out and replaced. Old soda glass apparatus is very liable to devitrify when repaired: F. C. FRARY, C. S. TAYLOR and J. D. EDWARDS (1928) advise, in such cases, a preliminary washing of the glass with dilute hydrofluoric acid to remove the surface layer.

Thermal Strain in Glass

When a block of glass is suddenly heated on all its faces, the outer layers are under compression and the inner layers are under tension, as explained on p. 12. This strain is temporary, however, since it vanishes as soon as the temperature gradient vanishes. Temporary strain is similarly produced when the surface of a block of rigid hot glass is cooled. Above the upper annealing temperature (p. 11) a stress can only exist in glass for a short time, because the glass flows to relieve the stress. Stress is relieved only very slowly at the lower annealing temperature. Thus over a certain temperature range glass changes from a viscous to a rigid body. Consider a block of glass to be rapidly cooled through this temperature range, so that there is always a temperature gradient. In the viscous region the glass is strain-free, and thus when it first becomes rigid it is also strain-free. Thus there is a rigid block of glass containing a temperature gradient but free from strain. When this temperature gradient is removed, the inner layers of the glass are in tension and the outer layers in compression. There is then a permanent strain in the glass. Clearly, when

a complex glass object is cooled rapidly there will finally be a complex distribution of permanent strain, and this can be great enough to cause fracture of the glass.

The drops of glass produced by Prince Rupert of Bavaria by dropping molten glass into oil become rigid when there is a great temperature gradient and the surface is consequently in strong compression. This makes the drops very strong, but they disintegrate violently as soon as the tail of the drop is cut off, when the internal stresses are no longer balanced.

Annealing of Glass

The object of this process is to prevent permanent strain arising from the cooling of glass. The glass must be cooled slowly through the critical temperature range in which it becomes rigid and ceases to relieve internal stresses by viscous flow. The rate at which these stresses are relieved in the annealing range of temperatures (*A* to *B* in FIGURE 2) depends on temperature; when this is such that the viscosity is $10^{13.4}$ poises, the glass will become practically stress-free in 15 minutes (A. E. DALE and J. E. STANWORTH, 1945). Below the lower annealing temperature the glass can be cooled quickly without introduction of permanent strain, but the temporary strain could become great enough to fracture the article.

Annealing is carried out most satisfactorily in an oven (p. 45). Complex articles of *Pyrex* glass can be annealed at 560°C for 30 minutes, followed by slow cooling with the oven door shut. Articles of *Firmasil* glass should be annealed at 575°C, but even at 475°C strain is very slowly removed. For *Phoenix*, the upper annealing temperature is 600°C and the lower annealing temperature is 520°C. Annealing at 560–580°C is therefore satisfactory for this glass. The annealing temperatures of these borosilicate glasses are not at all critical. The article must not, of course, be made too hot, or it will deform. Annealing is of great importance for articles made of a soda–lime–silica glass. *Wembley X.8.* soda glass should be annealed in the range 520–400°C, and the General Electric Company, which makes this glass, recommends annealing at a high temperature of 520°C for 5–10 minutes, followed by cooling to an intermediate temperature of 460°C at a rate dependent upon the glass tubing thickness. These rates are :—

3°C per minute for $\frac{1}{2}$ mm wall thickness
2°C per minute for 1 mm wall thickness
1°C per minute for 3 mm wall thickness.

The glass should be cooled from the intermediate temperature of 460°C to a low temperature of 400°C at double the above rates. The

article can then be cooled to room temperature at any rate possible without cracking it by temporary thermal strain. The corresponding high, intermediate and low temperatures for *Wembley L.1.* lead glass are 430°C, 390°C, and 340°C. The same annealing schedule can be used. For *Wembley M.6.* 'white neutral' glass the temperature range is 480–450°C. Again the same schedule should be used.

Complex apparatus assembled on the bench must be annealed by flame, and this method must also be used when no oven is available (see p. 170). In our experience, very complex apparatus of *Pyrex* glass can be flame-annealed satisfactorily. Usually with *Pyrex* the apparatus either cracks in a day or two after making, or else not at all. Complex apparatus of soda–lime–silica glass can be annealed by flame, but we do not find this satisfactory. For research apparatus it is best to avoid this kind of glass. With a complex vacuum apparatus of borosilicate glass a fracture can often lead to unfortunate consequences, especially when there are many mercury cut-offs present; and in such cases it is well, before evacuating, to wait for a few days after a repair or alteration has been made in a position where flame annealing is difficult.

Some Types of Glass for General Use

Many different kinds of glass are made. In this Section and the following Sections we mention only a few of these which are useful in the laboratory.

Wembley X.8.

This is a soda–lime–silica glass, containing magnesia and boric oxide (less than 1 per cent), made by the General Electric Co. It is often described as *GEC X.8.* or simply as *X.8.* The linear coefficient of thermal expansion between 20 and 350°C is $9{\cdot}65 \pm 0{\cdot}10 \times 10^{-6}$. This glass is available as tubing and rod in a wide range of sizes.

BTH No. 94

Made by the British Thomson-Houston Co., this is a soda–lime–silica glass. The linear expansion coefficient is $9{\cdot}5 \times 10^{-6}$ (50–400°C). The glass is available as machine-drawn tubing for bench working etc.

Monax

This is a borosilicate glass, free from arsenic, antimony and lead, made by John Moncrieff Ltd. The coefficient of linear expansion is $3{\cdot}4 \times 10^{-6}$/°C. The glass is available as tubing and rod of various sizes; in addition many standard pieces of laboratory glass-ware constructed in *Monax* are available.

Pyrex

Pyrex is a borosilicate glass free from alkaline earth metals, zinc and heavy metals, made by James A. Jobling & Co., and in the U.S.A. at the Corning Glass Works, where *Pyrex* was developed. The linear expansion coefficient (20–400°C) is $3{\cdot}2 \times 10^{-6}$. *Pyrex* tubing is supplied in a wide range of sizes, with standard wall, heavy wall, or as extra heavy tubing. Capillary tubing and rod is supplied in a range of sizes. Precision-bore tubing with very accurate inside dimensions is also made. A very wide range of laboratory apparatus is manufactured in *Pyrex* glass.

Phoenix

Phoenix, also, is a borosilicate glass made by the British Heat Resisting Glass Co. The linear expansion coefficient (50–450°C) is $3{\cdot}24 \times 10^{-6}$. Tubing and rod are supplied in a wide range of sizes. The tubing is made with either standard wall or heavy wall.

Firmasil

Laboratory glassware made from this glass is available. It is a borosilicate glass manufactured by Wood Bros. Glass Co. The linear expansion coefficient (0–400°C) is $3{\cdot}2 \times 10^{-6}$.

Some Types of Glass for Sealing to Metals

Again we mention only a selection from the many sealing glasses which are made.

Lemington W.1.

This is another borosilicate glass, and was developed to make strain-free seals with tungsten. It is made by the General Electric Co., and often described as *GEC W.1.* or simply as *W.1.* The linear expansion coefficient (20–350°C) is $3{\cdot}75 \times 10^{-6}$.

BTH No. 9

A borosilicate glass for sealing to tungsten, and is made by the British Thomson-Houston Co. It is often described as *BTH C.9* or as *C.9.* The linear expansion coefficient (50–400°C) is $3{\cdot}65 \times 10^{-6}$.

Dial 36

Made by Plowden & Thompson Ltd .*Dial 36* is a borosilicate glass for sealing to tungsten. The linear expansion coefficient (20–300°C) is $3{\cdot}6 \times 10^{-6}$.

Corning No. 7720 (*Nonex*)

Corning No. 7720, known commercially as *Nonex*, is a borosilicate glass containing lead and made at the Corning Glass Works. It is used for sealing to tungsten metal. The coefficient of linear expansion of *Nonex* is $3{\cdot}6 \times 10^{-6}$/°C. This is relatively low, although slightly higher than that of *Pyrex* brand glass (*Corning No. 7740*), and matches sufficiently with that of tungsten, $4{\cdot}5 \times 10^{-6}$/°C, to cause the glass to make satisfactory seals to this metal.

Nonex begins to soften, and can be worked, at temperatures a little below those required for *Pyrex*. Care must be taken when working it to avoid reducing the lead and causing the glass to blacken. The tip of an oxidizing flame must therefore be used. *Nonex* will seal well to *Pyrex*, though sometimes uranium glass is used between the two.

BTH No. 40

A borosilicate glass for sealing to alloys of the Kovar type (p. 111); it also seals to some electrical porcelains. It is made by the British Thomson-Houston Co., and is often referred to as *BTH C.40* or as *C.40*. The linear expansion coefficient is $4{\cdot}85 \times 10^{-6}$ from 50 to 400°C.

Lamington FCN

Made by the General Electric Co. and often called *GEC FCN* or *FCN*, this is a borosilicate glass for sealing to Kovar-type alloys. The linear expansion coefficient (20–350°C) is $4{\cdot}75 \times 10^{-6}$.

Kodial

A Kovar-sealing borosilicate glass made by Plowden & Thompson Ltd, this has a linear expansion coefficient (20–300°C) of $4{\cdot}9 \times 10^{-6}$.

Dial 444

This is a soda glass for sealing to platinum, and is made by Plowden & Thompson Ltd. The linear expansion coefficient (20–300°C) is $9{\cdot}8 \times 10^{-6}$.

Some Types of Glass for Special Purposes

Here we mention only a few of the glasses which are made for special purposes.

Wembley L.1. Lead

Made by the General Electric Co., this glass was developed primarily for pinches and exhaust tubes in lamps and valves. It seals directly

to platinum and to copper-clad wire (p. 111). The coefficient of linear expansion is $9{\cdot}05 \times 10^{-6}$ from 20 to 320°C. A lead glass, such as *L.1.*, is useful as an intermediate glass for joining soda glasses of different thermal expansions.

BTH No. 12

BTH No. 12 is a lead glass for lamp and valve manufacture, made by the British Thomson-Houston Co. The linear expansion coefficient (50–400°C) is $9{\cdot}1 \times 10^{-6}$.

Wembley M.6, 'White Neutral'

This is made by the General Electric Co., and is used for medical ampoules. It loses very little alkali to aqueous solutions. The linear expansion coefficient is $7{\cdot}3 \times 10^{-6}$ from 20 to 350°C. This is intermediate between the soft and hard glasses, and *M.6.* is sometimes used in graded seals.

Wembley 'Amber Neutral'

This glass, made by the General Electric Co., is similar to *white neutral* above, but is coloured amber by addition of iron and manganese. The linear expansion coefficient is $7{\cdot}5 \times 10^{-6}$ from 20 to 350°C. It is useful for ampoules when the contents need protection from ultra-violet radiation.

Lemington H.26X.

Lemington H.26X., made by the General Electric Co., is a very hard borosilicate glass of high softening temperature. The Mg point is 780°C. It is used in high pressure mercury vapour lamps. The linear expansion coefficient is $4{\cdot}6 \times 10^{-6}$ from 20 to 580°C. Sodium and potassium are absent and alumina is present in quantity in this glass.

Sodium Resistant, NA.10

This type, made by the General Electric Co., is resistant to sodium vapour and is used in sodium vapour discharge lamps. It has a very high boric oxide content, a low softening temperature, and a low electrical resistance. It is used as an internal layer in soda glass tubing (*X.8.*).

Dial 43

A borosilicate glass with an expansion coefficient intermediate between that of the tungsten-sealing and Kovar-sealing glasses. It is a useful sandwich glass for joining between these glasses. The linear expansion coefficient (20–300°C) is $4{\cdot}2 \times 10^{-6}$. It is made by Plowden & Thompson Ltd.

SOME TYPES OF GLASS FOR SPECIAL PURPOSES

Fused Silica

The Thermal Syndicate Ltd make fused silica (*Vitreosil*) tubes of several kinds. Translucent and transparent *Vitreosil* are manufactured; the former is supplied with 'sand', 'satin' or 'glazed' surface, and the latter usually has a glazed surface. A glazed surface should be used in vacuum work; the transparent tubing is best. *Vitreosil* tubing is available with a wide range of sizes and with several wall thicknesses. Rods, bars and capillary tubes are available. Many items of laboratory equipment are manufactured in *Vitreosil*. They are specially valuable when high temperatures and high thermal endurance are needed. The linear expansion coefficient is $0{\cdot}54 \times 10^{-6}$.

Graded Seal Glasses

The General Electric Co. makes a range of sealing glasses. For joining silica to hard glass, and hard glass to soft glass, the sealing glasses given below can be used. The coefficients of linear expansion are also given.

Sealing glass type	Expansion coefficient	Purpose
WQ.31	$1{\cdot}0 \times 10^{-6}$	for silica to hard glass
WQ.34	$2{\cdot}1 \times 10^{-6}$	
H.428	$3{\cdot}2 \times 10^{-6}$	
G.S.1	$5{\cdot}2 \times 10^{-6}$	for hard to soft glass
G.S.2	$5{\cdot}8 \times 10^{-6}$	
G.S.3	$6{\cdot}6 \times 10^{-6}$	
G.S.4	$7{\cdot}2 \times 10^{-6}$	
G.S.5	$7{\cdot}8 \times 10^{-6}$	
G.S.6	$8{\cdot}4 \times 10^{-6}$	

The G.S.1 can be joined to a hard borosilicate glass using as intermediates a Kovar-sealing glass, a sandwich glass such as *Dial 43*, and a tungsten-sealing glass.

The Ease of Working Different Types of Glass

The working of fused silica is described in Chapter 10. *Pyrex*, *Phoenix* and *Firmasil* require either a gas–oxygen flame or a gas–air–oxygen flame. All the operations we describe for *Pyrex* can also be carried out with *Phoenix*. These two glasses are very easy to work, and very suitable for research work. They join together well. *Monax* can be worked without oxygen, and is a most valuable glass for

laboratories, such as those in schools, where oxygen is not available. The methods we describe for *Pyrex* can be used for *Monax*. When a hand torch is used for working *Monax*, it should give a flame with a definite inner blue cone, and it may be necessary to work the *Monax* with the hand torch in small portions at a time, especially if the air pressure is low and the tubing is large. Finished articles in *Monax*, even when badly made, do not usually crack after a flame annealing. Soda glass is very easy to work in a gas–air flame, but skill is necessary to make apparatus which does not crack on cooling. Complex apparatus of soda glass cannot be made satisfactorily with a hand torch by many of the methods we describe for *Pyrex*; the bench torch methods are far better for soda glass, but they are far more difficult. We do not recommend soda glass for research apparatus unless this is of very simple construction.

The working characteristics of the other glasses mentioned can usually be deduced from their expansion coefficients. A glass of lower expansion than *Monax* needs an oxygenated flame, and one with higher expansion needs only a gas–air flame.

Distinguishing between Different Types of Glass

In a bunsen flame, soda glasses give a yellow flame and are softened, borosilicate glasses keep their hard edges, and a lead glass is blackened. When a joint is made between two glasses of different composition and pulled out, the softer glass will pull out further. The softer glass has the lower softening temperature.

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Chapter 3

GLASS-WORKING EQUIPMENT

The Glass-Blower's Table

THE usual practice is to have a special table for the bench blowpipe. This laboratory blowpipe table can be 2–3 feet square for most purposes; but for the construction of large apparatus a good size is 6 feet long and 3 feet wide. This size will permit the use of rollers for large tubes. The table should be placed so that long pieces of glass can extend beyond the ends. The top of the table should be of heat-resisting material. The blowpipe is placed near one edge, which should be flat. The other three edges should have a raised rim to prevent articles rolling off the table. Gas, oxygen and compressed air must be available; the latter is often obtained from bellows fixed under the table. Drawers for tools are desirable. A rack of some sort should be available in which hot glass objects can cool; a large block of wood bored with holes of different sizes to hold the ends of the objects is very useful for this purpose. A small flame should be present on the table for relighting the blowpipe if it is extinguished during work.

The table should be placed so that direct sunlight does not fall on the blowpipe flame. If this can happen, there should be a blind to cut out the sunlight. It is difficult to work glass in a flame in sunlight because the flame is then so difficult to see and the temperature of the glass cannot be judged properly.

The height of the table and the accompanying stool should be such that the glass-worker can rest his elbows on the table. A stool of adjustable height (like a music stool) is very convenient, and was advised by R. THRELFALL (1898). Both he and W. E. BARR and V. J. ANHORN (1949) advise a table of height 38 inches. A table of slightly less height is also satisfactory.

Blowpipes

The term 'blowpipe' is used for both the flame generator employed in glass-working and the iron tube used in blowing glass (p. 1). In the glass trade the term 'glass-blower' describes a person who uses glass melted in a pot, and the worker of tube or rod in a flame is a 'lamp-worker', who uses a blowlamp; but in the laboratory this usage is not

customary. There a 'glass-blower' is a person who works tubing in a blowpipe flame.

Satisfactory blowpipes or bench burners are available from laboratory furnishers. They usually have a range of jets to give different flame sizes. The changing of jets during work is avoided in a burner with a turret head. The burners normally burn coal gas with either compressed air or oxygen, or air–oxygen mixtures. Crossfire burners produce a number of small flames which heat both sides of a tube at once, as shown in FIGURE 3. They are very useful for large tubing. Special burners producing a long thin flame ('ribbon burners') can be

FIGURE 3. Two crossfire burners

used for glass tube bending; they are made giving lengths of flame of 50, 100, 200 and 300 mm. A cracking-off burner giving a thin line of intense heat is also made. All these special burners can be obtained from Stone-Chance Limited.

I. C. P. SMITH (1947a) has given an account of the construction of burners for the glass-worker. The adjustment of the flame size and temperature with a given burner is best found by experience.

Hand Torches

A hand torch is a moderately light blowpipe designed to be held in the hand and moved around the apparatus. Various hand torches are available. We find the *Flamemaster* hand torch, made by Stone-Chance Limited, to be very useful. This can be fitted with a number of different jets. One gives a wide range of flame sizes with air–coal gas mixtures. Three jets give different flame sizes of oxygen–coal gas flames. With the largest of these, 4-cm diameter *Pyrex* tubing can be worked with a single hand torch; with two hand torches as a crossfire, 6-cm bore tubing can be worked. A double-tipping device can also be put on the torch in place of the usual nozzle; this gives two small flames at an angle to each other of about 120°. This is useful in ampoule sealing. Controls are provided on the hand torch for

regulating the flow of gas and air or oxygen. The *Flamemaster* can also be clamped to the bench and used as a bench torch.

Cylinder Heads and Valves

The working of glass such as *Pyrex* and *Phoenix* requires either an oxygen–coal gas flame, or an oxygen–air–coal gas flame. The oxygen is obtained from cylinders. For glass-working it is desirable to use a cylinder head which has both a regulator for controlling the pressure of the oxygen and keeping it at a steady value, and a fine adjustment valve for regulating and turning off the gas flow. In operating a blowpipe or hand torch, the oxygen is frequently turned off at the blowpipe or hand torch. The regulator then automatically stops the flow of oxygen from the cylinder. If only a valve is used the oxygen continues to come out of the cylinder, and the pressure in the rubber tubing to the blowpipe becomes so great that the tubing is forced off the blowpipe or the cylinder head; this can be very inconvenient when one is working in an awkward position with a hand torch on a complex apparatus.

Cylinder heads are liable to be damaged when they are attached to cylinders left free to roll about on the floor, and to avoid this a cylinder stand should be used.

Bellows

Foot-operated bellows fitted with a rubber disc enclosed in a string net, to provide an air reservoir, give a steady pressure of about 20 oz/in.^{2} They are suitable for air–gas flames of moderate size. Bellows with a spring-controlled reservoir can also be used, but they do not give such a steady pressure.

Air Blowers

A compressor driven by a $\frac{1}{4}$ h.p. electric motor and giving a pressure above atmospheric of 10 lb/in.^{2} is adequate for all normal glass-working operations, including those with a small lathe (Chapter 6). A filter should be fitted on the suction side of the compressor.

A filter pump discharging into an aspirator, fitted with an outlet at the top for air and an outlet at the bottom for water, will give enough air for small-scale operations of glass-working. W. A. SHENSTONE (1889) described this device.

Glass-Working Tools

The tools necessary for most glass-working operations are simple and can best be described in relation to their uses. Probably the most useful general-purpose tool is an iron or steel spike about $1\frac{1}{2}$ inches

long set in a wooden handle (FIGURE 4). This spike can be used for manipulating the hot glass into the required place and in many instances can also be used in the same operations as the more specialized tools described below, although it may not be so convenient. Throughout the book this tool will be referred to as a spike.

Tools designed for various operations are marketed by a number of companies dealing in laboratory supplies. FIGURE 4 shows a selection of such tools. Triangular flaring tools are intended for flaring out the

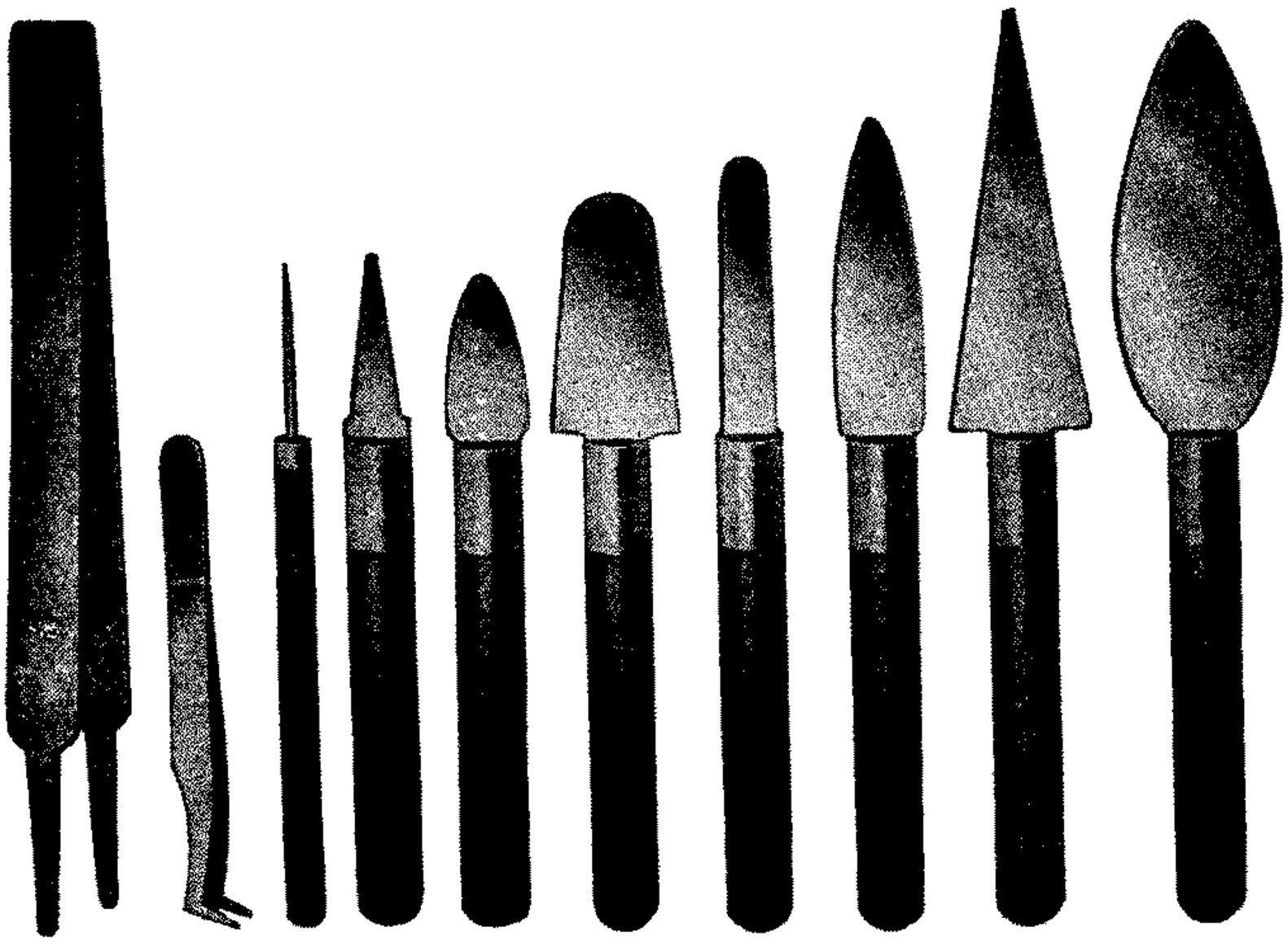


FIGURE 4. Some tools used in the working of glass

ends of tubes. They are usually made of brass sheet or of thin carbon plates, as are the other tools for shaping glass by means of pressure of the flat surface of the tool applied to the glass as it is rotated. The metal tools should be lubricated to prevent the glass sticking to the tool. Carbon tools require no such lubrication but achieve the same effect by wearing away in use. Hexagonal tapered reamers are used for working tubes to standard tapers for the purpose of making stopcocks or ground joints. These reamers may either be made of carbon or of metal—usually aluminium alloy. Similar tools with a much sharper taper and mounted on a handle may be found more convenient than the normal flaring tool for opening out the ends of tubes. We have found that the mounted needles from a set of dissecting instruments are useful in some cases with capillary tubing since they are fine enough to be inserted into the bore of the tube.

These needles have the disadvantage that, owing to their fineness, they tend to oxidize away readily and may also be melted if held in a hot flame for any appreciable length of time.

Forceps will be found useful in many circumstances. These are usually made of steel and should be designed so that there is no tendency for the soft glass to slip out easily. This will be avoided if the points are bent inwards so that they meet at an angle of about 25°. The forceps should either be long enough not to get too hot in use (about 10 inches long) or else they should be insulated in some way.

Flask clamps are made to fit a wide range of sizes of flask. These are devices for holding flasks while the necks are being worked and enable the whole flask to be rotated easily. They usually consist of a handle with three or four sprung prongs which hold the body of the flask. Some clamps are made so that they can be adjusted to fit a range of flask sizes.

Many operations require only a very modest set of tools, and too great a stress can be laid on the variety of tools required for any operation. As an example, forceps are useful for drawing off small pieces of glass or for pulling out excessively thick parts of the wall of a joint, but both these operations can be carried out by fusing a length of glass rod to the part to be drawn off and using this as a handle for the drawing operation. As in many other fields of craftsmanship, a skilful operator can work wonders with inadequate and makeshift tools, but his job would be much easier if he had all the equipment he needed, although for much of the time he may only use a very few of the tools at his disposal. There are times when the less commonly used tools will be useful, but on the other hand the glassworker should not allow himself to be deterred by the fact that he does not have a vast array of tools.

I. C. P. SMITH (1947b) has reviewed some uses of tools in lampworking.

Wax for Tools

Tools can be used without lubrication, but care is necessary in this case to prevent the tool oxidizing and the oxide entering the glass, which is then liable to crack on cooling. A lubricated tool slides more easily over the hot glass. Beeswax and paraffin wax have been used as lubricants; a thin film only is used. A better lubricant, advised by I. C. P. Smith (1947b), is paraffin wax mixed with some colloidal graphite. The colloidal graphite is not rapidly burnt off the tool, and it retains its lubricating properties up to 600°C. An over-heated tool can also be dipped into distilled water (5 oz) containing colloidal graphite (½ oz of *Aquadag*). The tool picks up a layer of graphite.

When soft asbestos is used on the bench, waxed tools are likely to pick up shreds of asbestos which can cause trouble. It is best to avoid soft asbestos in this case.

When constructing high vacuum apparatus, waxed tools should only be used when the apparatus under construction can be thoroughly cleaned afterwards. Otherwise wax may be present in the vacuum system, and will be removed by pumping only with extreme slowness.

Carbons

Carbon Plates

A few thin, rectangular plates of carbon can be very useful on the glass-working bench. The faces of these plates are of use in flattening the ends of tubing, in blowing flat-bottomed bulbs, and in shaping

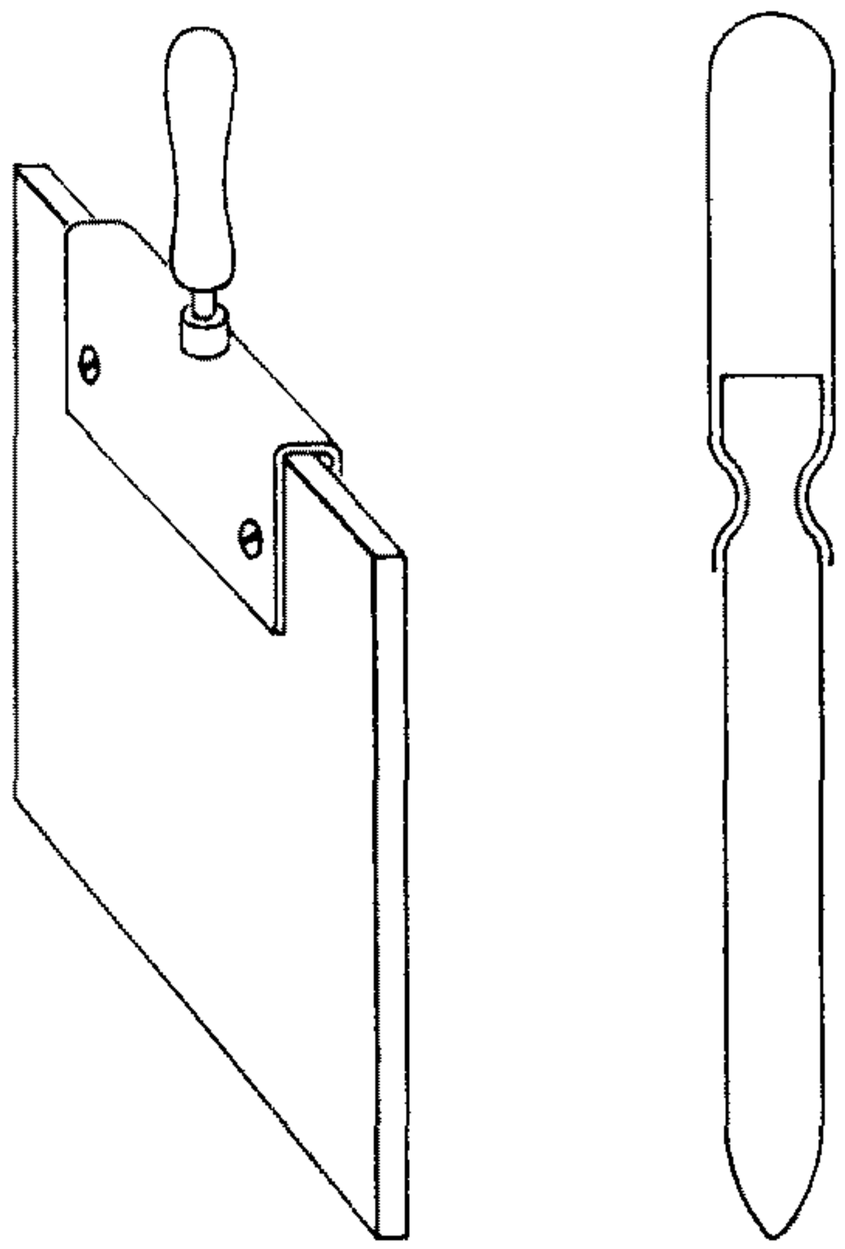


FIGURE 5. A carbon plate and a carbon rod. Both are attached to a handle

pieces of molten glass as, for example, in making the paddle for a glass stirrer. For shaping purposes it is sometimes preferable to have a handle attached (FIGURE 5). Plates ranging from $\frac{1}{4}$ to $\frac{1}{2}$ inch in thickness and of the order 3 by 6 inches are convenient sizes.

Carbon Rods

Carbon rods with one end filed to a conical shape make very good

flaring tools. It is useful to have a few of these handy, ranging from $\frac{1}{4}$ to $\frac{1}{2}$ inch diameter. Glass handles for these rods can easily be made by shrinking the end of a piece of glass tubing into a groove filed near to one end of the carbon rod (FIGURE 5). When working silica, carbon rods become a necessity as flaring tools, etc., because no metal will withstand the working temperature of this glass for long enough for much tooling to be done.

Gauges for Measuring

Some apparatus must be made to a definite size. The external diameter can be measured by an ordinary calliper gauge (FIGURE 6, *I*). The internal diameter can be measured either by the prongs at the

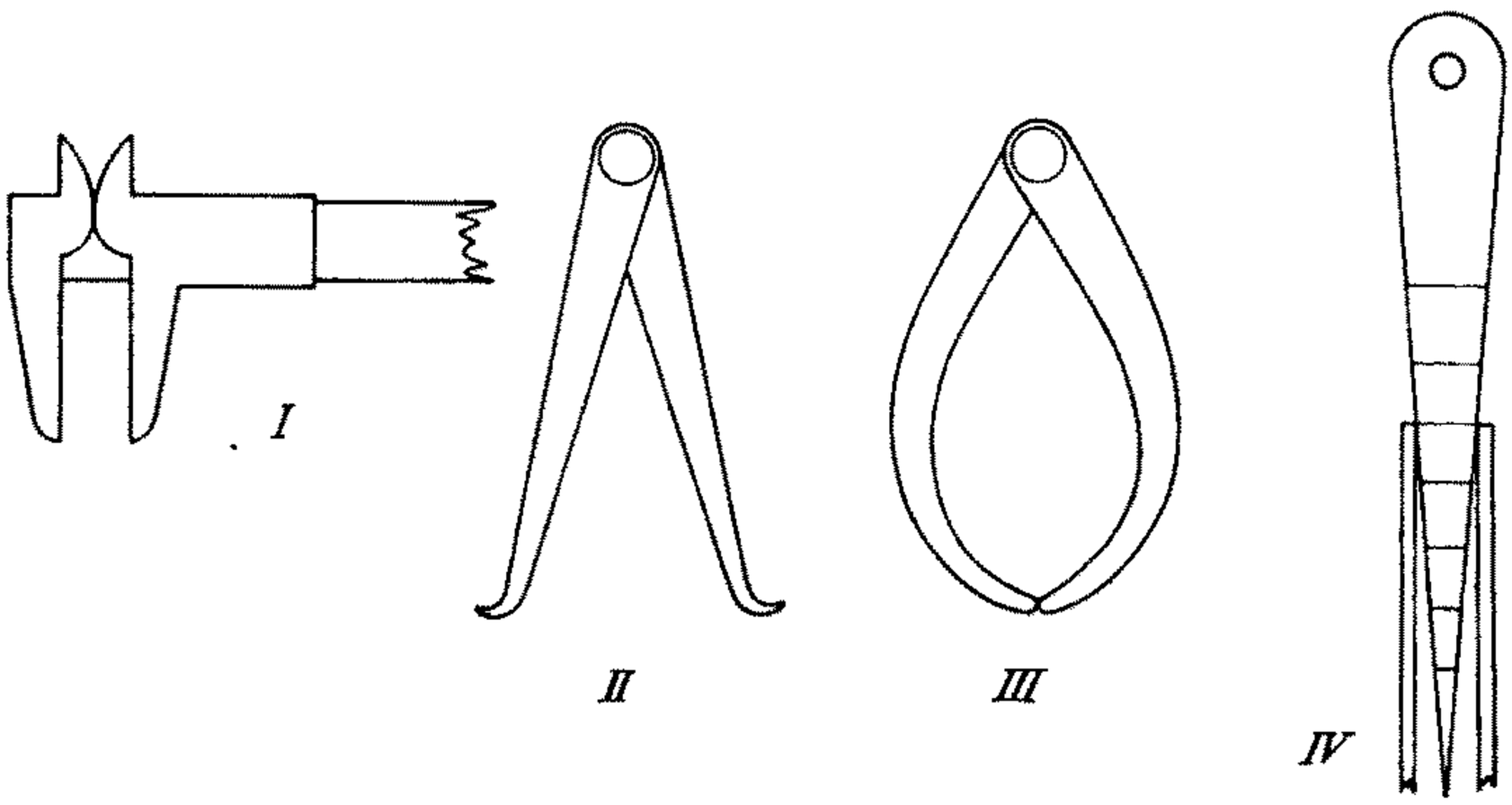


FIGURE 6. Gauges useful in measuring glass, and for working glass to fixed dimensions

back of the calliper gauge, or, for greater distances inside a tube, by an internal pair of callipers (FIGURE 6, *II*); the separation of the feet of this gauge is measured after it is withdrawn from the tube. The internal diameter at the end of a tube can be measured accurately with a cone or taper gauge (FIGURE 6, *IV*), which also reveals deviations from a circular cross-section. A pair of callipers of the type shown in FIGURE 6, *III*, is also useful in measuring the outside of a tube. The gauges in FIGURE 6, *I*, *II* and *III* can be used on hot glass; in working glass to fixed dimensions it is convenient to set a gauge at the required dimension and use it to measure the glass when it is hot.

Rubber Caps

A very convenient way of closing the ends of glass tubes for blowing, particularly with very small bore tubing into which it is difficult to fit

a cork or stopper, is to employ rubber caps. These are made in a variety of sizes to fit different diameter tubing, ranging up to about 30 mm, and when of good quality rubber can easily be slipped over the open end. They create a compression rather than a tensile strain in the glass tube and are therefore useful for closing tubing with ragged ends when it is desirable to avoid fire polishing. These caps are usually supplied made from rubber approximately 1 mm thick.

Thin Rubber Sheet

Pieces of very thin sheet rubber, of the order of 0·1 mm thick and of very elastic quality, as, for example, pieces cut from toy rubber balloons, can be very useful in closing ragged ends of large tubing. They are of particular use with tubing larger than 20 mm diameter. A small piece, of about the same diameter as the glass tube itself, can be stretched over a ragged end of the tube and will be found readily to stay in place by means of its own tension—provided excessive pressure is not built up when glass blowing. For detailed use of rubber caps and pieces of sheet rubber see Chapter 9.

Rubber Stoppers

Rubber stoppers are used extensively for making a closed system for blowing out. A complete overlapping range is a necessity to the glass-worker. The rubber should be of good elastic quality since poor quality stoppers often will not 'give' sufficiently to be squeezed easily into the ends of glass tubing. The stress on the glass is less and an air-tight seal is more easily obtained when good rubber is employed.

Two sets of stoppers are very useful, one set unbored and a second set with a single hole bored in them to carry blowing-tube connectors. A longer piece of glass tubing inserted into the hole will also form a very adequate handle when working small pieces of apparatus.

An assortment of small corks for closing small bore tubing can, in addition, be very useful. If the flame is to come near to a cork or a rubber stopper it is best to wrap either in thin asbestos paper before inserting into the tube. When pyrolysis of the cork or rubber stopper seems probable it is best to use a cork and not a rubber stopper, because the pyrolysis products from a cork can be cleaned off more easily than those from rubber. Sometimes a plug made from damp asbestos paper is adequate for closing a tube of small bore, and this plug can be heated quite strongly.

Blowing Tubes

Rubber tubing is necessary for blowing when the piece being worked cannot be brought to the mouth. A length of about 80 cm is convenient

and the size most useful is that of normal condenser tubing, or approximately 5 mm bore with 1 mm wall thickness. Lightweight rubber is advisable since weighty tubing tends to drag. It is occasionally helpful to have a finer blowing tube, and for this a length of bicycle valve tubing, about 2 mm bore, is quite satisfactory.

Mouthpieces

Mouthpieces for these blowing tubes vary with individual inclination. Some workers dispense with a mouthpiece and use the bare end of the rubber tubing. By gripping this in the teeth the flow of air can be controlled. Others desire a firmer mouth piece and use a short piece of glass tubing which must, of course, be fire polished at the end. It is perhaps also safer to strengthen the tube by thickening the end. A short piece of rubber tube, about 2 cm long, attached to the glass mouthpiece, is another modification desired by some. It combines the features of a firm mouthpiece with one where the air flow can be controlled with the teeth. A glass tube inside a rubber tube can also be used as a mouthpiece.

An assortment of glass connectors for joining blowing tubes and for connecting blowing tubes to rubber stoppers is desirable. For blocking holes in stoppers short pieces of rod or closed tubing are useful.

A holder for a blowing tube can be made out of wire. This fits round the operator's neck, and holds the mouthpiece near the mouth. It is useful when engaged in complex operations which require the use of both hands for a long time.

The Uses of Asbestos

Asbestos in one form or another finds many uses in laboratory glass-working operations. Asbestos paper or tape is very useful for blocking the ends of tubes which are so placed that they get too hot to allow the use of corks or rubber stoppers. A satisfactory blocker can be made by winding asbestos tape round a length of glass rod until it fits the tube. It may be found necessary to moisten the asbestos to make it adhere to itself. Wound round a length of tubing in a similar way, asbestos tape provides a convenient method of inserting a blowing tube into a system. Asbestos paper may be pulped with water to form a pulp which can be used for blocking irregularly shaped holes while glass-blowing is carried out on some other part of the system; but if it is used in this way care must be taken to prevent shreds of asbestos falling inside the apparatus, and one must not leave any shreds adhering to the glass when it is subsequently worked. When it is necessary to work glass under cramped conditions and

there is a likelihood of the flame being played on to other parts of the apparatus, it is advisable to screen these parts with sheets of asbestos board or by binding them with asbestos cord or paper. The bench is also liable to suffer from the effects of hot glass and hot tools being laid down unless it is protected by a sheet of asbestos board or similar material. Hard asbestos board is better for this purpose than the soft type as the latter has a tendency to contaminate the tools with loose fibres which may be transferred to the glass being worked.

Asbestos gloves protect the hands both from glassware which would otherwise be too hot to hold and from the effects of radiant energy. But they suffer from the disadvantage that, owing to the nature of the material, they tend to be clumsy and do not allow small objects to be picked up easily. Unless large sizes of tubing are to be worked there seems to be little advantage in the use of these gloves. But with operations with a hand torch on large *Pyrex* tubing, asbestos gloves are often essential—after a time the torch becomes too hot to hold, because it is heated by radiation and convection from the hot glass. The gauntlet type of asbestos glove is desirable for these operations.

When new asbestos paper and tape is used on apparatus, it may be desirable first to heat it in a gas–air flame to remove the binder.

Glass-Blowing Spectacles

It may be found that in operations in which it is necessary to observe the glass deforming or flowing whilst it is in the flame, vision is obscured by the sodium glare from the glass. Special glasses are made to filter off this yellow light and also much of the infra-red radiation. Spectacles or goggles made from such glasses are available commercially. The glass itself resembles the cobalt glass used in qualitative analysis flame tests, and may also contain rare earth oxides. Didymium goggles contain neodymium and praseodymium, and selectively absorb light in the sodium D region. The effect of the radiant heat from the glass is tiring to the eyes and may be reduced considerably by wearing ordinary sun glasses made with Crookes glass. These glasses reduce the light and heat intensity but do not significantly cut out the sodium glare. If ordinary sun glasses are used, care must be taken to see that the frames are not easily inflammable in view of the possibility of accidents.

Glass Knives and Diamonds

Glass knives are of two main kinds. The inexpensive ones are made of hardened carbon steel, and the more expensive ones are made of a very hard alloy. The latter kind keep their edge for a long time, but

the former soon lose their edge. Both kinds can be sharpened on a carborundum hone. For the expensive type a fine hone is desirable; for the other kind a coarse hone may be used and a scythe stone is satisfactory.

A glazier's diamond is very satisfactory for cutting sheet glass. It is not satisfactory for cutting the outside of glass tubing; the inside of glass tubing can be cut with a diamond by the device described below. A triangular file can often be used instead of a glass knife.

Tube Cutting Device

A very useful device for cutting tubing too large to break by hand is shown in FIGURE 7.* It can very easily be made in the machine shop.

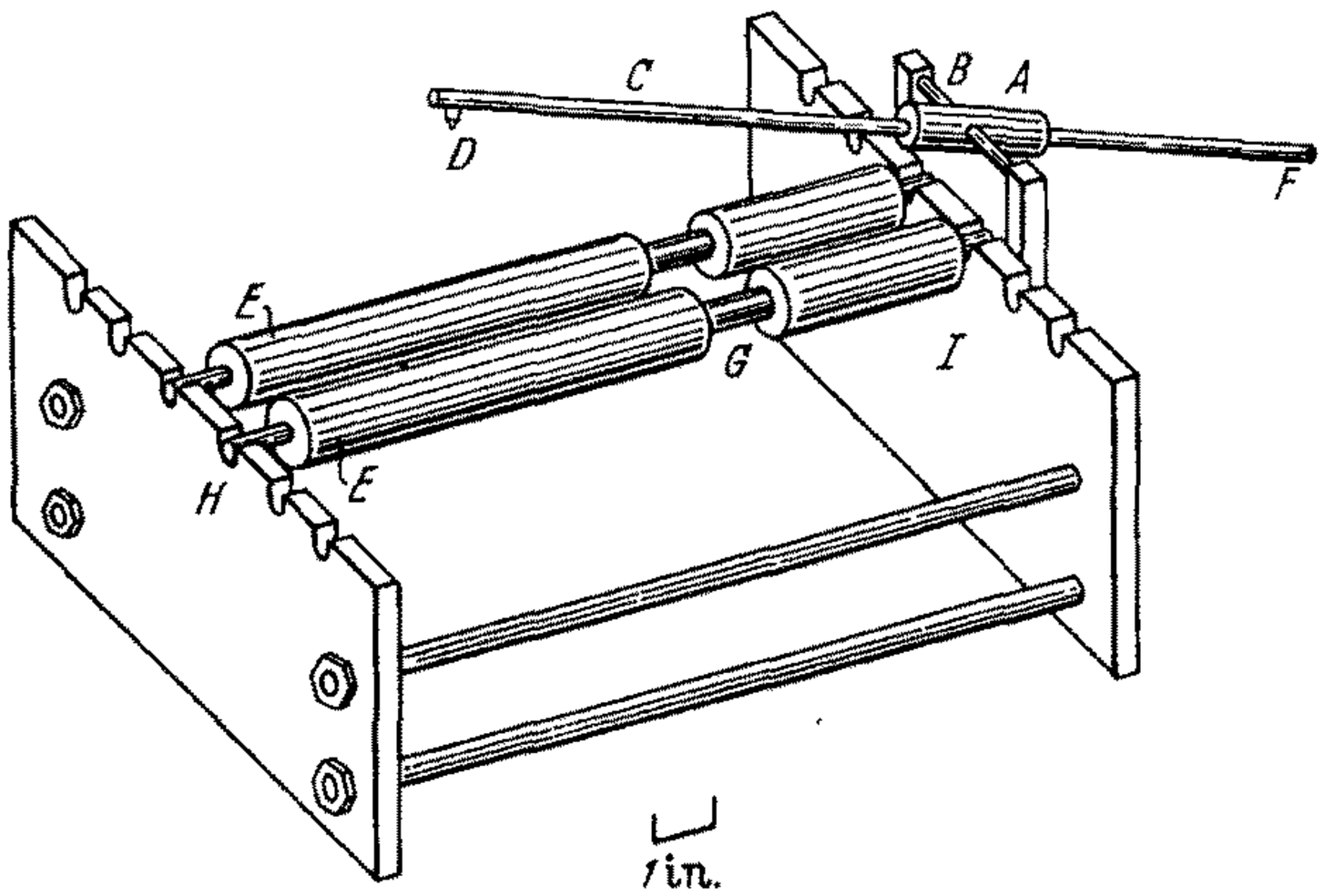


FIGURE 7. Tube cutting device

The bush *A* is hinged at *B* and carries a rod *C* whose position with respect to *B* can be adjusted. At one end of *C* is set a tiny diamond *D*. The tubing to be cut is placed over *C* so that the diamond is in contact with the inside of the glass wall and the tube is lowered on to the rollers *E*. It is then rotated through 360° while upward pressure is exerted at *F* causing the diamond to scratch the glass. A very clean break is obtained either right away, or on local heating of the glass (p. 50). The rollers are reduced in diameter over a short length at *G* to take a tube whose end is flared, as, for example, a test tube. Notches in the end-plates *H* and *I*, in which the ends of the rollers sit as shown, are cut at equal intervals on either side of the central point,

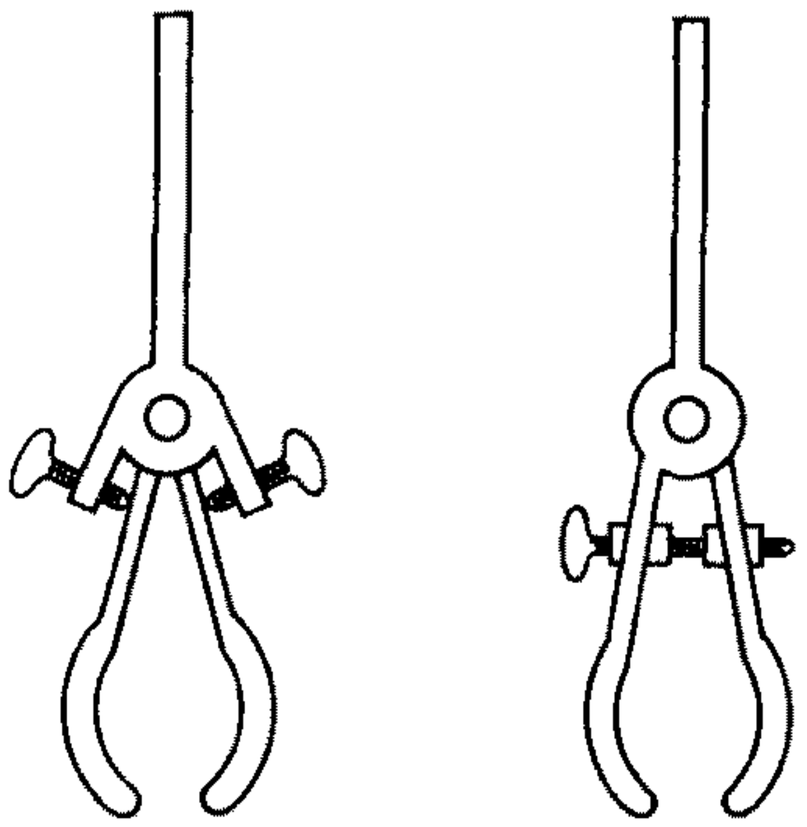
* We are grateful to Mr A. J. Hawkins, glass-blower at the University of British Columbia, whose design we have shown here.

so that the distance between the rollers can be varied according to the size of the tubing to be cut.

Clamps

Good clamps for holding glass apparatus in position are essential to the scientist. Also, in many of the operations described in this book the glass-working scientist will need to hold the glass tubes in position with clamps.

The best form of clamp has both arms of the claw independently movable with a screw to tighten up each (FIGURE 8, *I*). Many standard forms have only one tightening screw, and one movable arm is



I *II*

FIGURE 8. Two types of clamp

drawn towards the other whose position remains fixed (FIGURE 8, *II*). These are less convenient. Two prongs in each arm of the claw are desirable and lead to more stability than a two-and-one prong arrangement. A claw lining of asbestos or cork is preferable to rubber, and a machined boss makes for ease of manipulation and uniformity of pressure.

Abrasives

Glass may be ground to shape by using one of five abrasives. These are (*1*) grinding sand, (*2*) emery, (*3*) carborundum, (*4*) pure alumina or corundum, and (*5*) diamond powder. Each abrasive has its own particular property. Grinding sand is used for very rough grinding. It is mixed with water and the glass is ground to shape against a suitable surface. Grinding sand is very coarse and unsuitable for refined work, for which emery, carborundum or corundum are used. Emery is graded from coarsest to finest as 1, 0, 00, 000, 0000. The

grading is done by shaking the emery with water and leaving the suspension to stand. After a specified time the liquid suspension is removed and dried, so the longer the standing time the finer the emery. Emery can also be graded by particle size, by giving the size of a mesh which will just retain the particles. A 100-mesh sieve, for example, has 100 square holes per linear inch. (Often in a sieve only about 25 per cent of the area consists of holes; the remainder is the wire network. The wire size is equal to the aperture size when 25 per cent of the sieve area is holes.) When grinding glass with increasingly fine grades of emery it is essential to clean it so as to remove the coarser emery before using the finer. The glass is ground with emery in water either on a metal former or, when flat surfaces are required, on another flat piece of glass, with the emery between the two glass surfaces.

Carborundum is used mainly for fast work when much glass needs to be removed, and for grinding quartz. It is cheaper and harder than emery. It can be graded by particle size; the coarsest normally used is 90 mesh. The medium size is about 180 mesh, and the fine about 300 mesh, with very fine of 600 and 900 mesh; this last is usually unnecessary since better results can be obtained with jeweller's rouge. Carborundum is also often graded in F numbers: F consists of 240 mesh and finer; 2F of 280 and finer; and 3F of 320 mesh and finer. Carborundum is used in the same way as emery, and has the advantage that carborundum wheels and blocks can be obtained.

Corundum or alumina is again faster than emery, but it is much more expensive. Finally, diamond powder can be used, and is very fast but by far the most expensive. Once the glass has been ground it can be polished with jeweller's rouge or *Cerirouge* (see p. 83). With these polishes an optical finish may be obtained.

Glass Holders

One often needs to manipulate a piece of tubing so short that the normal method cannot be employed because the glass becomes too hot to hold. Various types of holder can be used. A rubber bung, covered with asbestos paper and bored with one hole containing a glass rod or tube, can be inserted into the tube as in FIGURE 9, *I*. Asbestos paper can be wrapped round a small tube until it fits into the tube to be worked (FIGURE 9, *II*). The smaller tube may now be manipulated with comfort and hence any desired operation can be performed on the larger tube. A variation of these methods is to employ a glass holder of the type shown in FIGURE 9, *III*. The holder is made by joining a small tube to a large tube; the two tubes must be coaxial. The large tubing is then cut about 2 inches from the joint.

In use, the tube to be worked is wound with asbestos paper and fitted into the large tube of the holder; a rubber bung can often be used instead of the paper.

Holders for spherical glass bulbs are mentioned on p. 32. BARR and ANHORN (1949) give many examples of the use of holders of the type of FIGURE 9, *III*; in many laboratories, however, they are rarely or never used.

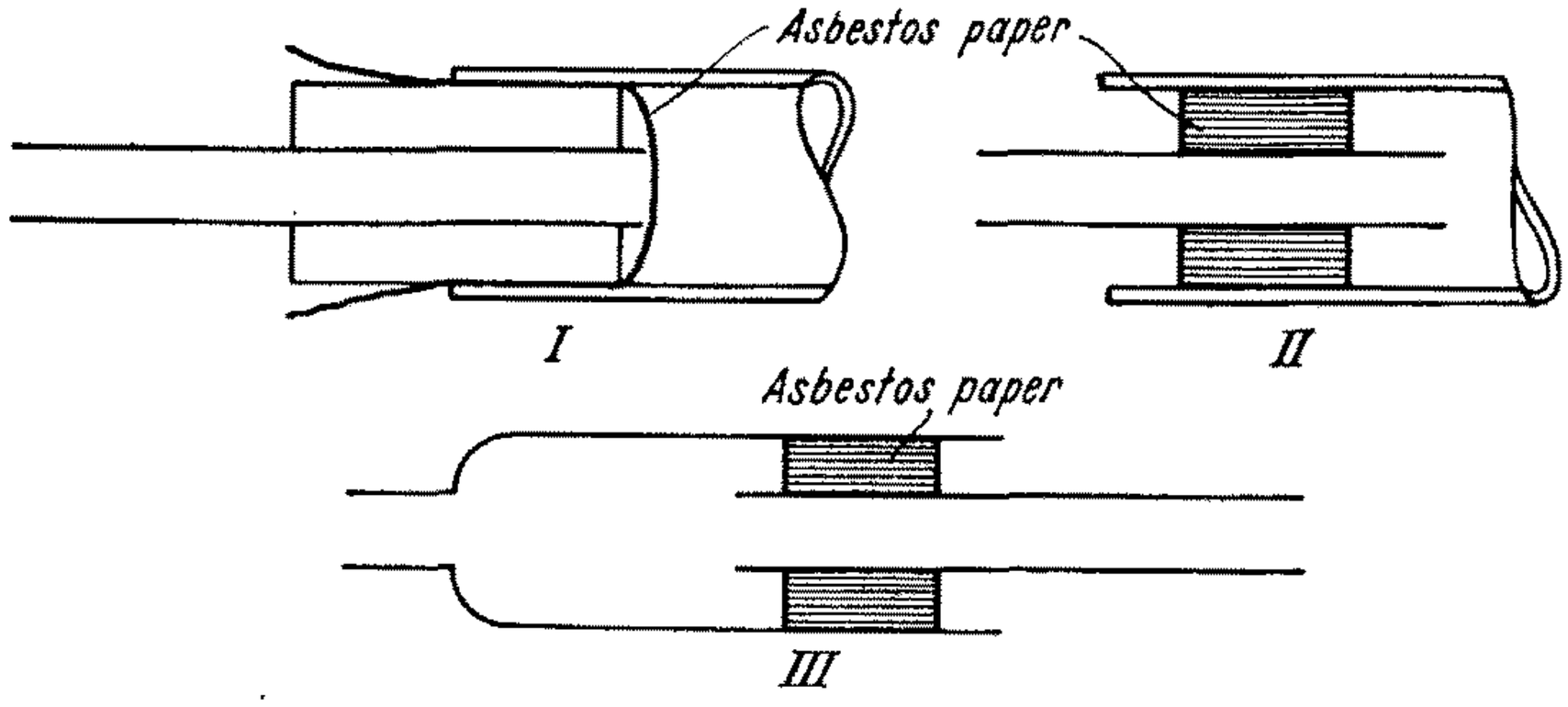


FIGURE 9. Some ways of holding glass tubes

Large Tube Supports

Large tubing is often too heavy to rotate as well as support with the left hand, so a support is used to take some of the weight. The very simple device shown in FIGURE 10 consists of a piece of wood with a 'V' cut in the top, and a slit to let a thumb-screw slide up and down.

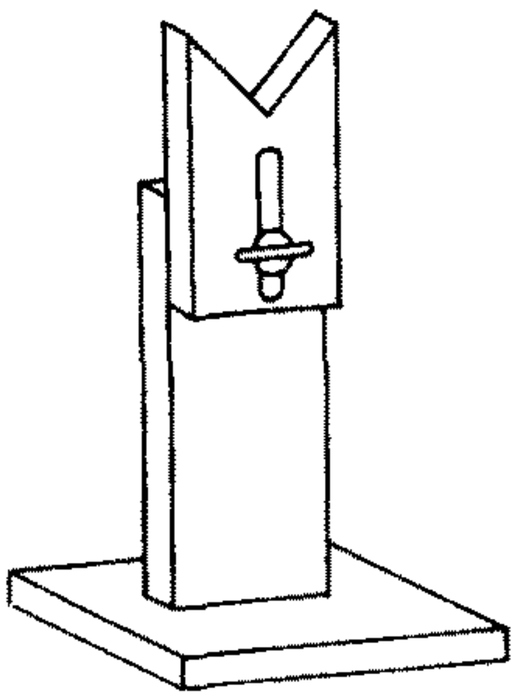


FIGURE 10. A support for large tubing

It is fixed to the stand by the thumb-screw so that the height can be varied. A few freely rotating wooden balls (e.g. small atom models) can be fixed along the 'V' to increase the ease of rotation. A clamp

and stand is often useful; the glass can be rested on the rod of the clamp, between it and the boss.

Adjustable Rollers

The effort required to work long heavy pieces of tubing, even with supports as described in the previous section, is tiring and may prevent adequate rotation of the glass in the flame. This effort may be reduced by the use of adjustable rollers (FIGURE 11), which may be in the form of two pairs of rollers on one stand or two sets of single rollers. This latter arrangement is rather more versatile, since they can be placed far apart to support long tubes, whereas the two pairs

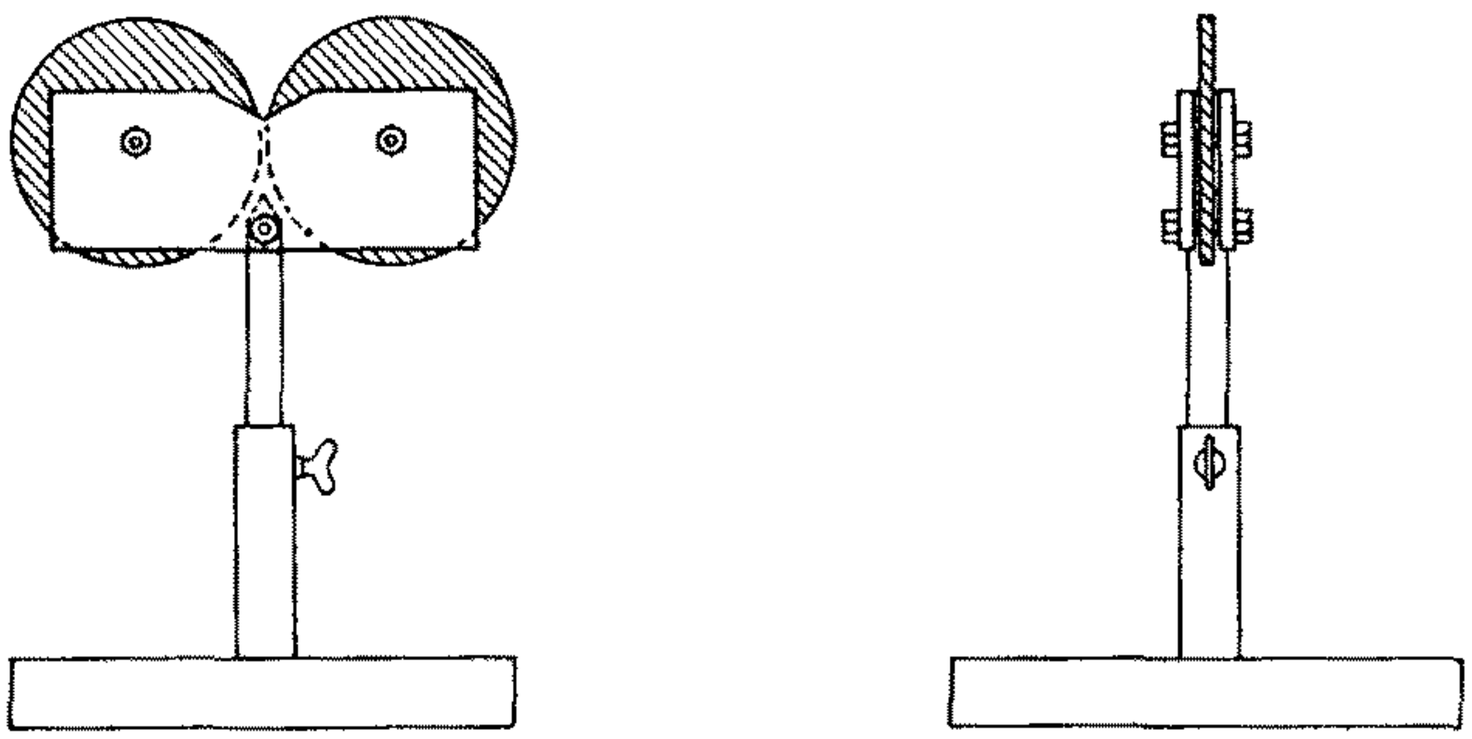


FIGURE 11. Adjustable rollers for rotating glass tubes

on one stand would have to be placed near the point of balance. When the glass is placed on the rollers it can be rotated easily with the palm of the left hand whilst the right hand is left free to work the glass at the end of the tube. If glass-blowing is to be carried out on the rollers, as distinct from flaring or reaming, the blowing tube must be connected by a swivel connection. These rollers can be driven by an electric motor, but for most purposes this is an unnecessarily elaborate device.

Glass-Blower's Swivel

For the amateur glass-worker it is very helpful if the rotating movement of a piece of work held horizontally in the flame can be continued in a horizontal line, with the work lifted slightly out of the flame for blowing, instead of having to lift one end to the mouth. The glass-blower's swivel enables him to do this.

This small device is shown in FIGURE 12, *I*. It is a small union, to which a rubber blowing tube can be connected, and inside which an

inner tube can rotate. It can be machined in metal or can be made quite satisfactorily from glass (FIGURE 12, *II*). The inner tube has holes around its perimeter directly in line with the union T-piece. If made from glass these holes can be formed by puncturing molten glass with a tungsten needle, or by drawing out tiny spindles and breaking these off. The inner tube must fit closely into the tube in which it rotates, to minimize air leakage.

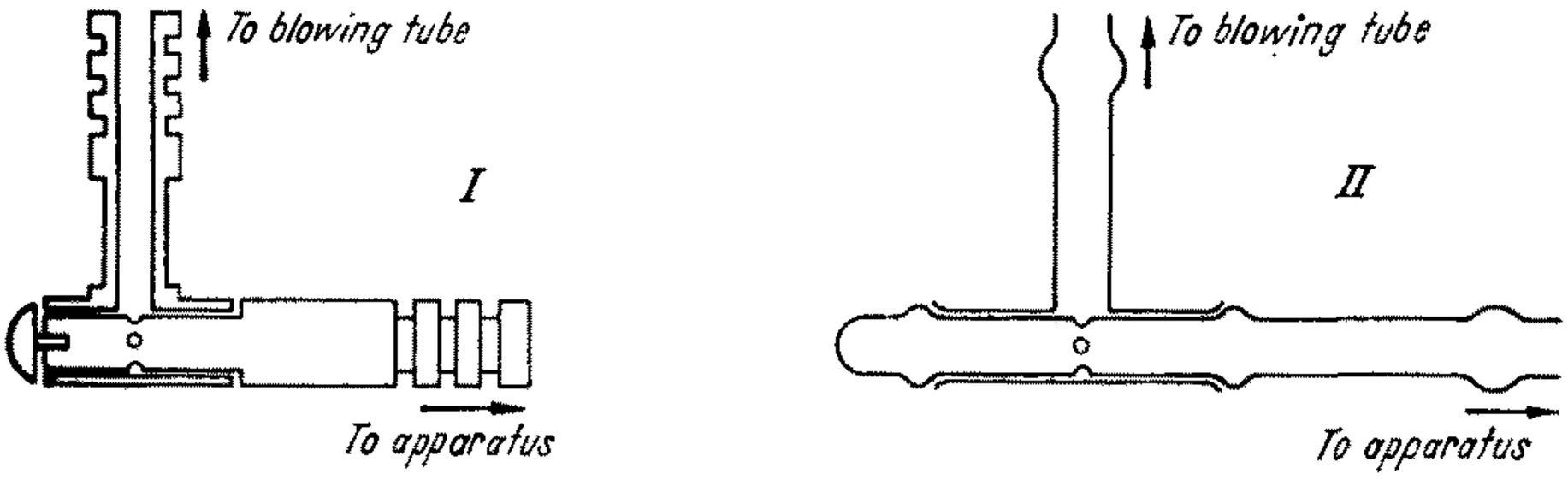


FIGURE 12. Two designs of glass-blower's swivel. *I* is of metal and *II* is of glass

Polarized Light Strain-Viewer

When intricate pieces of apparatus have been constructed it can be useful to observe the presence or absence of strain with a polarized light viewer. A. L. REIMANN (1952) gives descriptions of various systems and explains the optics of these systems.

The most sensitive form has a diffused white light shining through a polaroid screen, then a mica tint plate and a second polaroid screen crossed with respect to the first. The piece of glass apparatus under examination is held between the first polaroid screen and the tint plate. Unstrained glass appears coloured violet-red, and the presence of strain is indicated by change of colour in the strained portions through red or blue-green to yellow, depending on the nature and amount of strain. The orientation of the work must be varied to give maximum colour change, otherwise quite a large strain can be missed. This can happen because glass subject to simple axial tension or compression behaves as a uniaxial crystal with optic axis along the direction of stress. Along this direction, therefore, no double refraction occurs.

The tint plate produces a retardation of about 570 millimicrons, so that the yellow components of white light vanish. The resulting colour of violet-red (or 'sensitive violet') is changed when an extra retardation is introduced by the strained glass. The eye is very

sensitive to colour changes in this region. J. H. PARTRIDGE (1949) has given a table of colours observed in a strain-viewer without and with a tint plate.

Glass-Cutting Wheels

Large glass-working laboratories may be equipped with a glass-cutting machine. This usually consists of a high-speed, power-driven, fine abrasive wheel, or, better, a steel wheel in whose perimeter are embedded fine diamond particles. A lubricant—water, or water and cutting oil emulsion—is played on to the faces of the wheel from jets on either side. The glass to be cut is held on a movable steel table mounted on rollers.

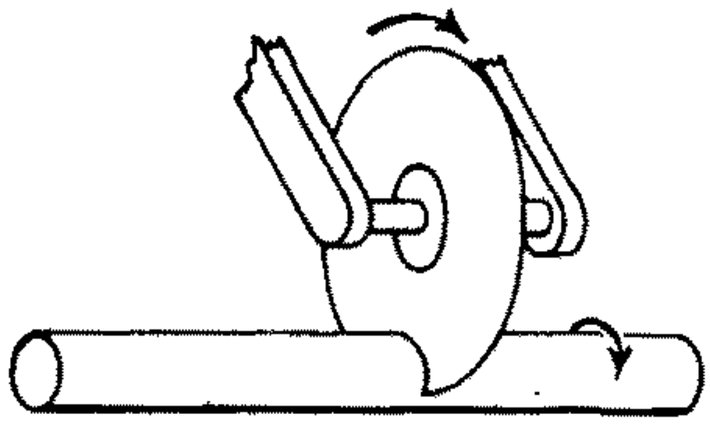


FIGURE 13. Cutting through a glass tube with a high speed wheel

For an oblique cut the glass can be clamped in position. When a straight cut across the length of the tubing is required the tube is carefully rotated during the last stages of the cut, in the direction shown by the arrow in FIGURE 13; this keeps the wall of the tubing, at the point of cutting, perpendicular to the cutting blade and prevents a portion of the wall from breaking away at the completion of the cut. By varying the manipulations the ends of tubing can be slotted and small holes or slots can be cut in the walls. A small hole, for example, is produced when the cutting operation is stopped just as the wheel breaks through the wall.

Lapping Wheel

For many grinding operations a lapping wheel can be most useful. A horizontal metal table rotating at about 30 r.p.m., sprinkled with carborundum powder and moistened with water or cutting oil–water emulsion, forms a very satisfactory grinding table. A raised rim (see FIGURE 14) prevents the grinding paste from being thrown from the circumference of the table while an inclined baffle, as shown,

made of wood with a lower strip of leather, keeps the paste from piling up on the outside.

In most cases the glass being ground can be held by hand, but for making accurately parallel faces, for instance, some form of adjustable mounting is useful. This can usually be built to suit the particular operation.

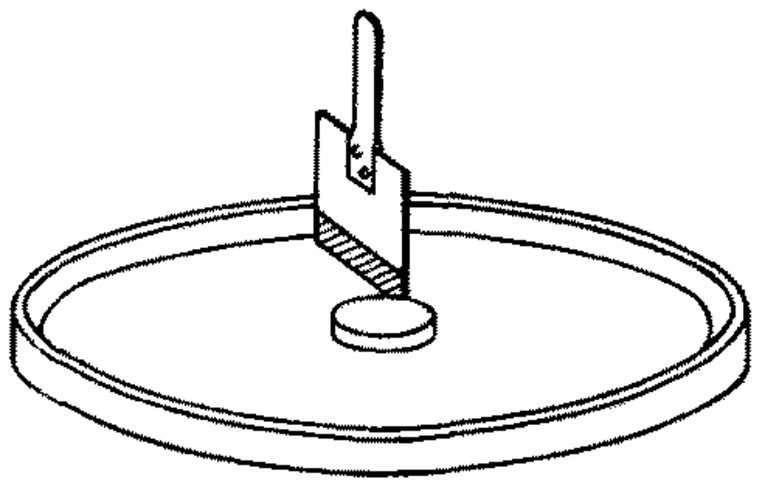


FIGURE 14. A lapping wheel

Carborundum Grinding Wheel

A very fine, high-speed, carborundum wheel—of the sort employed for grinding very hard metals—can be used for grinding glass. A delicate pressure must be used, particularly with tubing, to avoid shattering the glass. The wheel must be continuously moistened either with a jet of water or by holding a wet cloth against a portion of its face.

The method gives a very coarse finish but enables a large piece of glass to be ground down quickly. The work must be finished with carborundum powder by hand or using a lapping wheel.

Annealing Oven

Glass-working shops that are called upon to make large or intricate apparatus should be equipped with an annealing oven. With some ingenuity a home-made one can be built using *Nichrome* wire and fire-brick.

The glassware should be transferred to the oven as soon as possible and the temperature slowly raised to the annealing point of the glass used. This temperature must be held for from five to ten minutes or more and the oven then allowed to cool slowly. The maximum cooling rate depends on the glass and wall thickness, and data can usually be obtained from the manufacturer. For *Pyrex* brand glass, *Corning No. 7740*, and wall thickness not greater than 2 mm, the maximum cooling rate allowed should be 56°C/min. For a wall thickness of 5 mm the cooling rate should not exceed 9°C/min. Annealing schedules for some glasses are given on p. 20.

Sometimes it is possible to transfer the article straight into a hot oven. This depends upon the particular piece of apparatus. With a diffusion pump, for instance, where most of the joints have been allowed to cool completely before the final parts are assembled, the whole article is placed in a cool oven which must slowly be raised to the annealing temperature. On the other hand, it may be desirable to transfer a complicated part, a multiple glass–to–metal pinch seal, for example, directly from the glass-working flame to a hot oven already at, or near to, the annealing temperature. This is the case when cooling without careful annealing can set up large strains.

Treatment of Slight Burns

Trivial burns are sometimes produced by the accidental handling of hot glass. These may be smeared with vaseline or skin cream and covered with clean or preferably sterile linen. Blisters should not be broken. Tannic acid jelly is not recommended.

Amount of Equipment required for Different Purposes

The various items of equipment we have described are all useful in a laboratory where a great deal of difficult glass-working is carried out. Much difficult work can be done without a tube-cutting device, glass holders, large tube supports, adjustable rollers, a glass-blower's swivel, strain viewer, cutting wheel, lapping wheel, grinding wheel and an annealing oven. These items, however, together with a glass-working machine (Chapter 6), are desirable in a laboratory where all kinds of difficult jobs are tackled. In school laboratories only simple jobs will normally be done. The necessary equipment for such jobs consists only of a hand torch, an air blower or bellows of some kind, a glass knife, a spike, a flaring tool and forceps, and a set of rubber stoppers with a blowing tube. Stands and clamps will be available as ordinary laboratory equipment. An asbestos board is useful to protect an ordinary bench used for glass-working.

In research work a lack of the proper facilities may lead to a considerable loss of time because jobs are done far more slowly when the proper equipment is not available. For example, much time is lost when a complex apparatus cracks because an annealing oven is not available, and when large tubing is manipulated without a glass-working machine. We think that R. THRELFALL's (1898) remarks are still worthy of note. He wrote 'Though, no doubt, a great deal can be done with inferior appliances where great economy of money and none of time is an object, the writer has long felt very strongly that English physical laboratory practice has gone too far in the direction of starving the workshop, and he does not wish, even indirectly, to

give any countenance to such a mistaken policy. Physical research is too difficult in itself, and students' time is too valuable for it to be remunerative to work with insufficient appliances.'

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Chapter 4

BASIC GLASS-WORKING OPERATIONS

The Preliminary Preparation of Materials

BEFORE any glass-blowing is started it is highly desirable that the tools and all pieces of glass rod, tubing, etc., should be collected together and laid out systematically. At times this may seem unnecessary and a waste of time, but it will be found to be worth-while in the long run. When deciding what will be required, the complexity of the job in hand must be borne in mind: the more complex the job the more care required in preparing the materials and in working out mentally the way in which it is to be done. If this is not done, sooner or later it will be found that some tool is missing when it it required and that the work cools and cracks while the tool is being sought. B. D. BOLAS (1921) wrote 'A large amount of glass-blowing is spoiled through carelessness in arranging the work beforehand'.

The need for system and order in all practical work has been stressed by M. FARADAY (1842), who wrote, in giving general rules for young experimenters, 'They all relate to *method*, that great source of facility and readiness which is equally influential in the performance of the most common and the most difficult processes.' His remarks are as pertinent today as when they were made, particularly as the tradition of the handing on of practical knowledge is not as strongly maintained as it was in the latter part of the nineteenth century. (R. W. Bunsen used to teach his students glass-blowing and other practical arts and even went so far as to make the more difficult pieces of apparatus himself.)

There seems to be a general psychological tendency in doing practical work with glass (and other things) to try and take short cuts and save time by using improper and hazardous methods, and in our experience this tendency in the end leads to more waste than saving of time. One must also avoid the temptation, in any given case, to think that just for once a risk can be taken.

The Cleaning of Glass Tubes

Before any glass-working is started care should be taken to make sure that the glass is thoroughly cleaned. This cleaning is necessary for

two main reasons. One is that if inorganic material is left on the glass it will not burn off in the flame and will become embedded in the glass. This can, under some circumstances, lead to local strain in the joint and may also prevent the two pieces of glass fusing together properly, leaving a pinhole in the joint. The second reason why thorough cleaning is important is that it is essential, if the apparatus is to be used for high vacuum work, for the inner surfaces to be grease-free. It is much easier to do this cleaning before a complex piece of apparatus is assembled.

Normal chemical cleaning procedures are satisfactory for cleaning glass tubing for glass-blowing. Much of the dirt can be removed by the use of water, a detergent and a long brush such as a burette brush. It is desirable not to scratch the inside of the tube with the wire of the brush. The use of a brush can be avoided by pushing or pulling a piece of soft cloth through the tube. Chromic acid is satisfactory for cleaning glass, after removal of the gross dirt, provided care is taken to see that the glass is free from mercury. The usual laboratory cleaning solution, known colloquially as 'chromic acid' contains about 50 ml of saturated aqueous sodium dichromate in a litre of concentrated sulphuric acid. If mercury is present a residue is precipitated which is difficult to remove. Thorough washing with water is necessary after cleaning in chromic acid. Organic solvents are adequate for removing grease from the inner surfaces of tubes where no other dirt is present; while acetone or alcohol may be useful for drying glassware, care should be taken that liquid or vapour from these solvents does not remain inside the glass when glass-blowing is commenced, because of the risk of explosion. When glass which has been in contact with mercury is worked, great care should be taken to clean the mercury from all parts of the system likely to become hot, since mercury vapour is toxic.

It is very difficult to remove the last traces of chromium from glass cleaned with the usual chromic acid mixture. The remaining traces may cause error in oxidation studies. R. A. WILKINSON (1955) found serious errors from this cause in fat oxidation studies, and he cleaned greasy pipettes in a boiling, ½ per cent aqueous solution of "*Teepol*" (sodium secondary alkyl sulphate). Glass can also be cleaned in boiling aqua regia.

Breaking Glass Tubes

Tubing up to about 15 mm diameter can be broken by scratching the surface in one place with a file or glass knife, making the scratch at right angles to the axis of the tube and then placing the thumbs on the side of the tubing opposite to the scratch; the thumbs are pressed

forward and the rest of the hands are slightly pulled apart (FIGURE 15). This is a gentle process and not a test of strength. The scratch on the tubing can, with practice, be made with one stroke of the glass knife; it is not necessary to try and cut through the wall of the tubing, and the knife is soon blunted when it is used like a saw. Most glass-workers have the impression that wetting the initial cut makes the glass break more easily. Soda glass is easier to scratch with the knife than the borosilicate glasses.

We find that tubing in the range of 15–30 mm diameter (approximately) can be broken by a variation of the above method: The tubing is scratched fairly strongly, and placed, with the scratch uppermost, so that it rests on a copper wire, about 3 mm diameter, which is opposite to the scratch and parallel to it. The scratch is moistened,

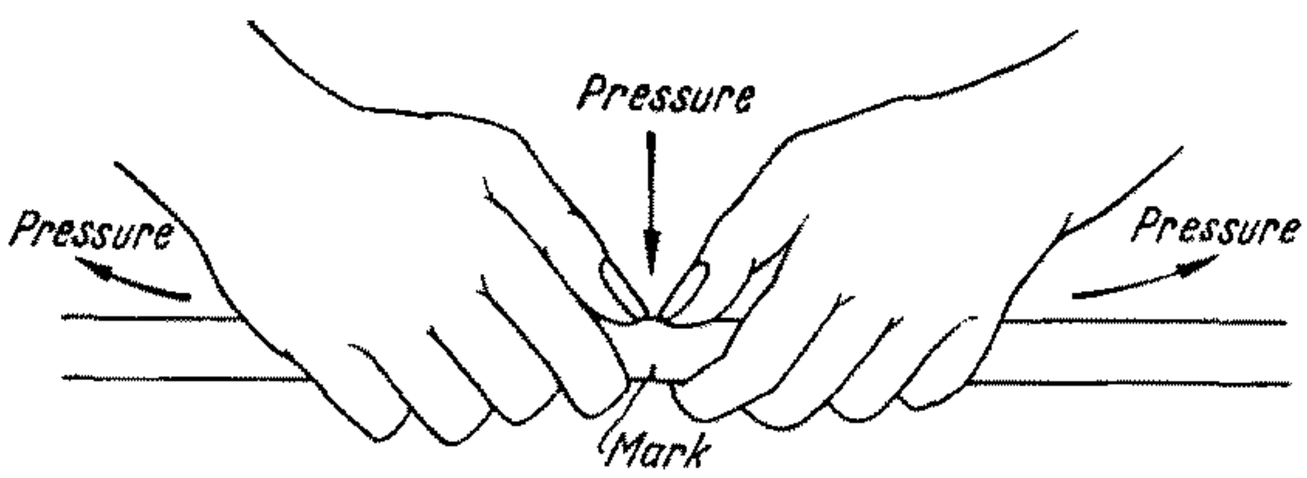

FIGURE 15. Breaking a small glass tube

and the ends of the glass tube are forced down strongly while the tube is balanced on the copper wire: a square break is then usually obtained. This method works best with moderately thin-walled tubing.

Tubes exceeding 15 mm diameter (and smaller) can be broken by the 'hot-spot' technique: A fairly deep scratch is made in the glass in one place, and wetted. The point of a piece of glass rod is then heated white hot, to give a hot spot a few mm in diameter, and this is placed on the end of the scratch. The glass may crack right away, or it may crack when the hot spot is removed and damp cotton wool is applied to the hot glass. Usually, with borosilicate glass, the crack goes only part of the way round the tube; a soda tube often cracks completely. The crack can be continued by placing another hot-spot against its end. This is continued until, with luck, the ends of the crack meet With borosilicate glass the crack usually wanders, and a square end is not obtained. A cracking-off burner (p. 29) can be used in place of a hot spot.

A square end is more easily obtained by the hot wire method. The tubing is first scratched all the way round. This may be done by

hand, a line being first drawn round the glass with a wax pencil—the line can be checked to see that it is perpendicular to the tube axis—and the scratch made along the pencil mark. Without the pencil mark, the scratch may deviate from the perpendicular. A peripheral outside scratch may also be made by rotating the tube against a rotating steel wheel. An internal scratch may be made with a diamond by the method described on p. 38.

After scratching, 26 s.w.g. (or thicker) *Nichrome* wire is wrapped once round the scratch, which is wetted. The wire is heated to redness electrically, and kept taut round the glass—for this purpose it is convenient to have insulated handles on the wire. The tubing may then crack smoothly; if not, it may be induced to crack by touching the scratch with wetted asbestos when the wire has been hot for about half a minute. An internally scratched *Pyrex* tube may not crack until it is put in a flame.

Sometimes a large bottle is cut to make a heavy beaker or a bell jar. Big bottles are generally of soda glass, and can be cracked with a flame. The bottle is first fairly heavily scratched all the way round, and then a very small flame is directed on to the scratch. A crack should start, and it can be led round by the flame. A cracking-off burner (p. 29) is specially useful here. This operation is quite difficult for beginners, because the glass often just shatters; but with practice it can be mastered. Increased safety is obtained by wrapping strips of wet blotting paper or asbestos paper round both sides of the crack, leaving a gap, about $\frac{1}{8}$–$\frac{1}{2}$ inch wide, containing the crack. An alternative method for large bottles, etc., is to spin them on a turn-table and to have a fine pin-point flame playing at the level of a small initial scratch; after a few minutes the flame is removed and a piece of wet cotton wool pressed on the scratch.

I. C. P. SMITH (1947) has described a simple method of grinding through glass tubing with a hacksaw: the blade is replaced by soft wire (such as iron, copper, or *Nichrome*) of 24 to 18 gauge, and this is ground through the glass, using 180-mesh carborundum powder and water and an ordinary sawing action. A glass-cutting wheel is described on p. 44.

Knocking off Small Bits of Tubing

Sometimes there is not enough tubing left beyond the scratch to hold in the hand, so that the normal method for small tubing (p. 49) cannot be used. The easiest method to use in this case is to scratch the tube and then place it on the top of a triangular file, with the scratch opposite to and above the edge of the file (FIGURE 16). The end of the tube is then knocked off by giving it a sharp blow with a heavy object.

Often the end of a broken tube is not quite square. This can be somewhat corrected, especially in larger tubes, by using a piece of wire gauze: The gauze is stroked against the end of the tubing, when small pieces of glass are broken off the protruding edges until the end assumes a roughly square shape. Another method is to heat the end of the tube strongly and cut off the protruding parts with scissors.

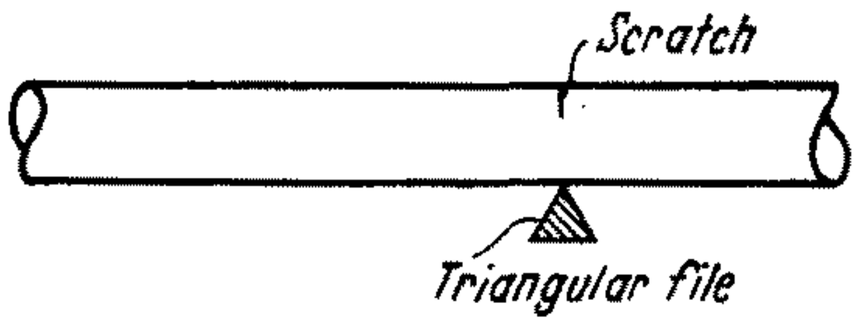

FIGURE 16. Knocking the end off a small piece of tubing

Holding and Rotating Tubes in the Bench Flame

Holding and rotating tubing in the flame is a most important part of glass-blowing with small tubing. Usually the weight of the glass is supported by the left hand, which is held over the tubing, and the right hand, under the tubing, maintains only a light grip. Although this may feel odd to the beginner, it will be found satisfactory because the right hand is free to do the manipulations and to bring the tube to the mouth for blowing. However, the right hand may also hold the weight with an overhand grip while the left hand has the underhand grip. In research work it is convenient to be able to use both methods.

The more usual disposition of the hands is shown in FIGURE 17.

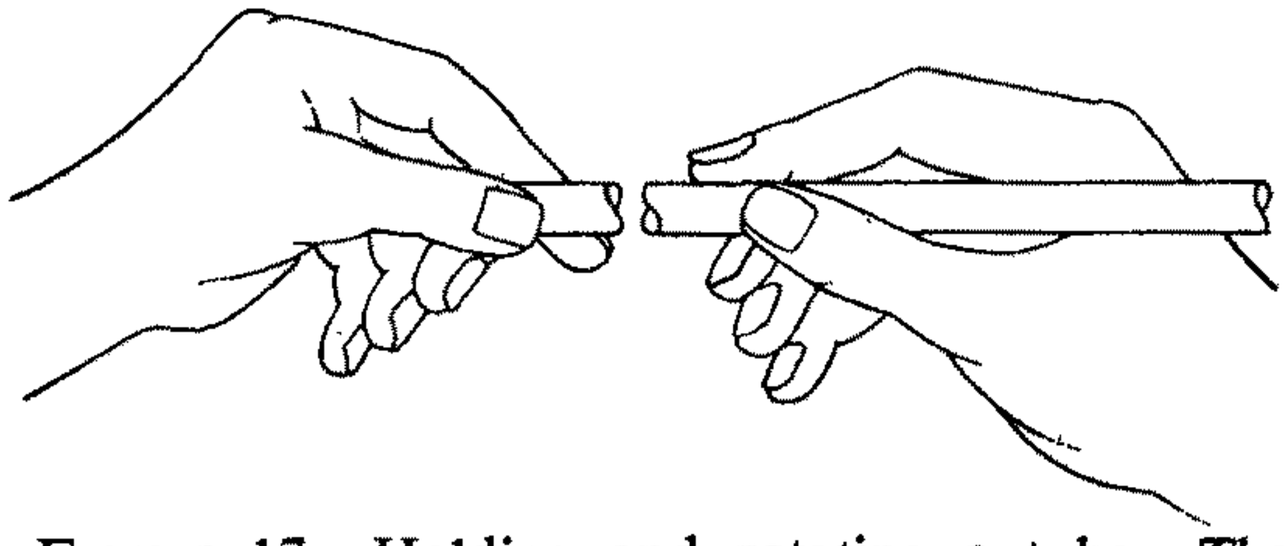

FIGURE 17. Holding and rotating a tube. The disposition of the hands is as seen by the operator

The tubing is rotated by the forefinger and thumb of the left hand, and rotates in the groove between the thumb and forefinger of the right hand, and can rest lightly on the middle finger of the right hand. It is easy enough to get a smooth rotation of the glass when it is rigid, but when it is soft both hands must rotate the glass without distorting

it, and this is difficult (p. 2). One must get the 'feel' of the glass. Its softening is a gradual process, and its 'feel' during working is determined by viscosity, surface tension, thickness etc. Practice is necessary here.

The glass should be rotated horizontally, unless it is desirable for it to flow towards one end. Usually it is rotated with the upper side moving away from the operator, but this seems a personal preference. When the glass becomes very hot and plastic, it is essential for the right hand to keep the tubing rotating at the same speed as the left-hand piece, or streaks and ridges will appear. This operation is found difficult by some people, but others master it with practice.

With soda glass the rotation of the article in the flame is of prime importance, since if part of the work is allowed to cool, re-heating may cause cracking and the destruction of the article; but with *Pyrex* and similar glass it is often possible to heat only a portion of the article at a time.

When the tubing is too heavy to hold and rotate, one end can be rested on a support (p. 41), or rollers can be used (p. 42).

The beginner should not be disappointed if at first these operations produce a hopeless mess; with practice the facility is gained.

Bends and Spirals

To bend a piece of tubing, the left-hand end is first closed. The tube is rotated in a fairly big flame with not very much air (for soda) or oxygen (for *Pyrex*) in it. A special flame from a ribbon burner (p. 29) is best, but not essential; an ordinary bench blowpipe gives an adequate flame. The tube is held as in FIGURE 17. When soft over the desired length it is brought, held horizontally, up to the chest. The right hand then brings its end vertical, and gentle blowing into this end brings the tube into shape with no kinks or flat portions. If a bad bend is made it can be corrected by heating and blowing. Many other methods of making a bend have been described.

Spirals

To make spirals of uniform size throughout is difficult; but useful spirals can be made fairly easily by a beginner even though the product may not look too elegant: Longer sections than usual must be heated, and care must be taken not to flatten the tubing. It is easier to make spirals with smaller tubing because the ratio of wall thickness to diameter is highest.

Spirals can be made either free-hand or with a mandrel or former. For the free-hand method a right-angle bend is made (FIGURE 18, *I*), with a length of tube (between *A* and *B*) of about 6 inches left to act

as a handle. The tube is next bent into a right angle at *C* (FIGURE 18, *II*) so that the length *BC* is equal to the desired radius of the spiral. It is then heated near *C*, both by rotation and lengthways, over a fairly large area—the major part of the heating being on the outside. When the glass is soft enough, the length *AB* is held horizontally in the left hand and the length *CD* is slowly bent to form the first arc of the spiral (FIGURE 18, *III*). The tube is next heated adjacent to the first arc, and, when sufficiently soft, is bent to continue the arc previously made. The bending is continued until a complete turn is made. The next step is to ensure that the tubing *AB* is axial. This is

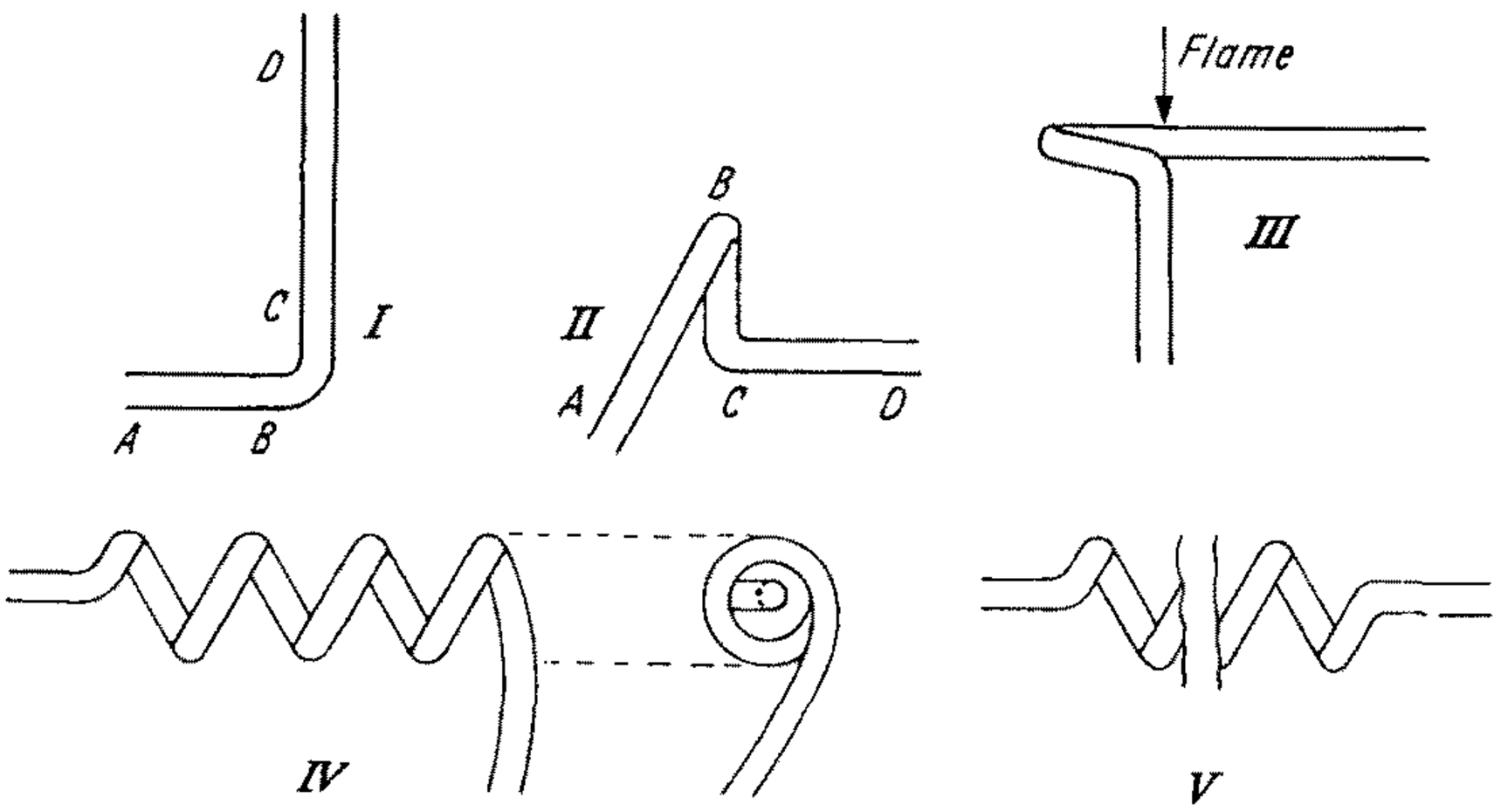

FIGURE 18. The production of a spiral

done by heating at *B* and bending *AB* into the correct position. All the bending should be done outside the flame and it is advantageous to look at the spiral from various angles to check the uniformity of the coils. The spiral is completed by continuing as before.

To make an elegant-looking product it is necessary to pay attention to the uniform diameter of the coils and to keep the spacing between them constant. The final product (FIGURE 18, *IV*) usually has to have the two ends aligned. To do this, the last turn is heated at a distance from the end of the last arc approximately equal to the spiral radius, and a right-angle bend is made. The end of the last arc is then heated, and this right angle is pushed inwards. Final adjustments can be made after rotating the spiral to check alignment. The spiral should appear as in FIGURE 18, *V*.

It is often easier to wind a spiral on a mandrel or former of carbon, metal tubing or glass tubing wrapped with asbestos paper. A right-angle bend is made in the tube to give a short portion at the end which

is strapped to the mandrel. The tubing is heated in sections of about 1 inch and wrapped round the mandrel. The asbestos can be used as a guide to ensure that even turns are made—one way is to wrap

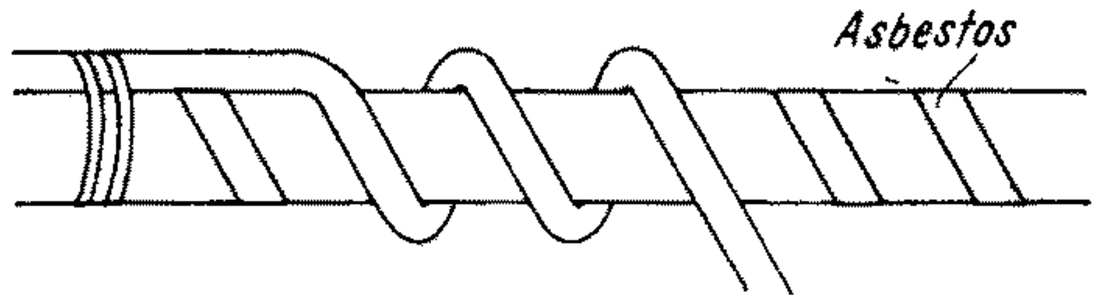

FIGURE 19. Production of a spiral with a mandrel

asbestos tape round the mandrel in a spiral path (FIGURE 19). Finally the asbestos is softened in water and the spiral is removed from the former.

A flat circular spiral can be made by the free-hand method: a right-angle bend is made, and the glass near the bend is bent round the short section used as a handle. By repeated heating and bending a flat coil is produced.

Pntting a Handle on the End of a Tnbe

When it is necessary to work comparatively near to the end of a piece of tubing it usually becomes too hot to hold. It may be inconvenient to support the whole weight of the glass in one hand, and the unsupported end will almost certainly drop under its weight when the tube is hot. A handle on the end of the tube is therefore useful.

First let us consider the case of a tube which is to be drawn off near the end. In this case the end need not be airtight. The tube to have the handle attached is held in one hand and the piece of rod or tubing to be used as a handle in the other. The end of the tube is rotated in the flame until the glass is soft for a length about equal to its radius. The handle should be heated until its end is molten and then attached to a selected point on the end of the tube. The tube should be taken out of the flame and the handle pulled across until the point at which it is attached to the tube comes into contact with the far side of the tube. This should be heated so that the two sides of the tube and the handle are thoroughly fused together and the handle should then be pulled approximately central on to the axis of the tube. The whole mass of glass should be kept heated for a short while by rotating in the flame to allow the strain in the glass to be reduced. Such a handle should not crack off when the glass cools, and is very useful when the tube has to be drawn off or bent near to the end. It does, however, leave the end of the tube in such a state that it has to be either drawn off or cut off and no blowing can be carried out as it is not air-tight.

A handle can also be fused on to the closed end of a tube to support it while glass-blowing operations are carried out: This is done by heating the end of a piece of rod for the handle, heating the centre of the closed end of the tube, and fusing them together. When the handle is to be removed after the operations are finished the point where it is fused on can be heated, the handle drawn off, and the end of the tube blown out again to its original shape.

With big tube it may be convenient to prepare a handle from rod shaped at its end like a Y, so that it sticks to two points along a diameter at the end of the tube. It is then not necessary to push in the ends of the glass tube. The Y-shaped handle is specially useful for very big tubes.

Drawing Tubes off to Spindles

Drawing a tube off to a spindle is the simplest of the basic glass-working operations. It is a convenient alternative to cutting glass tubing when short lengths are required and it is the preliminary stage to many other operations.

To draw off a tube using the flame of a bench torch, one places it in the flame and rotates it, so that heating is uniform, until the tube starts to collapse under the surface tension of the molten glass. The glass should then be removed from the flame and the two parts drawn steadily apart at a rate which gives a strong spindle as the glass cools. The spindle can then be cut by melting it in the flame and pulling the two bits apart. A more axial spindle is obtained when rotation is continued as the glass parts are drawn apart.

It may be convenient (specially for big tubes) to shape the glass somewhat in the flame before drawing the spindle. When the glass starts to soften, the two parts of the tube can be drawn apart slightly to maintain an approximately uniform thickness of glass as the tube is drawn down. In this way a tube may be tapered or constricted. If the two parts of the tube are drawn apart too rapidly, before the glass has collapsed sufficiently, thin walls are obtained, and, if they are not drawn apart enough, thick walls result. The rotation of the two parts of the tube must be the same and the tube must be kept approximately straight during this process if a symmetrical taper or constriction is to be obtained. Continuation of the process greatly reduces the size of the tube at the point of heating.

The spindle makes a very useful handle for holding the tube while other glass-blowing is carried out, and owing to its much reduced diameter it does not become hot when the tubing is worked near to the end. The tip of the spindle can be cut off to attach a rubber tube for blowing. If for any reason an accurately central spindle is

required it may be found convenient to put in a second spindle using the first one as a handle, since short lengths of tubing are normally more easily worked than long pieces which are unevenly balanced about the point of working. The operations involved in putting a central spindle near the end of a piece of tubing are shown in FIGURE 20. In many glass-blowing operations, a long spindle is extremely useful. Spindles can be closed very quickly in a flame, or opened again with a glass knife; we find it preferable to use them, when possible, instead of rubber stoppers.

Experience of the feel of the molten glass as the tube is rotated and

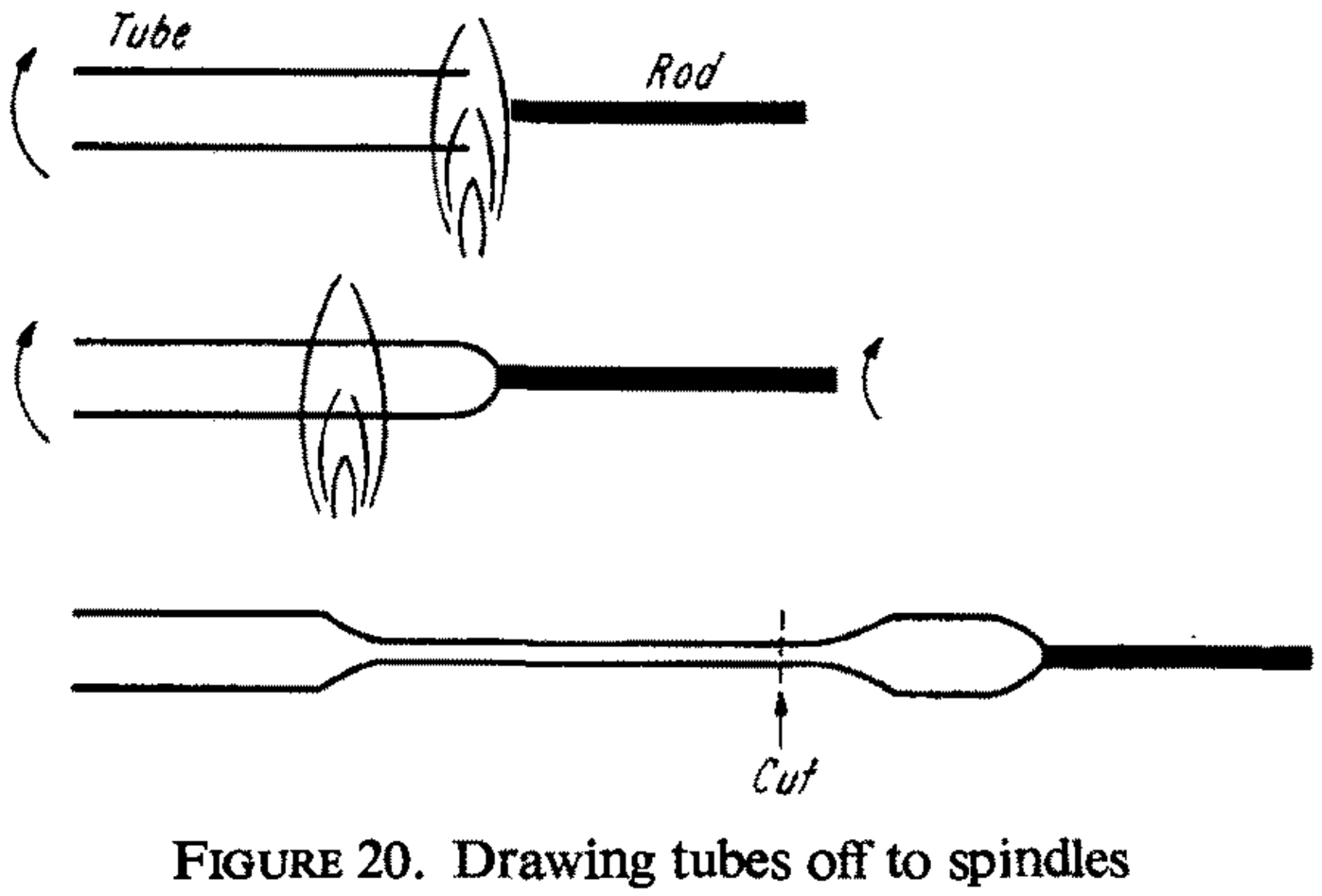

FIGURE 20. Drawing tubes off to spindles

drawn down will enable a happy medium, between the extremes of thin-walled spindles and spindles of glass rod, to be obtained.

The operation of putting a spindle in a tube is often described as 'pulling a point' or 'pulling a spear'. This is a most important operation in bench flame work. The spindle must be central, otherwise the rest of the work will not be central.

Round-Bottomed Closures of Tubes

When a good spindle is obtained and a rounded end is required the glass should be heated at the end of the taper (as shown in FIGURE 21): the glass here should be collapsed down in a small flame and pulled out comparatively slowly so as not to leave a blob of glass on the end of the tube. The whole end of the tube should then be heated by rotating it in a fairly large flame until the glass at the end of the tube has started to collapse a little. The tube should be removed from the flame at this stage and should be blown into gently. The glass should be heated relatively strongly—most beginners tend to try to

work the glass before it is hot enough which leads to an uneven finish. It is advisable in the first place to blow gently so that the end of the tube is stretched slowly. In this way the blowing can be stopped at any required stage, and this is desirable since if any part of the glass is thinner than the rest this part will start to blow out first because of its lower viscous drag but will cool faster than the thicker parts which will continue to blow out after the thinner glass has become too cool to extend much more. In this way a more even rounded end will be produced than if the glass had been blown rapidly and then allowed to cool.

The most usual fault in making a rounded end is to leave a lens of thicker glass at the point where the spindle was drawn off. This may be because either insufficient glass was drawn away with the spindle—if this is the case more can be pulled off with a piece of rod—or else

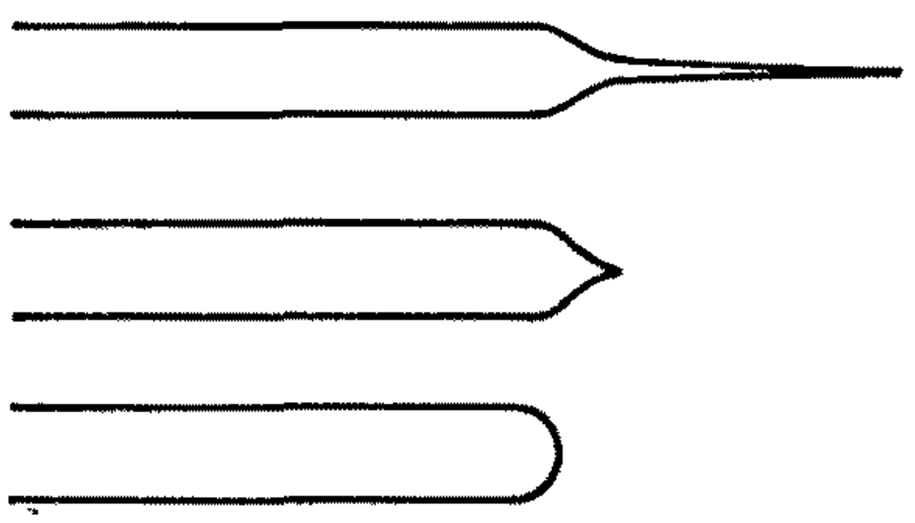

FIGURE 21. Closing a tube with a round end

the glass was not heated enough to allow the unevenness to even itself out by flowing under surface tension. A lens of thicker glass left in a round-bottomed closure can be removed by heating it with a small flame and blowing it into a small bulb. The whole end of the tube should be re-heated by rotation in a medium-sized flame until the glass starts to flow. The end is then blown out to a hemispherical shape and annealed.

Fabrication of Thin Glass Rod

In some of the following sections the use of thin 'spills' of glass rod (welding rod) is described. They are most easily made from glass rod by heating a portion and pushing the ends together until a reasonable amount of molten glass is accumulated. This is drawn out at a rate which gives a rod of 1–2 mm diameter.

If no rod is available the thin glass rod can be made from tubing: thick-walled capillary tubing is the most convenient but normal 8 mm tubing will do quite well. A length of tubing, about 10–15 cm for

thick-walled capillary or 15–20 cm for 8 mm tubing, is rotated in the flame until a part collapses down and fuses to a solid mass. This process is continued, pushing the ends inwards slightly to build up a mass of fused glass with no fine capillary through the centre. Such a capillary is easily seen while the glass is hot. The molten glass should be drawn down in the same way as for rod, taking care that the tube next to the fused mass does not draw down to form a length of fine capillary. This will be prevented if the initial stage of the drawing down is carried out very slowly, Straight rod is obtained by rotating during the drawing-out.

Blowing Holes in Glass Tubes

There are basically two sites for blowing holes in glass tubing, the first where the hole is to be made in the end of a tube which has been drawn off, and the second where the hole is required in the side of a tube. The principle is the same in both cases: the glass to be blown out is heated until it is soft enough to flow easily and is then blown out into a very thin bubble. The bubble is then removed. If the glass is too hot or it is blown too hard, the bubble will not be formed, but a small irregular hole will be blown and a cloud of very fine glass flakes produced. This is undesirable.

In the case in which the end of the tube is to be blown out care should be taken to see that the tube is heated evenly over the whole end, otherwise the hole produced in it will be skew. If the hole is to be smaller than the bore of the tube only a small area of the rounded end must be heated and the blowing must not be continued too long, since once the bubble of glass is formed it is enlarged principally by drawing more glass from around the edge of the hole in the original tube, thus making the hole larger. This also applies to the blowing of holes in the side of a tube.

The second case, the blowing of a hole in the side of a tube, is a necessary preliminary to making a T-joint, and therefore it is essential to be able to produce the right-sized hole in the correct place. The procedure is very similar to that for blowing out the end of a tube, except that a small area on the side of the tube is blown out. Generally speaking it is more convenient to heat a comparatively large area on the side of the tube and blow a slight bulge, and then heat the smaller area to be blown out more strongly with a rather smaller flame. This should give a hole, if blown out correctly and not for too long or too hard, which is surrounded by shoulders so that the rim of the hole stands clear of the general line of the tube walls. In many cases it is desirable to have these shoulders present (e.g. making T-joints).

The removal of the thin glass bubble from the hole does not present any problems. It can be easily knocked off, but this scatters flakes of glass which may get inside the glass being worked. The glass bubble may be crushed in a sheet of paper, which certainly causes less mess and may be preferred in the interest of general tidiness when the work is being carried out on fixed apparatus. A solution to the problem, which does not cause flakes of glass and leaves a supply of glass for further working about the hole, is to fuse the bubble down into a rim around the hole. This usually happens as a matter of course when a fairly cool flame is played on the thin glass, but if too hot a flame is used the glass is boiled which does not improve its properties (see Devitrification, p. 18).

To blow a very small hole the method described for multiple perforations in bulbs is useful (p. 72).

Joining Tubes of Similar Size

In this section detailed descriptions of the operations will be given for the benefit of the complete novice. It is convenient to deal with the subject in three sections, (*1*) the case in which both tubes can be rotated in the flame of a bench torch, (*2*) the case of one fixed tube and the other movable, and (*3*) the case of two fixed tubes.

(*1*) *Joining Two Tubes in the Flame of a Bench Torch*

The first step in producing a satisfactory joint is the preparation of the two ends to be joined. The most satisfactory method is to draw the tubes off and to blow the ends out. If this is done correctly the ends should be square. Cutting the tube with a file scratch leaves a line of fine bubbles in the joint due to the remains of the original scratch. This spoils the appearance of the joint when finished and may also cause a weak spot at which a pinhole may develop when testing for leaks with a Tesla coil. Jagged ends of tubing can be heated until the glass is quite soft and trimmed with scissors.

The second step is to block the end of one of the tubes and heat the ends of the two tubes in the flame until the glass is quite soft and starts to collapse. Uniform rotation is necessary for this, as in all the heating operations for this method. When this stage is reached the glass is removed from the flame and the ends pressed lightly together; initial contact is best made at some point by holding the two tubes at a slight angle (FIGURE 22). The ends are then brought together by bringing the two pieces of the tube into line. A slight pressure is then applied during the heating of the join so that the glass is thickened around it. This glass is worked thoroughly by alternate heating and blowing, care being taken to see that the two

parts of the tube are rotated at the same speed during the heating process. When the glass has been worked sufficiently, so that the original junction of the two tubes cannot be distinguished, a slight bulge is blown in the tube and it is drawn down until the walls are of uniform thickness and the tube has a uniform bore. The whole operation is sometimes described as 'butt sealing' or 'butt welding' and the joint as a 'butt seal'.

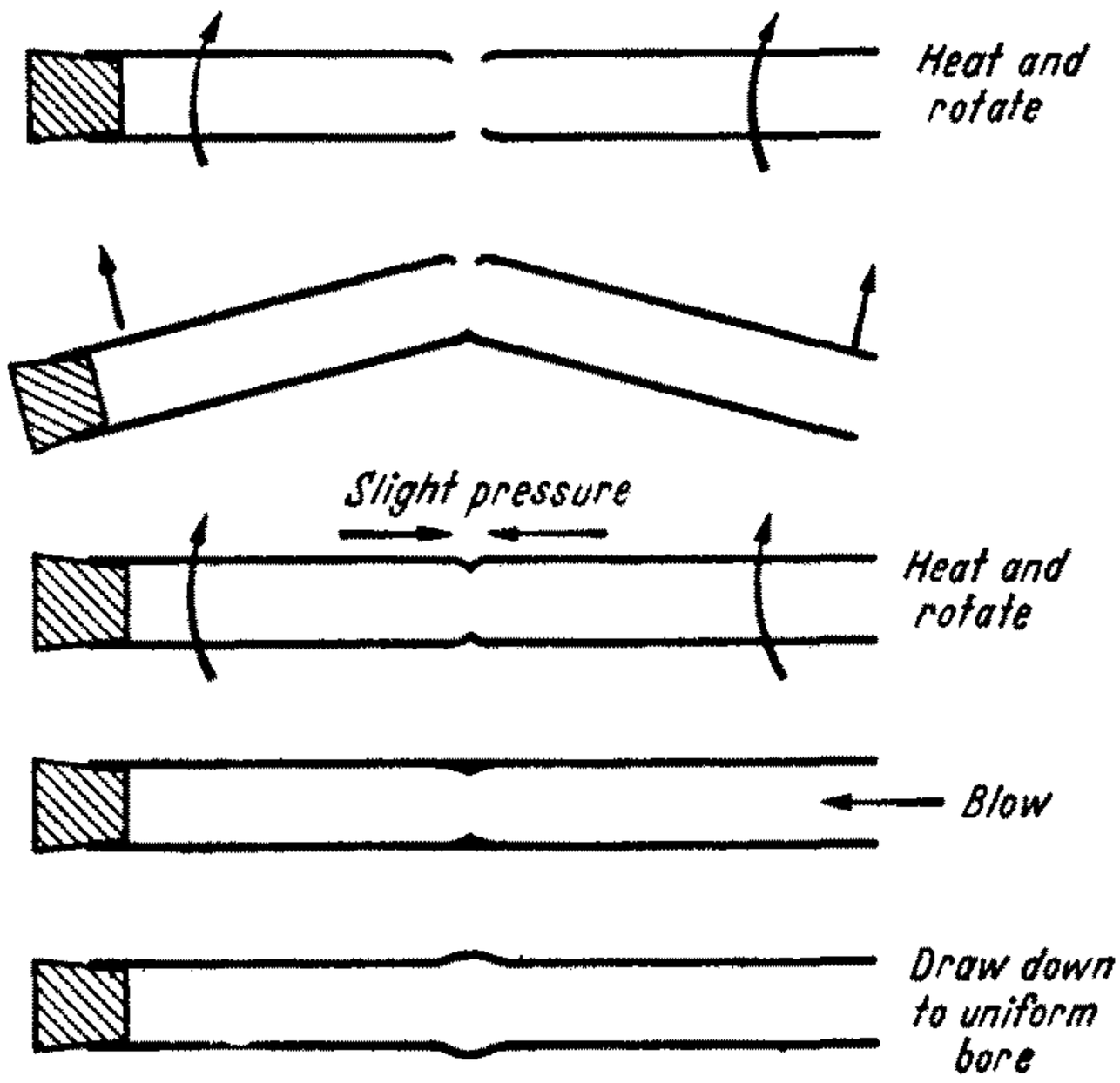

FIGURE 22. Joining two tubes

It will probably be found that much practice is necessary before good joints can be made in this way. A common difficulty is that the two halves of the tube must be rotated at the same speed, and to avoid this many novices tend to work round the joint in segments once the two pieces of tube have been pressed together. This method, while capable of producing vacuum-tight joints, leaves an irregular wall thickness which is much more liable to crack from strains induced on cooling, even though it may have been carefully annealed by cooling slowly in the flame. However, it is satisfactory for *Pyrex* and similar glass.

(2) *Joining Two Tubes, one of which is Fixed*

The necessity of joining a tube to another which is fixed arises frequently in the construction of laboratory apparatus where the

main part of the apparatus is either fixed to a stand or too large and unwieldy to allow the use of a bench torch. It is very often easier for a novice to produce a good joint by clamping the glass in position and working with a hand torch as described below, since it does not require the same degree of manual dexterity.

The two ends to be joined should be prepared carefully. The best method is by sealing off and blowing out, but in some cases this may not be possible. If the end of one tube is jagged, resulting from a break in the apparatus, and it is not practicable to close it and blow it out, the end should be smoothed by heating and working to a flat surface with a tool. This is done by removing protuberances with forceps or scissors, and then pushing the surface flat with the tool. The importance of working the ends of the tubes so that they are a good fit when pushed together cannot be overemphasized at this stage, since a good fit eliminates much work in the later stages, and overworking of glass leads to devitrification of the surface.

The working of the joint is carried out by first clamping the movable tube loosely in position so that the ends of the two tubes to be joined are nearly touching. The clamp should be left loose so that the two tubes can be pushed together later, but it should be tight enough to support the tube and not allow it to sag. The prepared ends of the tubes are heated, gently at first, with a hand torch, taking care to heat more or less evenly from all sides. Asbestos screens may be useful in joins of this type for protection of other apparatus. When the ends of the two tubes are hot enough to flow, the loosely clamped tube is pushed gently into place so that the glass is in contact all round the joint. The clamp is then tightened to prevent further movement. The glass is worked by heating and blowing in much the same way as described in the preceding section except that blowing is carried out by a blowing tube attached to one or other of the two tubes. In making fixed joints such as this it is undesirable to overheat the glass so that it becomes very liquid, since it flows under gravity. This flow should be kept to a minimum since it leads to uneven wall thickness.

The effects of this flow under gravity can be mitigated in a number of ways. If the tubing being worked is vertical it may be drawn down a little just below the join to reduce the wall thickness in this region; then, as the glass flows, this thin section will thicken so that a uniform wall thickness is achieved. Another method which can prove very useful to the amateur glass-blower is to make use of a spike to push the glass back to its original position. If the glass is heated until it is fairly soft, the point of the spike can be used to lift the glass which has dropped down. It is best to lift this glass above the position of the joint as it has to be worked to remove the local thickening of the wall

produced by lifting, and during this working it will tend to flow down again. This also applies to a horizontal joint where the glass tends to collect at the bottom of the tube.

It is very often possible to produce a better finish to a joint if the glass is actually blown while it is in the flame, but if this is done great care must be exercised to blow very slowly and with short puffs rather than a long steady pressure. It is also virtually impossible to do this in a system which has a volume in excess of two litres since the pressure response is subject to too great a time lag and when the blowing is stopped the pressure inside the system may not drop for several seconds, which may lead to a hole being blown in the glass.

An alternative procedure is to hold the movable tube in one hand while the other hand operates the hand torch: the tubes are heated and pushed together as before, and the movable tube is supported and manipulated with one hand. With tubing over about 1 cm diameter, the movable tube needs little or no support most of the time, as the side not being worked on is usually rigid enough to hold it. Hence it is possible to use welding rod for filling up any holes in the joint. The blowing tube should be attached to the fixed tube, to reduce the weight on the movable tube to a minimum. It is convenient to have the tubes vertical with the movable one held below. This general method is usually preferable to the method involving clamping the movable tube, unless the latter is very heavy.

An elegant joint can be obtained by heating the joint strongly all round, and as it thickens moving the lower tube up; then the whole is blown out and the lower tube is moved down, so that a smooth joint is made. This procedure resembles the joining of two tubes by Method 1.

(*3*) *Joining Two Tubes both of which are Fixed*

This situation may arise in the repair of apparatus or in making a closed system. If the joint is a good fit no great difficulty is to be expected as the case is similar to that just described, except that the ends cannot be pushed together unless they are in fairly close contact, when they can usually be pushed together with a spike or some other tool when the glass is hot.

If the tubes are so placed that there is a gap between them it is possible to close this gap by various stratagems. One of the two tubes may be drawn down slightly so that their ends are brought into closer contact. If this tube is vertical it is best to make the point of drawing just below the proposed joint and to use forceps to hold the short end of the tube just above the point of drawing. This will provide the constriction to take up the glass which flows down under

gravity. If the gap is not too great it may be filled in by building up a network of glass in it, using thin rod. This process is rather similar to darning the gap: a closely packed network of thin rod is built up in the gap and is then heated fairly strongly to fuse the individual rods together. The glass should not be heated too strongly while the original network is being built up, as strong heating very often leads to complete closure of the tubes. This method can be used for filling in irregularly shaped holes and finds many applications, particularly in the repair of apparatus which has been broken.

If the gap between the two pieces is too large for the above method to be attempted it may be possible to fit a sleeve of wider tubing over the two tubes and fuse its ends down on to them. This method inevitably leads to a very thick-walled joint, but the excess glass can be pulled off with forceps or by fusing a glass rod on to the thick part and pulling it out.

All joints, no matter how they are made, should be annealed with a relatively cool flame (p. 170).

When joints are made by the above methods, pinholes are very often left in them, particularly when they have been built up of thin glass rod. These pinholes are usually fairly easily visible when the glass is hot—at red heat they usually show up as fine orange lines running through it. They are sealed by placing a small spot of glass over their outer end using a thin glass rod for the purpose. The spot of glass is then fused in thoroughly. It is obviously more convenient to avoid such pinholes when the joint is made than to have to re-heat it later. It is desirable to wear glass-blowing spectacles when looking for pinholes.

Devitrified glass should be pulled out of a joint with forceps or a glass rod, and if this leaves a thin-walled joint extra glass can be built on using thin rod.

The aim in all these operations is to produce a joint with the same bore as the tube and with uniform wall thickness. Only practice will allow this to be achieved. However, with *Pyrex*, very bad-looking joints will hold together.

Joining Tubes of Different Sizes

There is very little basic difference in the procedures for joining tubes of different sizes and for joining tubes of the same size. The main difference lies in the preparation of the end of the larger tube: the end of this tube should be drawn down to a spindle and the spindle cut off in the flame. The closed end so produced should be heated locally so that when it is blown out the hole produced will be of the same size as the bore of the smaller tube. But it is usually better to

aim to produce a hole rather smaller than this, since it is a simple matter to open out the hole with a tool or to collapse the smaller tube down slightly; whereas it may be more difficult to close the hole in the drawn-down portion of the larger tube if this has relatively thin walls or uneven wall thickness. The above remarks apply equally to the use of a bench torch and to the use of a hand torch with fixed tubes.

When the joint is to be made using a bench torch it will usually be found easier to work so that the glass is blown through the smaller of the two tubes, when the joint is more easily visible. If the larger tube is used for blowing, the joint may be screened from view by it. When the novice starts trying to make this type of joint with a bench torch, difficulty may be experienced in rotating the two tubes of different

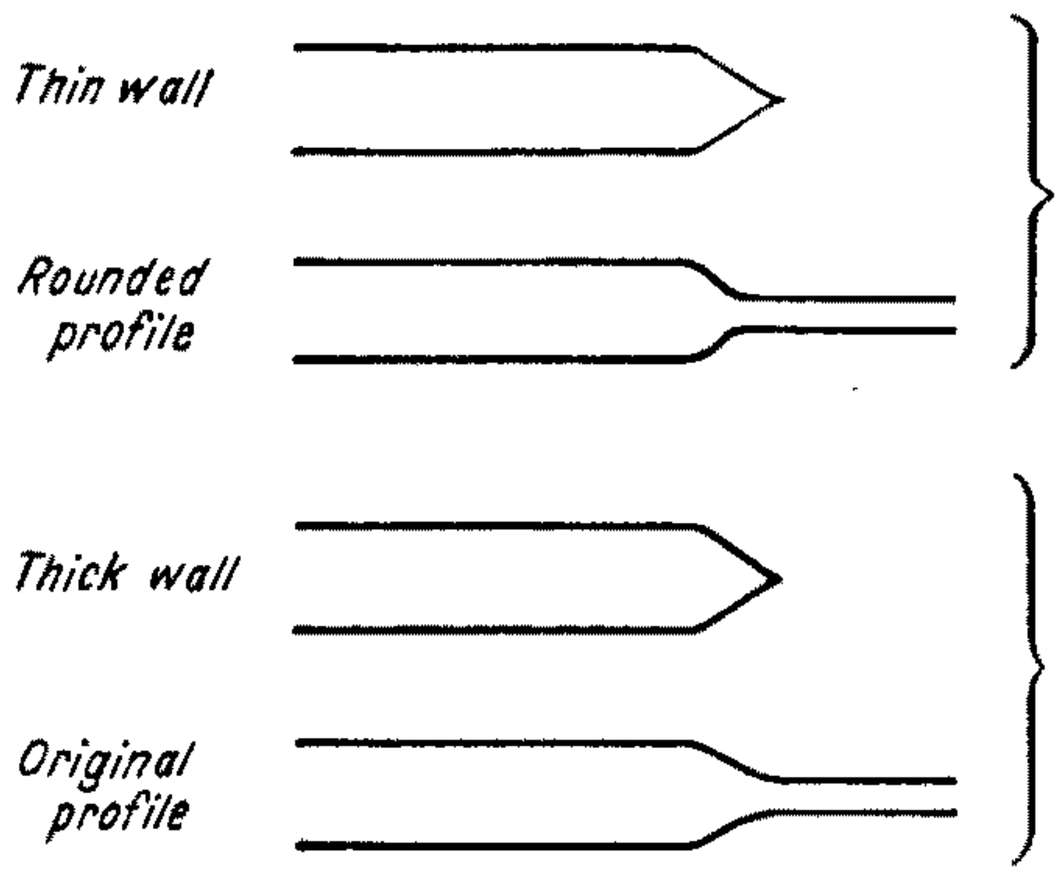

FIGURE 23. The effect of wall thickness on the joint, when tubes of different size are joined

bore at the same speed. The only remedy for this difficulty is practice, but it may be possible to avoid it by clamping the glass and using a hand torch. If only one tube is clamped, it should be the larger one.

The wall thickness of the taper produced by drawing down the larger tube can have an effect on the profile of the finished joint (FIGURE 23). If this taper has a wall thickness about the same as the unworked tube it will tend to retain its profile in working, but if it is thin-walled the joint will tend to have a rounded profile. This provides a method of controlling the type of joint produced. A rounded profile can be produced by starting with a uniformly thick rounded end.

T-Joints

The making of T-joints is probably the most common laboratory glass-working operation and therefore should be mastered thoroughly.

There are two basic methods of making these joints, (*1*) by the use of a bench torch and holding both tubes, (*2*) by the use of a hand torch and clamping the tubes (or one of them).

(*1*) *The Use of a Bench Torch*

Once again the first step is the adequate preparation of the surfaces to be joined. First the side arm should be prepared, as for butt-sealing two tubes, by blowing out the end (p. 59). A hole is then blown in the side of the tube at the position required (see p. 59 for details). Care should be taken not to make this hole too large. Both ends of the tube should then be blocked since it is best to blow through the side arm so that the whole joint can be seen while blowing. Some workers, however, prefer to blow through the cross-piece.

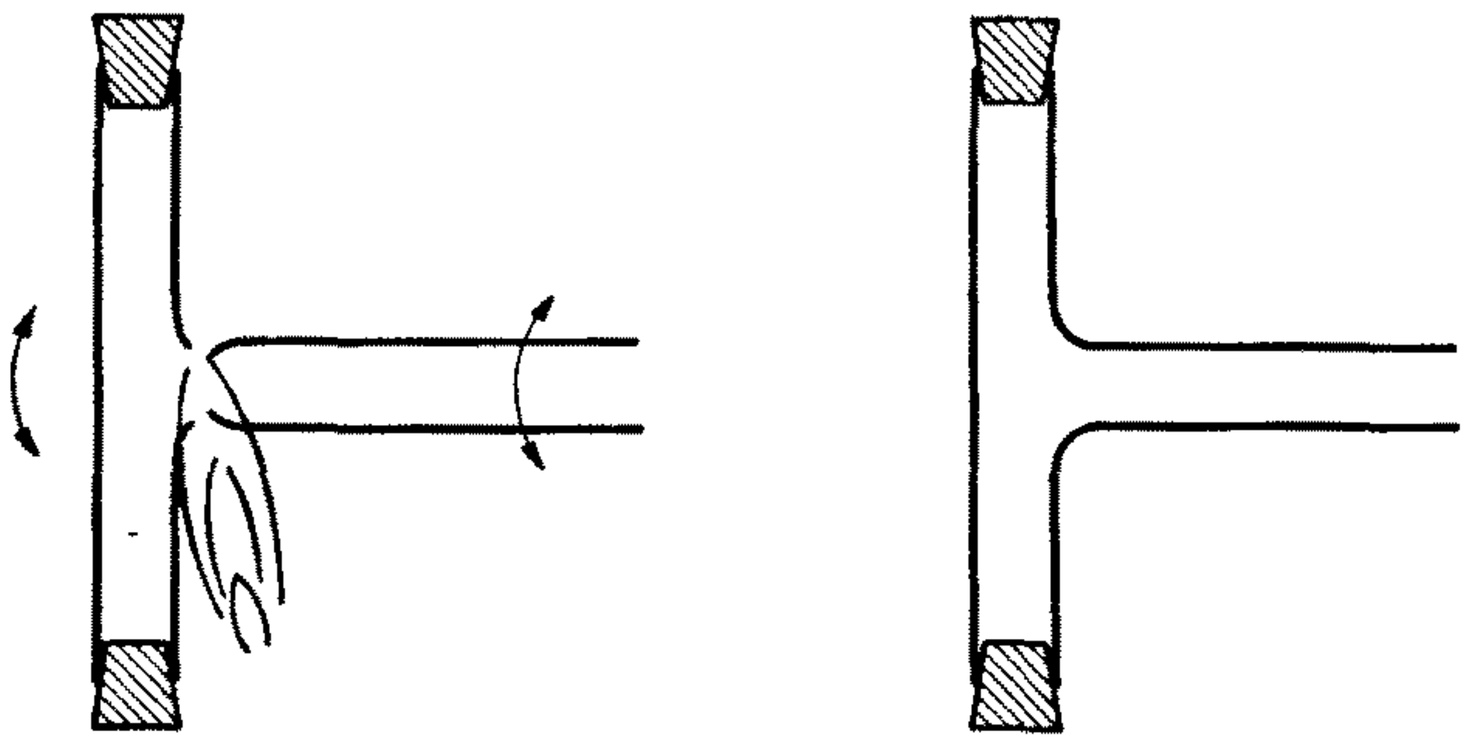

FIGURE 24. T-joints

The side arm is taken in the right hand and the cross-piece in the left and they are held in the flame so that the end of the side arm, and the area just around the hole in the cross-piece, are heated. This is done by directing the flame almost perpendicular to the side arm and swinging both pieces of glass through an angle of about 200° (FIGURE 24). These motions should be synchronized in order to obtain uniform heating. A great deal of practice is necessary before this can be done satisfactorily.

The two tubes are pressed lightly together when the glass is fused and the 'swinging' of the glass in the flame continued until the region of the join is thoroughly fused. The joint is then blown through the side arm. This is repeated until the glass has been worked sufficiently for a good joint to be obtained and it should then be annealed.

The above procedure can be simplified in two ways. First, it is not necessary to swing the cross-piece in the flame during the preliminary heating before joining the tubes, although this operation accustoms

the two hands to synchronous working. The side arm only is rotated in the flame, which is meanwhile directed on the hole in the cross-piece. Secondly, a T-piece can often be made by working only a portion of the joint at a time.

The procedure for this type of joint is the same for all sizes of tubing. In some ways it is rather easier to carry out for relatively large sizes (about 2 cm) than for smaller sizes (less than 8 mm). In the latter case it is very easy to heat the cross-piece in such a way that it bends at the joint. This may be due to several possible causes: too large a flame; failure to hold the glass at the correct angle to the flame; or overheating of the glass in the vicinity of the join, the heat being transferred by conduction.

A special technique for making T-joints, which does not distort the bore of the main tube if it is done correctly, is to make the joint by sucking: the end of the side arm, and a localized area on the main tube corresponding in size to the cross section of the side arm, are heated strongly until the glass is very soft. The end of the side arm is then placed on the heated area of the main tube and the heated area sucked up strongly into the side arm which must have a plug of cotton wool in it to catch any glass fragments. This produces a sound joint, but it requires very thorough annealing because of its very local heating.

(*2*) *The Use of a Hand Torch*

A casual study of any laboratory vacuum apparatus will show how commonly T-joints must be made with either one or both of the tubes clamped. Generally speaking this is a much easier technique than that previously described, as it removes the necessity for synchronized manipulation of the glass.

The glass is prepared in the same way as previously described, and a blowing tube is connected to some part of the system. The tubes are clamped in position and one of them left slightly loose so that they can be pushed into close contact at a later stage. The glass is heated in the vicinity of the join by working around it with the hand torch and the two tubes are pushed together. The glass is then worked by collapsing it down and blowing out until it has fused together thoroughly. The joint is then blown out to the desired profile.

If difficulty is experienced with the glass flowing under gravity the same stratagem may be used as described in the section on butt-joining two tubes (p. 62). If for any reason the tubes cannot be pushed together the two pieces should be as good a fit as possible before the joint is started and may be pushed into closer contact when hot with the aid of a spike, or if this fails the gap can be filled in with

thin rod. This may leave pinholes in the joint and a careful inspection should be made while it is hot. When larger size tubing is used it may be found advantageous to blow the holes in the tubes rather smaller than their bore, since this provides a means of thickening the wall of the joint as it is blown out to a smooth profile.

When making a T-joint with a hand torch it is often best to clamp the cross-piece and fix a blowing tube to it, and to manipulate the side arm with one hand. As with the joining of two tubes (p. 63), the side arm will be supported by the rigidity of the glass on one side of the joint most of the time, unless it is of small tubing. This method is usually preferable to that involving clamping both tubes.

The construction of big T-pieces is described on p. 94.

Y-Joints

Joining two tubes in the form of a Y is most easily effected by bending one of the tubes to the desired shape and then making a normal T-joint at the bend. If the T-joint is made first and then one of the arms is bent, the resulting Y-piece will probably not be symmetrical and the finish on the surface of the glass may not be good owing to volatilization of some of its components during the making of the joint. This causes the surface to wrinkle on bending the tube.

The most convenient order of carrying out the various steps is to prepare the side arm first, bend the main tube and blow out the hole for the T-joint, and then make the joint before the glass has time to cool.

4 and 5 Way Junctions

Once the simple T-joint has been mastered the manufacture of 4 and 5 way junctions presents no difficulty: the operations described for making T-joints are just repeated the required number of times. The various side arms are all prepared as the first stage, and sufficient corks, or other means of blocking the tubes as they are joined on, must be at hand. The only other step which requires extra care is to see that completed joints are not overheated in making others, since if any of the side arms are unsupported they will bend under gravity. Adequate annealing is, as in all other joints, essential.

Working Capillary Tubes

Working capillary tubing requires a slightly different technique from the working of normal tubing. This is principally due to the small bore of the tube and the very thick walls. Generally speaking it is necessary to use a smaller, hotter flame for capillary than would be

used for normal tubing. Three types of operation will be described in this section, (*1*) joining capillary to wider bore tubing, (*2*) joining capillary to capillary, (*3*) capillary T-joints.

(*1*) *Joining Capillary to Wider Bore Tubing*

The only difference between this type of joint and a normal butt joint is in the preparation of the capillary tube: first the end of this is sealed off and a small fairly thin-walled bulb blown equal in diameter to the hole in the wide tube. The end of this bulb is then blown out and the joint made by the normal method. The flame must be directed so that the capillary is not collapsed. If the capillary has a very fine bore some difficulty will be found in blowing through it. This may be overcome by connecting it to a compressed air line while the small bulb is blown; the end of the bulb can then be pulled out with a rod and cut off to give the prepared end.

If for any reason it is found to be impossible to blow out the end of the tube, the cut end of a capillary can very often be opened out by using a mounted needle from a set of dissecting instruments as a tool; but this is not recommended if the tube can be blown.

(*2*) *Joining Two Capillary Tubes*

Owing to the fact that capillary tubing is used in many systems to cut down the gas space it is often desirable for the finished joint to have the same bore as the tubes. Therefore it is undesirable to prepare the ends by blowing out; it is much better to join the ends of tubes cut at a file mark. They should be heated until the glass is soft, and the tubes then held at an angle to one another. The two tubes are brought into contact by touching their edges together, and then brought into line ensuring that the bores of the tubes are coaxial. The joint is then finished by heating as small a length of the tube as possible, collapsing the bore down to about half its size and blowing it out to about twice its size. This operation should be repeated until a good joint is obtained. These joints require careful annealing because of the heavy wall thickness.

(*3*) *Capillary T-Joints*

The side arm for the T-piece should be prepared by blowing the end out of a piece of capillary as described above for joining capillary to wider bore tubing. The cross-piece is prepared by heating a short length of the capillary in the flame and blowing a slight symmetrical bulb. This should not be more than twice the original bore of the tube. A very small hot flame is used to heat one side of this small bulb, which is then blown out as for a normal T-joint. If difficulty is

experienced here, it may be found advantageous to attach a glass rod to the point heated on the side of the small bulb and pull this out into a cone. The rod should next be cut away with the flame at the point of the cone, which can then be blown out much more easily. Lastly the joint is completed in the normal way and annealed thoroughly.

Fine capillary tubes may become blocked with water when blown into from the mouth; it may be desirable to put a drying tube in the blowing tube or to blow with a rubber bulb.

Blowing Bulbs in the Middle of a Tube

To ensure that the centre of the bulb lies on the axis of the tube from which it is made, the tubing, when in the flame, must be rotated continuously as described on p. 52; otherwise the glass will sag to one

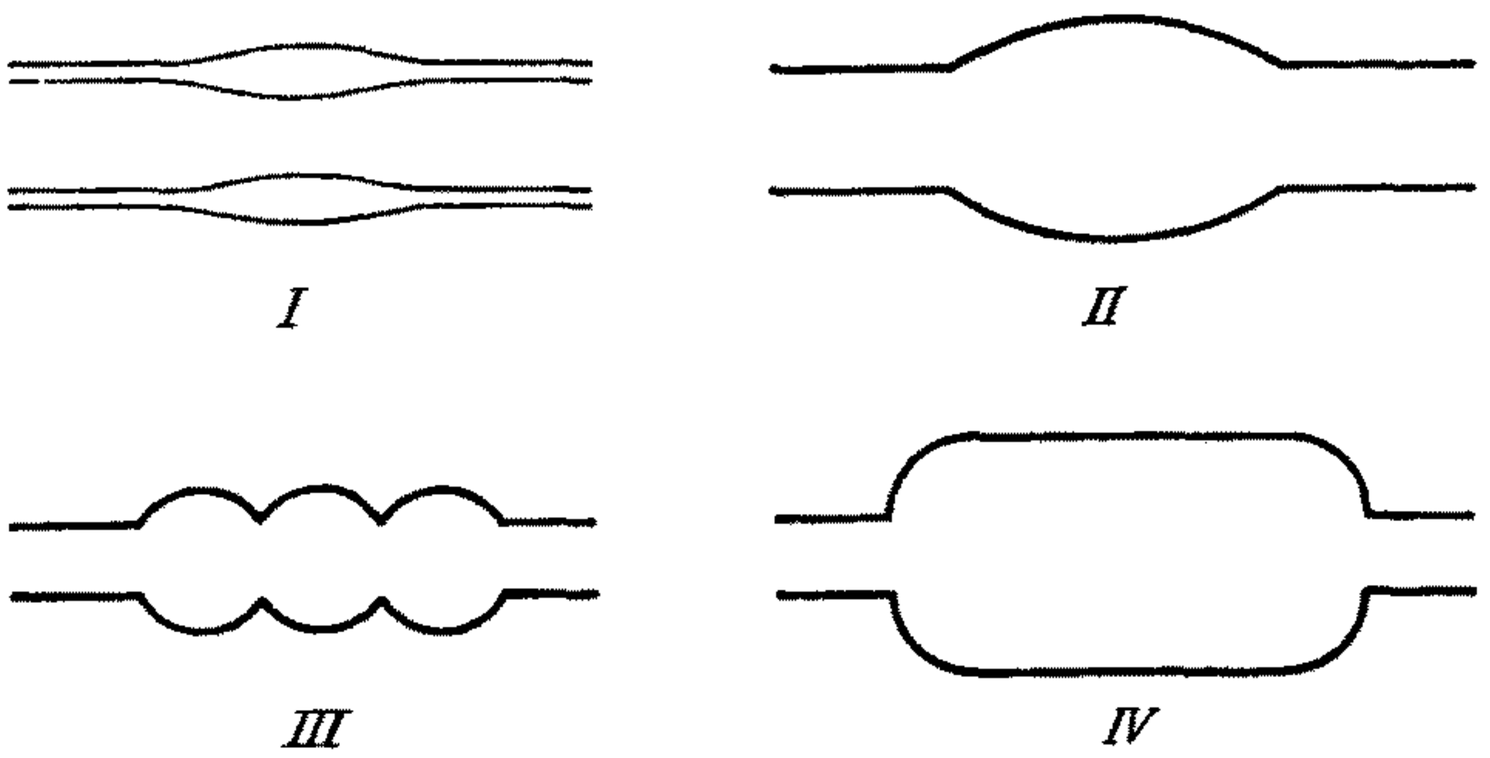

FIGURE 25. Blowing a bulb in the middle of a tube

side. Bulbs can be blown in the middle or at the end of a tube; the former operation is probably easier at first. A fairly large and relatively cool flame is used to heat a large portion of the tube, and as the tube is rotated in the flame the ends are pushed in to form a concentration of glass (FIGURE 25, *I*). The tube is taken from the flame and gently blown so that a slight bulge is formed (FIGURE 25, *II*).

The danger of distortion of the glass is much reduced when the blowing is done with a glassblower's swivel (p. 43): In this case the blowing tube is attached to the end of the tube—usually the left—which is held in an overhand grip. The process of accumulating glass is continued after the bulge has been re-heated, and the bulge is re-blown a number of times until enough glass has been accumulated for a final sphere to be blown. This may be blown by a series of puffs; the thinner parts blow out first, but they also cool first, and by blowing at the right moments a spherical bulb may be obtained. The

tubing should be rotated when it is being blown into; this is facilitated if the lips are moistened before blowing.

Instead of accumulating the glass in one bulge, an alternative method is to make three bulges by pushing the glass together (FIGURE 25, *III*) and then reduce these to a single bulge by repeated blowing and shrinking. This reduces the time required to collect enough glass for the final bulb to be blown.

If the bulb to be blown is of radius much greater than that of the tube, a large tube should be joined into the small tube, as in FIGURE 25, *IV*. The three portions of tube should be coaxial. The large one is then shrunk and blown a number of times, and finally blown to the required bulb. It should be longer than the required radius of the bulb, as its length will decrease with heating and blowing.

To blow an elegant large bulb is very difficult. It is much simpler for the scientist to join a round-bottomed flask into the tube.

Blowing Bulbs at the End of a Tube

Blowing bulbs on the end of tubing may be found difficult because there is no portion of tubing to help to keep the bulb central whilst it is being blown. A round end is first made on the tube, which is

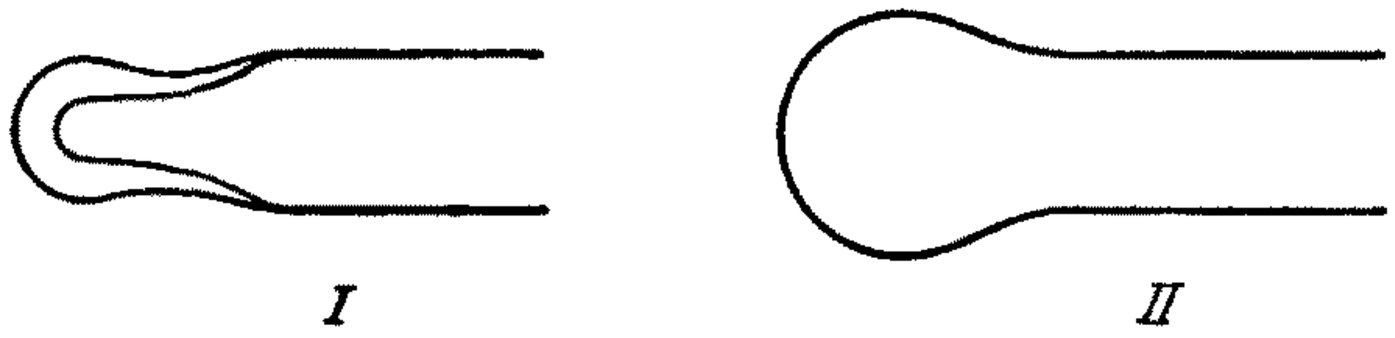

FIGURE 26. Blowing a bulb at the end of a tube

rotated continuously so that the glass thickens (FIGURE 26, *I*). The tube is taken from the flame, held near the vertical, and, with continual rotation, blown into until a slight bulb is formed (FIGURE 26, *II*). This is returned to the flame and collapsed and blown a number of times until enough glass has collected for the final bulb. When blowing this it is essential to rotate the tubing all the time, and to have a gradual air pressure, or a series of puffs, rather than a sudden pressure. The bulb must be watched as it is blown; if it is becoming off-centre it may be best to shrink it down and blow again. Beyond a certain size, however, a bulb cannot be shrunk, specially if it is thin.

Another method of blowing a bulb at the end of a tube is first to put a central spindle in it, and then accumulate glass just beyond the shoulder of the spindle by pushing the glass together with the spindle, and occasionally blowing through it. When enough glass has been

collected, the spindle is drawn off in a very small flame, and the bulb is blown as described above.

To blow a big bulb a larger tube must be joined to the end of the tube and shrunk to give the necessary accumulation of glass. The beginner should be able to make useful small bulbs which will become quite elegant with practice. For research, however, it is best to fuse a round-bottomed flask to the tube.

Multiple Perforations in Bulbs

A bulb with a number of small holes in it is useful in bubbling a gas through a liquid. These holes are easily made: a small bulb is blown; a very small area of the bulb is heated strongly, and the glass is pulled out by touching it with the fine point of a glass rod, and then

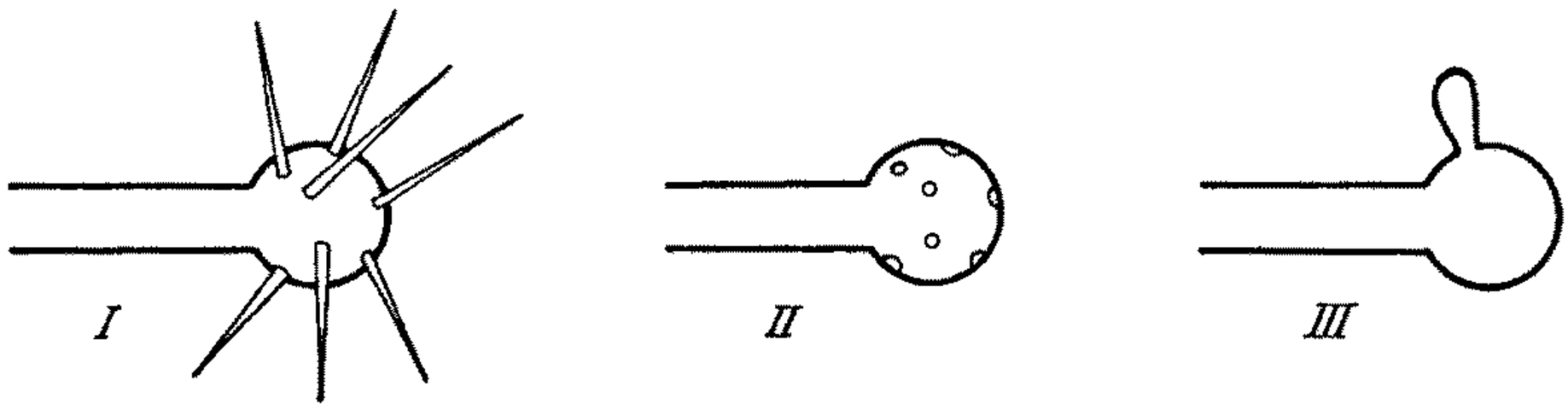

FIGURE 27. Making a number of holes in a bulb

drawing out the molten glass into a thin thread. This is repeated in a number of places, to give the result shown in FIGURE 27, *I.* The threads are then broken off near the bulb, and each hole so made is heated and rounded to give the finished perforated bulb shown in FIGURE 27, *II.*

An alternative method is to use a very small flame on the original bulb, and to blow a thin-walled bubble, as in FIGURE 27, *III.* A number of these bubbles are blown, then they are all broken off and each hole is smoothed off. If a bubble is broken accidentally, the hole can be blocked with damp asbestos paper so that more bubbles can be blown.

Flanging, Flaring or Bordering Tubing

A number of tools are useful in putting flanges of different kinds on the end of a glass tube. The operations are purely manipulative and require no blowing. Various tools which can be used in flanging are shown in FIGURE 4; perhaps the triangular-shaped tool is one of the most useful. Carbon rods are also useful (p. 33), and they have the advantage that they do not mark the glass; an overheated metal tool

sometimes leaves a small deposit of metal or oxide on the glass. The general method used in flaring is to heat the end of the glass tube over a length of 2–3 times its diameter, and when the glass has become soft and thickened (FIGURE 28, *I*) the tool is inserted and the tube rotated. The glass should be rotated fairly quickly backwards and forwards by rolling it between the forefinger and thumb of the left hand; or normal rotation may be used (p. 52). The glass should be *rested* on the tool, and the action should be gentle. The spinning, as this action is called, should be done with the flame impinging on the base of the cone formed (FIGURE 28, *II*). For a standard taper, a hexagonal

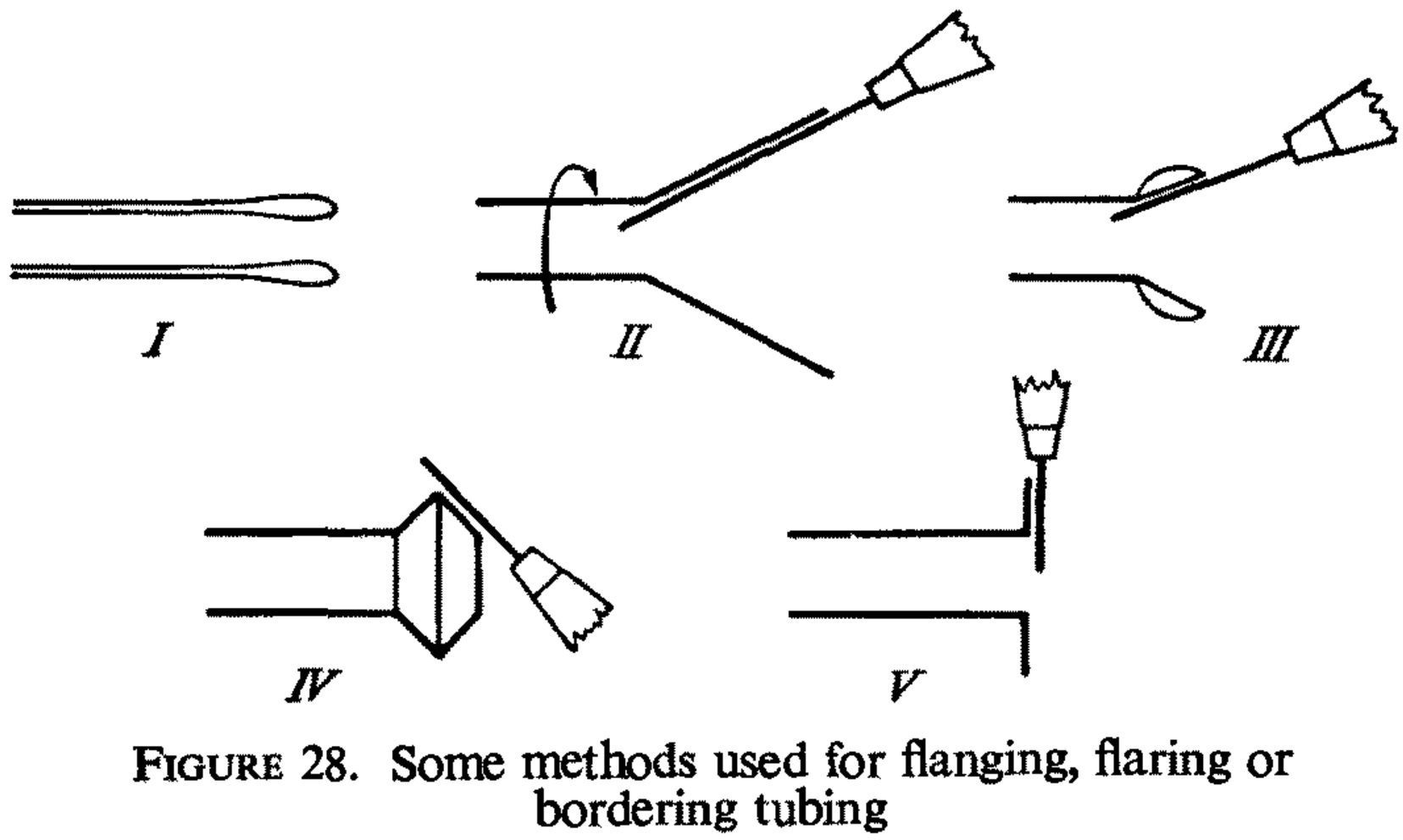

FIGURE 28. Some methods used for flanging, flaring or bordering tubing

reamer is used (p. 31). Lubrication is desirable to prevent the glass becoming twisted round the reamer. Various ways of using tools are shown in FIGURE 28, *III*, *IV* and *V*.

Internal Seals

Internal seals can be made by a variety of methods, which will be described; some indication will be given of situations in which they might be useful. Internal seals are also called ring seals.

Method 1

In this method a single piece of tubing is sealed into the larger tube. The first step is to form a slight ridge around the inner tube, at the point where it is to be sealed, by rotating it in a small hot flame and pushing its ends inwards slightly. If this is done correctly a symmetrical ridge should be formed.

The outer tube is then closed to a round end and a hole blown

centrally in this, large enough for the internal tube to be inserted but small enough to prevent the ridge on this tube passing through. The outer tube should be longer than the inner so that the open end may be closed by a cork or rubber stopper (FIGURE 29, *I*); the blowing is carried out by blowing through the projecting inner tube. It may be more convenient to close the other end of the outer tube with a spindle. This gives a useful handle.

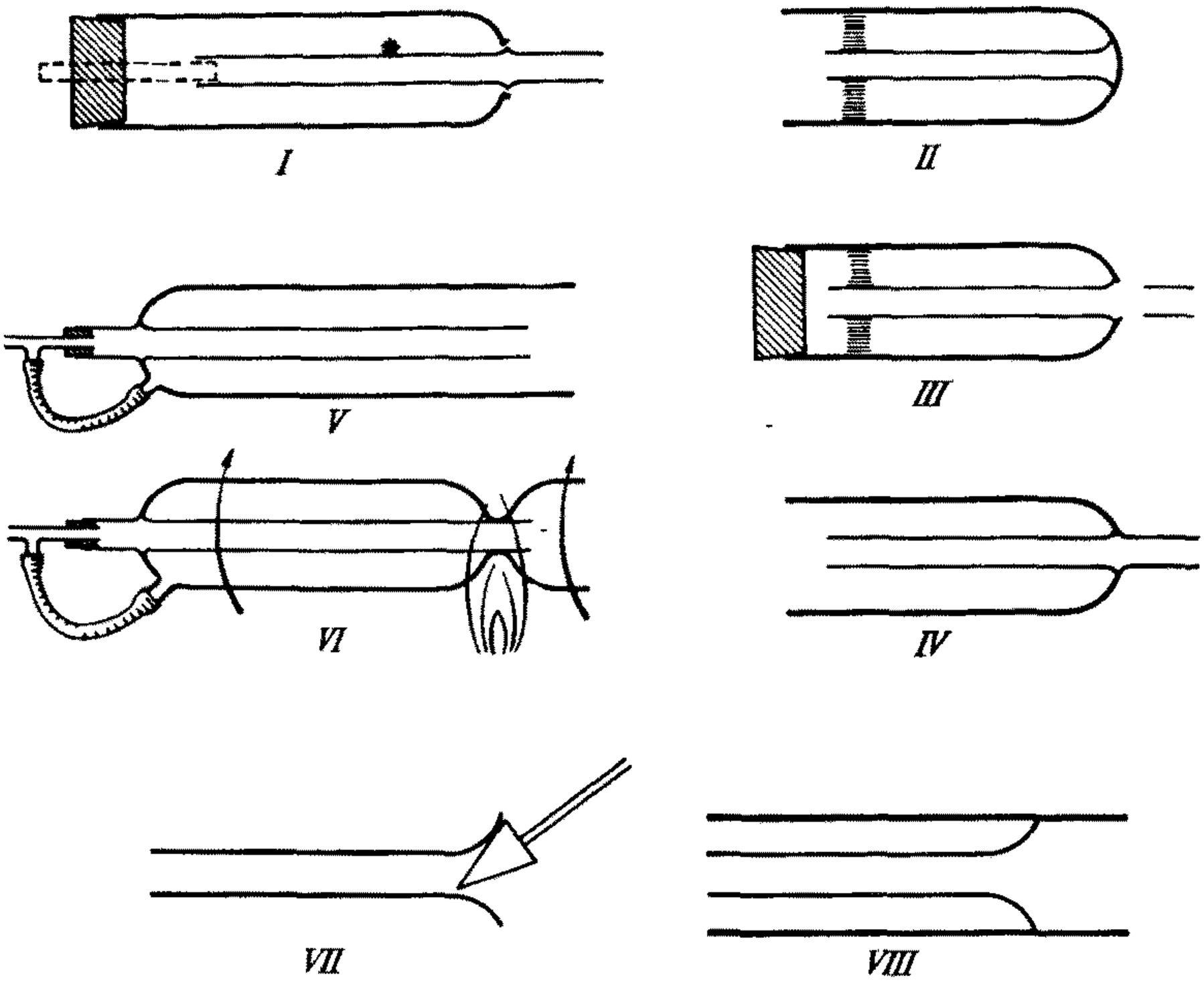

FIGURE 29. Various methods for making an internal seal

The glass is then heated by rotation in the flame so that the outer tube is fused to the ridge formed on the inner, and finally the joint is blown until a good-shaped joint is obtained. To get a good-shaped shoulder it is necessary to direct the flame mostly against the smaller tubing. The heating and blowing should be repeated until good fusion of the two tubes is achieved. Any pinholes present can be sealed as before by the application of a small spot of glass from a piece of rod. These spots of glass should be fused in thoroughly.

The unsupported internal tube will sag under gravity during the above operations, but if the rotation of the work is sufficiently uniform it can be centralized as the glass cools by allowing it to drop

to its central position and keeping it there by rotation. Alternatively it can be supported on a rod held by the cork blocking the end of the outer tube. If this latter method is used care should be taken to see that the flow of air from the inner to the outer tube is not hindered since it is essential for the air pressure to be equal in the two tubes at all times.

Internal seals require more thorough annealing than the more simple joints, and if any pinholes require subsequent repair great care should be taken in reheating the joint.

Method 2

In this method the internal tube is in two pieces and there is less likelihood of making a joint with pinholes in it. The first step is the preparation of the internal tube: a length of tubing is drawn off, the end blown out and polished smooth in the flame. The projecting part of the internal tube should be prepared in the same way. The outer tube should be prepared by closing one end to a round end.

The joint can be made most easily if the internal tube is supported almost in its final place by pads of asbestos paper or by a glass holder, but whatever support is used it must allow the air pressure to be the same on both sides of the internal tube. The inner tube is put in place and the rounded end heated. When this is starting to collapse inwards the internal tube is pushed forward slightly so that its prepared end comes into contact with the heated end of the outer tube. This glass is worked thoroughly by heating and blowing until a good fusion between the two parts is achieved (FIGURE 29, *II*). The end of the tube is allowed to cool slightly and then, with a fairly small flame, the area of glass covering the internal tube is heated and blown out. The other end of the outer tube is then blocked with a stopper (FIGURE 29, *III*) and the remaining part of the inner tube joined on as in a simple butt joint (FIGURE 29, *IV*). Once again it is important that the air pressure should be the same in both inner and outer tube and that the tube should be rotated uniformly. Finally the joint should be annealed.

These joints can be made with the internal tube unsupported by inserting the inner tube only after the outer tube is hot enough for it to stick firmly and then proceeding as above. This is rather more difficult and is seldom as satisfactory as wedging the inner tube, although at times one is tempted to avoid this added step.

This method is not on the whole satisfactory: with soda glass, the cold inner tube will probably shatter, and if a borosilicate glass is used to avoid this, the difficulty of getting the inner tube central still remains. A better method is the following: the outer tube is

rounded off and pulled to a slight taper. The inner tube is flared slightly so that it fits into the taper at the desired point and is thereby centralized. The tubes are fitted together when cold, and the operation is continued as before.

Method 3

The third method of making internal seals is not so widely applicable as the previous two, but it is very useful for certain joints. It is best described in terms of one of its uses—in the manufacture of a Liebig condenser.

Let us assume that the condenser is in the state shown in FIGURE 29, *V*: the first internal seal has been made by one of the two methods described above, and a side arm has been put into the water jacket. A connection should be made with rubber tubing, so that the water jacket of the condenser is connected to the inner tube, and then the outer tube is heated with a hot narrow flame to collapse it down on to the inner tube for the internal seal. When the tube has collapsed on to the second tube the heating is continued and both tubes drawn off together (FIGURE 29, *VI*). The system is then blown slightly to give a good round profile to the end of the outer tube; when this has been done the end of the inner tube is blown out and the joint finished as in Method 2. Another method of performing this operation is described on p. 130.

Method 4

This method involves the fusing of a flared-out narrow tube into a wider one. This type of seal has its uses in pieces of apparatus such as McLeod gauges. The first step is to flare out the end of the inner tube, using a flaring tool, until it will only just pass inside the wider tube. This is done by rotating the tube in a flame and using the flaring tool as shown in FIGURE 29, *VII*, or as described on p. 73. If a very large flare is required it will probably be necessary to fuse a wider tube to the narrow one as the initial stage in flaring. When the flare has been made to fit it is packed in place with asbestos paper or held with a rod, and the outer tube is collapsed down on to it (FIGURE 29, *VIII*). The outer tube is blown out again and the process repeated until a good fusion of the two pieces is achieved. This type of seal requires particularly careful annealing.

Use of the Hand Torch

Generally speaking it is best to make internal seals using a bench torch and rotating the glass in the flame. If this is not practicable it is possible to work with a hand torch with the glass clamped if Method

1 is used: in this case a rim of glass, left by the blowing of the hole in the outer tube through which the other tube passes, can be heated with a hand torch and pushed in against the walls of the inner tube with a spike. This can be done also in cases where the formation of a ridge of glass on the inner tube has been impracticable. Once an air-tight joint has been made by pushing the glass into place, the hand torch can be used to fuse the two tubes together by working round the joint. As before it is essential to have the blowing tube connected to both inner and outer tubes.

One great difficulty with this type of work is the force of gravity acting on the inner tube: If this is unsupported it is very difficult to obtain a good-looking joint, and even if it is supported the results are not always satisfactory, owing to non-uniformity of heating. It is best to clamp the tubes vertically. The inner tube may be held in one hand. Difficulties may possibly arise if the outer tube has thin walls, because any holes will enlarge as the glass is worked. In this case they may be filled with rod as in the other cases described. Care should be taken in inspecting these joints for pinholes and annealing the work when completed.

Thin Glass Windows

In recent years the need has arisen for very thin glass windows capable of withstanding a vacuum yet thin enough to transmit α and β particles. S. ROSENBLUM and R. WALEN (1945) have described the following method for putting very thin windows into capillary tubing of up to 2 mm bore. The process is very simple but may require some practice before the precise conditions necessary for a satisfactory window are achieved.

The end of the capillary tube is heated until the glass is soft, then before it has time to cool it is touched on to the surface of a thin bubble of glass and a slight suction applied. This forms the window into a concave shape and draws it slightly down into the capillary, whose ends then protect it from damage. The bubble of thin glass should be thin enough to show interference colours. These windows will stand a vacuum provided atmospheric pressure is on the concave side of the window. If they are subjected to a pressure difference in the other direction, failure occurs due to the reversal of curvature.

A method for making larger thin windows has been described by J. T. LLOYD (1949). For windows in the end of a tube he advocates the blowing of a thin-walled bulb at that end and then drawing it into the tube to form the window by the following method: the thin-walled bulb is blown at the end, the wall of the tube is then heated all round at a short distance from the end until the glass is fused, and then the

bulb is immediately re-heated and sucked inwards. The bubble is drawn into the tube and is fused to the walls in the region where the tube was heated. This gives a relatively robust window capable of withstanding a vacuum and protected by the projecting ends of the tube.

Lloyd also gives details of a similar method for the formation of windows in the sides of tubes. The bulb is blown on the side of the tube in the same way as it would be in the first stage of making a T-joint; it is then heated and sucked in. Lloyd's method requires more practice than the other method before satisfactory results can be obtained, but it has the merit of being more versatile.

Spinning Out Feet

The stands on measuring cylinders, wine glasses, etc., are called 'feet' by the glass-blower. They are made by an operation similar to flanging: the glass must be thick enough for spinning out. A piece of 3-cm diameter tubing about 10 cm long, with spindles (spears) at

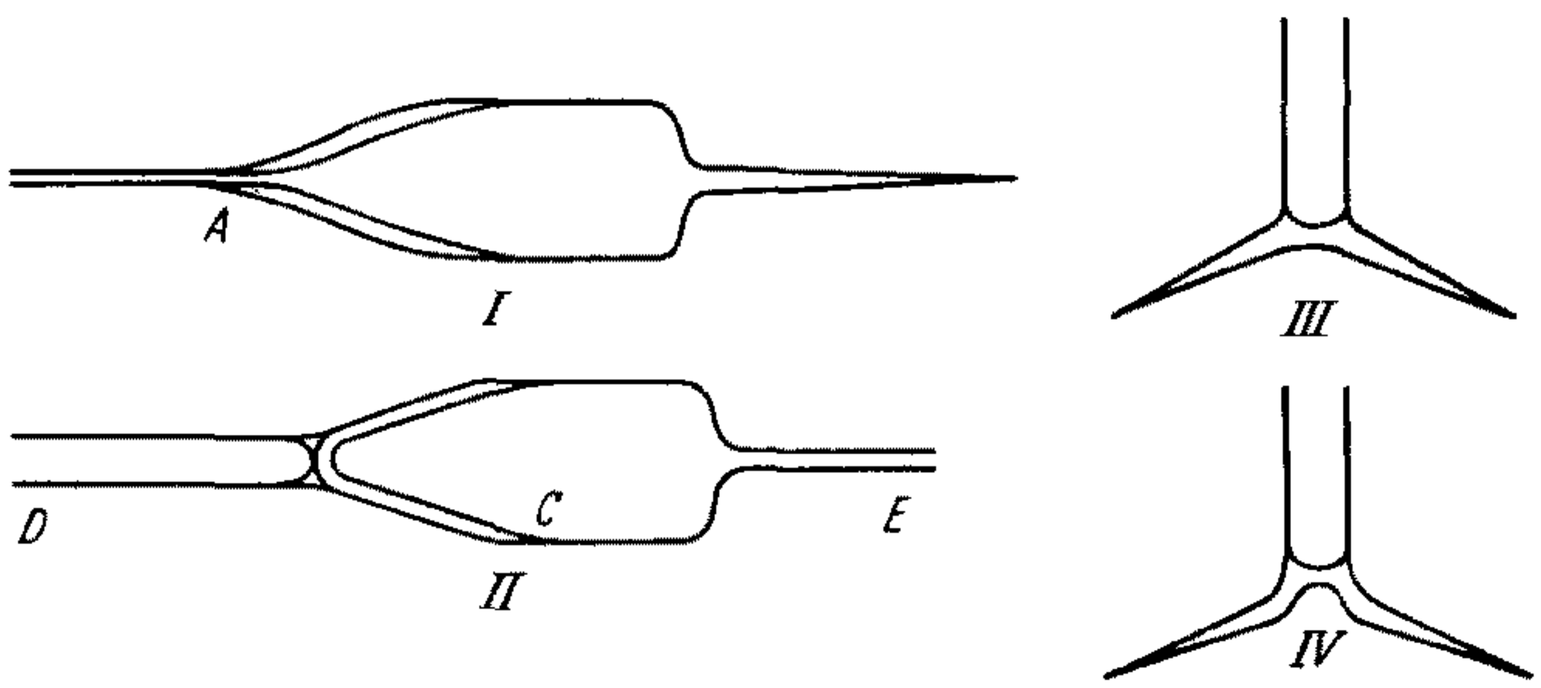

FIGURE 30. The production of 'feet' for vessels

each end, is prepared. One end is then heated in a large, relatively cool (soft) flame until it thickens, and, by pushing with the spindle and by blowing, the shape shown in FIGURE 30, *I* is produced. The spindle at *A* is removed and the end closed. This closed end is then joined to the closed and rounded end of the tube for which the feet are required. The two ends are simply brought together (FIGURE 30, *II*), and, by blowing through both *D* and *E* and slightly pulling, a good joint is made. The tubing is next cut at *C*; and the feet are worked to shape with a rounded tool (FIGURE 4, *G*), using the same operations employed in flanging (p. 72). It is important, however, to get the glass very hot and soft, so that the feet can be spun out in one operation, and not to push too hard with the tool, to avoid producing buckled

feet. As the hot glass is rotated horizontally, the tool should be slowly brought from the horizontal to the vertical. The spinning out should be done along the whole width of the glass to produce the shape in FIGURE 30, *III*; if only the outside is spun out the shape in FIGURE 30, *IV* is produced.

Tapering Glass Tubes

Glass tubing may be tapered by two methods, depending upon the wall thickness required. For a thin-walled taper, a central spindle is first put in the tube, and the shoulder is heated in a large soft flame. The tubing is rotated and slightly collapsed, and then taken from the flame and pulled out to the required taper, with constant rotation. This will give a fairly thin-walled taper, conical in shape; but by slightly pushing in the ends while heating the shoulder a thicker wall can be obtained.

For a thick-walled taper the operations are the same except that the tubing at the shoulder is shrunk down and blown out a number of times to accumulate glass. When enough glass is present, the shoulder is made hot, slightly blown out, and then drawn down out of the flame.

To taper a tube from one size to another, two tubes of the desired sizes are joined and worked to a taper at the joint.

When making tapers to fixed dimensions it is convenient to draw the required taper on asbestos paper and work the glass to match the drawing.

Sealing-in Sintered Glass Discs

The glass disc to be sealed into a tube must be made of glass having the same thermal expansion coefficient as the tube. Discs of different porosities can be obtained; they consist of a large number of particles of glass sintered together so that numerous channels remain. When

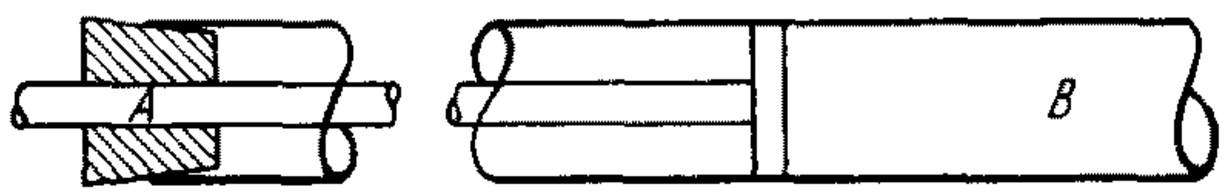

FIGURE 31. Sealing a sintered disc into a tube

working with a disc it should be warmed up carefully. One end of the tube into which the disc is to be sealed is closed with a rubber stopper carrying a tube *A* (FIGURE 31). The disc is rested against this tube, and should just fit into the outer tube. If that is too big, a constriction must be put in.

The tube and disc are carefully warmed until the flame is yellow; one side of the tube is then heated strongly and attached to the disc. The disc is next made perpendicular to the tube by pushing with a rod through the end *B*, and manipulation with *A* if necessary. The tube is allowed to cool slightly so that the disc is firm, and this is then attached again to the tube by strong local heating opposite the first point of joining. The whole tube is then shrunk down on to the disc. It will probably be necessary to blow the tubing out a few times, blowing through both ends. The tubing can finally be pulled down to a uniform diameter. The joint should be annealed with extreme care, preferably in an oven.

Mending Cracks

Unfortunately, mending cracks in laboratory glassware is one of the more common tasks. Cracks usually occur in glassware for one of two reasons—either failure to remove intrinsic strain from the glass by annealing, or else straining of the glass by careless handling of the apparatus itself. Whatever the cause of the crack the procedure for repairing the damage is the same.

The first stage in mending cracks and the one requiring most care is the initial heating of the glass: If this is done carelessly the crack will almost certainly spread much further. The glass should be warmed up slowly with a small flame, very little oxygen or air being mixed with the gas. This small flame should be played over the surface of the glass in such a way that it does not remain directed at any one part. The whole region surrounding the crack should be heated and not just the immediate area, particular care being taken if the crack is near an internal seal to see that this is heated evenly. As the glass warms up the size of the flame and the proportion of air or oxygen in it can safely be increased until the glass becomes soft and there is no danger of further cracking. For very hazardous repairs it is desirable to heat first with hot air, and then a flame with no air or oxygen until the article is covered with soot, which probably assists the uniform distribution of heat. When the soot starts to burn off, the most dangerous part of the operation has been reached, in our experience.

When the glass is soft the flame should not be made any hotter—a relatively cool flame is much more effective for sealing cracks. The flame is then played on to the crack, starting at one end, and the glass heated gently. It should be possible to see the crack sealing up under these conditions and the flame should be moved along it as it seals. If too hot a flame is used the crack will open as it is heated because the glass at its edges will become sufficiently hot to draw back under surface tension before the inner parts of the crack have had a chance

to fuse together: this is then a progressive process and the crack can only be sealed by filling in with glass rod or pushing the sides together with a tool.

When the crack has sealed, a hotter flame can be used and the glass worked by heating and blowing until the crack has been completely sealed. It is necessary to keep the glass in the region of the original crack, and all the surrounding parts, at a dull red heat for a short while (for *Pyrex*) and then to cool it slowly to allow sufficient annealing time. Care must be taken with this general annealing of a fairly large area of glass if further cracks are to be prevented.

Extremely unpleasant cracks can be sealed in *Pyrex* glass. With soda glass, however, the sealing of cracks is a hazardous undertaking, and the whole apparatus may be lost. Cracks can be sealed in *Monax*.

Detection and Removal of Leaks and Holes

Holes in awkward places are often difficult to find when one is assembling complex apparatus. If the joint in question is put in a luminous flame, or a flame put round the joint, and the apparatus blown into, the air will rush out of the holes and blow the flame away in a blue jet. The hole, when found, can then be repaired with thin glass rod (p. 58); or, if it is very big, it can often be filled in with a piece of glass. When a piece of glass falls out of an apparatus, it can be fused to a rod, to provide a handle, and put back again with the help of thin glass rod.

These methods work well with *Pyrex* glass, but not with soda glass, which cracks too easily on re-heating. A hole which has been mended with blobs of glass from a rod needs to be fused down and blown out a few times, to make it more homogeneous. Fusing down and blowing out glass often closes a small hole—this method works better with soda than *Pyrex* glass.

Small holes and leaks can often be found by covering the outside of the apparatus with soap solution and noting where bubbles appear when it is blown into. Very small holes can be found with a high frequency discharge: the apparatus is evacuated with a rotary pump (or a good water-pump) and the electrode of a Tesla coil is brought near it. A discharge appears inside the apparatus, and if the electrode comes near a hole or fine capillary in the glass, the discharge passes through it and a very bright spot of light appears at the leak. With a water-pump vacuum the method is not so sensitive, because only a poor discharge is obtained in the apparatus. When the pressure in the apparatus is about 0·1 mm, very fine leaks can be found.

However, the Tesla discharge may blast a hole in the tubing, and is not useful for glass–to–metal seals, because it goes straight to the

metal. If the seal is covered with cotton wool soaked in carbon tetrachloride or alcohol, this will pass through any leak, and should produce a greenish or bluish colour in the Tesla discharge. This method, however, is not reliable.

The minute capillary leaks found with a Tesla coil should be plugged with a blob of hot glass; the whole joint then needs heating and annealing. When assembling complex apparatus, parts which will become inaccessible to the flame must be tested for leaks before they are put in their final positions.

Grinding Glass

Glass surfaces which must fit closely are usually ground to shape. The most common example is the cone-and-socket joint: the surfaces are first worked to an approximate fit with fairly coarse carborundum (about 100 mesh) which is put between them and wetted. The cone is rotated by hand or in a lathe or drill chuck, or by a special machine, until the surfaces meet closely—the carborundum must be kept moist all the time. The coarse abrasive is then washed off, and the grinding continued a number of times with progressively finer abrasive. Various possible abrasives are mentioned on p. 39.

Surfaces which require regrinding, such as those of stopcocks, usually fit fairly well, and a fairly fine grade of carborundum can be used for the grinding. Stopcocks must not be ground too much, or the holes in the plug will no longer meet those in the barrel.

A flat surface can be put on the end of a tube by grinding. Such a surface may be required when a flat plate has to be cemented on to a tube. The flange is ground against a flat metal or glass plate using wet carborundum as an abrasive. The flange must be kept steady and not rocked, and is ground with a rotating motion of the whole tube about a point outside it. Progressively finer grades of carborundum are used. An operation of this kind is carried out very easily when a lapping wheel is available (p. 44).

Releasing Frozen Glass Surfaces

Ground surfaces are often found to be jammed together. If the parts are robust, a sharp tap will often release them. A glass stopper stuck in a bottle is often released by tapping with another glass stopper, or something solid. When the substance in the bottle has cemented the stopper in position, the joint may be put in a suitable solvent. Frozen surfaces are often more easily released after the joint has been immersed in a detergent solution for some time.

Fragile joints which cannot be struck may often be released by

quickly warming the outer member. This may be done with hot water, or, less safely, with a flame. If the surfaces are made too hot they are melted together and the apparatus must be discarded.

Polishing Glass

For many years, the standard method for polishing glass in the laboratory has been by rubbing the glass over a sheet of brown paper covered with a mixture of flour emery and a solution of camphor in turpentine. C. V. Boys (1927) has stated that he can find no reason other than tradition for the use of the solution of camphor in turpentine rather than pure turpentine for lubricating grinding media.

When the best possible polish has been achieved with the above mixture, rouge and water should be used for improving it. During the polishing only a fairly light pressure should be applied and the specimen should be moved fairly rapidly. If the polishing is being done manually, pressure should be applied evenly over the whole surface being polished. It is possible, however, to mount small samples in pitch and use a rotating polishing surface.

Cerium oxide (*Cerirouge*) has been recommended as being superior to rouge as a polishing material (*J. sci. Instrum.* 22, 1945, 224) and special cloths are marketed impregnated with cerium oxide. These are useful for polishing glass and other materials such as fluorite. For details of the methods of polishing optical surfaces, works on that subject should be consulted.

Drilling Holes in Glass

The problem of drilling holes in glass has occupied the attention of scientists since the days of Faraday and many methods have been described. Articles reviewing the subject have been published by C. V. Boys (1927) and more recently by P. Grodzinski (1953).

The method which has been used most widely is probably that involving a copper or brass tube in conjunction with a grinding mixture of carborundum and water; a slotted copper rod is also effective for small holes. To obtain the best results by this method a fairly high drill speed should be used and the copper tube drill should be raised from the cut periodically to allow a fresh supply of the grinding medium to enter the hole. It is also desirable to have some slots in the end of the tube to hold the grinding medium. The pressure on the drill should be fairly light, as too great a thrust may splinter the glass and may also slow up the cutting by preventing the grinding medium from getting between the rotating drill and the glass. Grodzinski favours the use of diamond powder in place of the carborundum,

but in most laboratories this is not obtainable. It is easy with a tube to obtain holes 3 cm in diameter.

Many variations of the shape of steel drills for glass have been described—from Faraday who used the ground end of a triangular file to C. V. Boys who designed a special drill of tool steel—while other workers (B. BROWN, 1927; N. G. HEATLEY, 1938) advocate the use of a normal twist drill. These drills are used in conjunction with some lubricant—usually the traditional camphor in turpentine, although pure turpentine or other mobile liquid seems to be as good, while Heatley states that dilute sulphuric acid is effective. To avoid splintering, the hole should be drilled from both sides.

Grodzinski, commenting on the use of sintered carbide drills, states that high speed is necessary for their use and strongly favours diamond drills. He gives a full account of their use and gives details of the mounting of diamonds for this purpose. Probably the simplest method of preparing a diamond drill for small diameter holes was described by Heatley (1938) who broke off the end of the elongated eye of a sewing needle and soldered a small diamond chip into this. He was able to drill quite deep holes, of diameter less than 1 mm, in glass with this type of drill.

Cementing Glass

Standard cones and sockets of metal and glass can be joined with *Picien* or *W* wax; such joints are vacuum-tight. Windows can be put on metal apparatus with these waxes. *Picien* is a black wax of low vapour pressure, and flows freely at about 80°C. Three useful *Apiezon* waxes are *W*, *W100* and *W40*, which are applied at temperatures of 100°C, 80°C and 40–50°C respectively. When a large glass socket is waxed to a metal cone, the strain set up on cooling can easily crack *W* wax, but *W40* is satisfactory for large joints. These waxes can also be used to seal two glass surfaces together, and a vacuum-tight joint can be obtained without ground surfaces.

Aero Research Ltd make two resin cements, *Araldite type 1* and *Araldite 103* and *Hardener 951*. They are epoxy resin cements, the former being thermal setting and the latter cold setting, and make transparent seals. To use *Araldite type 1*, the surfaces are cleaned, degreased and heated to 100°C, and the cement in solid form is applied. It melts and runs smoothly over the surface. The surfaces are pushed together and held together by a clamp while the whole is heated for a curing time dependent upon temperature, varying from 10 minutes at 240°C to 7 hours at 140°C. These are minimum times, and should be increased for stronger joints.

To use *Araldite 103*, 8–10 parts of *Hardener 951* are added to 100 parts of *Araldite*—they are both liquids. The mixture is stirred and allowed to stand before use for 20–30 minutes at room temperature. The pot life of the mixture is about 3 hours at 20°C, so it is made up as required. The glass is degreased and the cement applied with a spatula or brush as a thin, even coating on both surfaces. The surfaces are immediately joined and clamped lightly. The cement cures at a rate depending on temperatures. 16–24 hours are required at 20°C and 10–20 minutes at 100°C. It is claimed that *Araldite* can be used for vacuum-tight, glass–to–glass seals.

Mica windows can be fused to glass using a special powder of a lead borosilicate glass softening at 450°C (J. S. DONAL, 1942). A suitable glass for solder seals of this kind is *BTH No. 93*.

Silvering of Glass

Glass surfaces may be silvered by chemical precipitation of silver on to them. This is most conveniently done by reduction of a silver salt; the speed of precipitation depends on the reducing agent used. The glass must first be cleaned: it is degreased with benzene, washed with acetone and water, and left in 'chromic acid' (see p. 49) for an hour or more at room temperature. It is then washed with tap water, followed by distilled water. Some workers prefer to then clean again in concentrated nitric acid, followed by the water washings. The glass must on no account be allowed to dry after these washings.

The speed of precipitation of silver required depends on the surface to be silvered. If the rate of precipitation is too great, the silvering agent may be spent before it can be removed from the surface, and a 'bloom' will appear because the silver is in contact with the spent solution. This is a discolouration of the silver. Thus if a rapid silvering agent is used for Dewar flasks, it may be difficult to avoid spoiling the silvering.

The silvering agent usually consists of two solutions, sometimes three, which are mixed immediately before use and poured on to the object to be silvered. Various recipes have been proposed (I. C. GARDNER and F. A. CASE, 1931). The following are useful for slow silvering (R. B. SCOTT, J. W. COOK and F. G. BRICKWEDDE, 1931):

Solution A—50 gm silver nitrate in 2l. distilled water
Solution B—90 gm potassium hydroxide in 2 l. distilled water
Solution C—(80 gm cane sugar in 800 ml. distilled water) plus (100 ml. ethanol and 3·5 ml. nitric acid sp. gr. 1·42).

Solution A should be kept in the dark, and C improves with age. The solutions are used in the proportion A : B : C = 16 : 8 : 1 by volume. To use, concentrated ammonia is added to A drop by drop,

with stirring, until the precipitate first formed is redissolved. Th requisite amount of B is next added, and a dark brown or black precipitate forms. (But if enough ammonia has not originally been added to A, a yellowish-green precipitate will be formed; the solution should be discarded in this case). Ammonia is then added until the brown-black precipitate is almost redissolved; and to the resulting solution the correct amount of C is added to give the final, prepared solution. The intermediate solution prepared from A plus B can be used immediately, or kept up to an hour, after which it loses its activity. If allowed to stand for about half an hour it will become practically clear.

To silver a mirror a bath should be used just larger than the article to be silvered. The glass should be cleaned and under no circumstances allowed to become dry. The prepared solution is poured over the glass and the bath is rocked steadily. The silvering must be stopped at the right time or the mirror will be tarnished. The end-point is when a flocculent precipitate is formed. The time for this to happen depends on a number of factors: old silver nitrate solution deposits silver more slowly; temperature alters the rate, and a test run to find the approximate time may be desirable. To see how the silvering is proceeding one may take the mirror out of the solution, but it should never be exposed to the air for more than 1 or 2 seconds. The rocking during the silvering prevents precipitate adhering to the surface and spoiling the mirror.

The mirror can be silvered a second time if a thicker coating is required. Brighter and thicker coats are obtained by using a more dilute solution twice instead of a more concentrated solution once.

Front-silvered mirrors can be polished with optical rouge on a pad of chamois leather over cotton wool. Care is necessary because it is easy to polish completely through the silver. Back-silvered mirrors can be protected by first varnishing the silver and then painting the varnish. Front-silvered mirrors can be protected with a thin coat of lacquer, such as a dilute solution of cellulose nitrate in amyl acetate. The lacquer should not be too thin, or interference colours may be produced.

In order to silver Dewar flasks the jacket is cleaned and filled about two thirds full with the prepared solution. The Dewar should be rolled or shaken during the silvering. The end-point is determined by occasionally pouring out a little of the solution, and when a flocculent precipitate is formed the solution should be removed from the flask as quickly as possible to prevent blooming. The flask is then cleaned and dried. If the glass is first etched with a dilute solution (about 0·7 molar) of hydrofluoric acid before its final cleaning, the

stability of the silvering with respect to peel-off is increased, and spots are less likely.

A simple method for filling and emptying Dewar vessels is mentioned on p. 123. F. E. J. OCKENDEN (1938) has given some hints on silvering glass, and E. A. BAKER (1931) has described a method for electrolytically removing silver from one face of a small mirror. P. B. G. UPTON and E. F. G. HERINGTON (1950) have described a method for depositing a heavy silver film by reduction of a silver ammonionitrate solution by hydrazine sulphate. The deposition of silver other than on the desired surface is prevented by pyridine. The effect of the pyridine is due to the presence of surface-active impurity, and the process becomes controllable by adding cetyl pyridinium bromide to 'inactive' pyridine.

Depositing Copper on Glass

Copper can be electro-deposited on to a silvered article: the plating bath consists of 10 gm of copper sulphate in 100 gm water. The article is taken from the silvering bath, washed, and put wet in the plating bath—on to a copper cathode so that electrical contact is made to the silver. A copper anode is placed 1–2 cm above the article. The electrodes should be about the same size as the article. The current density should be about 0·05 amp/cm², and in 2–4 minutes a thin copper film should be made. If the current density is too high a granular deposit will be formed which will rub off, and if it is too low a non-uniform deposit will be formed.

Copper can be chemically deposited in the same way as silver, using phenylhydrazine as the reducing agent. F. D. CHATTAWAY (1908) found this to give good results with blown glass but not with glass that had been polished. For plane surfaces E. A. H. FRENCH (1924) obtained good results with hydrazine sulphate.

Many metals can be deposited on glass in a vacuum, by vaporizing the metal and condensing it on the glass. J. YARWOOD (1955) has given a general account of this method.

Metallizing Preparations for the firing on of Metal Layers on Glass

Preparations of silver, platinum, gold and palladium are available which consist of a suspension or solution of the metal, or a compound, in an organic material; adhesion-promoting materials are also present. The cleaned glass is coated with the preparation by brushing, spraying or dipping. It is then heated fairly slowly to a high temperature (the firing temperature), when the organic materials are oxidized or volatilized and the metal becomes strongly bound

to the surface. The preparations mentioned here are all made by Johnson, Matthey & Co.; they also supply thinners for them.

A glass surface to be silvered by a firing-on process should be cleaned with soap and water or detergents. For metallizing with platinum, gold and palladium, improved results are sometimes obtained with glass which has been stored when, after cleaning, it is immersed in 5 per cent acetic acid (cold) for six hours. The special preparation is then applied. If necessary it is mixed before use with a suitable thinner. The preparation is allowed to dry; this may be accelerated by warm air. Dust should not be allowed to settle on the surface before firing.

After drying, the coated glass is put in a furnace below 100°C and then slowly raised to the firing temperature. The exact firing temperature is best found by experiment, but data provided by Johnson, Matthey & Co. give good guidance. A temperature 20°C below the softening point (Mg point) of the glass is often used. The furnace is kept at the firing temperature for about 20 minutes, and well ventilated so that the organic constituents burn away. With palladium, however, oxidation of the metal occurs at 400°C and the supply of air to the furnace should be restricted during the final stages of the firing operation. The furnace can be cooled at any rate which does not introduce permanent strain into the glass (p. 19). The cooling to the lower annealing temperature of the glass should therefore be at the same rate as that for annealing the glass, or at a lower rate.

Some further details concerning the particular metals are given below.

Silver

The silver pastes give a thickness of silver of about 25–30 microns after a single application and firing. Successive applications and firings can be made up to a certain limit. *Silver Paste Y.40* gives a thick and strongly adherent silver film. One application of *Silver Paste X.351* followed by one of *X.353* gives a thick, matt, silver deposit. Several other silver pastes are made by Johnson, Matthey & Co. *Silver Paste O.182* is for mounting quartz crystals. Their liquid burnish silvers are much less viscous than the pastes, and with one application give a silver film about 3 microns thick.

The fired-on silver films on glass can be soft-soldered. They should be 'tinned' with a solder containing some silver; ordinary soft solder can bring about complete dissolution of the silver film. *Matthey Ceramic Solder No. 1* is a suitable solder for the films. A resin-type flux should be used. Tinning can be carried out by dipping into the molten solder.

Platinum, Gold and Palladium

The films of these metals are much thinner than those of silver. A single application of *Liquid Bright Gold* (12 per cent), followed by firing, gives a gold layer of about 0·12 microns thick; one application of *Liquid Bright Platinum* gives a layer of about 0·08 microns; one application of *Liquid Matt Platinum* a layer of about 0·35 microns; and one application of *Platinum Paste* gives a layer of about 2 microns. These are too thin for soldering, except with considerable difficulty. Thicker films can be built up by successive applications and firings. The *Liquid Bright Platinum* preparations give brilliant mirror-like surfaces on glass; the films deposited by the *Liquid Bright Golds* are useful as optical reflectors and filters. Films of gold on glass can be prepared that reflect nearly all the infra-red radiation and transmit most of the visible spectrum.

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Chapter 5

THE MANIPULATION OF LARGE TUBING

General Observations

We apply the term large tubing to tubing with an external diameter of about 35 mm and greater. The problems encountered in working this size of tubing differ from those connected with the working of smaller tubes. These problems usually arise from either the greater weight of the tubing or from the greater heat losses from the site of working by conduction along the glass and by radiation from its surface. The weight poses no problem that practice in manipulation and the use of supports or rollers cannot overcome, but the increased heat losses cause the tube to become too hot to hold for a considerable distance along its length, and the radiation from the large mass of hot glass may prevent the tubing from being manipulated with unprotected hands.

The obvious solution to the above problems is to protect the hands with asbestos gloves, but even this has its disadvantages. Asbestos gloves prevent the same degree of manipulative skill as can be attained without them, which increases the difficulty of working the heavy tubing, and if the radiation is great the air inside the glove may become unbearably hot. For the amateur glass-blower it is frequently more convenient to clamp the tubing and carry out the work with hand torches.

Many operations with large tubing can be performed easily with a glass-working machine (Chapter 6). The following pages describe some methods for hand-working which are applicable to hard borosilicate glass tubes, e.g. *Pyrex* and *Phoenix*.

Bending Big Tubing

Method for the More Skilled Worker—Shrinking down a Short Portion

Bends cannot be made in tubing of greater diameter than 12 mm without blowing. Skilled workers can bend tubing up to about 25–30 mm diameter by heating it with rotation over a length equal to about twice the diameter, shrinking it over this length to about half

its diameter until the glass is beginning to sag, removing it from the flame, bending and blowing it out with one puff. The unskilled glass-blower has, however, to rely on heating and bending a short portion at a time.

Making Sharp Bends and Bending a Portion at a Time

A good method of producing the desired result is that of sealing two separate pieces of glass held at the required angle, or, in effect, making a T-seal at the end of a piece of tubing. This, in fact, is the only way, for the unskilled worker, of producing good sharp bends in tubing greater than 15–20 mm diameter.

The method of heating and bending a portion at a time will give a satisfactory bend whose radius, for the outside wall of the bend, is between five and six times that of the tube. The method has been used satisfactorily on tubes up to 50 mm diameter. The tube, closed at one end and with a blowing tube attached at the other, is clamped horizontally near one end, and heated, where the bend is required to start, over a length equal to about twice the tube diameter, with a large but not too hot flame from the hand torch. As the glass heats up it will begin to bend under its own weight, and the free end is supported until the glass reaches a dull red heat when it is allowed to fall a short way but prevented from sagging too far. The glass on the outside of the bend tends to flatten and it is heated fairly strongly with a hotter flame and blown out evenly. Kinks on the inside of the bend are heated even more strongly, first with a pin-point flame until they shrink in, and then with a larger flame which enables them to be blown out smoothly. The clamp is then rotated in its boss until the free end of the tube is again horizontal, and the procedure is repeated on the neighbouring portion and so on until the required bend is obtained.

With tubing up to about 30 mm in diameter the whole bend can be annealed by flame at the finish, but above this diameter it is advisable to anneal each portion for a few minutes after it is bent and shaped, and before one moves on to the next portion. With extra large tubing it is better to have two workers, one on either side of the tube, each with a hand torch.

Drawing Off Large Tubes

The need for a strong spindle is avoided if the end of the tube is to be blown out to make a joint or if it is to be drawn off by way of cutting the tube. In this case the tube may be drawn off under less well controlled conditions than if a strong spindle is required. It is possible to clamp the tube, preferably in a vertical position, and work

the glass with hand torches. It may be necessary for two persons to carry out this operation so that the lower part of the tube can be supported to prevent too rapid drawing off as it falls under gravity. The lower part of the tube will remain much cooler than if the glass were worked in a horizontal position.

Drawing large diameter tubing off to spindles can be accomplished by a simple extension of the methods previously described. It is almost essential to support the tube on a stand or set of rollers since the great weight of a relatively long length of tubing will prevent accurate rotation of the two pieces. It may also be found that the normal bench torch does not produce a hot enough flame for the purpose. If this is the case it is possible to arrange two hand torches in laboratory clamps so that the tubing is rotated between them. Then, if the tubing is not drawn down too rapidly, a strong spindle will be produced. Asbestos gloves may be necessary to protect the hands from the hot glass and the radiation if the tube is to be kept in the flame for long enough to collapse down sufficiently to form a strong spindle.

Cutting a length of tubing can be a useful initial stage in putting-in a strong spindle. This reduces an unwieldy length of heavy tubing to a more manageable size which can be held in one hand while a handle is put in preparatory to drawing off the end to a strong spindle. If an uneven end has been produced by faulty cutting and it is not intended to draw the tube off, the end may be tidied up considerably by local working with a hand torch and the use of forceps and scissors for removing excess glass. Any glass so worked should be annealed carefully to prevent cracking on cooling.

Closing Large Flasks

The necks of large flasks (about 5 litres capacity) are usually of thick glass, and they must be heated slowly. Before the top is closed a side arm must be put on the flask, and the neck is usually the best place for it. The mouth of the flask is closed with a rubber stopper wrapped in asbestos paper and carrying a blowing tube. A considerable area of the neck of the flask is slowly heated, using initially a large smoky flame and finally a hot flame. When the flame is tinged yellow a hole is blown in the side of the neck, of the right size to fit the side arm. The end of the side arm tube should previously have been thickened by heating, flanging and running down, otherwise a bad joint will be made because the thin, side arm glass runs so much more easily than the thick glass of the flask neck. The side arm is joined to the neck by the normal T-piece procedure, and the neck all round the joint is annealed carefully. The top of the neck is then heated and pushed in

with a spike until a handle can be attached as in FIGURE 32, *I*. The neck is pulled off near the top, giving a spindle which will probably be very thin (FIGURE 32, *II*). This is removed and the end rounded by running down and blowing out a number of times—to blow out all at once requires a very large flame, and the end must be blown out piece by piece with all ordinary flames.

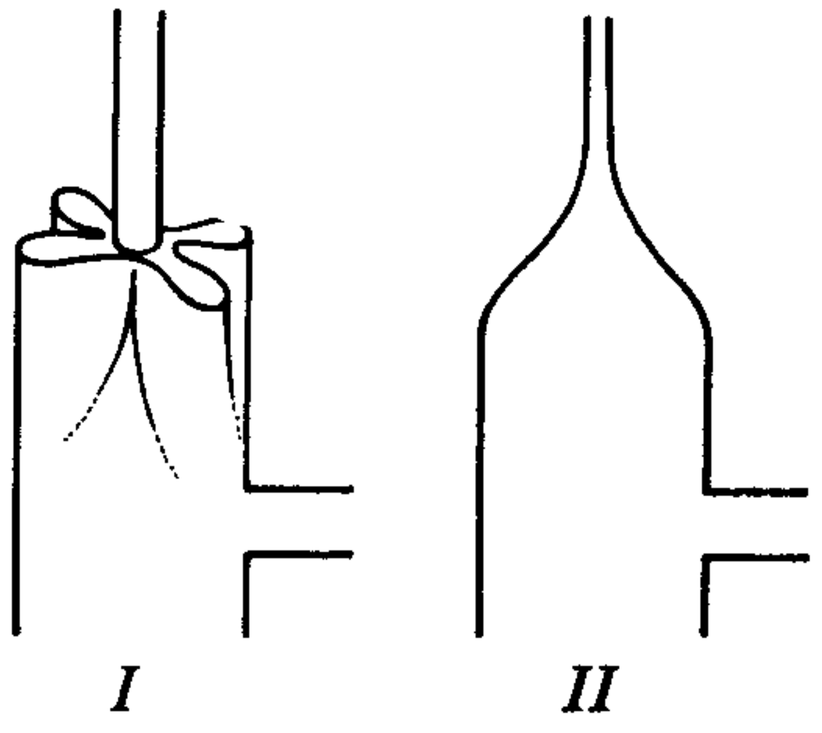

FIGURE 32. Closing the neck of a large flask

Joining Big Tubes

Once again the methods previously described for joining two tubes are applicable provided the limitations imposed by the size of the tubing are borne in mind. Joining two large diameter (say 4 cm) tubes in the flame of a bench torch follows the same course as joining smaller tubes by the same method. Care must be taken to see that the tubes are long enough not to become too hot to hold. Tubing of 4 cm diameter may become too hot to hold for about ten inches on either side of the site of working, since not only is more heat conducted along the glass but also a much larger flame is necessary.

Working large diameter tubing clamped is very often more convenient than attempting to work it with a bench torch. Joining two large diameter tubes by this method follows the same pattern as the joining of smaller tubes in similar circumstances: first an adequate supply of tools and thin glass rod to meet any emergency is collected. Then the two tubes should be prepared by blowing out the ends. It is best to blow holes of only about three quarters of the diameter of the tube (FIGURE 33) as the ends of the drawn-off tubes are liable to have rather thin walls and this allows them to be thickened as the joint is made. The tubes are heated and pushed together as before, or, if this is not possible, the flanges of glass left by blowing out the ends are heated and pushed together with a spike, or the gap is filled with glass

rod. While this is being done it is necessary to heat the joint more or less evenly and not just on one side. If this is difficult two operators, each with a hand torch, may be found an advantage.

All other operations—the working of the glass by alternate collapsing and blowing, sealing pinholes, and finally blowing the joint to an acceptable shape—are the same as previously described. The flow of glass under gravity will be more noticeable when large tubes are joined, principally because the time required to make the joint is greater. This flow can be combated as before.

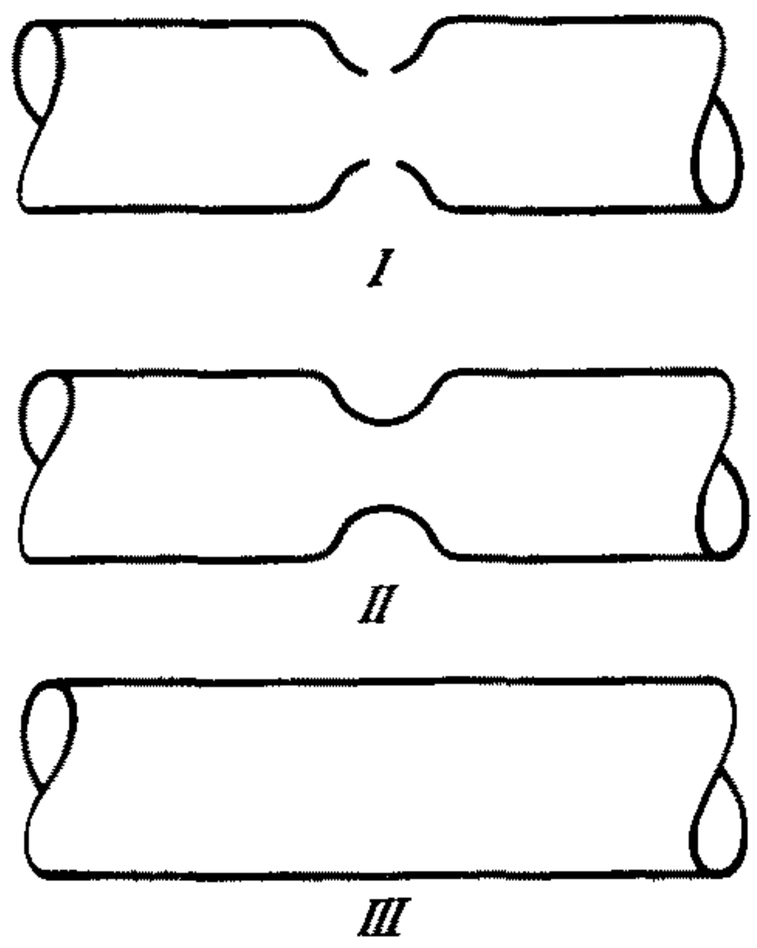

FIGURE 33. Joining large tubing

The final annealing of the joint and adjacent tubing is of the utmost importance with large tubes. If this is neglected there is a high probability that the joint will crack as a result of the strains induced by working and cooling the glass.

Big T-Joints

Large sized T-joints can be made using a bench torch and manipulating the glass in precisely the same way as previously described. This is difficult since the whole weight of the glass must be carried by the hands, because the way in which the tubing must be turned in the flame precludes the use of supports. The simplest way of making these joints is to clamp both tubes in such a way that they can be pushed together at the required time.

The two parts should be prepared by blowing a hole in the end of the side arm smaller than the bore of the tube and then blowing a similar sized hole in the cross-piece. These parts should next be clamped so that the tubes are nearly touching and can readily be

pushed together. The two tubes are then heated as uniformly as possible around the join, and when the glass has become soft the two tubes are pushed together. If they do not make contact at any point it may be possible to push the glass in this region together with a spike or else to bridge the gap with thin rod. The joint is then worked, by the usual method, to fuse the two tubes together. When the joint is blown out to its final form it is advisable to heat a fairly large segment of it at a time; and blowing the glass carefully while it is in the flame very often leads to a better finish. Mending any pinholes in the joint is much more easily accomplished at this stage and therefore a careful inspection should be made before the final annealing, which should be carried out thoroughly. For work on very large diameter tubing it may be necessary to use two hand torches for the above operations.

Very often it is found more convenient to replace a right-angle bend by a T-joint made right at the end of the drawn-off cross-piece. This makes a very neat right-angle join in tubing which has too large a diameter to bend easily.

Big Internal Seals

Big internal seals can be made by any of the methods described in the previous chapter although by far the easiest method is to work with the glass clamped and to keep the internal tube in one piece.

Preparation of the materials is one of the most important phases of the operation, since once the glass-blowing is started it is virtually impossible to leave the work until it is finished and annealed thoroughly. The inner tube should be prepared as before with a slight ridge round the tube at the position of the joint. The outer tube should then be closed off to a round end and the hole to take the internal tube blown. This should be left with a slight rim of glass and not flame-polished. Before this can cool the internal tube should be inserted and clamped in position and work started on the joint. If the glass is not in good contact initially it may be heated and pushed on to the internal tube with a spike. Care should be taken in so doing to see that the flame is not concentrated too much on one side of the partly made joint, allowing the other side to cool and crack—one of the dangers of pushing the glass in with a tool. The attention is concentrated on the localized area being worked and the rest of the join is allowed to cool, thus straining the glass excessively. The localized working itself tends to strain the joint, but if care is taken to see that the whole joint is kept hot the strain is dissipated by a continuous annealing process.

Once the two tubes have been pushed into close contact and the

joint is air-tight it can be finished as previously described. These internal seals require very thorough annealing and it is best to heat them to a dull red heat for a short while before cooling slowly in the flame. The use of two torches facilitates uniform heating during the annealing process, and since no other operations are necessary one can be held in each hand if they are both prepared before the commencement of the glass-blowing. This use of two flames is probably unnecessary except for very large tubing.

Mending Cracks in Large Tubing

When large tubing has been worked and has been annealed in a flame it is usually strained to a considerable extent, which sometimes causes it to crack. Mending these cracks without spreading them is an operation requiring a considerable amount of patience. It is one of the cases in which it is necessary 'to make haste slowly'. The use of two flames is an advantage as they allow the heating up of both ends of the crack at the same time. The glass should initially be warmed uniformly and slowly with small cool flames. When it has started to heat up, the rate of heating is increased, concentrating the flame slightly on the ends of the crack so that these parts warm up just a little faster than the rest of the glass. Once the glass has heated up sufficiently to flow slowly the crack is allowed to seal itself, using only a cool flame as previously described. Finally, when a complete repair has been effected, the glass is annealed.

Mending Cracks near Complex and Large Seals

The above remarks apply equally here, except that extreme care is needed to prevent the crack spreading into the complex region. If the crack spreads to an internal seal and the inner tube cracks, it will be virtually irreparable. This spreading can be prevented if the whole of the complex joint is heated at the same time and at the same rate as the glass around the crack. The heating must be slow to allow time for the inner parts of the joint to be heated adequately by conduction.

Chapter 6

SOME OPERATIONS WITH A GLASS-WORKING MACHINE

WE have mentioned (p. 2) the difficulty of holding and rotating a piece of glass tube in each hand and operating on a semi-molten mass between the two pieces. A very satisfactory way of resolving this problem is to use a machine—the *Edwards Model G 3 Glass-Working Machine* (made by Edwards High Vacuum Ltd) is of great value. The glass to be worked is held in two chucks which grip the tubing and rotate it synchronously at a speed which is variable over a considerable range (120–500 rev/min). One of the chucks can be moved towards or away from the other. The glass may also be held in only one chuck.

The glass can be worked both by blowing into it as it rotates and by the use of tools which can be mounted in various ways or held in the hand. Thus operations involving the production of articles with circular symmetry can be carried out with great ease, and a skilled operator is by no means necessary. The advantages of using this machine, when possible, for work involving tubing exceeding 3–4 cm in diameter are, we think, particularly manifest. The operation of putting a spindle in a large tube, for example, is done by hand only with difficulty, whereas with the machine it can be done rapidly and effectively. In this Chapter we attempt to give merely a general idea of how such a machine can be used; the detailed manipulations involved soon become clear after a little practice. The machine is valuable either for the production of special parts in research and development work or for continuous operation on repetition work by unskilled personnel.

General Description

Two views of the glass-working machine are shown in FIGURES 34 and 35. It consists of a substantial cast base fitted with a fixed headstock and a movable tailstock. This can be moved towards or away from the headstock by operating the tailstock traverse control. Rotating mandrels in the headstock and tailstock are synchronously driven; the tailstock mandrel drive can be disconnected and locked

in position so that the mandrel does not rotate, and it can then be employed as a tool holder for tools used to work single lengths of tubing held in the headstock chuck. The tool can be moved towards or away from the glass using the tailstock traverse as usual.

In the front of the machine there is a burner mounting bar which normally carries either a cannon burner, operating with gas and air or gas–air–oxygen mixtures, or a crossfire burner, operating with gas–oxygen mixtures. Both burners can be used together and their flames directed on the same region. The burner mounting bar has a

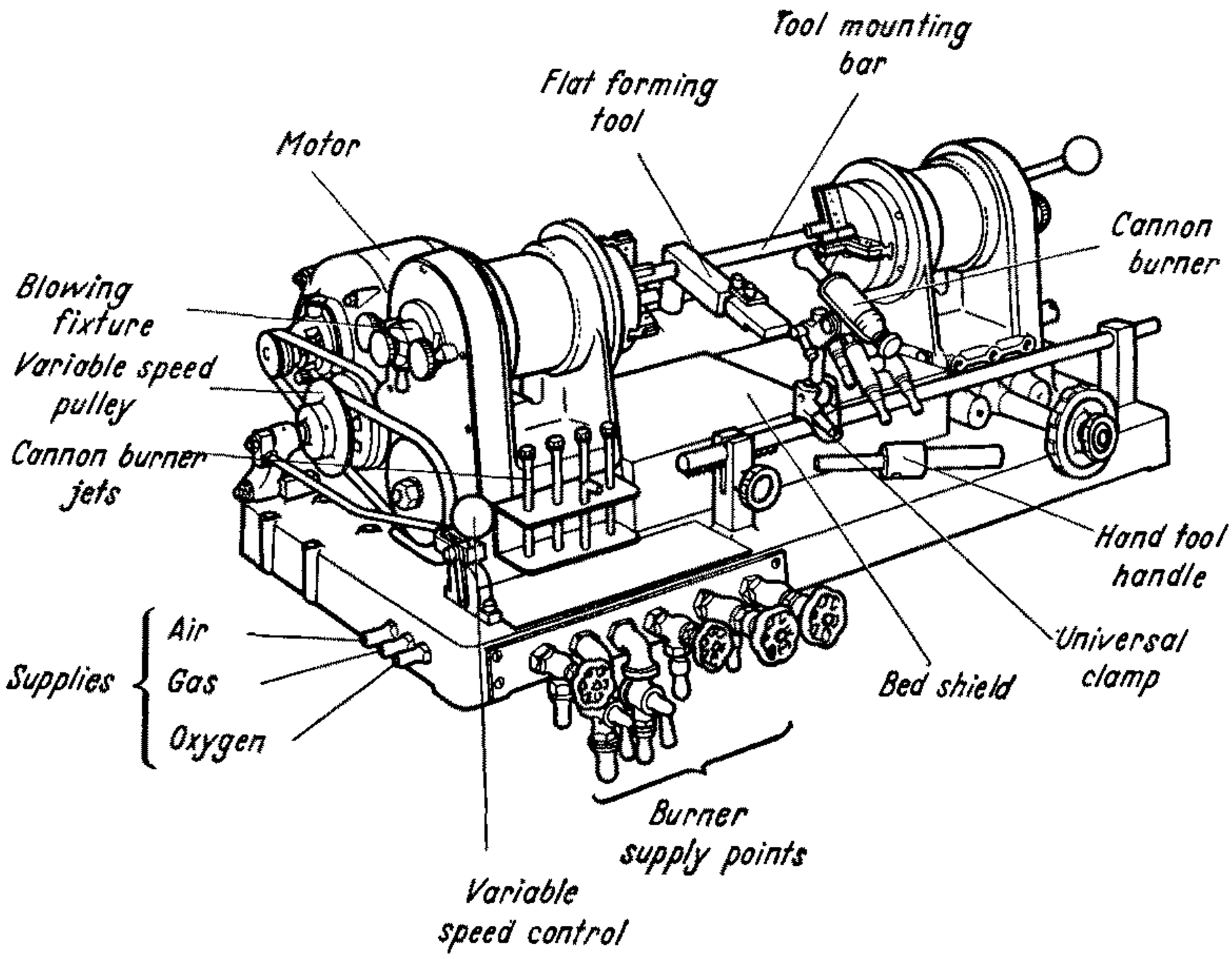

FIGURE 34. A glass-working machine

rack-and-pinion mechanism so that the burner or burners can be traversed a limited distance along the glass and be made to follow up the work.

At the rear of the machine there is a tool mounting bar. A flat forming tool can be fixed to this, as shown in FIGURE 34; and by using the operating lever (FIGURE 35), the tool is brought into contact with the lower side of the rotating tubing, which, softened in a flame, is pushed into shape. A socket for a cone-and-socket joint is easily made by setting up the flat forming tool at the proper angle. The tool mounting bar is fitted with an adjustable mechanical stop which

is also an electrical contact for giving visual indication on a lamp when pre-set limits are reached. This arrangement is useful for repetition work. Generally, when constructing specialized apparatus for research work, operations with tools are performed by holding them in the tailstock chuck (not rotating), or in the hand tool holder (FIGURE 34), which is held in the hand.

The mandrel bore size is 32 mm, so that tubing of this size will pass right through the headstock and tailstock. The maximum jaw opening is 63 mm with the jaws in their normal position; but they are

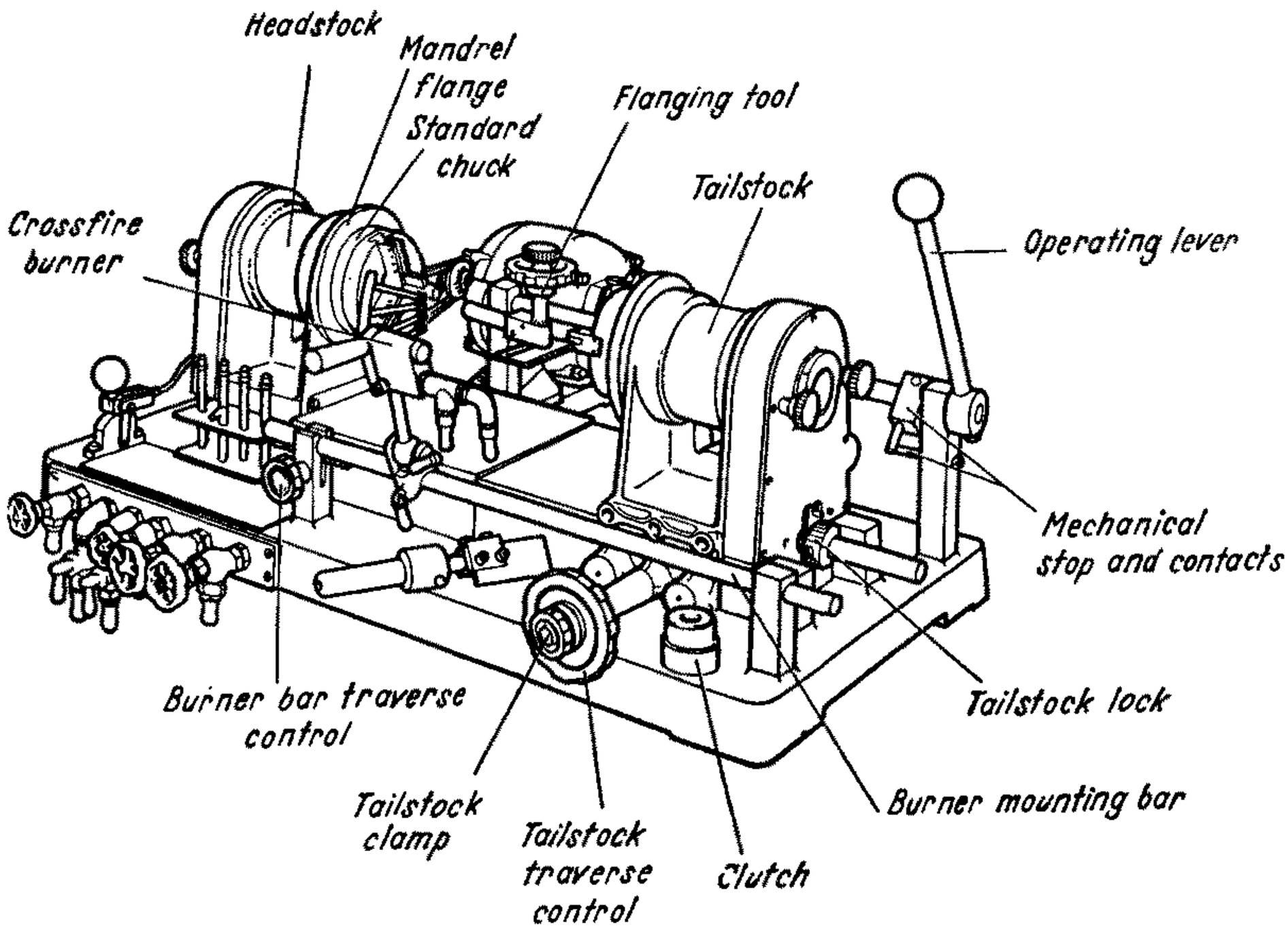

FIGURE 35. Another view of the glass-working machine

reversible, and by reversing them tubing up to 115 mm diameter may be held.

There are various ways of blowing into the rotating work. If the tube, or a portion of it, is long enough and less than 32 mm diameter it may be passed right through the headstock and blown into using a glass-blower's swivel (p. 42). Otherwise spindles or rubber stoppers carrying glass tubes may be used in conjunction with a swivel. The machine itself can be used to put a short spindle in a tube, using, in principle, the method described on p. 56. A blowing fixture can also be fitted to the headstock or tailstock mandrel (see FIGURE 34); this

remains stationary while the work rotates, and so it is only necessary to prepare a sufficiently air-tight joint between the tubing to be used and the mandrel with the help of tape or rubber stoppers, or stoppers and tape together.

Sketch of Some Possible Operations

A tube held in the headstock chuck can be flared by any of the methods previously described (p. 72). Flares can be made very easily with a cylindrical carbon rod attached to a special holder fitting into the tailstock chuck, which is not rotated during the operation. The flare is made by pushing the carbon rod, held at the desired angle, into the end of the rotating and heated glass tube, for which purpose the tailstock traverse is used. With this method the end of the tube can be opened out until it resembles a funnel.

To put a spindle in a tube (p. 57), both ends are held in the rotating chucks. It may be desirable to straighten the tube, by heating it in a gas–air flame as it rotates, if it is so bent that it is not held well in both chucks. A spindle is put into the tube by the method described on p. 56, except that the operator's hands are replaced by the two chucks. The length of the spindle which can be obtained is limited by the length of the tailstock traverse.

A tube with a spindle in it can be worked to a tube with a round end by removing the spindle just beyond the shoulder (see p. 57) with a small flame, and then shrinking down and blowing out the end. The blowing can be done while the glass is in the flame. A hole can be blown in the end of the closed tube by heating the extreme end in a small flame. The hole can then be opened out with a tool, using an ordinary flaring method. Two tubes with holes of the same size in their ends can be joined very easily by bringing their hot ends together as they are both rotated, one tube being in each chuck. The joint is finished as described on p. 61. To join big tubes it may be convenient to grind the two ends flat; no holes need then be blown, since the tubes will meet well enough.

To blow a bulb in the middle of a tube, it is held in both chucks; glass is accumulated by pushing the tailstock to the headstock as the glass rotates, and the tube is blown into gently at the same time. The bulb is blown as the glass accumulates; an elegant bulb can be obtained very easily.

An internal seal can be made by Method 1 of p. 73. The hole in the outer tube and the ridge or bulge on the inner tube are both put in on the machine. The ridge can be put in by heating a small portion of the tube as it rotates in both chucks, and quickly pushing one end towards the other with the tailstock traverse. Alternatively, a small

bulge can be blown until it is the required size. This is conveniently done with the help of a calliper gauge. When making the final internal seal, the inner tube of the seal can be supported in the outer tube with corrugated cardboard. An internal seal can also be made by Method 4 of p. 76. The correct size flange or flare is put on the inner tube with a tool, and a calliper gauge is again used to check the size of the hot glass.

A *Dewar* seal (p. 122) can be made without blowing by flaring the smaller tube outwards until it will nicely meet the larger tube (as in FIGURE 50, *III*). The smaller tube is mounted in the larger tube with the help of corrugated cardboard, and the final joint is made by pushing the flare of the inner tube on to the outer tube with a tool as the glass rotates in the flame. If the tubes are first ground flat at the ends to be sealed, an elegant joint can be obtained very easily.

Graded seals must be used for joining together glasses of such different thermal expansions that a direct seal cannot be made (p. 25). The best way of making a graded seal with the machine is to use the various intermediate glasses as rod. The first piece of glass tubing is held in the headstock chuck, and the first intermediate glass is held in the tailstock chuck; both are rotated. The end of the tube is closed with the rod, which is then drawn off just beyond the closure. The closed tube remaining then has its end worked to a rounded shape. The next glass is attached as a globule on the extremity of the hemispherical end and is worked to another hemispherical end; the process is repeated with the next intermediate glass, and so on. The final tube is joined as a tube.

A graded seal can also be made by fusing together rings of the intermediate glasses, or by fusing on each intermediate glass in the form of tubing, then drawing a spindle, making a round end, and blowing a hole.

The making of glass bellows is an operation which is very greatly facilitated by the machine. The glass is rotated in a small hot flame and blown out to give a thin protruding portion of glass. More of these thin portions are then introduced. Practice is necessary to make bellows with the required degree of flexibility. The flexibility of course increases with the number of thin portions of glass; the fragility likewise increases.

Tapering can be carried out by the methods described on p. 79. When a precise angle is required the flat forming tool on the tool mounting bar (FIGURE 34) can be used.

Chapter 7

METAL–TO–GLASS SEALS

ALTHOUGH the sealing of noble metals through glass has been known for a great many years, even centuries, the extensive use of metal–to–glass seals in laboratories has developed only in recent years. This recent development has, however, been extremely rapid and a great deal of detailed literature now exists. An excellent collation of much of the data has been made by J. H. PARTRIDGE (1949) in his monograph devoted entirely to glass–to–metal seals. We, in this Chapter, attempt to supply the reader with information and detail sufficient to be useful to the average laboratory experimentalist.

Matched and Mismatched Seals

Where a direct fusion of glass–to–metal or metal oxide is obtained (sometimes termed the "wetting" of metal by glass) two types of seal can result—one matched, the other mismatched, depending on how the relative coefficients of thermal expansion of the glass and the metal compare.

In a *matched seal* the stress is kept within safe limits by close correspondence of the two thermal expansions throughout the temperature range over which the seal has to be worked during its manufacture.

A *mismatched seal* is one in which the glass and metal have differing coefficients of expansion. Here the resultant stress is reduced to within the limits required to avoid fracture by special shaping of the metal at the junction. In some cases relief to a small extent is obtained by the use of a ductile metal which gives slightly, so reducing the stress in the glass.

When it is required to seal a metal into a glass with which its coefficient of thermal expansion is greatly mismatched, graded seals are often employed from the given glass, through one or more intermediate glasses, to a glass which will form a matched seal with the given metal. *Kovar*–to–*Pyrex* seals (see below) are a good example of this type.

A further combined characteristic that a glass and a metal must have before they will form a seal, over and above that of the provision

by some property or other for the relief of stress in the glass, is that the glass must wet and adhere to the metal or its oxide.

Another important consideration that must be given when employing a matched seal in the laboratory is the temperature range to be encountered during use of the seal. The absence of stress relies on the matching of thermal expansions throughout the temperature range encountered when the seal is worked, and this matching must continue over the temperature range in which the seal is to be used, else fracture may result. This factor must be watched when, for instance, the metal–to–glass seal is to be immersed in, or is near to, refrigerants.

Sealing Tungsten into Pyrex

Pyrex (*Corning No. 7740*) adheres well to tungsten oxides and will give a vacuum-tight seal with this metal provided the joint is made carefully. The seal is mismatched and an upper limit to the diameter of tungsten rod that will give a satisfactory joint is 0·5–1 mm. The exact figure varies; the most important factor is probably the severity of the service requirements.

For vacuum work tungsten rod is superior to wire because its method of manufacture leaves the metal with less surface cavities and less fine channels running within it. Centreless-ground tungsten rod is advised. The rod is first beaded, and for this a small length of well-fitting, thin-walled, *Pyrex* capillary is drawn; a piece about 1 cm long and approximately 0·5 mm wall thickness is required. The portion of the tungsten rod to be beaded is cleaned by heating to dull red heat and bringing it immediately into contact with crystalline potassium or sodium nitrite, either in the form of a stick or as a small heap of powder balanced on a spatula. A strongly exothermic reaction occurs during which the metal continues to glow red hot and is rapidly dissolved. The reaction is allowed to proceed for a few seconds, then the rod is washed free of the nitrite and wiped dry. The surface of the metal is left clean. It is then reoxidized slightly in a just non-luminous flame until it appears coloured a second order green or blue.

The piece of glass capillary is immediately slipped over the prepared portion of the rod (FIGURE 36, *I*) and heated progressively from one end to the other. The rod must not be allowed to cool before this is done or the oxide layer may break away from the surface of the metal due to differing thermal expansion coefficients. As the glass shrinks on to the metal and adheres to it the air is slowly driven out ahead of the shrinking portion (FIGURE 36, *II*). The metal, when in contact with the glass, appears larger in diameter and assumes a reddish colour so that the progress of the beading is easily observed.

The finished bead, shown in FIGURE 36, *III*, will, when made correctly, be coloured something between a straw yellow and a reddish tan. If desired, the bead may be enlarged at one end or in the middle to assist in fusing it into a small bore tube as indicated in FIGURE 36,

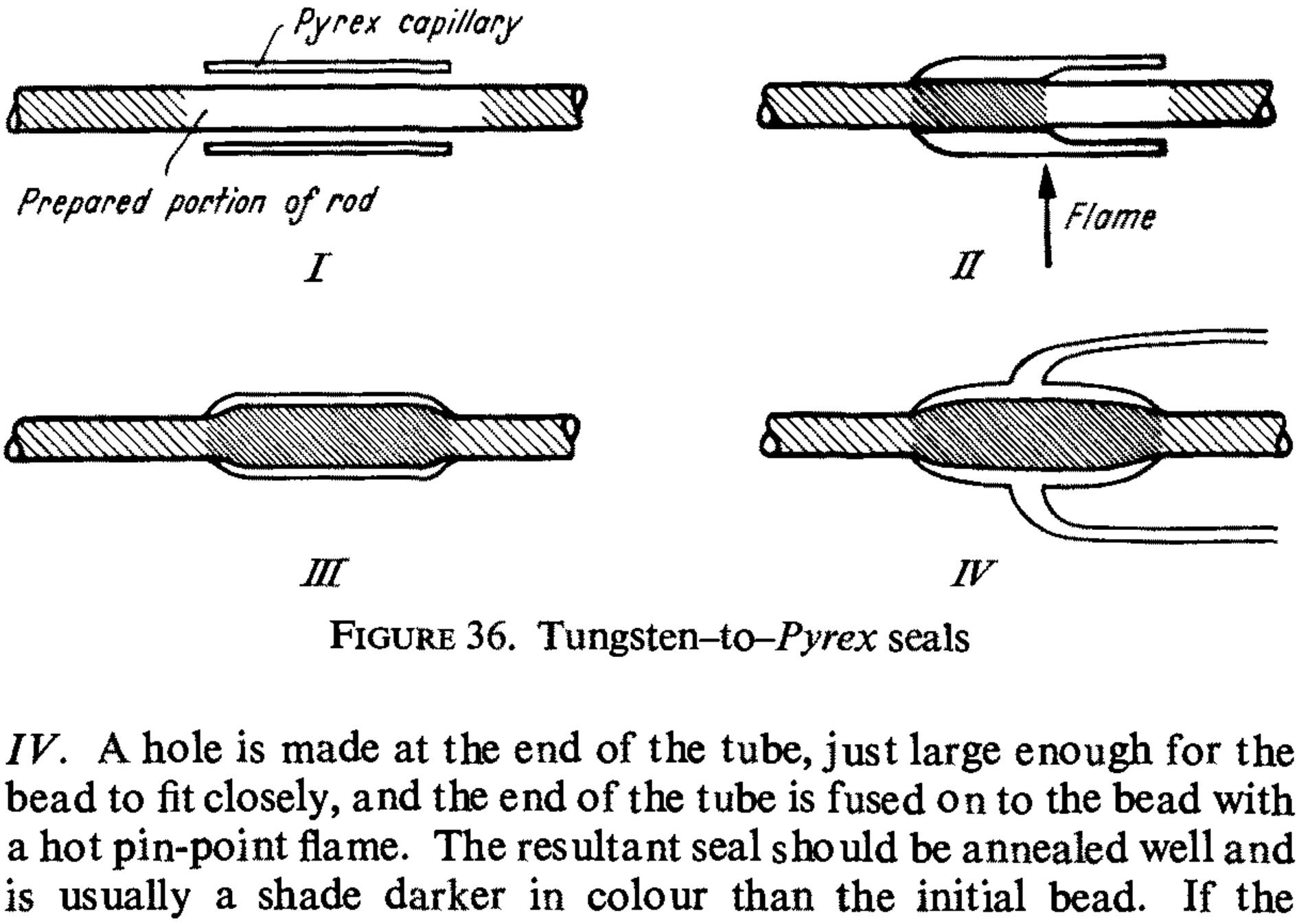

FIGURE 36. Tungsten–to–*Pyrex* seals

IV. A hole is made at the end of the tube, just large enough for the bead to fit closely, and the end of the tube is fused on to the bead with a hot pin-point flame. The resultant seal should be annealed well and is usually a shade darker in colour than the initial bead. If the tungsten wire or rod has to be cut to length, this is best done by grinding or dissolving in molten potassium or sodium nitrite. Any attempt to cut tungsten results in the metal being torn and in the opening of fine channels which may cause leaks.

Wire Holders

The tungsten rod during the operations described above can, when of sufficient length, be held in the fingers; but with short pieces of rod

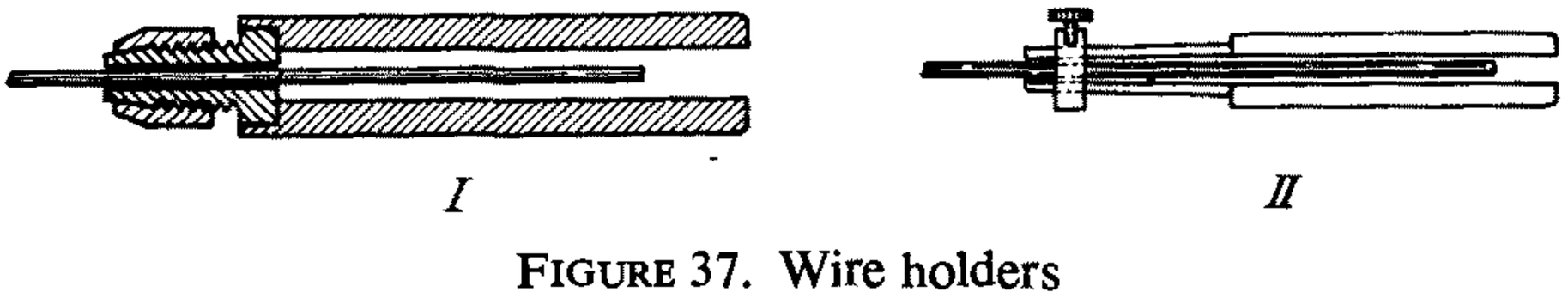

FIGURE 37. Wire holders

some method of gripping is necessary. An ordinary pair of workshop pliers will suffice, but if the metal–to–glass seals are made frequently it is convenient to have at hand some form of small wire holder. A useful tool is seen in FIGURE 37, *I*. A small metal collet head, for

gripping the wire, is set into a hollow handle—preferably of wood. Another type of holder, simpler to make, is shown in FIGURE 37, *II*. With wire holders such as these a very even rotation of the wire during beading is possible.

Sealing Tungsten into Special Glasses

With tungsten rods of diameter 0·5–1 mm it is desirable to use special sealing glasses for vacuum work, while with rods larger than 1 mm diameter this becomes a necessity. *Nonex* (*Corning 7720*), uranium glass (*Corning 3320*), *G.E.C. W.1*, *B.T.H. C9*, *Dial 36* and *B.T.H. C14*, all form a more matched seal with tungsten than does *Pyrex* (*Corning 7740*). *Phoenix* glass, which has an expansion coefficient almost the same as that of *Pyrex* to which it fuses excellently, seals more

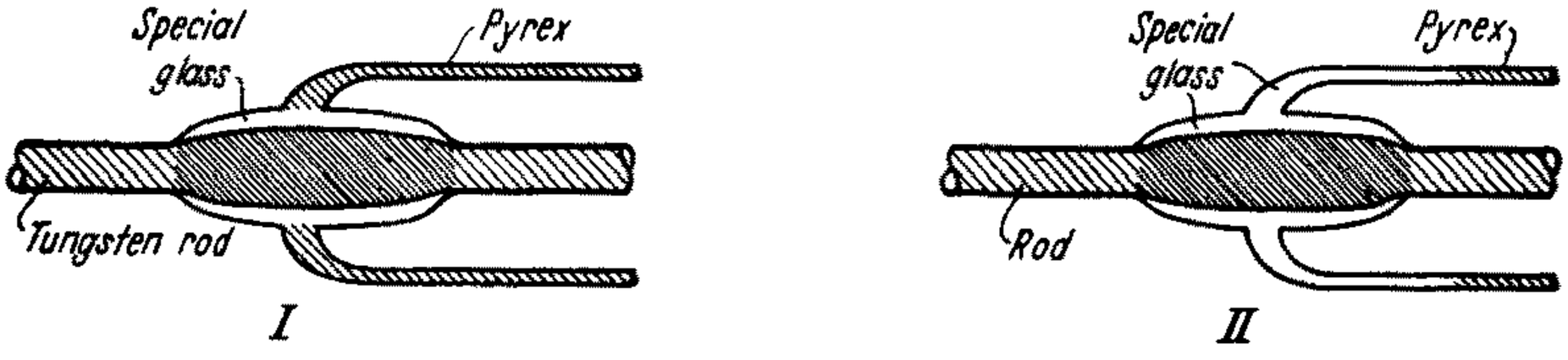

FIGURE 38. Tungsten–to–*Pyrex* seals with sealing glasses. *Phoenix* can always replace *Pyrex* in these seals

satisfactorily to tungsten than does *Pyrex*. It is therefore very useful as an intermediate glass for *Pyrex*–to–tungsten seals with rod up to about 1·25 mm diameter, and also in making pinch seals.

Of the above special glasses we find that *W.1*, *C9* and *Phoenix* are easily worked and give very satisfactory seals. Very great care is necessary when using *Nonex* to prevent the lead in the glass from reducing with resultant blackening; the tip of an oxidizing flame must be used. The characteristics of sealing glasses are given in Chapter 2. Those mentioned above seal well to *Pyrex*, though with *Nonex* uranium glass is sometimes used as an intermediate.

When making a seal the tungsten rod is first beaded as with *Pyrex*. The bead of the special glass may either be fused directly into *Pyrex* (FIGURE 38, *I*) or first fused into a tube of the same glass and this tube cut near to the seal and fused to a *Pyrex* tube (FIGURE 38, *II*).

Multiple Wire Seals

When a number of mutually insulated wires have to be carried into a piece of apparatus through one glass tube, a multiple wire seal is necessary. There are several methods for achieving this end, and the one to use depends upon the particular purpose and circumstances.

Multiple Wire Seals when Special Sealing Glasses are not available

One way of making a multiple seal is indicated in FIGURE 39, *II*. This method is extremely useful when it is required to seal several tungsten rods or wires through *Pyrex*, and special sealing glasses are not available. Each of the wires is first beaded with *Pyrex* as described above. One end of the tube, into which it is desired to seal the wires, is rounded off and the smaller tubes are added one at a time. Tubing of 3–5 mm bore is used for these separate sheaths. Using a hand torch, and employing a fine pin-point flame, it will be found with practice that making a number of small joints, very close together in this manner, is quite straightforward. The central sheath should be added

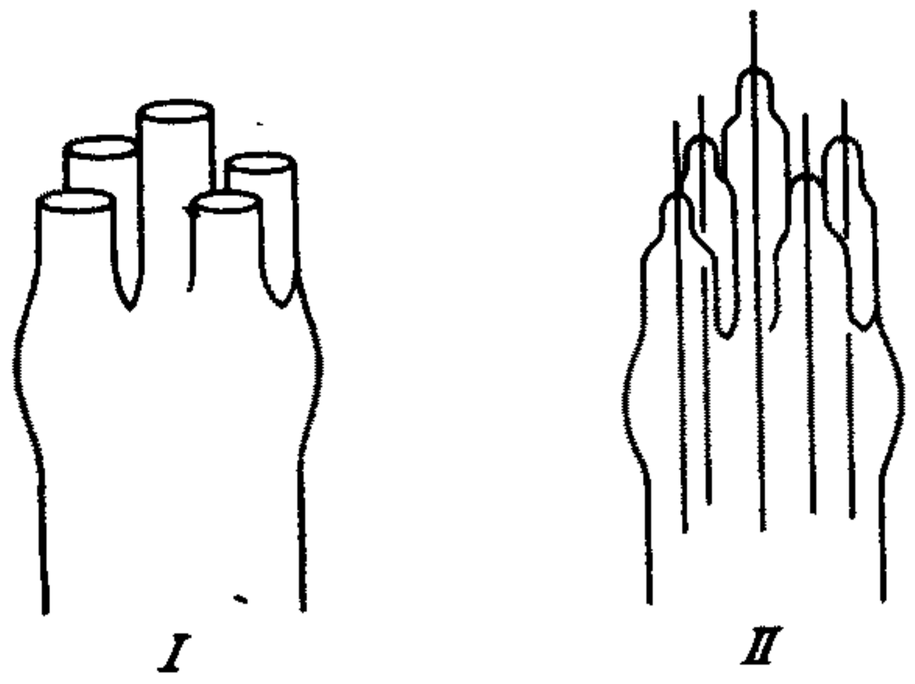

FIGURE 39. Multiple wire seals

first and the remainder positioned around it. As each tube is sealed in position it can be truncated to the required length as shown in FIGURE 39, *I* and closed with a small expendable cork.

When the multiple sheath is completed each cork is removed in turn and replaced by a beaded rod which is fused into place after careful scraping away of any burnt cork. The finished work should be well annealed. We find the method described here to be very serviceable and useful even when beads of a special sealing glass can be employed.

Pinch Seals

A higher degree of stress exists in pinch seals than when the wires are given separate sheaths. Direct *Pyrex*–to–tungsten pinch seals, therefore, are not satisfactory. However, serviceable multiple pinch seals can be made with most of the special glasses mentioned above. When using *Phoenix* glass a special sealing glass for beading the wires is not necessary.

The end of the *Pyrex* tube that is to carry the wires is heated and shaped with tongs as shown in FIGURE 40, *I* or *II*. The desired number

of wires, beaded with one or other sealing glass, is held in a clamp of the form shown in FIGURE 40, *III*, and the latter is supported so that the wires are introduced to the prepared tube with the beads enclosed in the pinched section. The whole of the prepared portion of the tube is then heated strongly and evenly and the glass pinched hard on to the beads with a pair of tongs. The initial beads should be prepared large enough for neighbouring ones to be touching when the wires are clamped in position; this ensures that sufficient of the special sealing glass is present. During the working of the pinch seal all trapped air must be carefully squeezed out. With most glasses flame annealing is adequate.

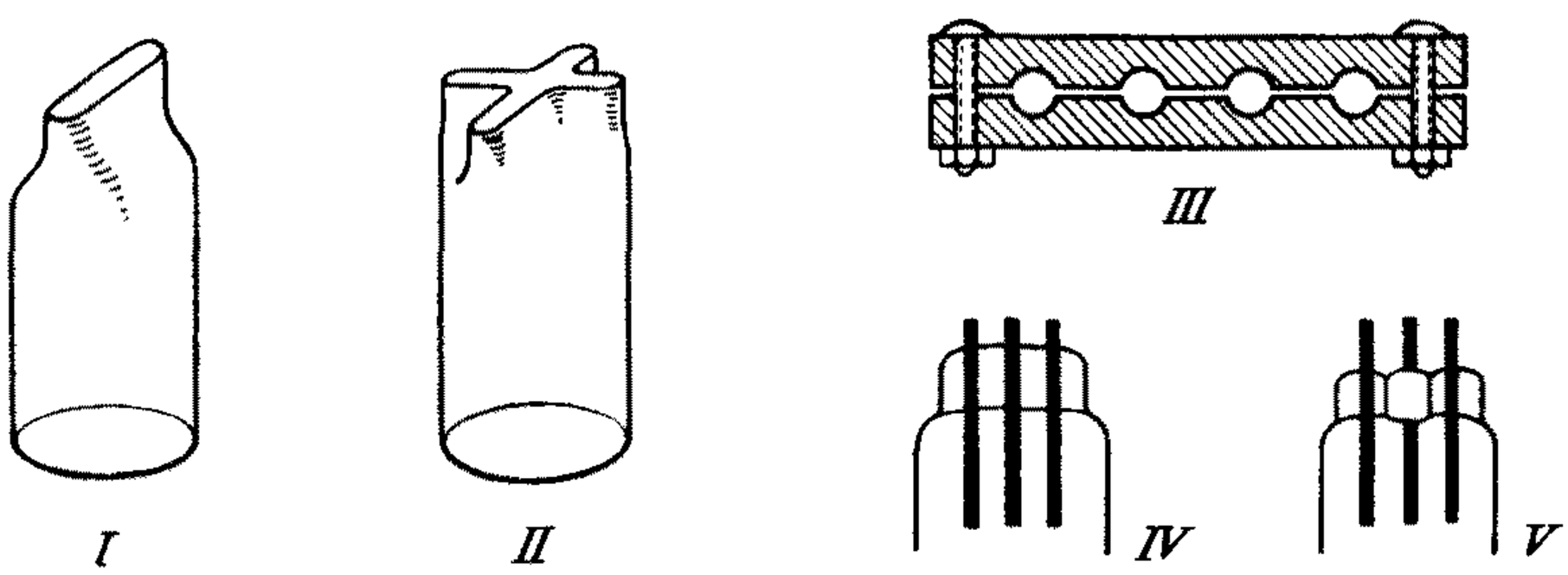

FIGURE 40. Pinch seals

Finished multiple seals of this type are shown in FIGURE 40, *IV* and *V*. The outer tube may be sealed directly to the apparatus for which it is required, or a *Dewar* seal may first be formed as shown in FIGURE 50, *VII*, making the multiple seal re-entrant. The re-entrant form is very useful when, for example, the wires are directly attached to, and are supporting, electrodes (see p. 157).

Prepared Copper–Tungsten–Nickel Wires

Long lengths of tungsten rod beyond a metal–to–glass seal are often inconvenient because the metal is so brittle and will not give. Any lateral strain at the end of a long length of rod or wire can easily fracture the metal–to–glass seal. For this reason it is often desirable to use prepared copper–tungsten–nickel wires. These are obtainable, or can be made by butt-welding braided copper at one end of a short piece of tungsten and nickel at the other. Nickel also spot-welds well to tungsten.

These prepared wires are very useful for sealing through *Pyrex* and *Phoenix*. Care must be taken to see that potassium or sodium nitrite does not come into contact with the metal junctions or the tungsten

will not be left clean. When beading the tungsten the glass capillary must be kept short, so that the glass comes only into contact with the tungsten portion and does not overlap either junction, otherwise fracture will result.

Nickel Tipping of Tungsten

In the prepared wires described above, the copper and nickel butt-welds at either end of the tungsten serve to seal up any fine channels that might be present in this metal and to prevent leaks. If the tungsten rod or wire is to be used alone it is sometimes desirable to seal the ends by welding or fusing a coating of nickel over the tip. A fine

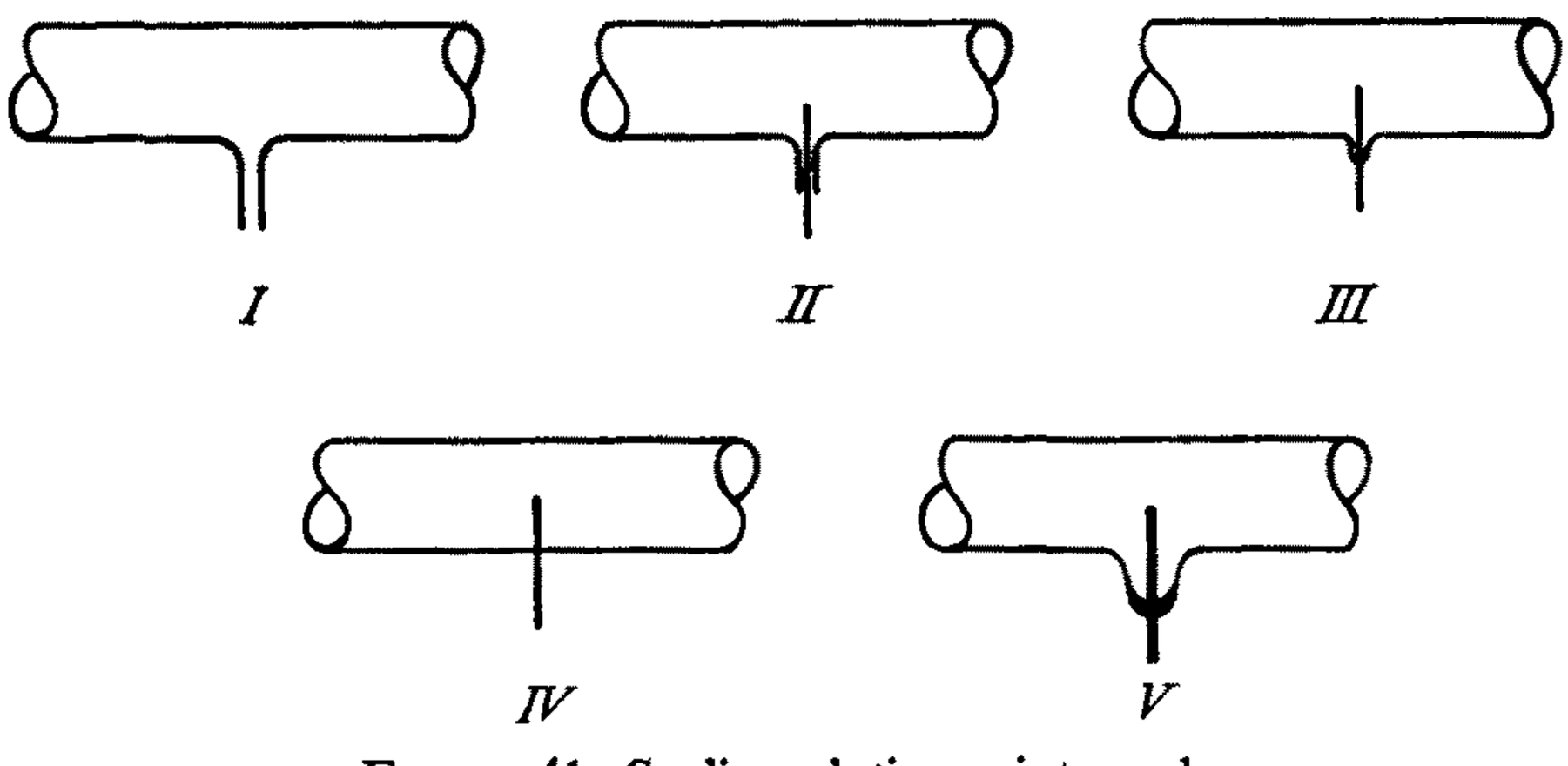

FIGURE 41. Sealing platinum into soda

channel is much more likely in tungsten wire than tungsten rod. With rod the nickel tipping is generally not required.

Sealing Platinum into Soda or Lead Glass

Platinum has a thermal expansion coefficient of 9×10^{-6}/°C, and therefore seals easily into a number of soft glasses. Fine platinum wire, of diameter less than about 0·25 mm, can be fused directly into soda glass to give a vacuum-tight seal. If wire above that diameter is to be used and the apparatus is required to hold a vacuum, then an intermediate lead glass that will fuse to soft glass should be used; alternatively *Dial 444* may be used as a sealing glass. Platinum oxidizes only slightly and thus no great care is needed when fusing glass to it.

When a wire is to be fused into the wall of a tube without employing an intermediate glass a small tail is first pulled out (FIGURE 41, *I*). This is done by heating with a pin-point flame, touching the tiny hot portion with the tip of a glass rod (or with the platinum wire itself) and drawing out a fine spindle. The spindle is broken off about 2 mm

from the wall and should at this point be of diameter just sufficient to accommodate the wire (FIGURE 41, *II*). The wire is inserted, the fine tail is collapsed and fused on to the platinum to form a small bead *in situ*, which is heated strongly so that it shrinks back into line with the walls of the tube (FIGURE 41, *III* and *IV*). In this manner the seal is made without any appreciable thickening of the glass around the platinum. When the seal is finished the whole circumference of the tube should be heated at this point and well annealed.

Use of an Intermediate Glass

If an intermediate lead or sealing glass is used then the wire is beaded separately and a slightly larger tail is drawn out—sufficient for the whole bead to be inserted. The tail is prepared first so that the hot bead can be inserted and fused into place without cooling. The seal is otherwise finished as above; care being taken not to form a lump in the wall of the tube around the wire. When platinum wire of diameter greater than about 0·5 mm is used it is preferable to add to the larger tube a small T-piece as a sheath for the beaded wire (FIGURE 41, *V*), as described above for tungsten–to–*Pyrex* seals.

Sealing Platinum into Pyrex

No satisfactory, single, intermediate glass for sealing platinum metal to *Pyrex* glass is readily available. Very fine platinum wire and very thin-walled platinum tubing can, however, be satisfactorily sealed directly to *Pyrex*, but such seals are not usually serviceable for vacuum requirements.

Light Platinum Tube

With thin-walled platinum tubing, of wall thickness up to 0·15 mm, the glass is fused around the outside of the tube for a length of 5–15 mm; slight blowing may be necessary. The metal in this form will give sufficiently to take up the stress caused by differing thermal expansions. If used for current carrying, the capacity of the platinum can be increased by inserting a suitable wire and soldering it at one end.

Housekeeper Seal

As described by W. G. HOUSEKEEPER (1923), platinum wire of 0·05 mm diameter can be successfully sealed through *Pyrex* if the wire is first flattened and the edges filed to a sharp V-shape as shown in FIGURE 42, *I*. The flattened portion of the wire is fused into a tube whose end has first been collapsed down until it will just take the broad part of the wire. Alternatively the wire may be beaded first,

but in either case a strong heat is required and the glass is pressed into good contact with the metal with tweezers. The entire section of the apparatus around the seal must finally be well annealed. The finished seal is shown in FIGURE 42, *II*.

Use of Mercury Cup

If the platinum wire is to be used as an electrode it may be preferable not to flatten and shape it as in the Housekeeper method, but to use a mercury cup on the outside of the apparatus (FIGURE 42, *III*). The fine wire is fused into a constricted part of a narrow bore tube and the

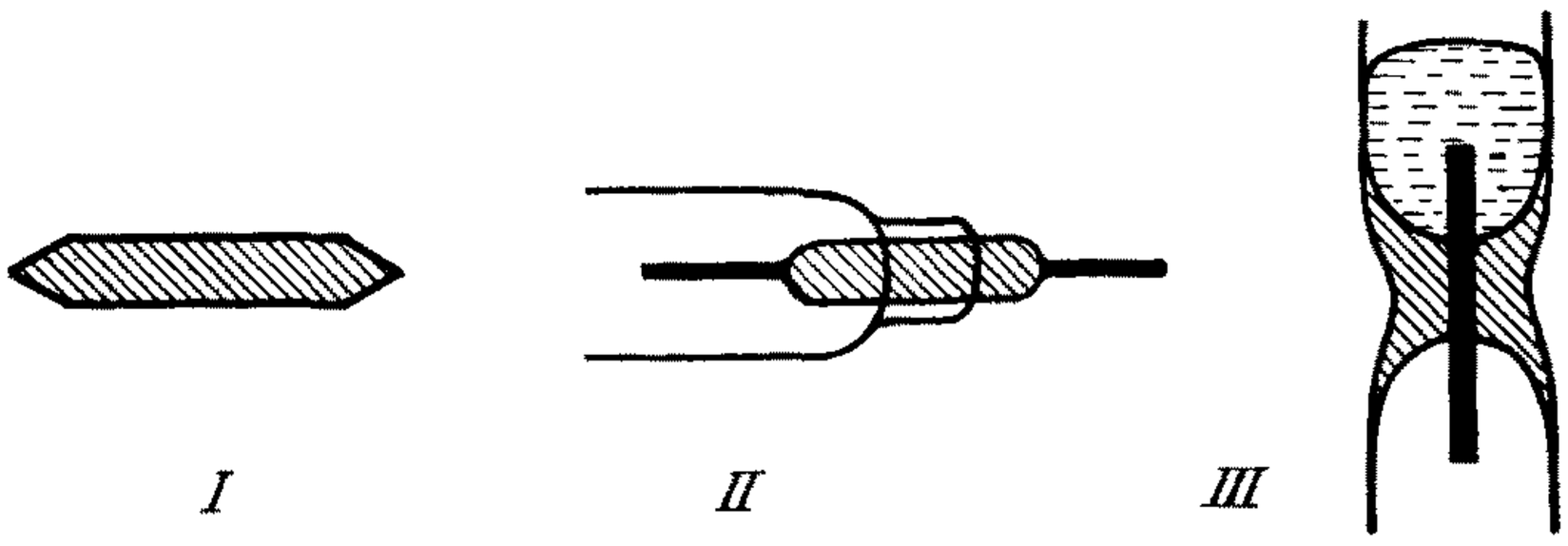

FIGURE 42. Sealing platinum into *Pyrex*

exposed end, on the outside, is covered with mercury which serves to seal up any leaks that might develop by the glass drawing away from the metal. Another wire dipping into the mercury cup can serve for electrical contact. Clearly this is of no avail for vacuum work, but is useful in making electrolytic cells.

Use of Platinized Glass Rod

A method for effecting a platinum–to–*Pyrex* seal by platinizing thin glass rod has been reported by G. F. TAYLOR (1929) and described in detail by C. C. COFFIN (1940). *Pyrex* rod of 1–2 mm diameter is coated with metallic platinum by dipping in a platinizing solution, allowing the excess to drain off, heating uniformly in a smoky flame until a shiny black film appears and then slowly heating to redness to burn the film into the glass (see p. 87). Three to four coats are required and then the platinized rod can be sealed into a *Pyrex* tube directly. The platinum layer gives electrical conductivity. Coffin reports that 10–15 applications will give a layer sufficient to carry several amps.

Special Alloys for Sealing to Glass

Tungsten and molybdenum, which have been used extensively for metal–to–glass seals, suffer from the disadvantages of being expensive

and difficult to fabricate into rod and of being non-ductile. Platinum is not so difficult to fabricate, but it is very expensive. An attempt has been made, therefore, to replace these metals with metal alloys, and extensive research in recent years has been aimed at finding alloys whose thermal expansion characteristics match those of glasses in the range from room temperature to the upper annealing temperature of the glass The expansion coefficient of glass increases rapidly in the range between the lower and upper annealing temperatures and it has been found that magnetic *Kovar*-type alloys of nickel, iron and cobalt, sometimes with a little chromium, have thermal expansions which behave in this manner. With alloys of only nickel and iron the increased expansion rate sets in at too low a temperature; however, such wires coated with copper, forming a *Dumet* alloy, are used extensively even to the present day in the lamp industry for sealing to soft glasses. A detailed survey of all the characteristics of the various alloys used for sealing to glass has been given by PARTRIDGE (1949).

Copper-Clad Nickel–Iron Wires for Sealing to Soda Glass

Seals for carrying small currents through soft glass can be made using *Dumet* alloy wires. These are drawn from rod which consists of a core of 43 per cent nickel–iron alloy on which about 30 per cent of its weight of copper has been electrolytically deposited. Wires up to 0·5 mm can be satisfactorily sealed into soft glasses. The seals are made in the normal manner but are left not fully annealed so that the glass remains in a state of compression in the longitudinal direction. The deposit of copper is sufficient to raise the total thermal expansion coefficient of the rod, in the radial direction, to the order of that of soda glass, but full annealing would result in a state of tension in the glass along the length of the seal.

If the seals are to be used for vacuum work it is advisable to tip the ends of the drawn wire with nickel in order to seal up any fine channels that may open between the copper layer and the central alloy core.

Nickel–Iron–Cobalt Alloys for Sealing with Intermediate Glasses to Pyrex

A large number of *Kovar*-type alloys of iron, nickel and cobalt of slightly varying compositions are obtainable and are marketed under different manufacturers' names. Common examples are *Fernico I* (54 per cent Fe, 28 per cent Ni, 18 per cent Co), *Fernico II* (54 per cent Fe, 31 per cent Ni, 15 per cent Co), *Nicosel*, *Telcoseal* and *Nilo*. The last three all have a composition of the order 54 per cent Fe,

29 per cent Ni, 17 per cent Co and expansion coefficient of $4{\cdot}5 \times 10^{-6}/°C$.

All the above alloys seal well to several special sealing glasses of which *B.T.H. C40*, *G.E.C. Lemington FCN*, *Kodial*, *Corning 7040*, *Corning 7052* and *Corning 7060* are common. The choice of alloy and sealing glass depends (other than upon availability) upon the service requirements. All give very satisfactory seals for normal purposes, the residual strain being slight and negligible; while, for instance, *Fernico II* and *Nicosel* (or *Telcoseal*) give practically completely strain-free joints with *Corning 7060* and *G.E.C. FCN* respectively.

For sealing to *Pyrex* glass a further intermediate glass is required in order that expansion coefficients become sufficiently matched. For example, when sealing *B.T.H. C40* or *G.E.C. FCN* to *Pyrex*, *C9* or *W.1.* can be used as intermediates. Another intermediate glass, such as *Dial 43*, may be desirable as a sandwich glass between the *Kovar*-sealing glasses and the tungsten-sealing glasses. A *Kovar* to *7052* or *7040* tubular seal can be joined to *Pyrex* (*7740*) via *Nonex* (*7720*) if the tubing is less than 1 cm diameter; while for larger tubing *7052* (or *7040*) to *7050* to *7070* to *7740* is necessary.

Metal–to–Glass Seals using Kovar Tubing

Metal–to–glass seals with *Kovar* tubing up to about 8 mm diameter can be made fairly comfortably by hand, but with tubing larger than this it becomes increasingly desirable to use a glass-working lathe so that uniform seals are obtained. Firstly, square edges on the metal must be rounded off, and for this machining or filing is advised rather than grinding, since particles of abrasive are difficult to remove from the metal and great care must be taken. The metal is degreased and then cleaned by dipping in a solution of 10 per cent hydrochloric acid and 10 per cent nitric acid at 70°C, followed by thorough washing and drying. Touching of the cleaned portion of the metal, before the seal is made, should be avoided.

The metal should be outgassed by heating in a stream of hydrogen for 3–4 hours at 900°C, otherwise bubbles will be formed in the glass and an unsatisfactory seal will result. With small size tubing, less than 8 mm diameter, this cleaning in hydrogen is not absolutely necessary but the resulting seal will be less strong hermetically and leaks may develop. A useful makeshift hydrogen furnace consists of a silica tube along which hydrogen is passed and which is kept glowing white hot with a bench torch. The hot hydrogen must not flow into the atmosphere and produce an explosive mixture.

Tubing of the glass to be used should be chosen of diameter such

that the *Kovar* tubing just slides inside. A short length of cleaned metal tubing is mounted on a glass rod handle with asbestos tape and oxidized by heating to dull red heat. It is allowed to cool slightly and inserted inside the glass tube to a depth of 3 mm. The glass is then fused directly on to the metal starting from one end so that occluded air is driven out. The flame should be played on the glass and not on the metal. The glass tube is finally pressed lightly towards the metal tube so that it builds up slightly over the inside edge of this tube, the flame being directed at this portion during the operation (see FIGURE 43, *I*). Slight blowing may be necessary. The resulting seal is grey in colour and is annealed in the same manner as a glass-to-glass seal. With larger tubing a small 3-mm long ring of glass can be fused on to the metal tube first (FIGURE 43, *II*) and the bulged end of the glass tube sealed to this (FIGURE 43, *III*). Much more satisfactory seals can be made employing a glass-working lathe, for then the glass can be

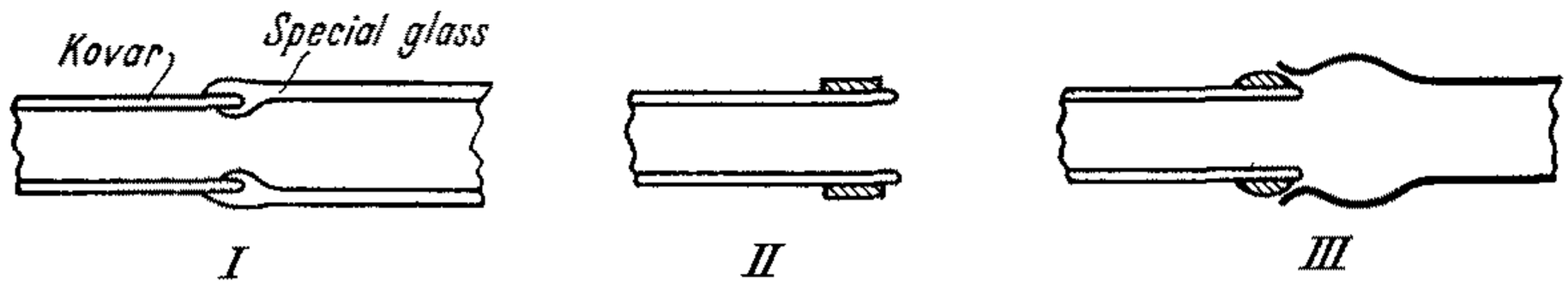

FIGURE 43. Sealing *Kovar* to a special sealing glass

tooled into good contact with the metal and all air carefully squeezed out. Several types of lathe are available; the machine described in Chapter 6 is satisfactory for these seals.

Copper-to-Glass Seals

Most glasses adhere strongly to copper, but the coefficient of expansion of the metal (17×10^{-6}/°C) is much higher than that of all known technical glasses, resulting in a greatly mismatched seal. Copper is so ductile, however, that when specially shaped the metal will yield, relieving much of the stress and making the formation of satisfactory copper–to–glass seals possible. The strong adhesion at the glass–metal interface results in a preference for the metal to give rather than to separate from the glass. A large contact surface between the glass and the metal is therefore provided. W. G. HOUSEKEEPER (1923) has reported in detail on the methods for sealing ductile metals to glass with special shaping of the metal at junctions. Oxygen-free high conductivity copper is the best type of copper for seals.

Copper to soft glass seals are most easily made since the difference in expansion rates is least here, but satisfactory seals of copper to *Pyrex* can be formed with a little more care. A further factor with

hard glasses is that a higher working temperature is required and therefore the copper is subject to excess oxidation and the seal to a larger cooling range during which the stresses are set up.

The method for sealing copper wire through any glass is the same as described for platinum through *Pyrex*. However, with copper the metal is very brittle after being worked and the seals are not frequently used. Copper wire can be sealed through glass if it is flattened to 0·05 mm thickness and the edges feathered. The flattened portion is cleaned, pre-oxidized by heating to red heat for a few seconds, allowed to cool, and then given a thin coating of borax. This is effected by dipping the prepared metal in borax solution and drying in the hot air above a flame. Borating of the copper prevents excess oxidation when the seal is worked and is therefore of much greater importance when employing hard glasses. With soft glasses it can be dispensed with but this is not advised if the seal is to be used in vacuum apparatus.

The prepared flattened section is inserted into a glass sleeve shaped to fit, and the glass is fused on to the metal, pinched with tweezers and strongly heated. The seal must be carefully annealed. With soft glasses the copper glass interface is coloured red, while with harder glasses the colour is more of a yellowish red.

Copper–to–Glass Tubular Seals

Tubular seals can be made by several methods reported by HOUSEKEEPER, of which three are described here.

In the first the copper tube is machined so that a cone-shaped flange is formed—30° to the axis of the tube—at the end to be sealed (FIGURE 44, *I*). The metal flange is oxidized and while still hot the molten end of the glass tube is pressed inside the cone and fused into contact. Care must be taken not to let any glass overlap the end of the copper tube, so that a seal is finally formed as shown in FIGURE 44, *II*. Gentle blowing helps to shape the join. That the junction is bulky is the only disadvantage of this type of seal.

In the other two kinds of seal the glass is allowed to overlap the edge of the copper tube, but this edge must be first machined to a fine 5° taper. After pre-oxidizing this feathered edge by heating to red heat for a few seconds a well fitting glass tube can be fused to the outside or to the inside without allowing the oxidized metal to cool. The glass is bulged slightly over the feathered edge thus affording it protection. The seal to use (FIGURE 44, *III* and *IV*) depends upon whether the seal is to undergo further heating or cooling in service, consideration being given to the fact that the copper will attempt to expand or contract much more than the glass.

Since tubular seals can be made in this manner to both soft glass and hard glass, an intermediate copper tube can afford a very useful method for making a soda–to–*Pyrex* seal. If *Nonex* is available a stronger join is obtained by using it between the copper and the *Pyrex*, because a lower working temperature is required for *Nonex* and thus, on cooling the worked seal, the copper has a shorter temperature range in which to contract at its greater rate.

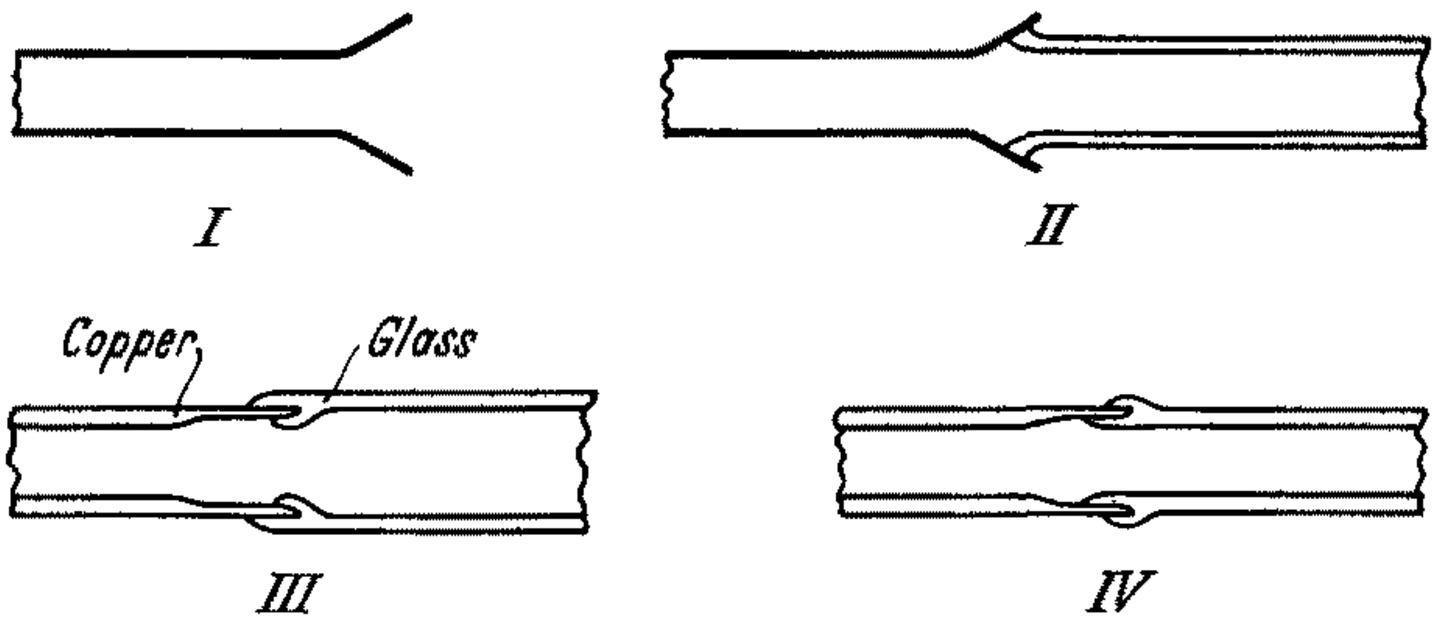

FIGURE 44. Copper–to–glass seals

Disc Seals

Copper disc seals can be used for carrying a wire conductor into a glass tube: two pieces of glass tubing are flared, each at one end, and a copper disc is sealed between the two flared ends. A suitable thickness of disc for tubes of diameter 2 cm is about 0·3 mm.

To make the seal one of the tubes is clamped vertically and the pre-oxidized disc is placed centrally upon the flare of this tube. Working round the tube with a hand torch the glass is softened and the disc fused into place. With the seal still hot, the tube is removed from the clamp and, rotating by hand in a bench flame, the second glass flare

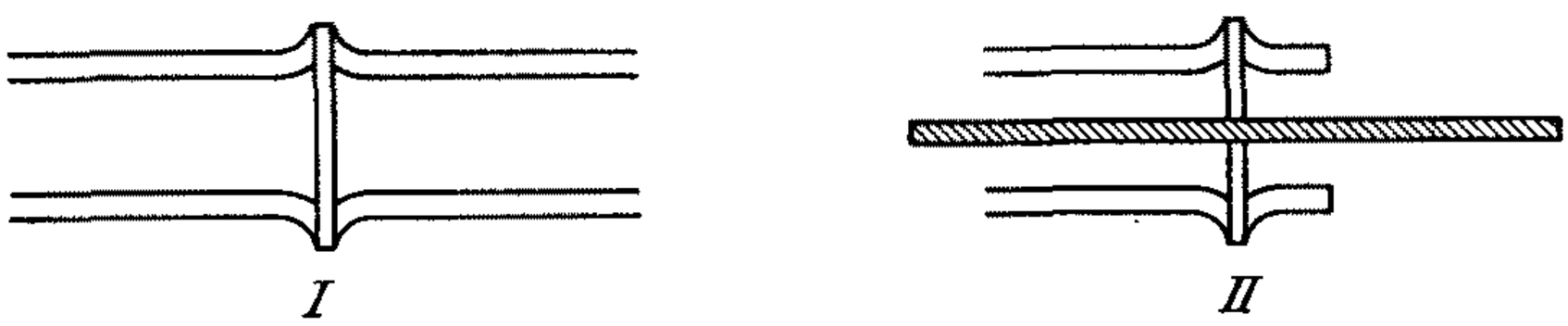

FIGURE 45. Copper–to–glass disc seals

is fused to the disc, centrally and directly opposite the first (FIGURE 45, *I*)—if desired, the lower tube can be left clamped. Great care must be taken during these operations to see that no glass runs over and bridges the edge of the copper disc, otherwise fracture will result. After annealing, one of the glass tubes may be cut off near to the disc and a conductor wire may be soldered through the disc (FIGURE 45, *II*).

Glass tubing must be sealed on either side of the disc in this manner so that stress is taken up by the disc contracting and not by it bending, which would fracture the seal. HOUSEKEEPER reports that a disc up to 2·5 mm thick has been used but the resulting seal would not stand heating or cooling.

The copper disc, if thin, will distort fairly easily if there is a circular groove in it. This stratagem may be found useful if difficulty is experienced in making a disc seal.

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Chapter 8

THE CONSTRUCTION OF SOME TYPICAL SINGLE PIECES OF EQUIPMENT

In this Chapter we describe the construction of various pieces of apparatus by the methods of glass-working already described. All the apparatus mentioned below can be made by quite simple methods and without the use of the expensive items of glass-working equipment such as lathes, ovens, various wheels and so on. Many pieces of complex-looking apparatus are made merely by a succession of simple operations.

Ampoules

Ampoules (Figure 46, *I*) are used when a material, such as a biological fluid, has to be stored in a sealed-off glass vessel. To make an

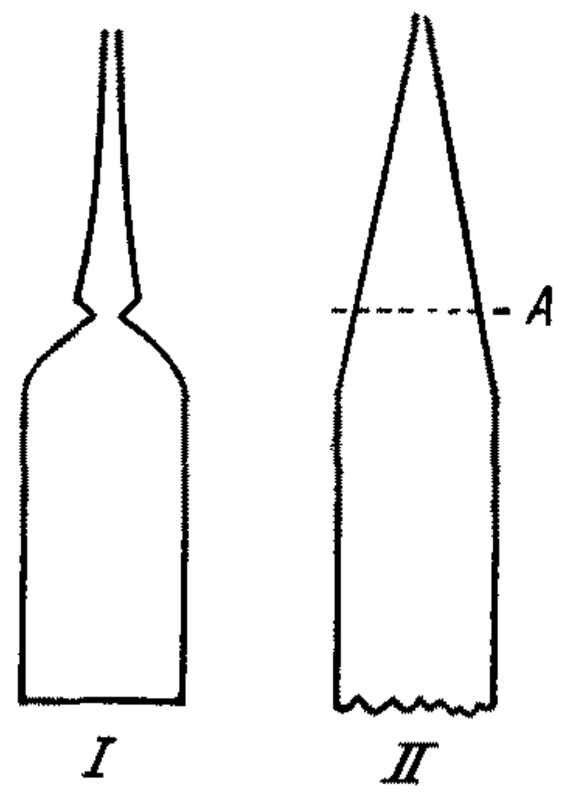

Figure 46. Ampoules

ampoule of normal size, a tube about 4 cm long and 1 cm diameter is prepared with a spindle at each end: a uniform taper is made at one end (Figure 46, *II*); the tube is then heated at *A* and either rotated against a metal object or else sucked-in so that a shoulder is formed as in Figure 46, *I*—this shoulder is not necessary, however, and the operation may be omitted. The spindle at the other end of the tube is

taken off and a test-tube end (i.e. a rounded end) is made. This is run down by heating in a flame, and either pressed against a carbon plate or blown out against a carbon plate to form a flat bottom on which the ampoule can stand.

Special glasses for ampoules are mentioned on p. 24; *Monax* is also a good glass for ampoules.

Sealed Tubes for Reactions

Tubes for 'sealed tube' reactions, such as the Carius determination of halogens and sulphur, can be made from *Pyrex*, *Monax* or soda glass. The mechanical strength of the glasses is about the same, but a soda Carius tube is much more likely to crack as a result of thermal strain than a *Pyrex* or *Monax* one. The Carius tubes are usually made from tubing of approximately 20–25 mm diameter and 3 mm wall thickness—*Pyrex* extra heavy tubing of external diameter 22 mm has a wall thickness of 2·5–4 mm and can be used up to 600°C.

To prepare a Carius tube, a length of tubing is cut off slightly longer than the furnace into which it is to go. A handle is attached to one end, which is then drawn off and rounded so that the round end has about the same thickness as the wall of the tube. The tube is cleaned, and the necessary chemical substances are introduced—for a halogen determination silver nitrate is added, using a paper cylinder so that none of the salt adheres to the walls except at the end of the tube. Nitric acid is added with a long thistle funnel, and finally the organic specimen in a small test tube is introduced. The open end of the tube has to be drawn off to a thick spindle and sealed while these substances are in the tube, so that the normal method of holding and rotating a tube horizontally cannot be used.

Two methods for sealing the tube can be used: in one, the tube is held in one hand at about 45° to the vertical, a glass rod handle is attached to the open end, and a thick-walled spindle or capillary is put in, using a bench torch, in such a position that the final sealed capillary will project about 2 cm from the iron tube which will contain the Carius tube in the furnace. Following this operation, the capillary is sealed at the proper place before the tube has cooled. This ensures that the tube is cooling at the moment when it is finally sealed, so that as the gas inside contracts this final sealing is assisted by the atmospheric pressure which pushes the glass in. In the other method the tube is clamped vertically and a handle is attached to the top, open end. A thick-walled spindle is then put in the tube, using a hand torch flame played round the glass; if at first the spindle is too wide and too thin it is slowly thickened by shrinking in a flame which is not too hot. The end of the capillary is sealed off as before.

The external diameter of the tube and its wall thickness determine the pressure which it will stand. Failure of a tube is due to the tension at the surface becoming too great. For a cylinder of internal radius r and external radius R, and with an internal pressure in the cylinder of P, the maximum circumferential (or hoop) tension S (see A. MORLEY, 1940) on the inner surface of the cylinder is

$$S = P\left(\frac{R^2 + r^2}{R^2 - r^2}\right)$$

Thus, if the maximum safe tension is 1000 lb/in.^{2} (see p. 10), a Carius tube with 3-mm wall thickness and 2-cm external diameter is suitable for reactions at 340 lb/in.^{2}. It is necessary to remember, of course, that the strength of glass under tension is a statistical quantity.

Break-Tip Seals

These seals separate two parts of an apparatus, and they can be broken by a slight mechanical blow. The simplest type is illustrated in FIGURE 47, *II*—the tip is broken off by a piece of iron, usually enclosed in glass, dropping under gravity. This weight is moved and lifted with a magnet.

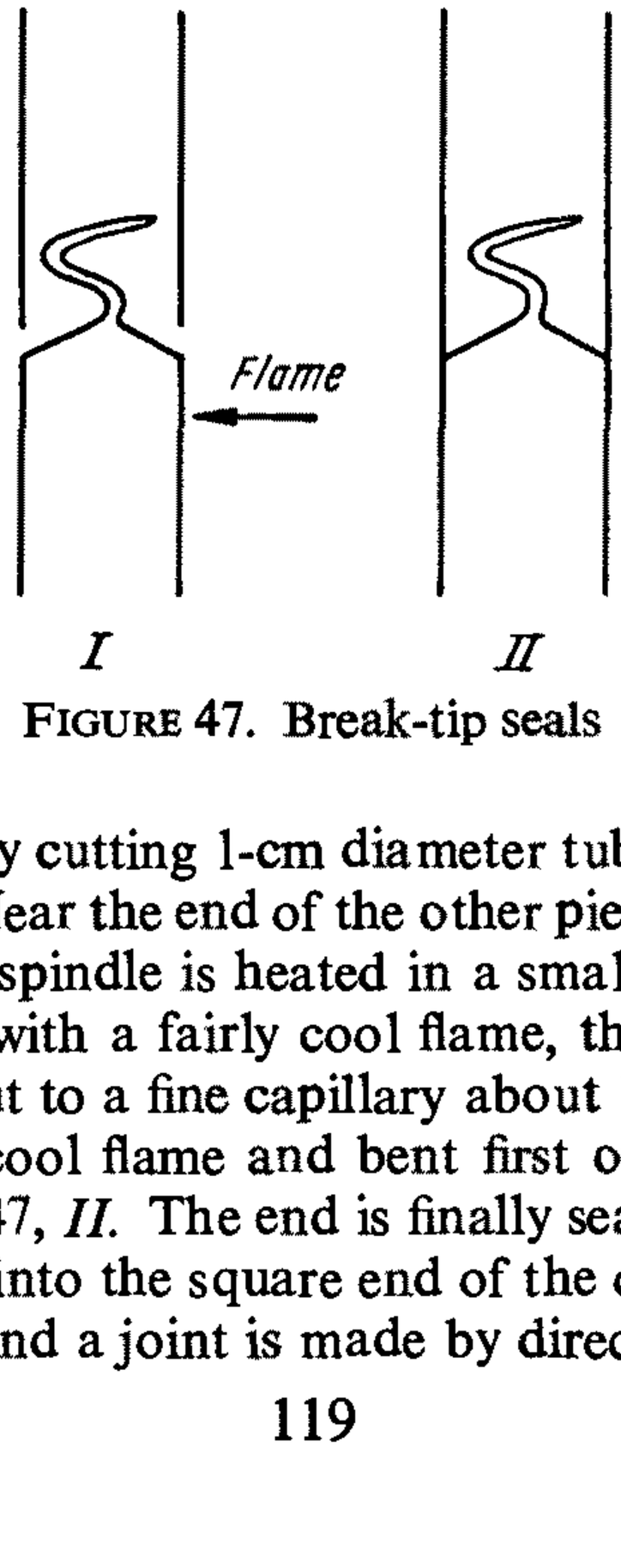

FIGURE 47. Break-tip seals

The seal is made by cutting 1-cm diameter tubing to give one piece with a square end. Near the end of the other piece a spindle is drawn. The shoulder of the spindle is heated in a small hot flame so that it thickens, and then, with a fairly cool flame, the spindle beyond the shoulder is drawn out to a fine capillary about 1 mm diameter. This is heated in a very cool flame and bent first one way and then the other, as in FIGURE 47, *II*. The end is finally sealed off. The tip thus prepared is inserted into the square end of the other piece of tubing, as in FIGURE 47, *I*, and a joint is made by directing a small flame at

the piece of tubing with the tip, and not at the square-ended piece. The joint is run down and blown out a number of times and then drawn out so that the outer tube has a constant diameter. It is essential to direct the flame only at the tube with the seal on the end of it, because if the other tube is heated strongly it will collapse on to the fine capillary.

Distillation Flasks

A round-bottomed flask can be made into a distillation flask simply by the addition of a side arm, which is added just as a side arm is added to a big flask (p. 92). The angle of the side arm to the neck is usually about 75° (FIGURE 48, *I*).

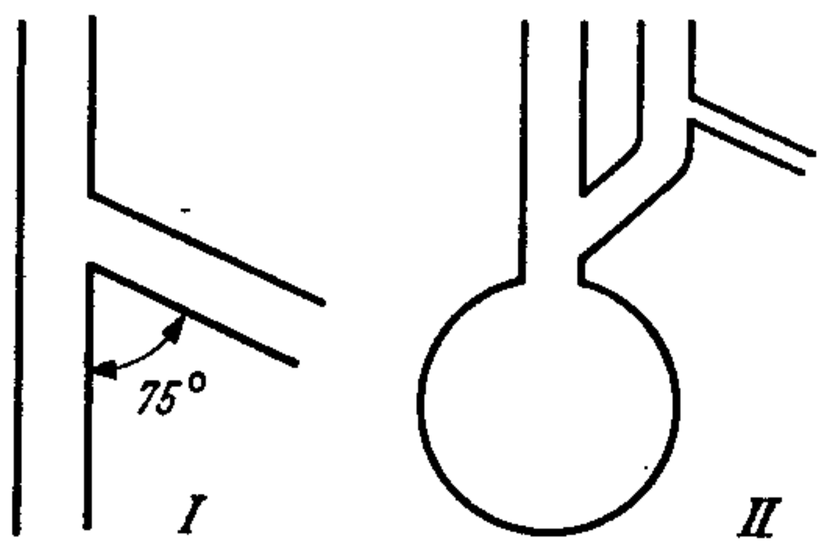

FIGURE 48. Distillation flasks and Claisen flasks

A Claisen flask of the type shown in FIGURE 48, *II*, has a wide side arm sealed into its neck at an angle which, for convenience, is about 45°. Any liquid in the side arm will then readily run back into the flask. A 45° joint of this kind can be made quite easily with a hand torch: a round end is put on the side arm, using a bench torch, and a hole is blown in this round end so that the plane in which the hole lies is at 45° to the tube axis. The wide side arm is joined to the flask neck, and the small side arm is then joined on.

The small side arms are very often broken off distillation flasks, especially in teaching laboratories; the most common place of breakage is near or at the actual T-joint, and the neck of the flask is often cracked. The crack can be repaired easily if the flask is of a borosilicate glass, as described on p. 80, and with great care a similar repair is possible for soda flasks. The remaining stumpy portion of the side arm is then removed with forceps and scissors, the hole is filled in with thin rod, and a new hole is blown for a new side arm. If the remaining part of the old side arm is long enough (over 1 cm)

it is only necessary to put a spindle in it and then blow a hole so that a new portion of tubing can be added.

Distillation Splash Heads

Splash heads are used in distillation to prevent the liquid in the distillation flask from splashing over into the condenser. Several types are used, but probably the simplest is the pear-shaped one shown in FIGURE 49, *VI*. A bulb is blown in the middle of a tube of about 1 cm diameter (FIGURE 49, *I*) and heated at *B* in a large flame, with rotation, so that, by blowing and pulling, the pear-shaped bulb of FIGURE 49, *II*, is produced. The tube at *A* is removed and the top of the bulb is rotated in the flame—this, with gentle blowing, should

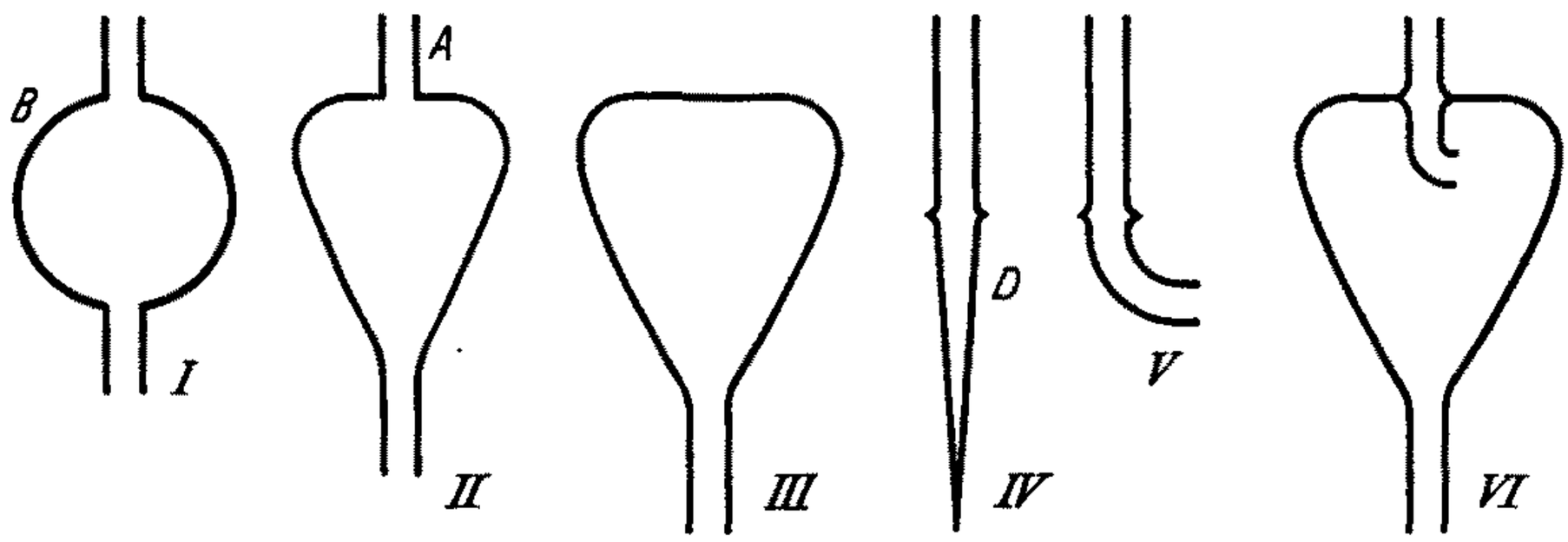

FIGURE 49. The construction of a distillation splash head

produce a fairly flat-topped bulb (FIGURE 49, *III*). The tube to form the top part of the splash head is heated in a large flame and tapered. The top of the taper is next heated strongly with a small flame, and gentle pressure applied at both ends of the tube so that a ridge is formed (FIGURE 49, *IV*). The taper is then heated at *D* in a soft flame and bent round. The curved part is cut off (most easily in the flame) about 1 cm beyond the ridge, and the end is fire-polished (i.e. heated in the flame to smooth it). In the centre of the flat top of the bulb a hole is blown, just smaller than the size of the ridge; into this the curved part is inserted, and an internal seal is made either with a bench torch (Method 1, p. 73), or with a hand torch (p. 76), the former method is preferable. The final splash head is shown in FIGURE 49, *VI*.

Dewar Seals

The Dewar seal is important and useful, although a ring seal can sometimes be substituted in simple apparatus—as, for example, in the cold finger liquid air trap shown in FIGURE 52, *III*. In the making

of a Dewar seal the preparation of the parts is of extreme importance. Basically, the inside tube is flared at one end, and the end of the outside tube is turned in so that with the two tubes coaxial the inward and outward flares meet as closely as possible.

FIGURE 50, *I*, shows the ideal starting point for obtaining a good Dewar seal. The flare in the inside tube should be just large enough for this tube to be held up by its flared end when sitting inside the clamped outer tube. With small diameter tubing the flange on the inside tube can be made by simply blowing out the end with the glass

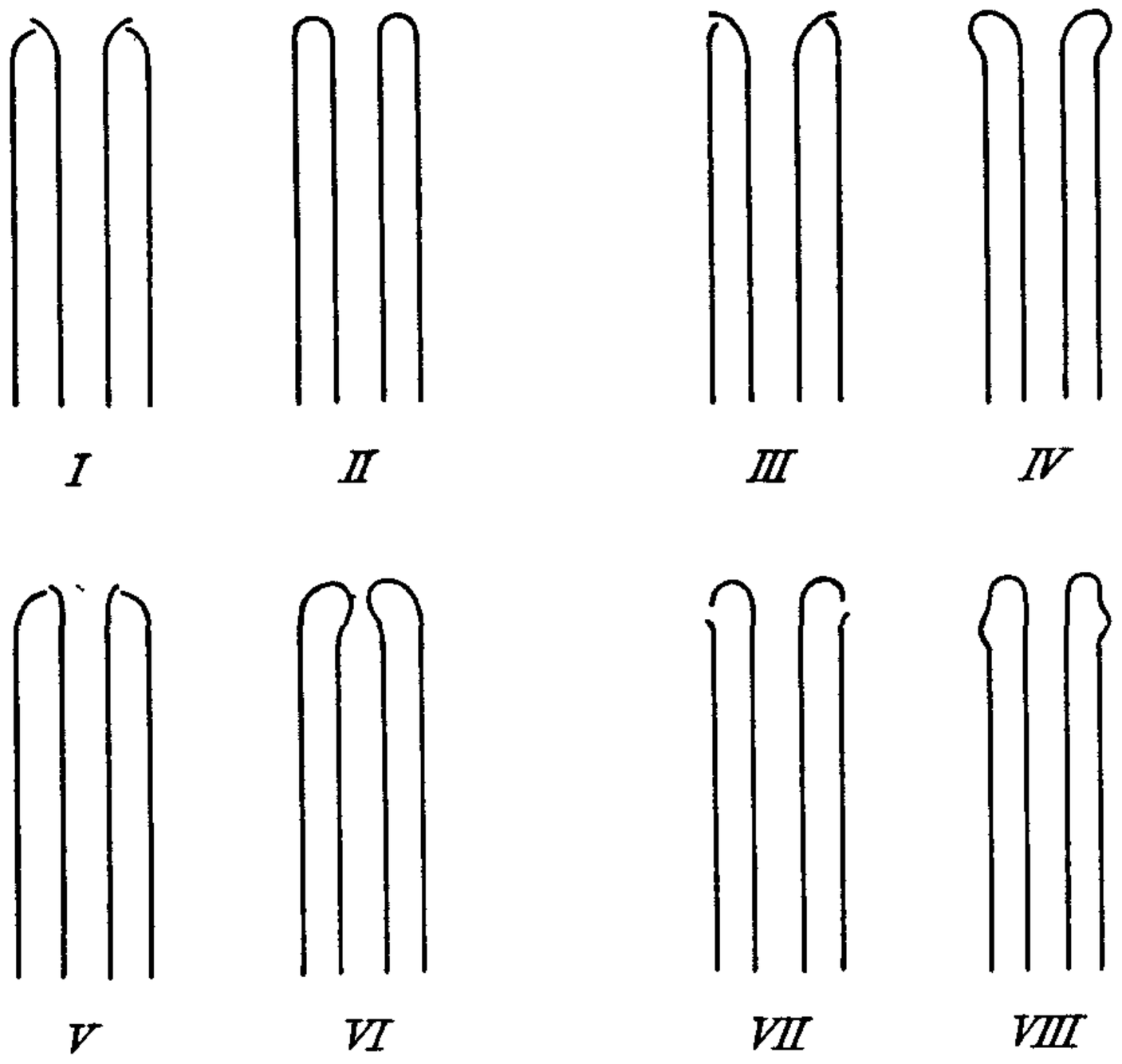

FIGURE 50. Dewar seals

just slightly less hot than normal. To make the Dewar seal using the hand torch, the whole joint is warmed and then a portion at a time worked with a largish flame and enough oxygen to make it just non-luminous. The glass of the inside flare is pushed down with a spike or similar tool on to that of the outside tube, gradually working round the seal, and when blowing becomes possible a pin-point flame is used for blowing out and shaping a portion at a time.

Welding rod is employed when necessary, but if the parts are prepared satisfactorily the flares should provide sufficient glass to alleviate the need for rod. With care it will also be unnecessary to hold the inside tube in position since only part of the joint is heated to

softening point at a time and, in the final annealing, if the seal is made well the inside tube will tend to hang centrally. However, the inside tube may easily be kept central with asbestos paper or corrugated cardboard or with copper wire that can be dissolved out with nitric acid. In making the seal care must be taken not to collapse the outer tube on to the inner just below the join; otherwise little difficulty should be encountered. The finished seal is shown in FIGURE 50, *II*.

If the starting parts are shaped as in FIGURE 50, *III* or *V*, the resulting seals will be bulged outwards or inwards respectively (FIGURE 50, *IV* and *VI*). These may satisfactorily serve their purpose but require extra careful annealing. If the flare of the inside tube is turned right back on itself (FIGURE 50, *VII*), then the final joint is most easily made.

Dewar Vessels

The work which led to the modern vacuum flask has been described by J. DEWAR (1896). Vacuum jacketed flasks are used extensively in scientific laboratories and can be constructed relatively easily: tubing is chosen to give a flask of the required size and, after the inside tube has been rounded off at one end, a Dewar seal to the outer tube is made at the other end (FIGURE 51, *I*). If a flask with a narrowed neck is required the parts must be prepared as in FIGURE 51, *II*, before the Dewar seal is made.

The open end of the outer tube is then drawn off, a 1-cm tube *B* is attached, and a 2-mm internal diameter constriction is formed at *A* as shown in FIGURE 51, *III*. This is best done by rotation in a flame, but, if the tubing is too large for the operator to manipulate, it can be performed with the apparatus clamped as described in Chapter 5. For efficient thermal insulation the inside walls of the jacket should be silvered, and the silvering is performed at this stage of the construction by the methods described on p. 85 and in some detail by W. E BARR and V. J. ANHORN (1949). FIGURE 51, *IV*, shows a simple stratagem for filling and emptying the jacket with the cleaning and silvering solutions respectively. After cleaning and drying, the silvered vessel is ready for evacuation and sealing off.

Evacuation of Dewar Vessels

Glass under vacuum slowly releases gas and vapours (p. 16) and therefore, in order that a good vacuum will be retained in the closed jacket, it is necessary to outgas the evacuated jacket before sealing off. As shown by I. LANGMUIR (1918) and described by Barr and Anhorn, the most effective outgassing is obtained when the evacuation is carried out in two or more stages of decreasing temperature.

The prepared flask is supported so that the tube *B* can be sealed to a vacuum system. If a suitable furnace is available the flask is surrounded by this, with the constriction *A* still within reach of the hand torch flame; otherwise the heating must be done by flame. A liquid air trap is included between the flask and diffusion pump. With a vacuum of below 10^{-6} mm of mercury the temperature of the flask is raised to near the softening point—the heating may be rapid

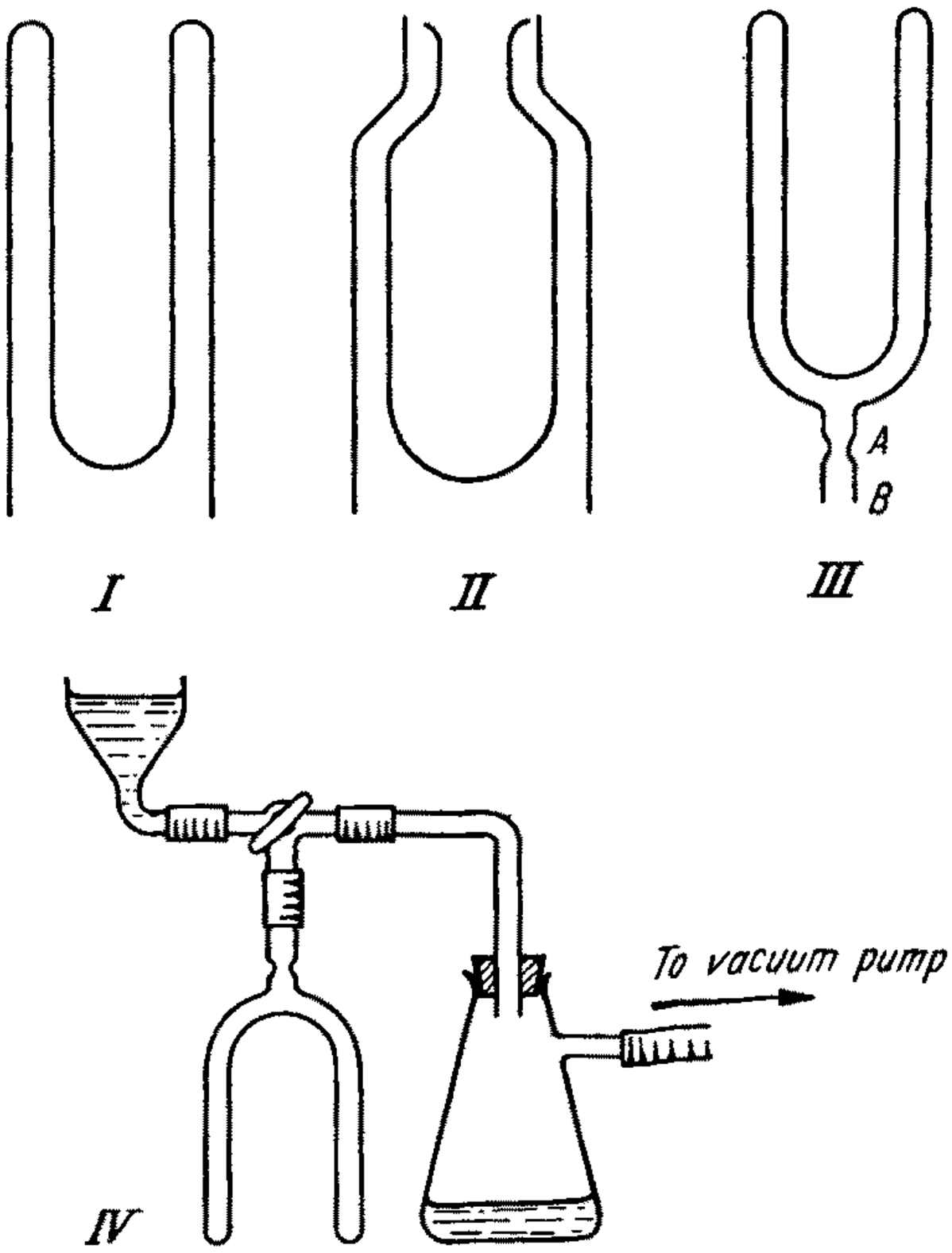

FIGURE 51. Construction of Dewar flasks

with unsilvered vessels, but must be very slow when the walls have been silvered. With *Pyrex* glass (*Corning No. 7740*) a temperature of 550°C is held for about one hour; the temperature is then reduced to about 400°C and this held for a further thirty minutes with continuous pumping. With the flask still at about 400°C the glass is slowly collapsed at the constriction *A*, allowing four or five minutes for this process to enable the heated glass in this region to outgas thoroughly. The tip on the bottom of the flask is rounded and annealed, and the flask then allowed to cool as quickly as possible without setting up strain.

Both commercial and home made Dewar flasks used in the laboratory should be wound along the whole of their length with adhesive

tape for safety. This prevents them exploding and shattering in the event of fracture which sometimes results from strain set up during pouring refrigerants, particularly liquefied gases, into or out of the flask.

Cold Finger Refrigerant Traps

Cold finger refrigerant traps are often used in vacuum lines as a substitute for the more efficient total immersion traps which, however, tend to cut down the pumping speed of the system. In constructing a cold finger trap a Dewar seal is first made, then, before

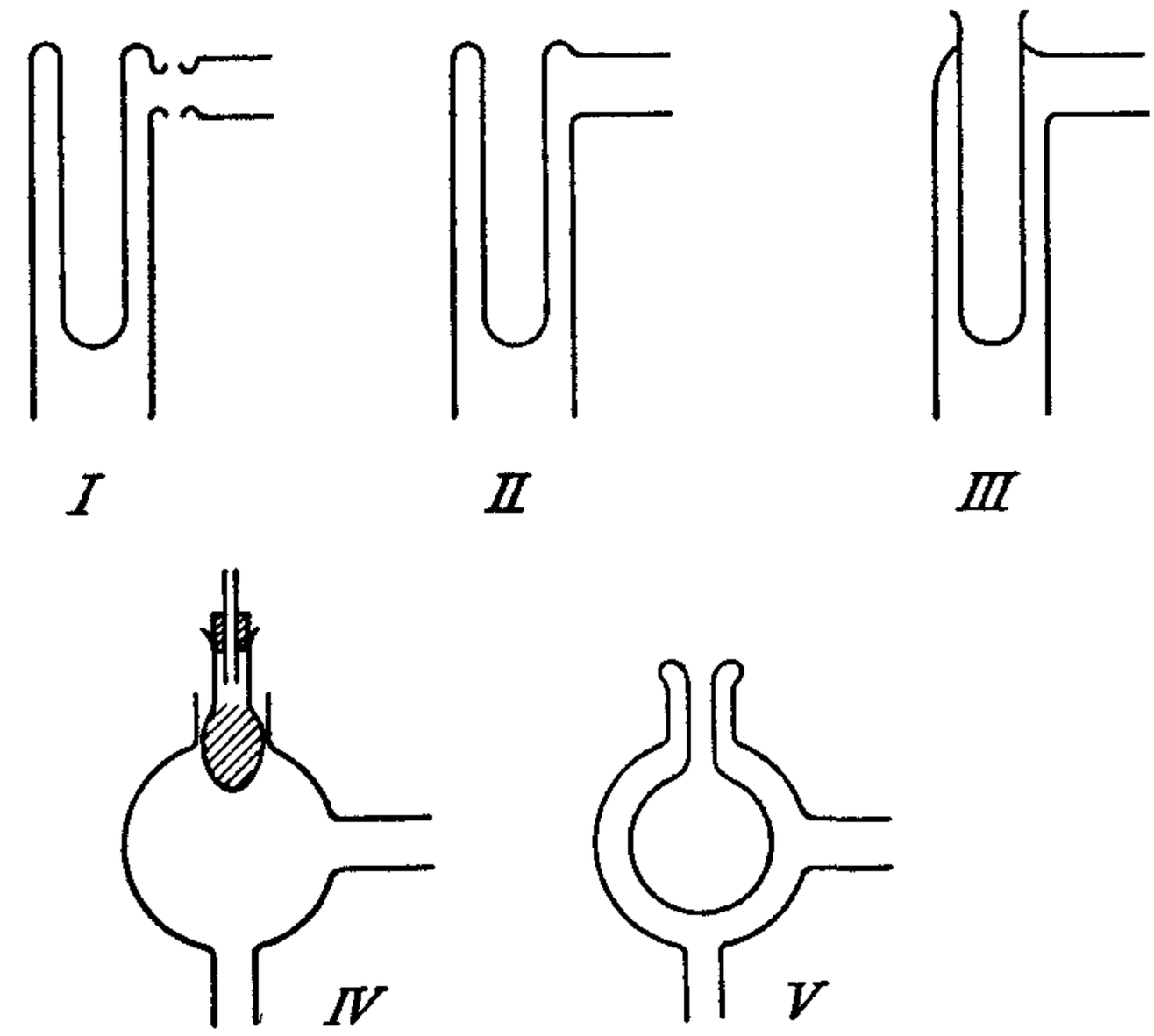

FIGURE 52. Construction of cold finger refrigerant traps

annealing, a small hole is blown out near to the seal (FIGURE 52, *I*) and a prepared side arm sealed in position. Care must be taken to keep the far side of the Dewar seal tube hot while the side arm is being added, to prevent cracking; finally the whole is annealed. The finished trap is shown in FIGURE 52, *II*.

FIGURE 52, *III*, shows how a quite satisfactory cold finger trap can be made with the Dewar seal replaced by a straightforward internal seal. As before, the side arm must be added immediately the internal seal is completed and the whole annealed together.

For high vacuum work, where it is desirable to shorten the pumping path to a minimum, a useful built-in cold trap is shown in FIGURE 52, *V*. The outer jacket is prepared from a round-bottomed flask to which the side arms are attached, as shown in FIGURE 52, *IV*. The inner tube is shaped with a flare at its upper end and its heated

lower end is thickened and collapsed as for making a bulb (p. 71). The lump of white hot molten glass is lowered through the top side arm of the outer jacket and the bulb blown steadily and firmly to the required size. Contrary to expectation it is found that the hot molten glass does not adhere to the cold outer tube when it comes into contact with it, provided the lowering operation is performed quickly. The trap is finished by making a Dewar or ring seal at the upper end. An unskilled worker will find, with practice, that it is possible to make small, bulbular, cold finger traps of this type for pumping lines up to 25–30 mm diameter. Anything larger requires a skilled hand.

With all these cold finger traps the rate of loss of refrigerant is much greater than from the Dewars whose inner surfaces are silvered to cut down radiation losses.

Filter Pumps

It is advisable before making a piece of apparatus such as a filter pump to prepare all the separate components, since the work, if left whilst a component is made, may crack on re-heating—in fact if soda glass is used it probably will do so. Hence for a filter pump of the type shown in FIGURE 53, *V*, the jet, the cup, the side arm and the tube for the jacket are first made separately.

The jet is made from tubing of about 1 cm diameter: the tubing is strongly heated and a ridge put in. Another ridge is made about 1·5 cm below the first, and a taper is made 4–5 cm long with the shoulder about 1 cm below this last ridge. A ridge is then made at the shoulder of the taper, and the end of the taper is cut off and fire-polished (FIGURE 53, *I*).

The side arm is made from tubing of about 0·5 cm diameter by drawing a spindle and making a ridge just above the shoulder. The glass is cut off 1–2 cm from this ridge (FIGURE 53, *II*).

The jet is sealed into the jacket with an ordinary internal seal (Method 1, p. 73), and the side arm is attached at the shoulder; the jet must be perfectly central (FIGURE 53, *III*).

The cup is then made: a piece of tubing of the same diameter as the jacket (about 1·5 cm) is joined to a tube of the same size as the jet tubing. A slight taper is made in the smaller tubing, and a ridge made at the shoulder of the taper. A bulb is blown at the end of the taper. This is not very easy since the glass is usually very thin at first—the bulb must be blown and shrunk a number of times until enough glass has been accumulated to blow a strong bulb. It should be 3–5 mm diameter (FIGURE 53, *IV*). The end of the bulb is blown out and spun (p. 73) to give a thistle funnel shape (*B*, FIGURE 53, *V*). The hole should be just bigger than the end of the jet.

The end of the jacket is rounded off so that when the cup is inserted the jet will be just inside the cup. The cup is next sealed in with an internal seal. The larger tube is then rounded off at *C* (FIGURE 53, *V*), and a side arm is put on at the bottom and bent round. Lastly, the two side arms are cut off and fire-polished to give the final pump seen in FIGURE 53, *V*.

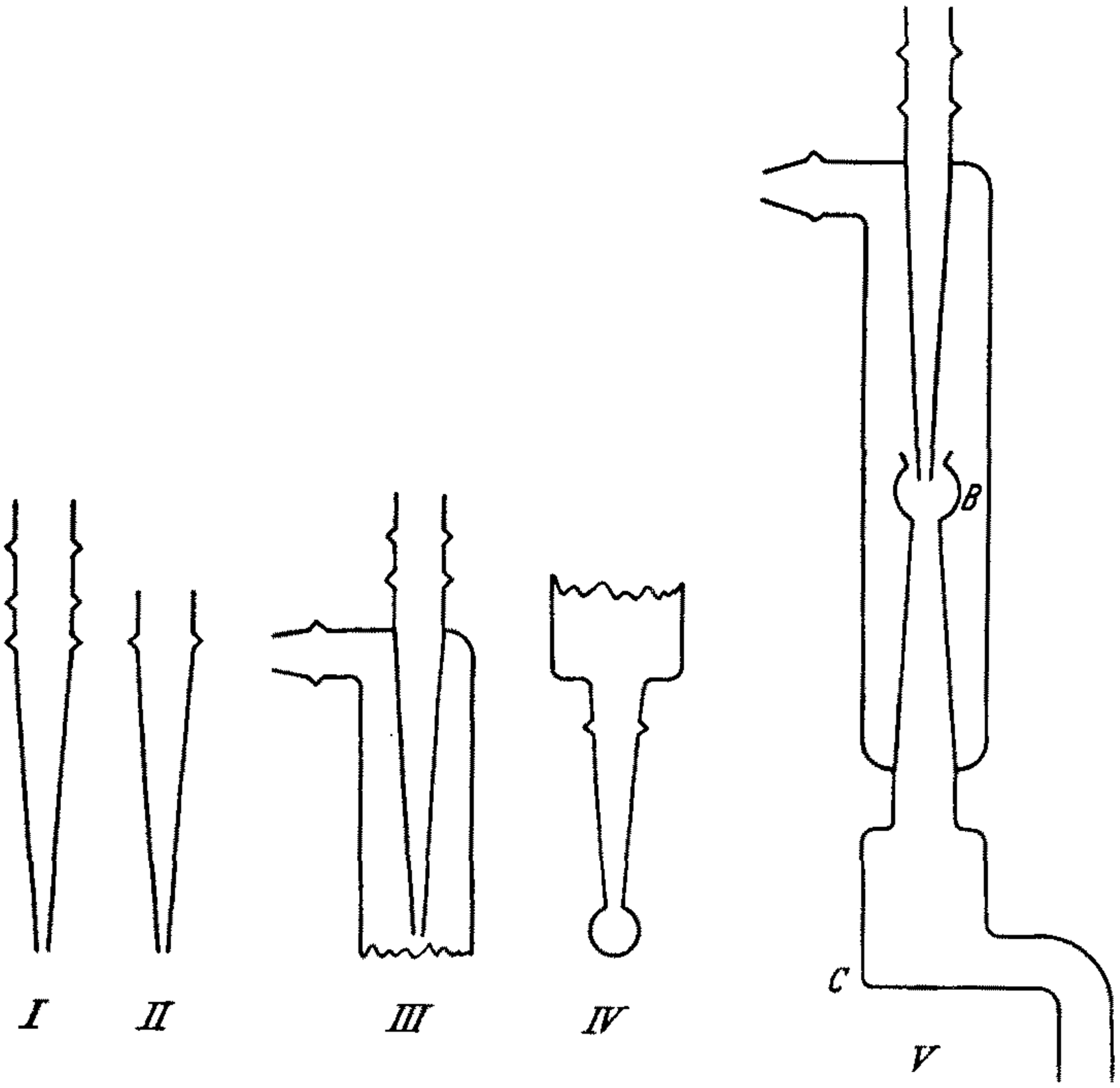

FIG. 53. The construction of a filter pump

Phosphorus Pentoxide Traps

Water vapour and some organic vapours are readily removed from a gas stream by phosphorus pentoxide traps. A simple trap is shown in FIGURE 54, *I*; *A* is the P_2O_5 boat.

In most cases a tube of 2 cm diameter will suffice for housing the boat, and to one end of such a piece of tubing is sealed a ground glass socket *B*. The other end is rounded off at the required length. At *C* and *D* two T-junctions are formed to tubing 8–10 mm diameter and these side tubes are bent to the desired shape. A stopper *E* is made from the appropriate standard cone as shown. Vacuum grease or

wax is used for sealing the stopper in place. In this way the P_2O_5 boat can be easily recharged. When building the trap into an apparatus, the side arms at *C* and *D* should point upwards so that any phosphoric acid syrup spilt from the boat will not get into the system.

Phosphorus Pentoxide Boats

A satisfactory P_2O_5 boat can be readily made from glass tubing: a short length of suitable diameter is cut along its length (FIGURE 54, *II*) either on a cutting wheel or by leading two cracks along the length of the tube on opposite sides—this latter operation is fairly easy with soda glass. Using one half, each end in turn is heated and

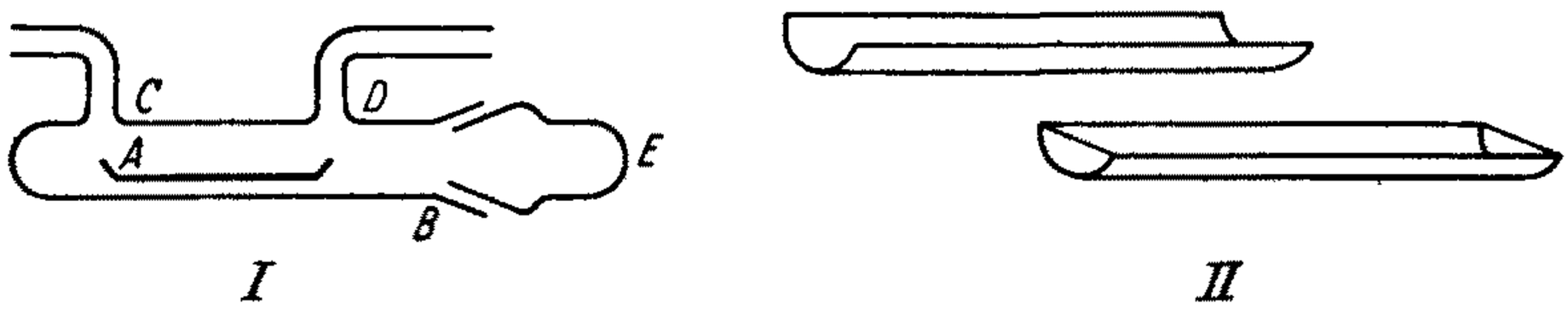

FIGURE 54. Phosphorus pentoxide trap

bent upwards, then finally shaped with tweezers to form a closed end as shown. A pin-point flame is used for sealing the seams. Finally the long edges are fire-polished and the boat annealed carefully.

A less elegant P_2O_5 boat can be made much more quickly by the following method: the glass tube to be used is closed at each end with a spindle so that the proper length is obtained. A hole is blown somewhere in the side of the tube, and the glass is strongly heated at one side of this hole and pulled away with forceps. This process is continued until the glass has been removed from one whole side of the tube. The ends are then worked with forceps and tweezers to give the final boat. This method avoids both the use of a cutting wheel, and the cracking of a tube along its length, an operation which does not always go well because the crack may wander in the wrong direction.

Condensers

The construction of a water-cooled condenser is a good example of a case where all the parts must be prepared, and at hand, before the final assembly is begun. There are several methods commonly in practice for setting about the construction; FIGURE 55 shows one of these.

Lengths of tubing are chosen of suitable diameter for the inner tube and the jacket. An internal seal (ring seal) is formed at one end and a side arm, bulged to take rubber condenser tubing, is immediately

added near to the ring seal while the latter is still hot. Thus, after annealing, the stage shown in FIGURE 55, *II*, is reached. Then, at a point giving the required length of the condenser, the outer tube is collapsed on to the inner tube and the two are pulled off together

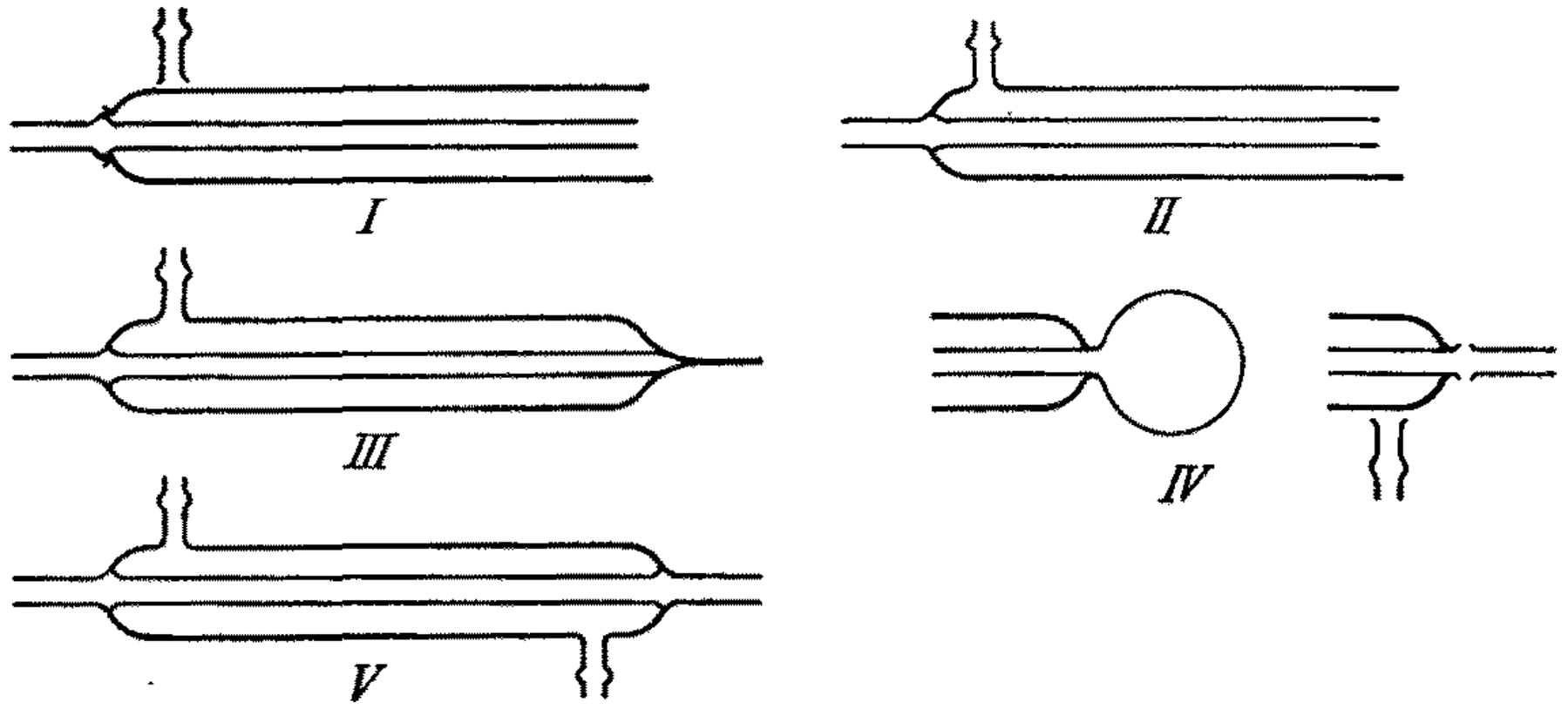

FIGURE 55. A method of making a condenser

(see p. 76) to a spindle (FIGURE 55, *III*). Excess glass is pulled away so that a rounded end is formed. Blowing through the side arm at the far end prevents the outer tube shrinking on to the inner behind the ring seal; while blowing down the central tube allows the end of this

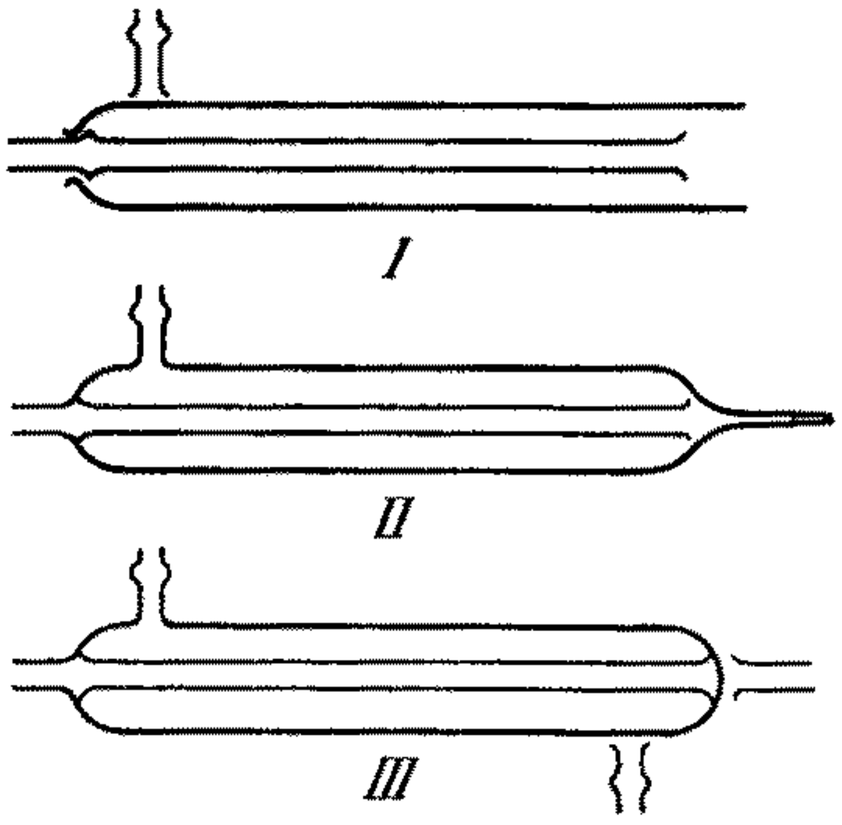

FIGURE 56. A method of making a condenser

to be thinned and finally blown out as shown. A prepared piece is then immediately added to the central tube (FIGURE 55, *IV*) followed by the side arm attached to the water jacket. Careful annealing of this whole end is required to relieve as much strain as possible from the apparatus. The finished condenser is shown in FIGURE 55, *V*. If

the outer tube has subsequently to be heated for any further glass blowing, very careful warming is required because, at either ring seal, the outer tube heats more quickly than the inner and therefore tends to draw away from the latter.

A second method of construction is to form the first ring seal and side arm from the prepared parts shown in FIGURE 56, *I*. The inner tube is slightly flared at the far end from the ring seal. In the second stage of the construction the outer tube is drawn off so that the shoulder is very close to this flared end (FIGURE 56, *II*). The shoulder

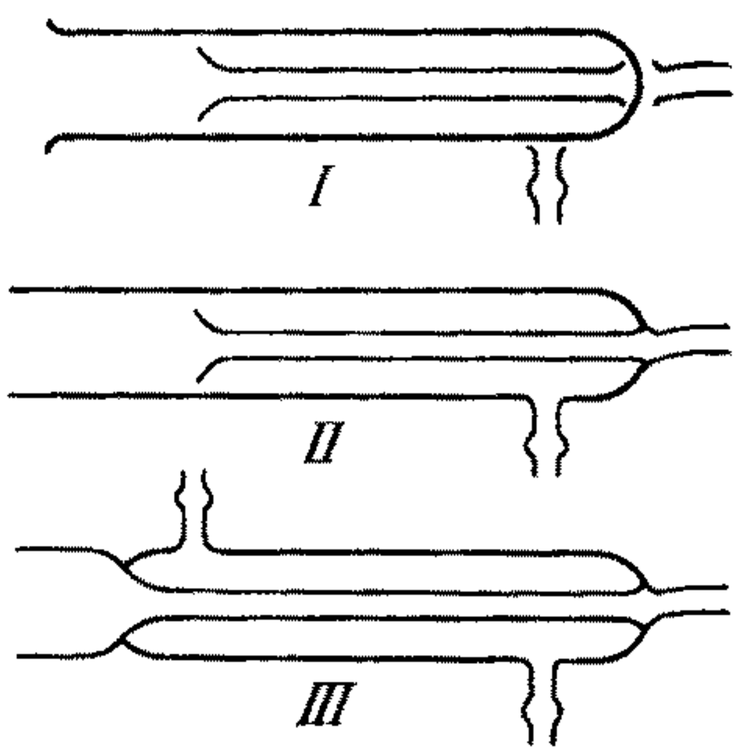

FIGURE 57. A method of making a condenser

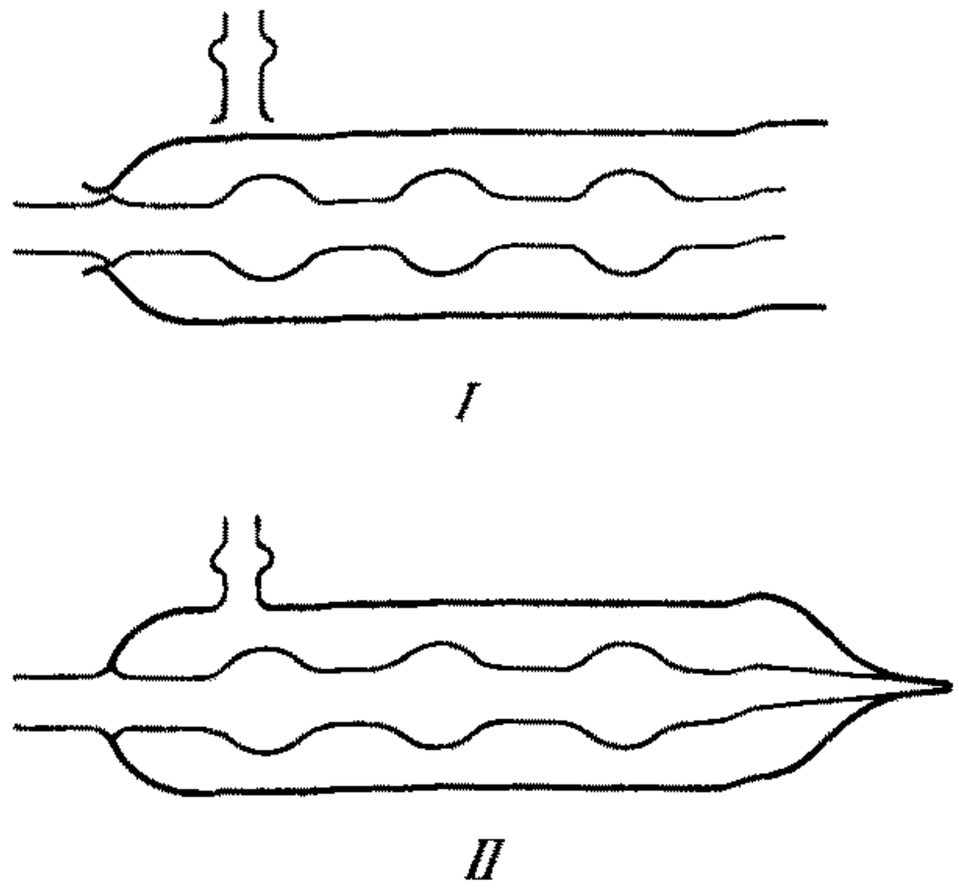

FIGURE 58. A condenser with a bulbular inner tube

is shrunk on to the inside flare and, again with blowing through the far side arm, a ring seal is formed. The condenser is then finished as before.

In the third method the outside tube is rounded off at one end and the inside tube prepared with a large flare and a small flare at opposite ends as seen in FIGURE 57, *I*. A butt ring seal is first formed at the closed end of the outer tube. This is blown out as shown in FIGURE 55, *IV*, a piece added to the central tube, and a side arm added to give the stage represented in FIGURE 57, *II*. In the last stage the outer tube is shrunk on to the inner flare forming a large ring seal and the second side arm added. This method gives a finished condenser as shown in FIGURE 57, *III*.

If the internal tube is required to be of some special shape, bulbular or in the form of a spiral for example, then the best method of approach is probably the first method described above: the inner tube is first shaped as required and the outer jacket made to fit as in FIGURE 58, *I*. The ring seal can be formed at one end and the side

arm added to give the stage shown in FIGURE 58, *II*. Finally both tubes are drawn off at the far end to give a ring seal and the condenser is finished as in FIGURE 55, *IV* and *V*.

Fractional Distillation Condensers

A condenser of the type shown in FIGURE 59 is often called a fractional distillation condenser and was advocated by H. Jackson, Professor of Chemistry at King's College, London—hence the condenser is sometimes called a King's College condenser. It can be used as an ordinary condenser and as a reflux condenser. To make it, a

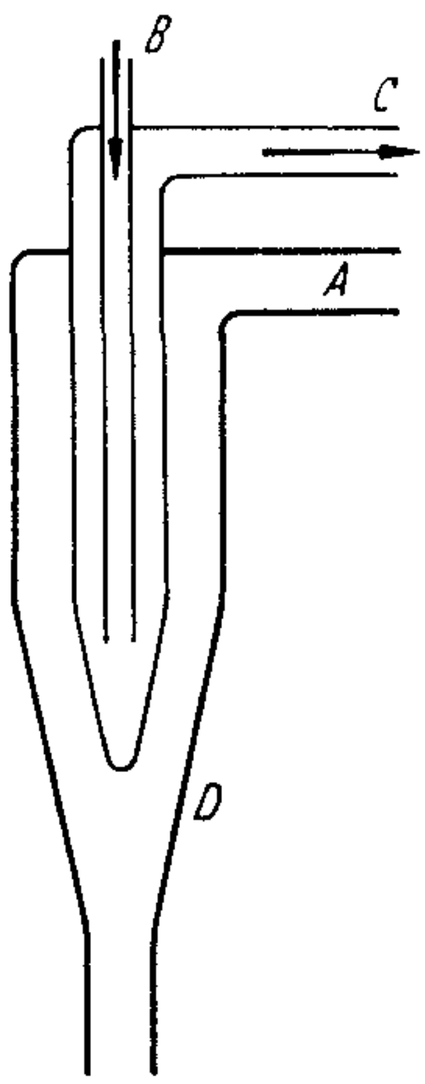

FIGURE 59. A fractional distillation condenser

piece of 3-cm diameter tubing is joined to 8-mm diameter tubing by a taper (*D*). 1·5-cm diameter tubing, for the water circulation, is drawn out to a taper at its end, and closed. 8-mm diameter tubing is prepared for the side arm *A* and 5 mm tubing for the water inlet and outlet tubes *B* and *C*. The 3 cm tubing is rounded off and a hole blown just large enough to let the 1·5 cm tube slide into it. A normal internal seal is next made, but in this case the joint must be blown from both ends. The side arm *A* is then added, followed by the side arm *C*. The larger tubing to which *C* is joined is then rounded off, blowing through *C*, and the central tube *B* is inserted in the usual way, but the internal seal is somewhat more difficult to make since the side arm *C* is already present. The whole apparatus requires careful annealing.

Alternatively, the whole water-circulating part can be made separately and internally sealed into the wide tube, using a hand torch, and

blowing from both ends. Another design employs a Dewar seal as in FIGURE 52, *II*, and the water-carrying tubes are inserted with a rubber stopper.

Fractionating Columns

These are devices for establishing contact between ascending vapour and descending liquid in a distillation. A variety of columns can be made. Perhaps the simplest (FIGURE 60, *II*) consists of a support in a tube on which a packing rests, made up of short glass tubes, glass rings or pieces of metal gauze. The support for this packing is made

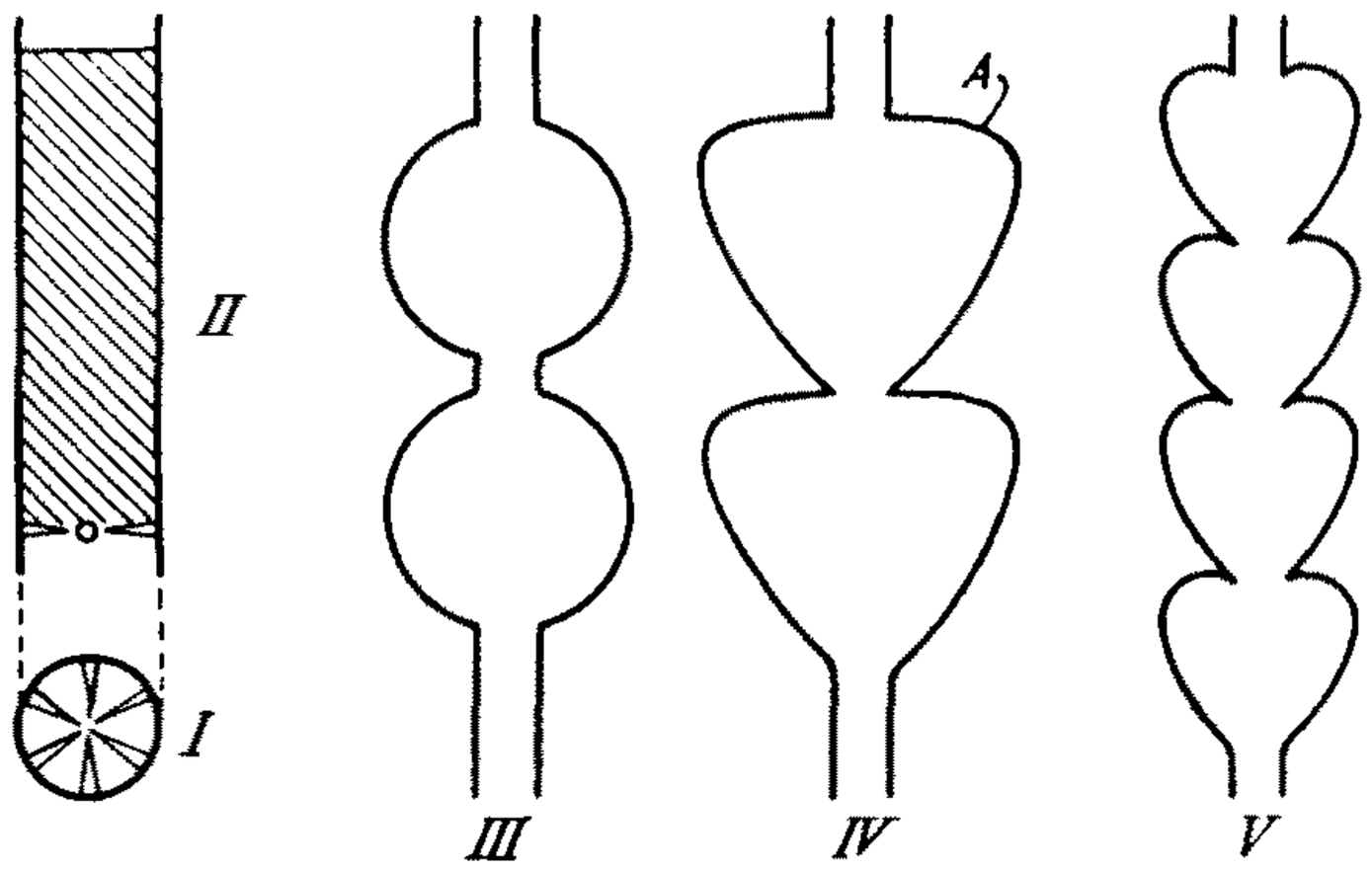

FIGURE 60. Two types of fractionating column

by heating the tube with a small hot flame and pushing the wall in with a spike almost to the centre of the tube; this is done a number of times to give the support shown in FIGURE 60, *I*. A thorough annealing should be given.

Another fractionating column consists of a number of pear-shaped bulbs (FIGURE 60, *V*). A series of bulbs is blown (FIGURE 60, *III*), and their ends are heated and blown and drawn so that pear-shaped bulbs are formed (FIGURE 60, *IV*). The ends of these bulbs are heated (at *A* etc.) and they are gently pushed in. The column should then be thoroughly annealed. The whole sequence of operations can be carried out very easily with a glass-working machine.

A Fractional Distillation Receiver

A receiver for low pressure distillations designed by G. A. R. KON (1930) is quite easy to make: the joints *D* and *C* (FIGURE 61) are both standard B.19 joints; the two taps at *A* and *B* are standard designs of three-way taps. For the part *G* a tube of about 3 cm diameter

is joined at both ends to tube of about 8 mm diameter. One of the small tubes is joined to the tap *B*. To prevent distortion of the barrel of this tap, the joint should be made 1–2 cm away from the barrel. An internal seal is made into the cone *D* and a side arm attached. The top of the internally sealed tube is then joined to the tap *B*. The two tubes *E* and *F* are joined to the tap *A*. The socket *C* is then joined to the larger tube.

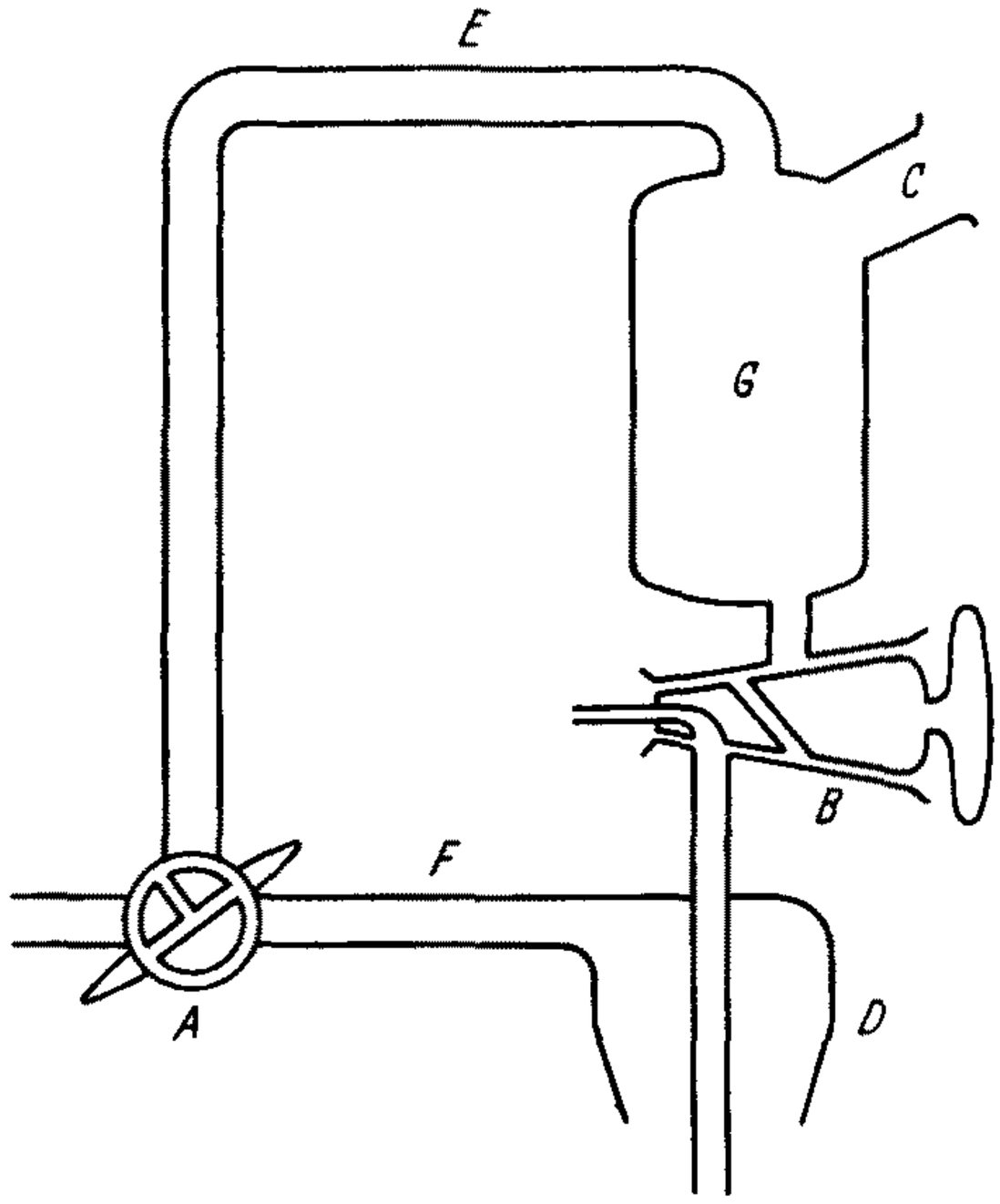

FIGURE 61. A fractional distillation receiver

An advantage of this receiver is that receiving vessels connected at *D* can be taken off and put on without altering the low pressure in the main apparatus. When tap *B* is shut during the operation of changing a receiver the distillate collects in *G*.

Double Surface Condensers

A simple form of double surface condenser that can be made without too great difficulty by an unskilled worker is shown in FIGURE 62, *IV*. The parts seen in FIGURE 62, *I*, must first be prepared.

The ring seal at *A* is made in the early stages and is partially annealed. This part of the assembly is then inserted into the prepared tube *B* which is held clamped, and, when seated well, is itself clamped at *C* (FIGURE 62, *II*). Using a hand torch and exerting care to warm

up the whole area slowly and uniformly the ring seal at *D* is now made by tooling, a little at a time, the glass flare of tube *B* on to the

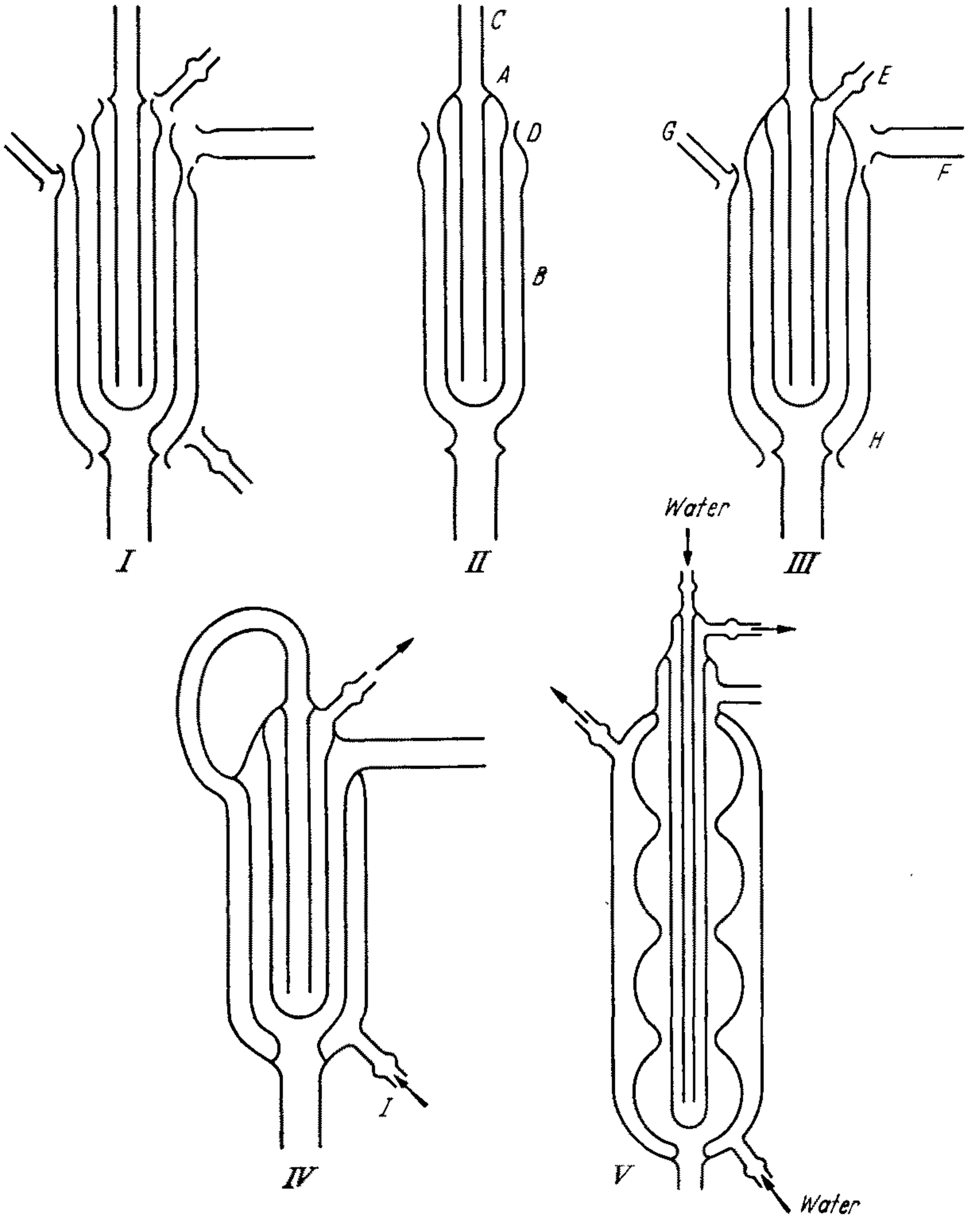

FIGURE 62. A simple form of double surface condenser

bulge of the inner tube. Care must be taken to keep the whole of both ring seals hot while this is being done; a large flame should therefore be used and only reduced to a sharp hot flame for the final blowing out. The water outlet tube *E* is added while the whole area is hot.

This tube can be held by hand and a pin-point flame used to seal it in position. Every few seconds the flame size should be increased and the far side of the assembly re-warmed before continuing the sealing of *E* in position. Finally the whole section must be very carefully annealed.

The assembly may now be clamped ready for the final large ring seal as in FIGURE 62, *III*. For this operation, and to a lesser extent for that described in the previous paragraph, it is advisable to have a second worker, also with a hand torch, who can keep the far side of the apparatus hot while one side is being worked. Extra careful warming up is required at this stage before the large final ring seal can be made. The warming should be commenced with hot air for about 10 minutes, then with the tips of large luminous flames and finally with slowly increasing oxygen content. It will be found with such a procedure that the initial double ring seal will survive without cracking. The large ring seal is then worked in the same manner as the seal *D*, and, with the entire section kept hot, the side arms *F* and *G* are added. This region with three ring seals needs extremely careful annealing. We have found, however, that flame-annealing is adequate.

The completion of the outer water jacket, with ring seal at *H* and water inlet tube *I*, is relatively straightforward. The tubes *G* and *C* may be joined with a short piece of condenser tubing, though a more elegant finish to the condenser is achieved if they are bent and sealed to give a closed circuit as in FIGURE 62, *IV*. If this is done the upper ring seals must be very carefully protected with asbestos paper, but otherwise the procedure is again straightforward.

The design of this double surface condenser can be changed as desired; for example, FIGURE 62, *V*, shows a double surface reflux condenser which may be made by modifying the above general procedure.

The type of double surface condenser most often found in laboratories is the 'Davies improved double surface condenser' which appeared in 1905, and was designed by J. Davies of A. Gallenkamp & Co. as a direct development from Thresh's modification of the Bidet condenser. All rubber bungs and tubing were eliminated in the Davies condenser, which has retained its popularity for 50 years. To make a condenser of this kind the inner water jacket is connected to the outer one by two internal seals which are made by a slight modification of Method 2 described on p. 75. The rest of the assembly is similar to that for an ordinary Liebig condenser.

Mercury Cut-offs

Mercury cut-offs are used in vacuum apparatus instead of taps when tap grease is undesirable. A frequently used type is shown in FIGURE 63. A two-way tap is attached at the top of a reservoir for mercury.

The internal seal at *A* can be made with capillary tube extending right down into the reservoir, but this necessitates very careful annealing. It is easier to first join the capillary to normal tubing of equal external diameter, and then to seal this normal tube into the larger tube. The top part of the cut-off consists of an internal seal at *B* and a side arm. This part is connected to the reservoir by about 70 cm of capillary

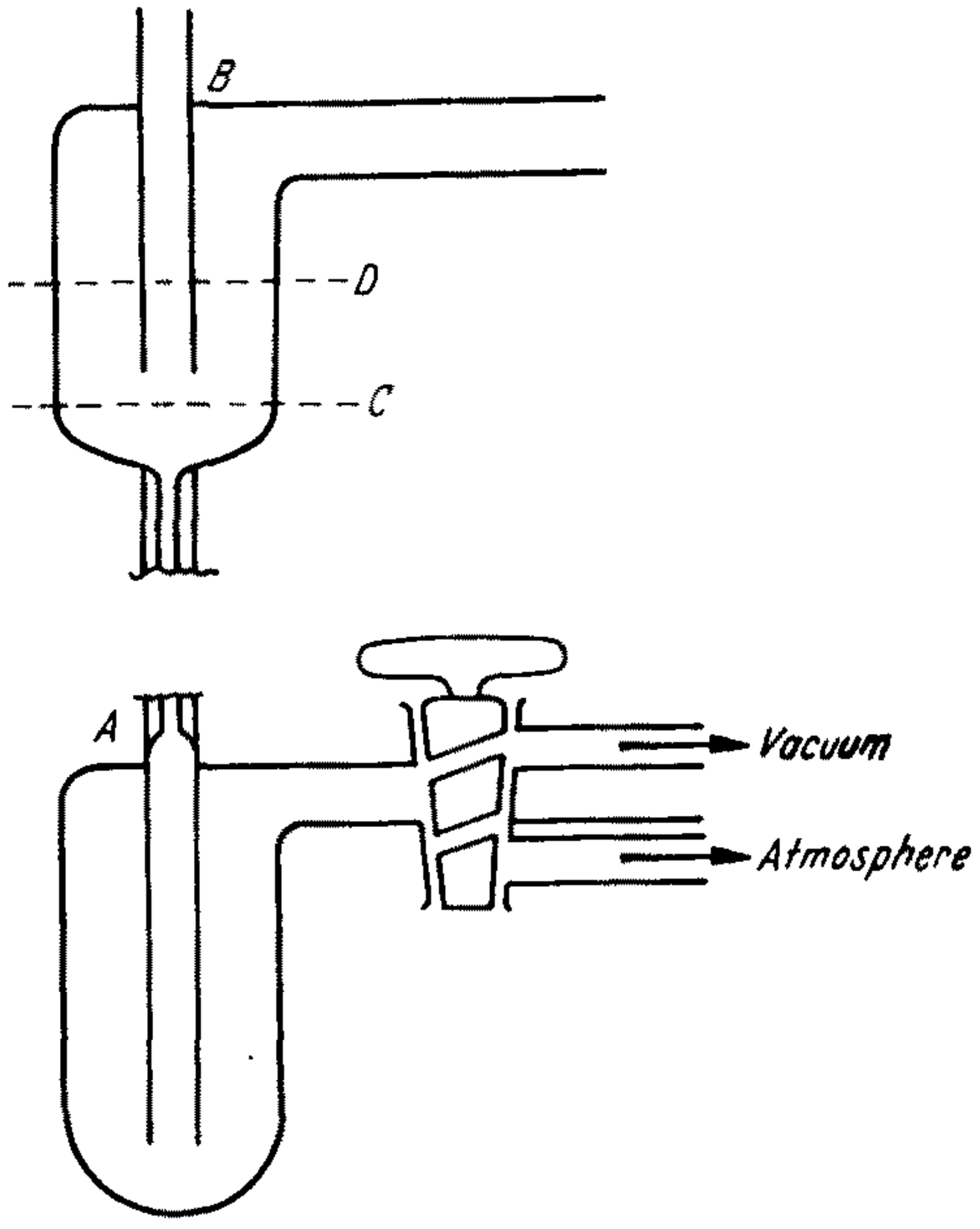

FIGURE 63. A mercury cut-off

tube, which gives strength and reduces the volume of mercury required. To use the cut-off the mercury level is raised and lowered between *C* and *D*.

W. E. BARR and V. J. ANHORN (1949) describe a mercury cut-off with mercury return lines which prevent mercury surging into a vacuum system.

McLeod Gauges

The McLeod gauge is one of the oldest instruments for the measurement of vacua and it has remained virtually unchanged since its introduction in 1874 (H. MCLEOD, 1874).

A simple bench type McLeod gauge is shown in FIGURE 64, *III*. Before starting to make such a gauge an estimate should be made of

the volume required to give pressure readings in the required range: a gauge with a volume of 300 ml and using 0·75-mm bore capillary may be used to read pressures of 10^{-5} mm of mercury with reasonable accuracy, but if relatively high pressures are to be measured it will be more satisfactory to use wider bore capillary, say 2 mm. This avoids difficulties encountered with mercury sticking in the capillary. The bore of the capillary should be determined accurately by direct measurement along its length to ensure uniform bore—unless

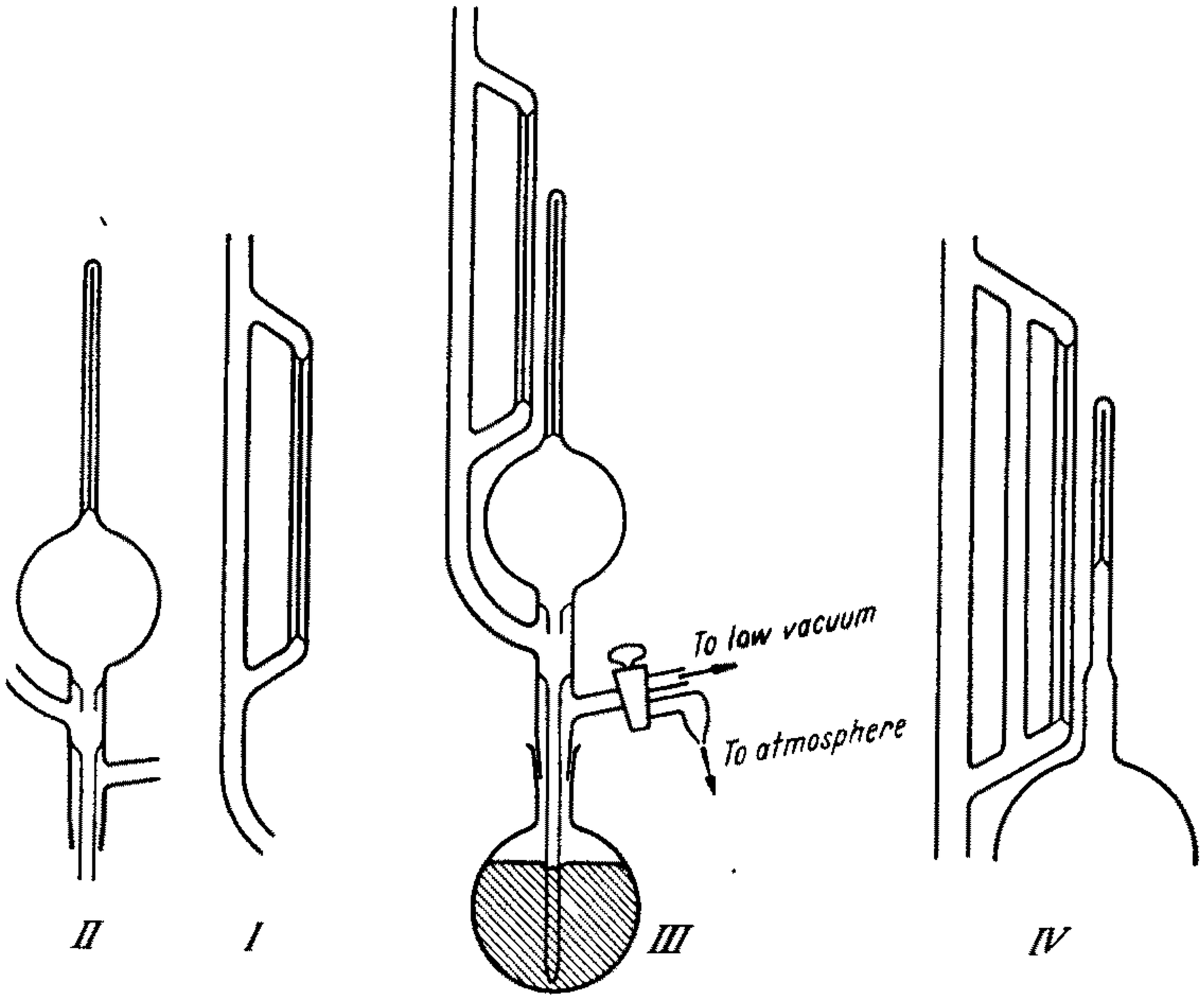

FIGURE 64. A McLeod gauge for bench use

precision-bore tubing is used. This precision-bore tubing may be contaminated by the lubricant used to prevent it sticking to the metal mandrel used in its manufacture, and consequently great care should be taken in cleaning it. Stripping the surface with hydrofluoric acid gives a uniform clean surface if normal cleaning methods fail.

The simplest way of making a McLeod gauge is to make it in two basic sections which are finally joined together. Since the capillary on both parts of the gauge should be identical in bore and in surface properties, it is best to make use of a single length of tubing so that both capillaries are subjected to identical cleaning procedures. The capillary should be carefully inspected for particles of dirt which may

not have been removed by the cleaning. FIGURE 64 shows a gauge fitted to the mercury reservoir by a standard ground joint; this is usually the easiest method of assembly.

The part in FIGURE 64, *II*, can be made either by blowing a bulb of suitable volume or by using a round-bottomed flask. If the latter is used a slight bulge should be blown centrally in the bottom so that a sharp shoulder is avoided at the capillary seal. A 15–20 cm length of capillary is sealed to this bulge and its upper end left open. This capillary can then be used as a handle and blowing tube for the subsequent work on the neck of the flask. The neck of the flask should then be drawn down, a smaller tube joined to it and cut off short to form the inner tube defining the cut-off volume. Alternatively, the cut-off volume can be defined by sealing a flanged tube into the neck of the flask, which then does not have to be drawn down. The standard cone is then joined to the mercury supply tube by a ring seal to the flared out end of the tube. This part is then joined to the end of the flask as shown, allowing sufficient room between the two seals for the attachment of the side arm to the vacuum system. The two side arms should be inserted while the glass is still hot from working the internal seals and the whole section annealed thoroughly.

The next step is the calibration of the cut-off volume: this can be done by filling with water and weighing the volume contained in the capillary and bulb up to the cut-off point; for small volumes mercury is a better medium but becomes unmanageable for larger volumes. Once a satisfactory volume calibration has been obtained the end of the capillary is closed to a square end. This can be easily achieved by drawing down a piece of glass rod until it just fits into the bore of the capillary and cutting a short length of this rod to plug the end of the capillary before fusing it. If this is done carefully the capillary will be closed with a square end and the walls of the tube will not be distorted at the end of the gas column, which introduces uncertainties in reading this position. It is important to avoid lens effects here if accurate readings are to be obtained.

The part of the gauge in FIGURE 64, *I*, is simply constructed by joining lengths of normal tubing to either end of the length of capillary, bending these to a suitable angle and blowing out the ends. One end of this composite tube is then blocked with a stopper, and the other end joined to the main tube at an angle by a normal T-joint. A hole is then blown in the main tube for the second T-joint and the shape of the bends in the tubing is adjusted by gentle heating and bending until the end is brought into close contact with the hole. This joint is then completed. The parts *I* and *II* (FIGURE 64) are then joined so that the two capillaries are parallel and a few millimetres apart.

A useful safety precaution is to draw the end of the tube dipping into the mercury reservoir down to a capillary to retard the rate of flow of mercury into the gauge if excessive air pressure is applied accidentally. The drawing down of the air inlet on the two-way stop-cock makes the gauge much easier to operate. Various other methods have been used for raising and lowering the mercury in the gauge but the most convenient and most widely used is that shown—the use of atmospheric pressure and a low vacuum which can be supplied by a normal water pump if necessary. If this method is used it is advisable to have the high vacuum connection at more than atmospheric height above the mercury reservoir.

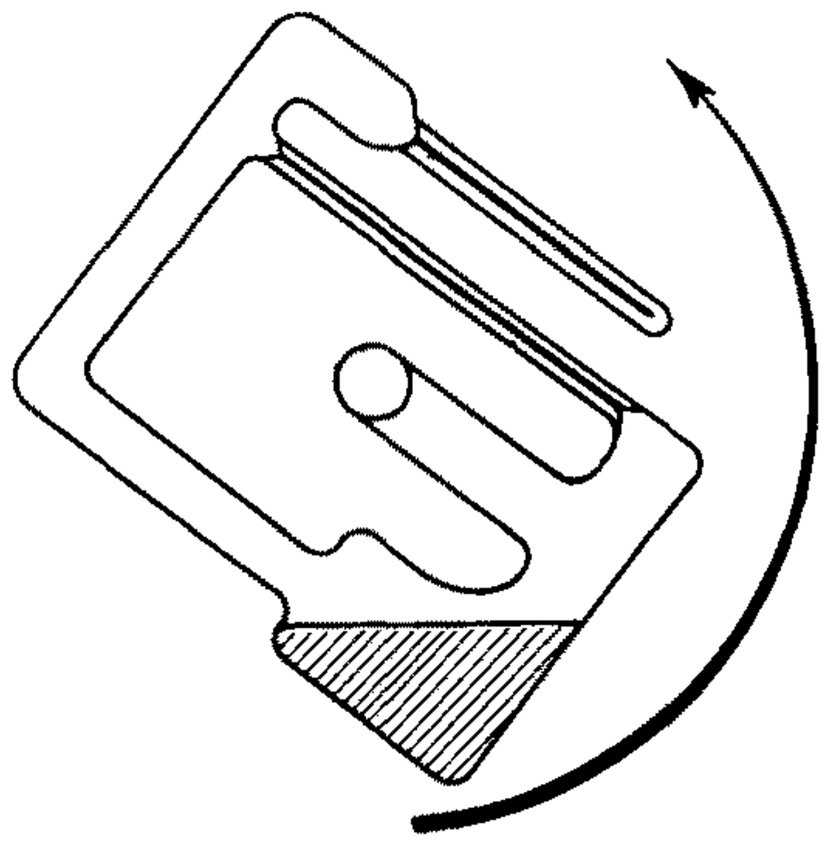

FIGURE 65. A small rotating type of McLeod gauge

The range of a McLeod gauge may be extended by using various sized tubes in conjunction with a small diameter capillary and similar sized compensating tubes (L. DUNOYER, 1926). In this way comparatively high pressures may be determined on the same gauge as may low pressures. The calibration technique is more complex as the volume is not a simple fraction of the length of the compressed gas column (see FIGURE 64, *IV*).

Small McLeod gauges have been developed for rapid pressure measurements: they avoid the tedious raising and lowering of a large volume of mercury by the gauge rotating about a ground glass joint in the high vacuum connection. A gauge of this type is shown in FIGURE 65. The volumes used are relatively small and can be made conveniently from short lengths of tubing; the reservoir bulb should be large enough to permit free passage of gas into the gauge when all the mercury is in the reservoir. The volume of mercury required is determined by the volume of the bulb and the volume of the tubing

—the mercury level in the compensation capillary should be level with the top of the closed capillary when the gauge is in a vertical position and under vacuum. These gauges are best calibrated against a normal type of gauge.

Traps to catch Mercury displaced in Apparatus

When parts of an apparatus contain mercury, as in cut-offs and McLeod gauges, it very often happens that air rushing in or out of the system will displace mercury in an undesirable manner. With cut-offs across a differential pressure, bubbling of gas through mercury is unavoidable, and in tubing of internal diameter less than 3–4 mm bubbling will not occur but the gas will raise a column of mercury in front of it. It is therefore desirable to insert anti-splash traps wher-

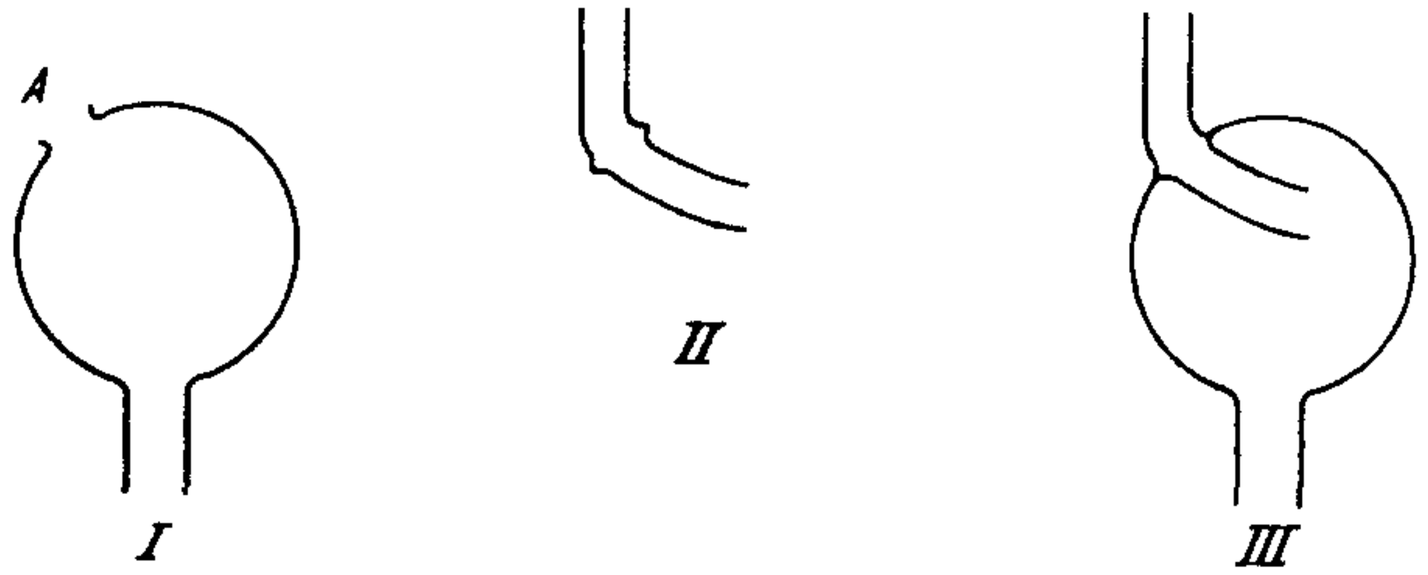

FIGURE 66. Trap for catching displaced mercury

ever bubbling is likely to occur. A simple form of trap is shown in FIGURE 66, *III*.

In the construction a bulb of the required size is first blown and then a bulge is formed at *A* (FIGURE 66, *I*): the tip of the bulge is blown out resulting in a hole which will just take the size of tubing being used. A short piece of tubing is bulged and bent to shape (FIGURE 66, *II*) and a ring seal is formed at *A*. Care must be taken to see that the lower end of the inside tube finally slopes downwards so that drops of mercury will not lodge in it.

Valves

Valves are of two kinds: those meant to close a system as far as possible, and those designed to operate as check valves which reduce the flow through a system.

A simple and effective check valve which impedes the flow of mercury is seen in FIGURE 67, *I*. It consists of a small plunger floating on mercury, which is pushed into a seating when the mercury level rises. The plunger *P* is made by blowing an elongated bulb at the end of a

tube and then drawing out a neck. The bulb is about half filled with mercury and the neck sealed off. The tube *C* is joined to the tube *A* and the edge of the join at *B* is heated in places and pushed in to form a few spikes to hold the plunger (FIGURE 67, *II*). The plunger is then inserted in *C*; the end of *C* is rounded off and *D* is joined. This must be done with the tubing held vertically, or nearly so, to prevent the plunger coming in contact with the hot glass; the joint can be made very easily with a hand torch.

Valves that are to close a system must have two surfaces which make intimate contact; this is readily effected by grinding the plunger

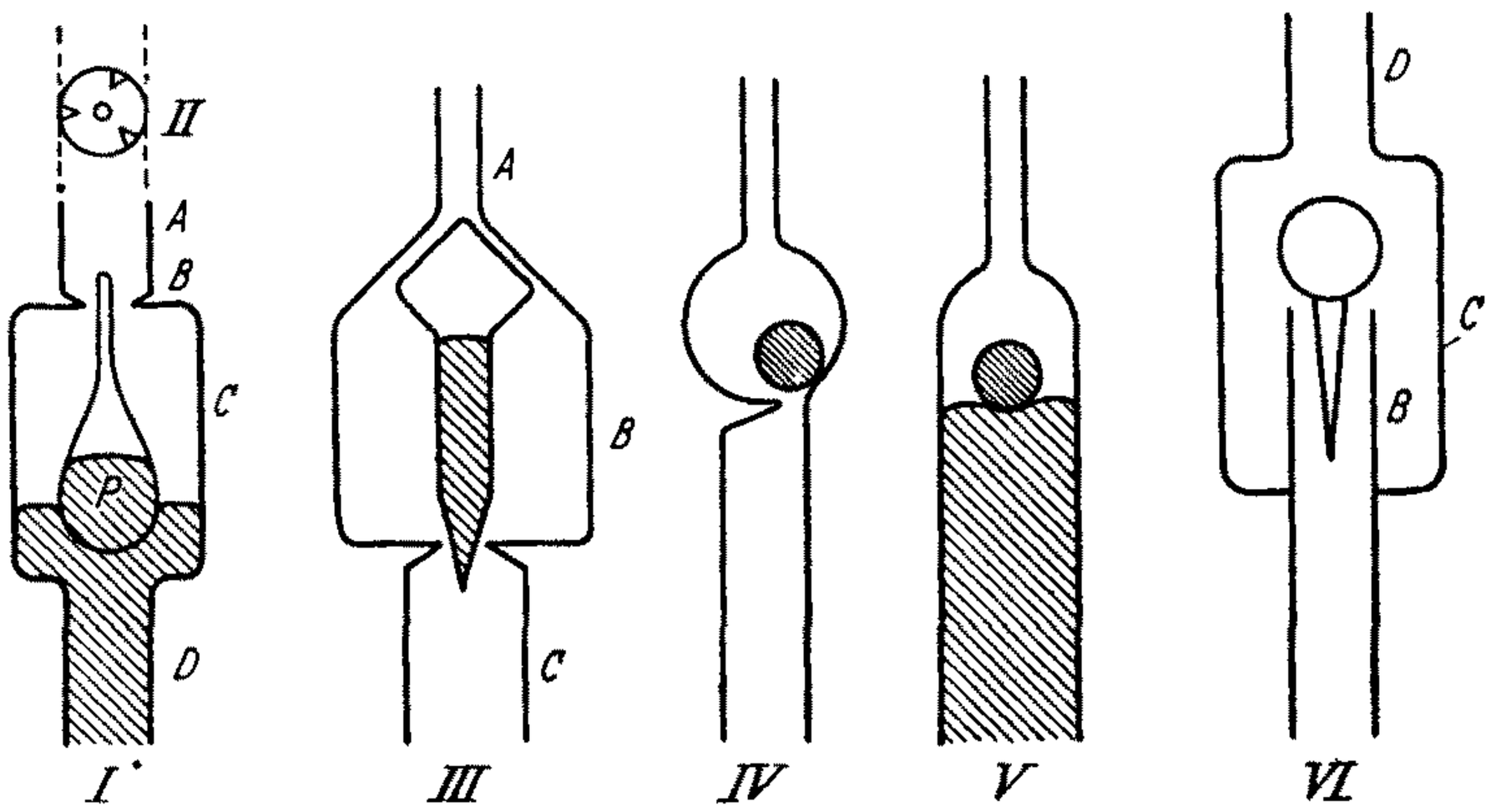

FIGURE 67. Some kinds of valves

into its seating. A simple design is illustrated in FIGURE 67, *III*: the plunger is made by blowing a bulb on the end of a small tube, heating the end of the bulb, touching it with a piece of hot glass, and pulling out into a conical shape. The plunger must be able to pass down the tubing to be used as *C*. The tubing *A* is joined to *B* by a taper with an angle equal to that of the head of the plunger, and the plunger is ground into this taper with fine carborundum until a continuous ground band 1–2 mm wide is formed on the glass. The cone is then washed and dried, and *B* is joined to *C*. The end of the plunger is tapered as shown, mercury is added, and the plunger is sealed off and put into *B* through *C*. The junction of *C* and *B* is then pushed in at a few places so that the glass spikes just allow enough freedom of movement of the plunger.

Instead of a glass plunger a ball bearing may be used, as in FIGURE 67, *IV* and *V*. In both of these examples a larger tube is joined to a smaller tube and the joint is ground to fit the ball bearing. The

grinding can be carried out with the ball bearing or with a glass grinding tool of diameter equal to that of the ball bearing. This tool is a glass rod with a sphere made on the end by rotating the end in a hot flame. The glass sphere is ground into the joint, and since both surfaces are ground away a slightly tapered seating is produced which is better than that produced by direct grinding with the ball bearing. The ball bearing can float on the mercury as in FIGURE 67, *V*, or be kept in place by a small indentation in the glass (FIGURE 67, *IV*) made by pushing it in with a spike. The former valve is closed by the mercury rising, and the latter may be closed by mercury also, or by a great enough flow of gas or liquid pushing the ball bearing into place. In this case the bulb shown in FIGURE 67, *IV*, may be replaced by a tube just bigger than the ball bearing.

Another form of valve which we find useful is shown in FIGURE 67, *VI*. To make the plunger a sphere is made on the end of a glass rod, and to this sphere a thin glass rod is attached, using a very small flame so that the sphere is not distorted. The sphere is then removed from the larger glass rod with a small and very hot flame, and it is rotated with the thin rod until it is spherical. The glass sphere is next ground into the end of the tube to be used as *B*; this can be done easily by rotating the tubing with a lathe chuck and keeping the sphere stationery. The tube *B* is then internally sealed into *C*, the plunger is put in and finally *C* is rounded off and *D* is added. The tail of the plunger should be long enough to prevent the plunger coming out from the tube *B*. A valve of this kind is useful for liquids and gases; it is opened by pressure of the fluid in *B*, but flow cannot occur from *D* to *B*.

Soxhlet Extraction Apparatus

The apparently complex Soxhlet apparatus (FIGURE 68, *III*) is made by three simple operations: first the large tube *A* is joined to the lower main tube *H*, then the vapour bypass is fitted, and finally the liquid siphon is put on. The main tube is usually made from 3-cm diameter tubing about 15 cm long, and this is joined to 1·5-cm diameter tubing *H*. There is not a continuous tube when these are joined, and hence the joint must be blown from both ends. The joint is made by touching the small tube against the rounded end of the large tube, and then directing the flame against the smaller tube, which is shrunk and blown a few times, and then straightened out. Directing the flame against the smaller tube prevents the larger tube becoming distorted. A hand torch can be used for this joint.

To make the vapour bypass, 1-cm diameter tubing is bent with two right angles, and the ends are cut off so that the tube just fits at *C* and

D and is parallel to the larger tube. Holes are then blown at *C* and *D*. One end of the vapour tube is closed with a stopper and the joint *D* is made; then, after removing the stopper, the upper right angle bend of the vapour tube is heated and the tube pushed until the lower end meets the hole at *C*. The end of the vapour tube is then heated and pushed with a spike on to *H* until there are only a few small holes; the joint is finished in the usual way. The top angle is then made smooth and the whole annealed (FIGURE 68, *I*). It is advantageous to have a bunsen burner handy with which the top joint is kept hot while the lower joint is made.

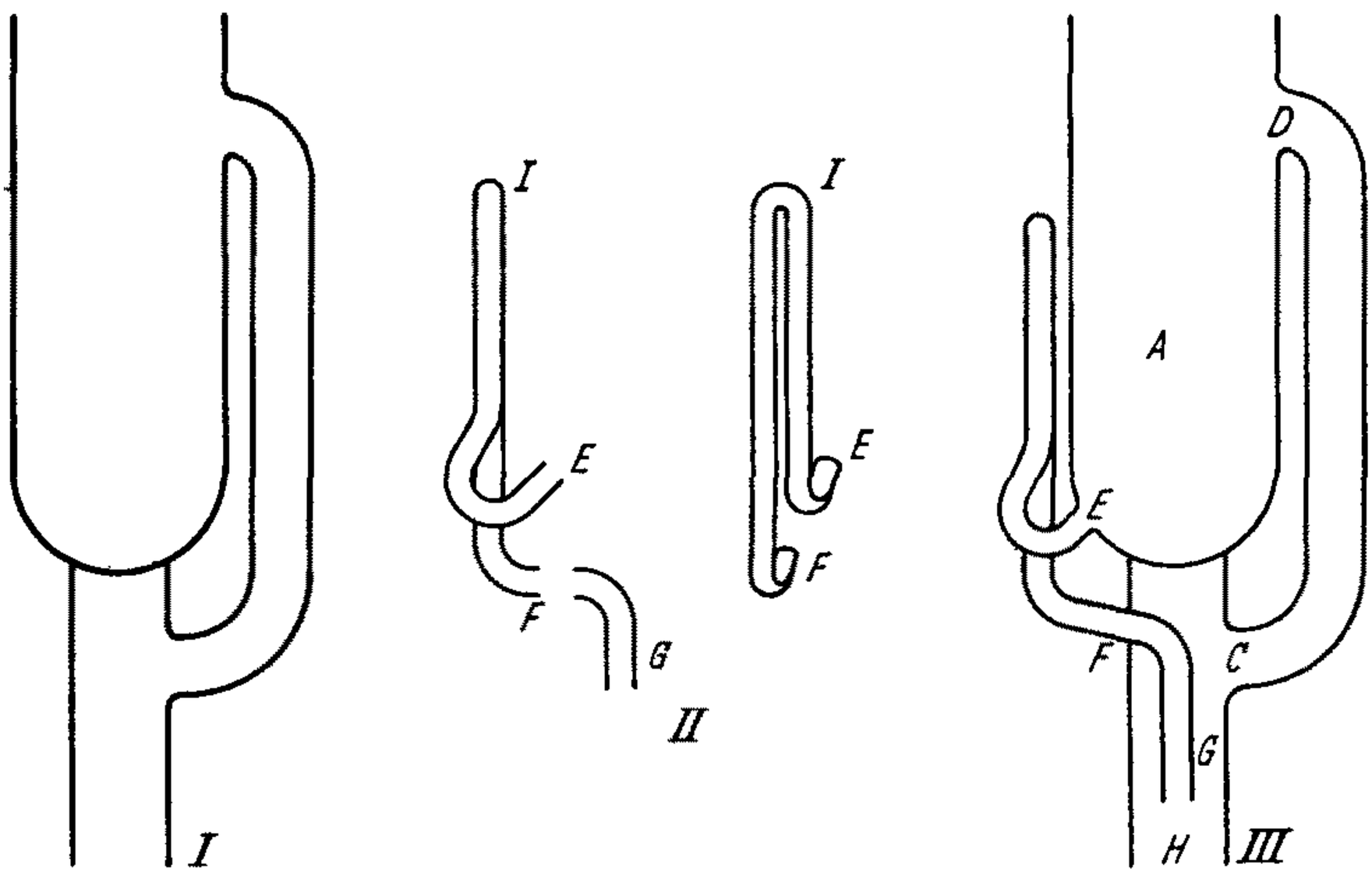

FIGURE 68. A Soxhlet extraction apparatus

For the siphon tube some 2–3-mm diameter tubing has a circular bend made at one end through about 180°, and then about 8 cm from this bend the tube is bent sharply again through 180° (FIGURE 68, *II*). The straight end of the tube is bent about 3 cm below the end of the circular bend, into a right angle in line with the circle. The end is cut off at *F* so that *E* and *F* will meet the larger tubes as shown. Another piece of the small tube is bent into a right angle to make *G*. This piece is internally sealed into *H*, and a fine hot flame is directed against the centre of the join, which is blown out to a bubble. *G* is made central, and the siphon tube *FIE* is joined to *G* by the appropriate internal seal procedure: *E* is closed with a stopper. This is removed, a hole is blown in the large tube *A* (for joining on *E*) and, with *F* heated, *E* is manoeuvred into position. The joint is then made with the help of a spike as usual. *F* is then made smooth and the whole annealed.

In making the second joint *E* it is helpful if the ends of the tube to be pushed to a hole with a spike are thickened and slightly flanged before the first joint is made. This is especially the case for small diameter tubing. The holes blown for both the bypass and the siphon should be so placed that both side tubes are parallel with the main tube.

Mercury Vapour Pumps

Mercury diffusion pumps are standard pieces of laboratory equipment and, for most cases, quite adequate ones can be constructed from glass. In general, *Pyrex* glass (or similar glass) is necessary because hot mercury vapour circulates and differential strain is

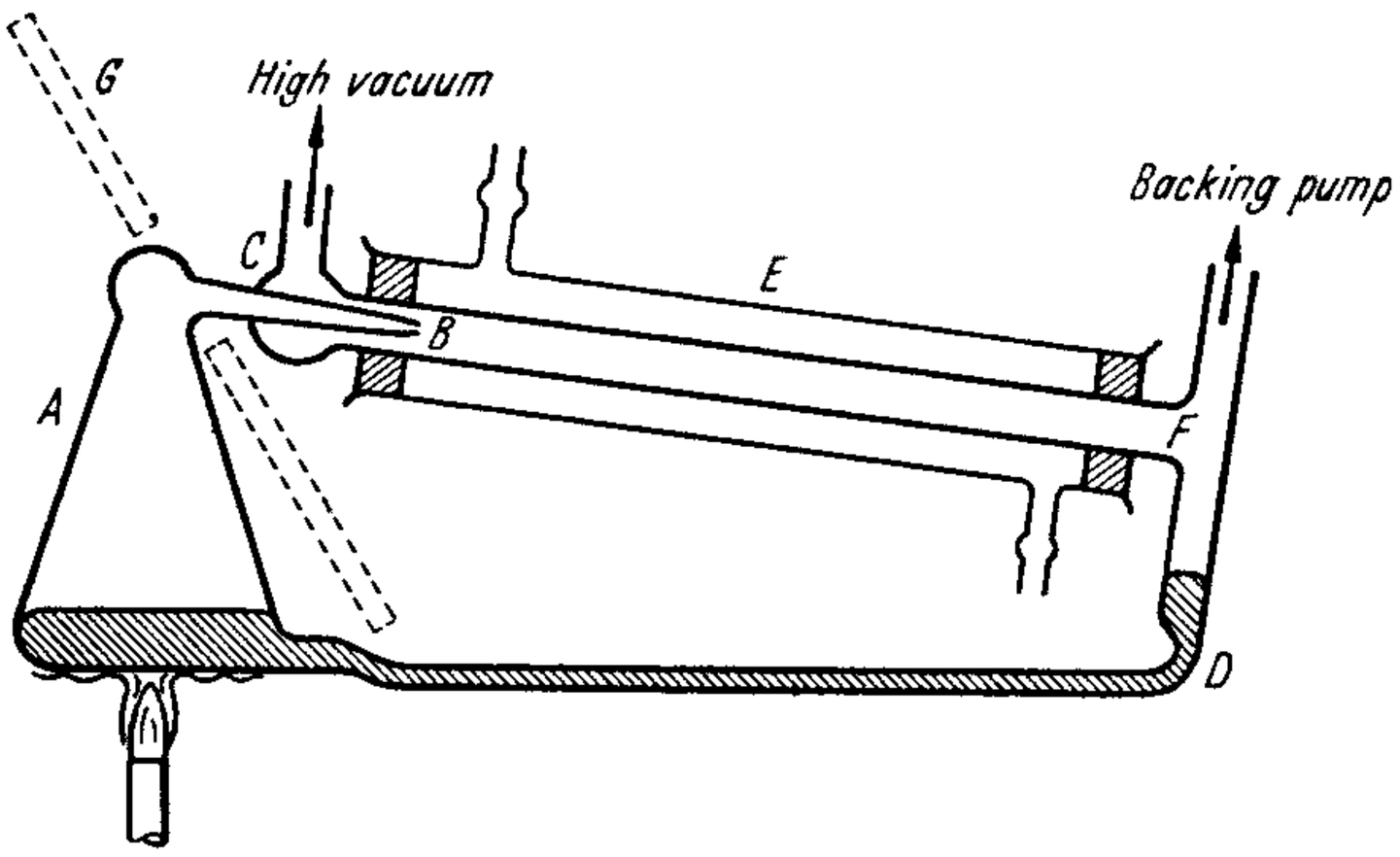

FIGURE 69. A simple diffusion pump

rapidly set up at various, sometimes complicated, joints. Below, however, is described a very simple glass diffusion pump which is quite satisfactory for many purposes, and which can easily be constructed from soda or other glasses.

Very Simple Glass Mercury Diffusion Pump

This pump (FIGURE 69) is due to H. P. WARAN (1923) and is designed such that it may be constructed by the most average of experimentalists. Soda glass is satisfactory because all joints are subject to a minimum, if not a zero, temperature gradient. *Pyrex* glass would, of course, be an advantage but is not a necessity.

The boiler *A* can be made from an ordinary laboratory conical flask: a side arm is added and then the top of the flask is drawn off and rounded—if a laboratory filter suction flask is available, and has sufficient thermal endurance for a boiler, the side arm already exists and need only be lengthened.

The side arm is next bent slightly downwards to form the nozzle *B* which can be tapered as shown if desired. The mouth of the nozzle should be of the order of 5–10 mm diameter, though the size is not critical provided a good rotary, oil, backing pump is to be used. The larger the nozzle the faster the pumping that can be obtained, while the higher the backing pressure employed the finer must be the nozzle and the higher the boiler pressure of mercury vapour.

The ring seal at *C* is made not too far from the flask so that the hot mercury vapour has to travel as short a path as possible. The top of the boiler and the exposed portion of the side arm may be lagged with asbestos rope or paper to reduce loss of heat. The remainder of the pump is easily constructed. The condenser *E* can be any reasonable length, for the length does not affect the pump performance. *D* is a 3–5 mm bore tube for returning mercury to the boiler, and must lie below the level of the mercury in the boiler. The condenser jacket is made separately and is fixed to the inner tube with rubber stoppers or corks which are waxed to give a watertight seal. The jacket must be positioned before the T-junction at *F* is made. If *Pyrex* glass is employed then the condenser jacket may, if desired, be joined to the inner tube with ring seals at either end in the normal manner.

The boiler can be heated with an ordinary bunsen burner but its base should be coated with a $\frac{1}{16}$ in. layer of asbestos paste—this is absolutely necessary with soda glass. It may be desirable to employ an asbestos board *G* for deflecting radiant heat from the boiler, thereby protecting the ring seal and the waxed cork of the water jacket.

Waran reports that an average pump of this design, working against a backing pressure of less than 1 mm of mercury, will pump a 1 litre volume at atmospheric pressure down to 10^{-3} mm of mercury in 2–3 minutes. When a rotary backing pump is not available, a diffusion pump with a 5 mm nozzle can be operated, with less efficiency, against a backing pressure of 7 mm which can be obtained with an ordinary water vapour pump. Sufficient heat must be supplied to the boiler, in the latter case, to produce a mercury vapour pressure of 1–2 cm.

Glass Mercury Diffusion Pump suitable for High Vacuum Work

A useful glass diffusion pump, due to J. Pollard, is shown in FIGURE 70, *VIII*. This pump is quite satisfactory for high vacuum work, and our measurements on pumps made by hand torch methods show that a pumping speed for air of about 10 litres/second at 10^{-5} mm of mercury can be obtained at the mouth of a pump of this design. FIGURE 71 shows some results for the speed of this pump communicated to us by J. Pollard.

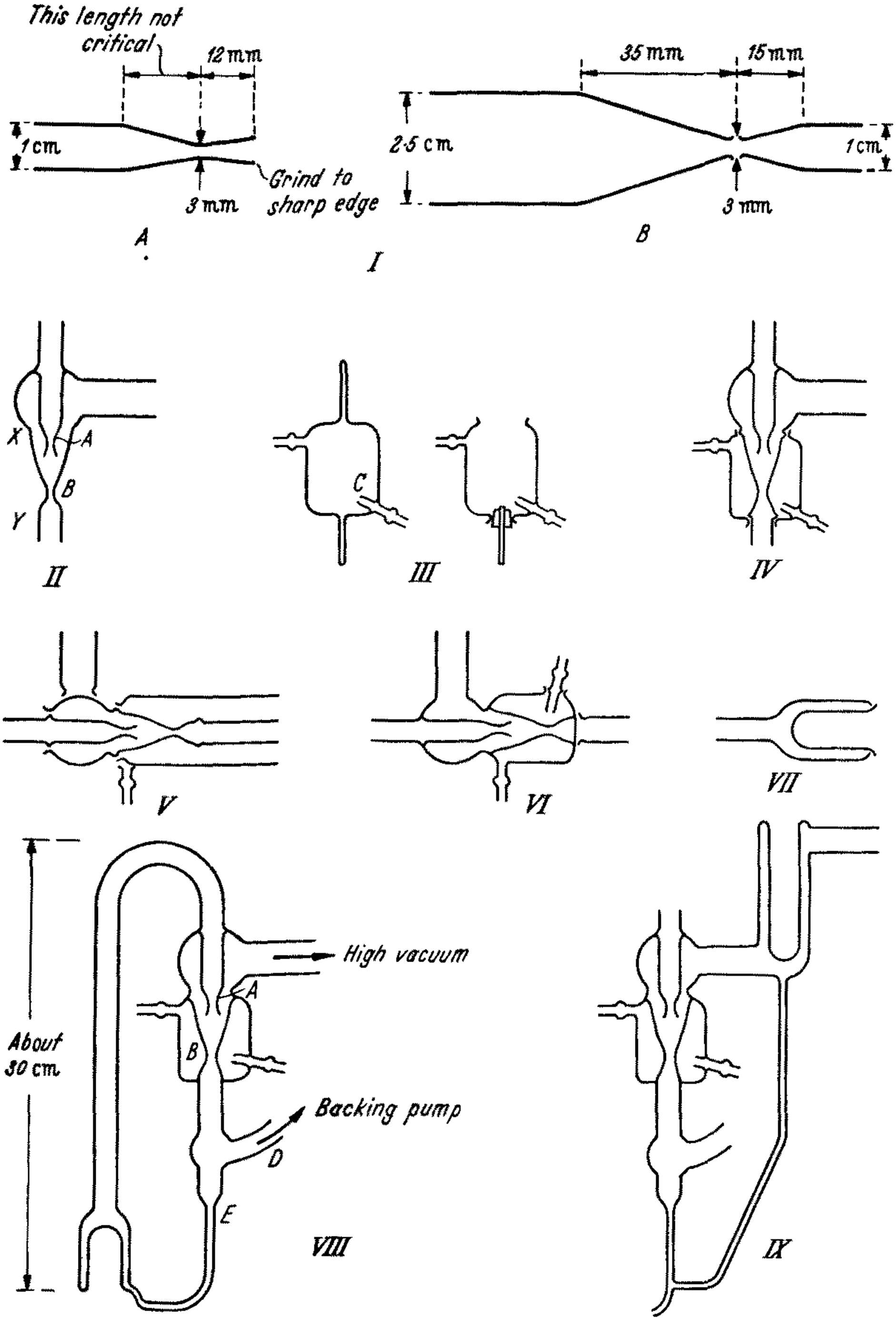

FIGURE 70. A mercury diffusion pump due to J. Pollard

Two 3-mm internal diameter constrictions at *A* and *B* form the pump 'jets'. These tapered jets are made in the first stages of the construction. The small jet *A* is drawn down from 1 cm tubing by careful rotation and shrinking in a not too hot bench flame. Care is necessary to avoid thickening the glass overmuch. The angle of the taper is not critical but should be approximately that shown in FIGURE 70, *I*. The open end of this jet is ground to a sharp edge.

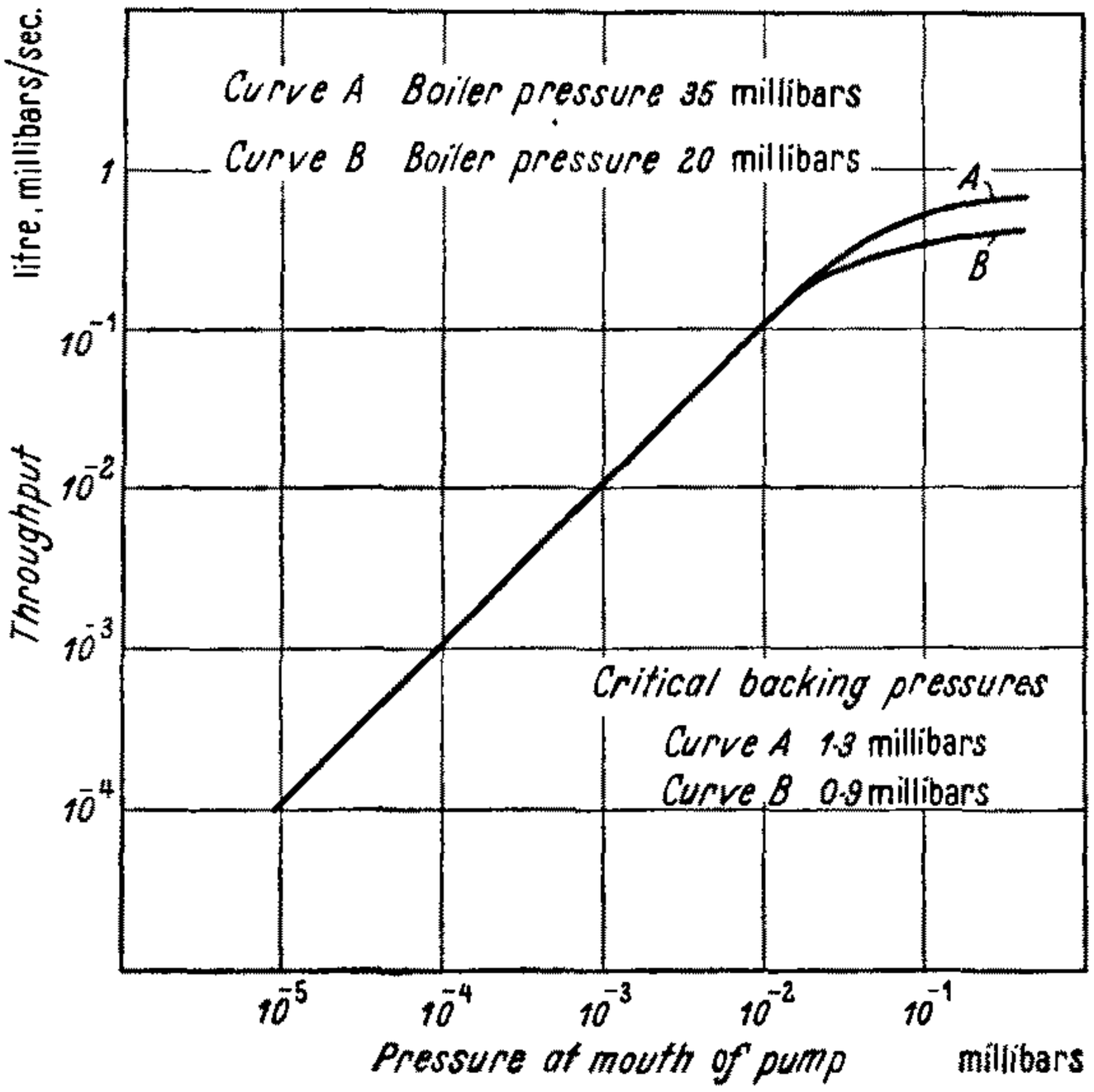

FIGURE 71. The pumping speed of a Pollard type pump

The larger jet *B* is more easily made in two parts. The larger, 25-mm diameter tube is drawn down to the required taper to give a cone-shaped end. As small a hole as possible is blown out at the apex of the cone with a pin-point flame. A 1 cm tube is similarly drawn down and its end blown out to a tiny hole. For joining these two tapers, to give the jet *B*, the tubes are best clamped, since extremely skilful handling is required to avoid buckling if they are rotated by hand. A relatively cool flame will be found adequate for this joint. The joint must be tested for leaks before the pump is assembled (see p. 82).

The larger tubing of the jet *B* is drawn off at the required length, the end enlarged by blowing a slight bulb, FIGURE 70, *II*, and the small jet *A* inserted into place by forming a ring seal as shown. The side arm is added while the seal is still hot and this portion is then annealed. The ground end of *A* should be 35 mm from the constriction in *B*.

Next the water jacket is prepared. A piece of tubing of internal diameter about 40 mm is drawn off to a spindle at both ends to give an intermediate piece of the required length. The two side tubes for in-flow and out-flow of water are added at this stage. The in-flow tube takes the form of a jet *C*, FIGURE 70, *III*, and is inserted by means of a ring seal. At the lower end of the water jacket is blown a small hole to take the 1 cm tube. A rubber stopper carrying glass tube will form a handle at this end for blowing the larger hole needed at the upper end of the jacket. The two jet assembly in FIGURE 70, *II*, is then bulged in the regions *X* and *Y* so that the water jacket fits as shown in FIGURE 70, *IV*. The top upper ring seal can be sealed with plasticine, or wrapped with a long strip of thin rubber sheet, so that blowing is possible for making the lower ring seal. If desired, the upper end of the jacket can be permanently plugged with asbestos paper and sealed with rubber strip or a high melting-point wax. However, by clamping the apparatus, and with careful initial warming, we have found it relatively straightforward to make the large upper ring seal of the water jacket at this stage. We advise a proper seal here because a waxed joint is liable to spring a leak.

A more skilled worker may prefer to do the operations in the order indicated by FIGURE 70, *V* and *VI*. Annealing is then only necessary at two stages: first, when the work of inserting the smaller jet, ring-sealing the upper end of the water jacket and adding the large side arm is completed; and secondly, when the work at the lower end of the water jacket is completed.

When the water jacket is in place, the tubing below the jacket is bulged and the backing pump side arm *D* is added—far enough from the ring seal to cause no strain in the latter. The return drain tube *E* for mercury can now be attached.

The boiler is made separately. The inner tube is chosen to fit the heater to be used, and the outer tube is of such size that the annular space has a volume of 30–40 c.c. to just above the top of the inner thimble. A boiler holding more mercury can also be used.

A piece of outside tubing is first drawn off and attached to a piece of 1·8 cm tube; then the other end of the large tube can be drawn off and blown out at the required length. The inner thimble is shaped separately so that a Dewar seal can be made as shown, FIGURE 70, *VII*. The annular space is usually quite small and care must be taken with this Dewar seal to keep the open end of the thimble from shrinking with the result that the heater cannot be inserted. The side tube for returning mercury is added while the Dewar seal is still hot. The completion of the pump after this stage is straightforward.

The heater can be made from *Nichrome* wire wound in the form of

a fine spiral which is itself spiralled round a cylinder of fire-clay. A thin piece of sheet mica serves to separate the glowing wire from the inner glass of the thimble. A 150 watt heater is adequate. The tube from the boiler to the first jet should be lagged along the whole of its length with asbestos rope.

If desired, a cold finger refrigerant trap can be built in, as shown in FIGURE 70, *IX*, with a draining tube for returning condensed mercury to the boiler. The point of return must be below the mercury level in the boiler so that the high vacuum is separated from the backing pressure by mercury in the draining tube. It has been found desirable to have some form of water-cooled condenser, preferably a double surface condenser, in the backing line *D*. This condenser can be positioned conveniently alongside the pump water jacket, and in this way the tube *D* bends upwards so that mercury condensing in the backing line runs back into the drain tube *E*.

Mercury used in diffusion pumps should be thoroughly cleaned by vacuum distilling. The glassware should be flushed with chromic acid, then rinsed and thoroughly dried before filling with mercury. Any grease left inside the glassware will have a most deleterious effect on the performance of the pump. If rubber tubes are used in the backing line these should be freed of sulphur by boiling in concentrated sodium hydroxide solution.

Electrolytic Gas Generator

A very convenient method of preparing small quantities of pure hydrogen is by the electrolysis of a 30 per cent solution of caustic soda (A. FARKAS and H. W. MELVILLE, 1939). A cell for this purpose (FIGURE 72) can be made simply. The cell itself is H-shaped with the top part of the 'H' elongated. The side piece should be of tubing approximately 2·5 cm bore and about 30 cm long, and the cross piece should be of the same size tubing and about 10 cm long. A narrower tube, 1 cm bore, for filling with the electrolyte extends from the mid-point of the cross-piece, and is closed with a rubber stopper when the cell is in use.

Nickel gauze or thin sheet make satisfactory electrodes, which are suspended from tungsten seals in the top of the sides of the 'H'. The gas outlet tubes should be of fairly small diameter (8 or 10 mm), and should include bulbs of about 20 ml volume to allow the gas to bubble through any electrolyte which is displaced into the outlet tubes by uneven gas pressure. Both sides of the gas generator should be connected to an overflow (see FIGURE 72) which allows excess hydrogen and the unwanted oxygen to escape by bubbling through liquid (dibutyl phthalate is convenient). The gas pressures then remain approximately

the same on both sides of the cell. We have found that this type of generator is not satisfactory when used on a very small scale.

The glass-blowing of the cell may be carried out in the order considered most convenient by the person making it, but we have found it most convenient to make the 'H' first by making one T-joint in a bench torch and then making the second T-joint with the glass clamped, since this makes it easy to get the two sides of the 'H' the same length. This means that the cell will stand firmly on the bench.

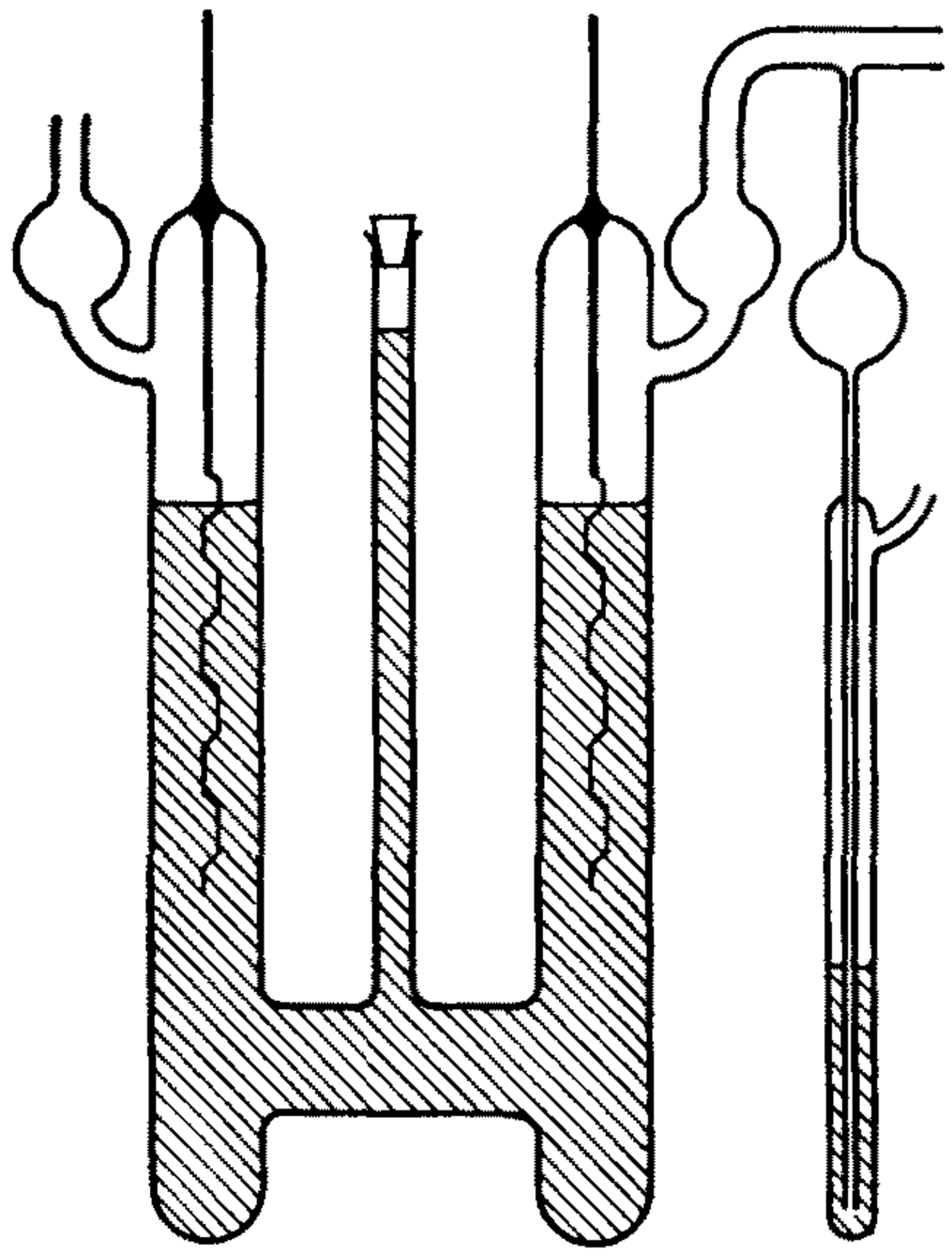

FIGURE 72. An electrolytic gas generator

The filler tube is next attached, followed by the gas outlet tube. The metal–to–glass seals with the electrodes are put in last.

Thermostat Regulators

The regulating device for the heating current of a thermostat may depend on the bending of a bi-metallic strip or on the expansion of a liquid. The latter is more sensitive, and we describe some of these regulators here. An apparatus represented diagrammatically in FIGURE 73, *I*, constitutes the 'make and break' device. When the liquid in the regulator expands, the mercury is pushed up and completes a circuit between E_1 and E_2, and a relay then operates to cut off the heating current. When the circuit between E_1 and E_2 is broken the heating current is turned on again.

For a sensitive regulator the surface area to volume ratio must be high, so that thermal equilibrium is rapidly approached. Toluene (or other convenient liquid) is used as the regulator liquid, with a mercury column for the electrical contact between E_1 and E_2. Various designs giving large surface area have been proposed (see J. REILLY and W. N. RAE, 1954). A bulky design is shown in FIGURE 73, *II*; the tap *T* facilitates filling and the adjustment of the regulator for the required temperature. The apparatus is made from 15-mm diameter tubing. The construction is simple: perhaps it is easiest to make four parts separately and join them between *A* and *B*, *C* and *D*, and *E* and *F*.

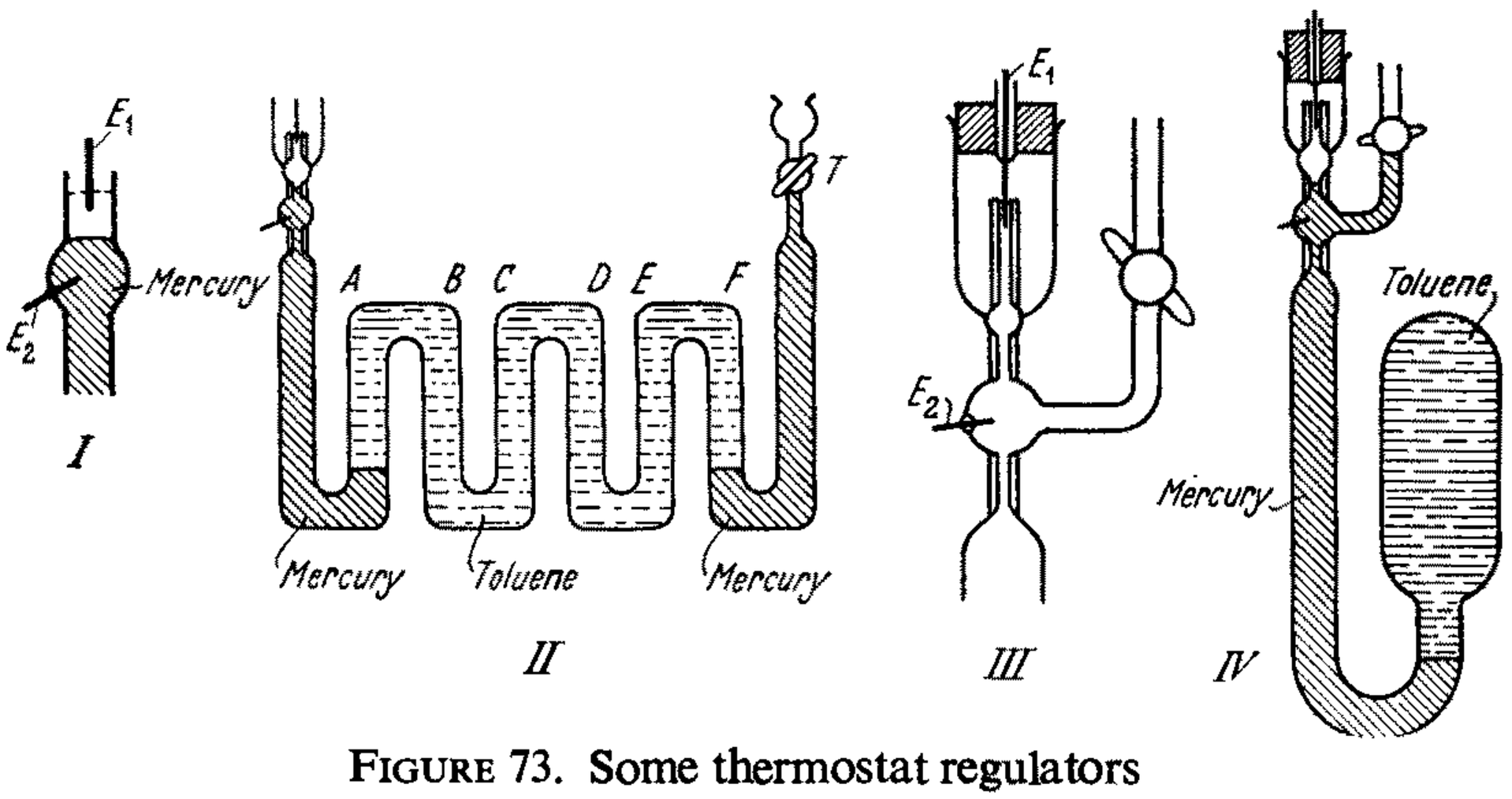

FIGURE 73. Some thermostat regulators

The regulating head is an important part of the apparatus; only a small current should flow, otherwise arcing will occur and the mercury will oxidize. This oxidation is also reduced by keeping the surface area of mercury exposed to the air a minimum by using capillary tube. A useful form of head is shown in FIGURE 73, *III*; the capillary is about 1 mm diameter, the upper bulb about 5 mm diameter, and the lower one about 1 cm diameter. An internal seal is made round the upper bulb; this avoids the thick accumulation of glass which would result from a direct internal seal to the capillary tube. The tungsten rod or wire E_2 of 1 mm diameter can be sealed in without a sealing glass (for a *Pyrex* apparatus). The electrode E_1 is held by a rubber stopper.

A convenient thermostat control for a beaker is simply a small bulb filled with toluene to which a U-tube full of mercury is attached (FIGURE 73, *IV*). This can be made with 8 mm and 15 mm tubing. The bulb may be filled by successive heating and cooling, with the head in toluene.

To keep to a minimum the current flowing through the mercury it is best to employ a valve circuit with a relay, so that the regulator current only controls the grid potential of a valve.

Gas Flow Meters

The usual laboratory gas flow meter is essentially a manometer which measures the pressure difference across a capillary tube through which the gas flows, as in FIGURE 74, *I*, showing a design described by W. BARKAS (1939). This is intended to prevent a sudden change in pressure from forcing the liquid in the manometer into the rest of the

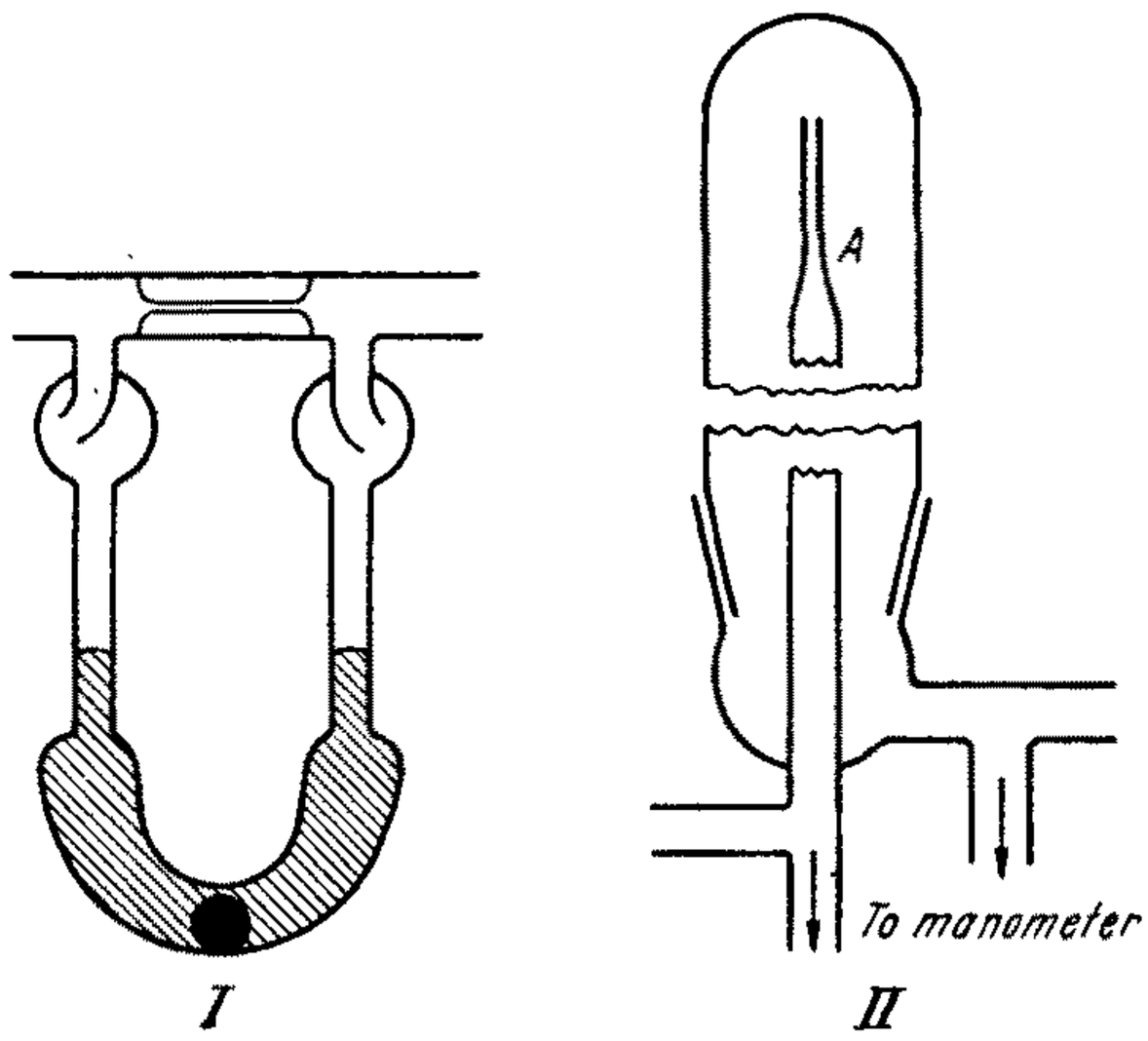

FIGURE 74. Two gas flow meters

apparatus. The bottom tube of the manometer is slightly larger than the rest of the manometer tube, and it contains a fairly closely-fitting ball bearing which is displaced by any sudden flow of liquid and thus blocks the narrower tube. The construction of this apparatus is straightforward. When the capillary tube is joined to the wide tube the join should be as sharp as possible (i.e. there should be a sudden change of internal diameter) if the flow rate is to be related to the pressure difference using an equation for viscous flow of the gas in question; but a direct experimental calibration can also be made, using, for example, a large Berzelius gas holder.

A convenient flow meter is shown in FIGURE 74, *II*. The construction is simple. Splash heads or bulbs should be incorporated in both manometer limbs (as in FIGURE 74, *I*). The capillary tube at *A* may

be drawn out as required, in which case calibration is necessary, or a precision-bore tube may be sealed on. With a drawn-out capillary, the correct adjustment is found by breaking off portions of capillary until the proper flow rate is reached; with the ground glass joint arrangement shown this is very simple. A broken thermometer often provides a useful capillary for small gas flows.

Spectrum Tubes

The spectrum excited by an electrical discharge through gas at low pressure can be studied with two simple types of spectrum tube seen in FIGURE 75. These tubes also act as weak sources of certain radiations, such as monochromatic light for an optical spectrometer. The

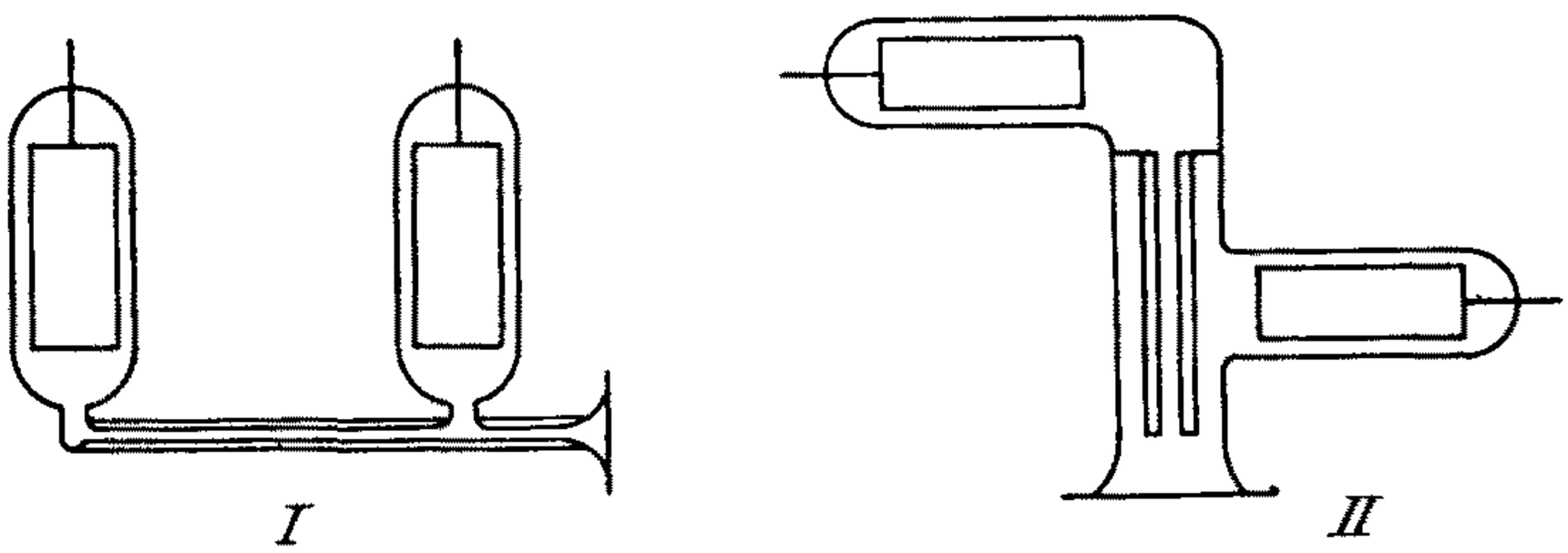

FIGURE 75. Two types of spectrum tube

two tubes shown can be made of *Pyrex* with fused quartz windows sealed on with *W* wax or a cement (p. 84); but for many purposes these windows are not required. In both *I* and *II* the electrodes are metal cylinders (e.g. aluminium or stainless steel) joined to tungsten rods sealed into the glass. Stainless steel cylinders 1 cm diameter and 3 cm long are suitable—the stainless steel is not so prone to splutter-ing as most metals. The cylinders are hard-soldered to the tungsten, or bolted on with a suitable connector. If the tungsten rod exceeds 1 mm diameter it should be sealed in with a sealing glass. 1 mm bore capillary is suitable for the tubes.

To fix on the window as shown in FIGURE 75, *I*, a bulb is blown in the capillary, the top of the bulb is blown out to a bubble, knocked off, and the end remaining is fire-polished and finally ground flat. The tube can be filled with gas through a side arm joined to one of the electrode chambers; the spectrum tube may be sealed off, or provided with a stopcock. The capillary tube in FIGURE 75, *II*, is sealed to the glass at one end only of the capillary. To do this, the capillary is joined to a wider tube, which is cut off and flared out to make the

necessary flare at the end of the capillary. The remaining operations are simple.

Discharge Tube Lamps

Discharge tube lamps for laboratory work usually consist of three basic parts: the tube to contain the discharge, the electrodes, and a water cooling system. Many different types of lamp have been designed, and while a complete review is outside the scope of this book a few of the more simple ones may be described.

High voltage hydrogen discharge lamps which give continuous radiation in the ultra-violet can be constructed quite simply and elaborate designs are unnecessary for many purposes. A very simple

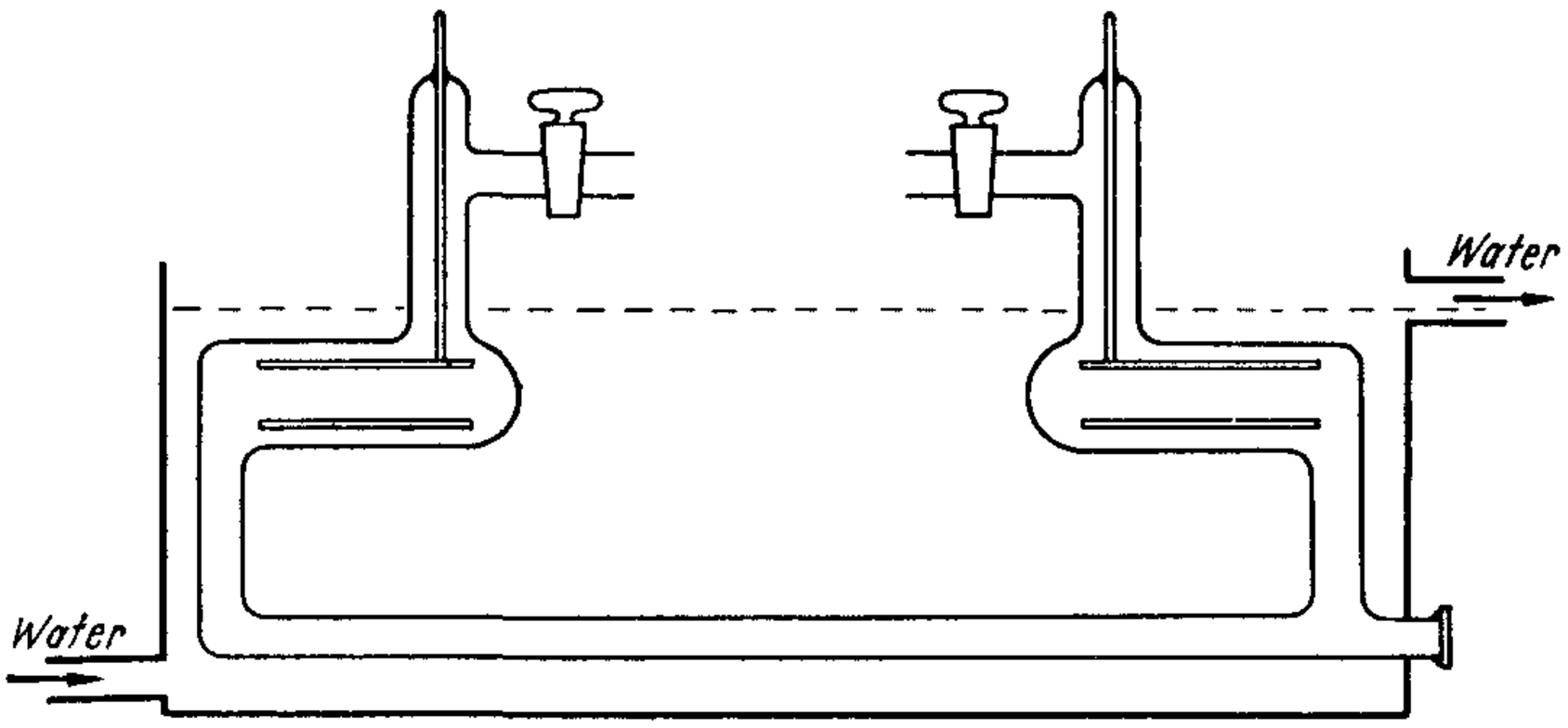

FIGURE 76. A hydrogen discharge lamp

design which should give satisfactory results is shown in FIGURE 76. The lamp consists of a straight tube with a fused quartz or fluorite window waxed to the end and side arms carrying the electrodes and connections for pumping out and filling with purified hydrogen. The lamp is immersed in a trough of water with only the window projecting through a watertight seal in the wall. The electrodes should be of aluminium or stainless steel as these metals are particularly resistant to spluttering under the conditions of the discharge. The tube containing the discharge should be lightly platinized (see p. 87) to facilitate recombination of the atomic hydrogen produced in the discharge.

The chief factors to be borne in mind in the design of these discharge tube lamps are that the electrode material will splutter in the discharge and, unless it is geometrically impossible, will coat the window with a metal film, and that a large amount of energy is consumed by the lamp, much of which is dissipated as heat. This

demands an efficient cooling system, particularly in the region of the waxed joint sealing on the fused quartz or fluorite window.

W. WEST (1949) gives a survey of some discharge lamps of different types in which he provides details of the operating conditions for such lamps.

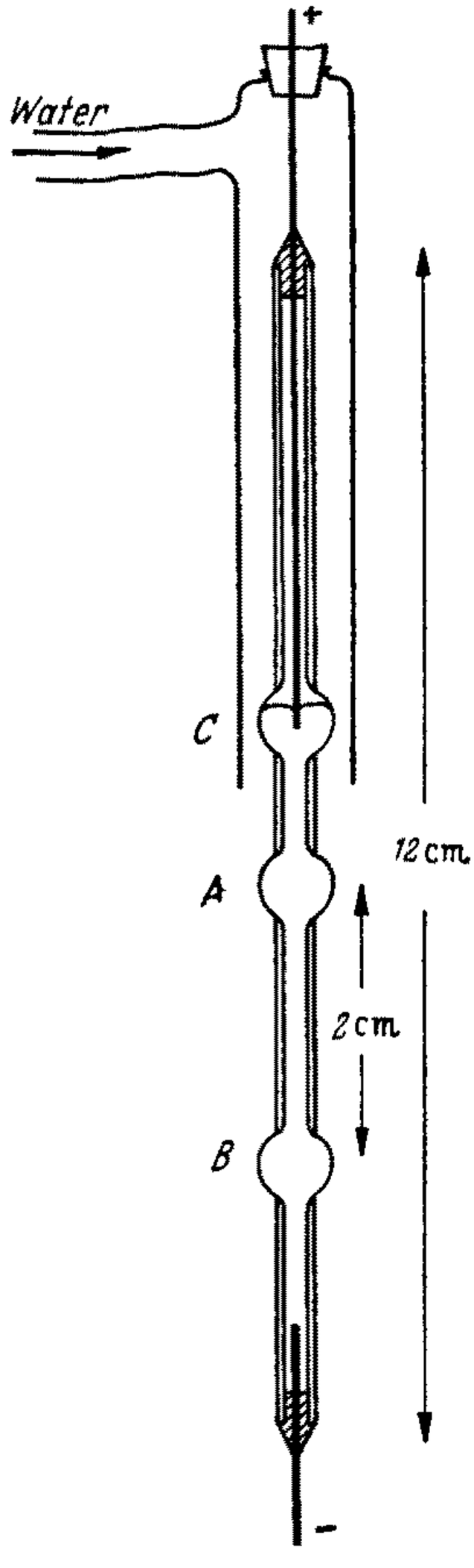

FIGURE 77.
A high pressure mercury lamp

Air or water cooled mercury discharge lamps find many uses, one of the more obvious of which is the study of photochemical reactions. These lamps are usually made of vitreous silica because of its low thermal expansion, high melting point and its transparency to ultra-violet radiation. Their operating pressure has a profound effect on the spectral distribution of the radiation produced and therefore it is important to consider the requirements in the design of such lamps.

E. W. R. STEACIE and N. W. F. PHILLIPS (1938) describe in detail an ingenious design of a low pressure mercury lamp with a rare gas carrier. This lamp was designed for the study of mercury-sensitized photochemical reactions sensitized by the 2537 A mercury line. The lamp itself in this design is of vitreous silica and *Pyrex*, joined by graded seals, and mounted in the centre of the reaction vessel, thus giving the maximum amount of reaction possible. Practical constructional details are given by BARR and ANHORN (1949). These authors also give details of the construction of rare gas discharge tubes and display signs.

A simple, high pressure, capillary, mercury arc has been described by F. DANIELS and L. J. HEIDT (1932), which, although it has only about twenty four hours useful life, may be useful on account of its very simple construction. The lamp (FIGURE 77) is constructed from silica tubing of 1–2 mm bore and 0·7–1 mm wall thickness. The bulbs *A* and *B* confine the discharge and their spacing governs the operating voltage. The third bulb constitutes the expansion volume and is not filled completely with mercury. The tungsten electrodes are sealed in with de Khotinsky cement (shellac and pine-tar), although it is probable that other stable cements would serve equally well. The lamp is started by heating the column of mercury between the bulbs *A* and *B* with a small bunsen burner flame until the mercury is vaporized. A rapid stream of cooling water is then turned on to prevent the silica tube melting as a result of the discharge. These lamps require a ballasting resistance since they have effectively zero resistance before starting, and 20–500 Ω resistance is generated when operating, depending on the operating pressure and other factors. W. WEST (1949) also describes the operating conditions for this type of lamp.

Apparatus containing many Electrodes

Hand made radio valves and other vacuum tubes containing several electrodes, as for example, mass spectrometer tubes, present special difficulties. In many cases, electrodes, plates and grids can be supported by spot-welding to nickel wires which in turn are spot-welded to tungsten rods that form metal–to–glass seals with the vacuum envelope. Various ways for making the metal–to–glass seals are described in Chapter 7. A pinch seal is often employed, as shown in FIGURE 78, *I*. Careful planning of the position of the final joints, at *A* and *B* for instance, allows for the electrodes to be bent first to any required shape and position. Clearly an infinite number of particular assembly arrangements is possible. Prepared copper–tungsten–nickel wires are often very useful in preparing such electrode assemblies.

APPARATUS CONTAINING MANY ELECTRODES

The above method is not generally suitable when precise spacing of electrodes is necessary. In electron and ion guns, for example, some form of rigid mounting with accurately ground spacers between electrodes is used. The whole mounting is supported in some way in its glass envelope and fine conducting wires make electrical contact between the various plates and their respective tungsten–to–glass seals. FIGURE 78, *II*, shows diagrammatically such an arrangement.

Copper connecting wires are generally used and must be connected to the electrodes before the mounted assembly is introduced into its envelope. The connecting wires can then be drawn one at a time out

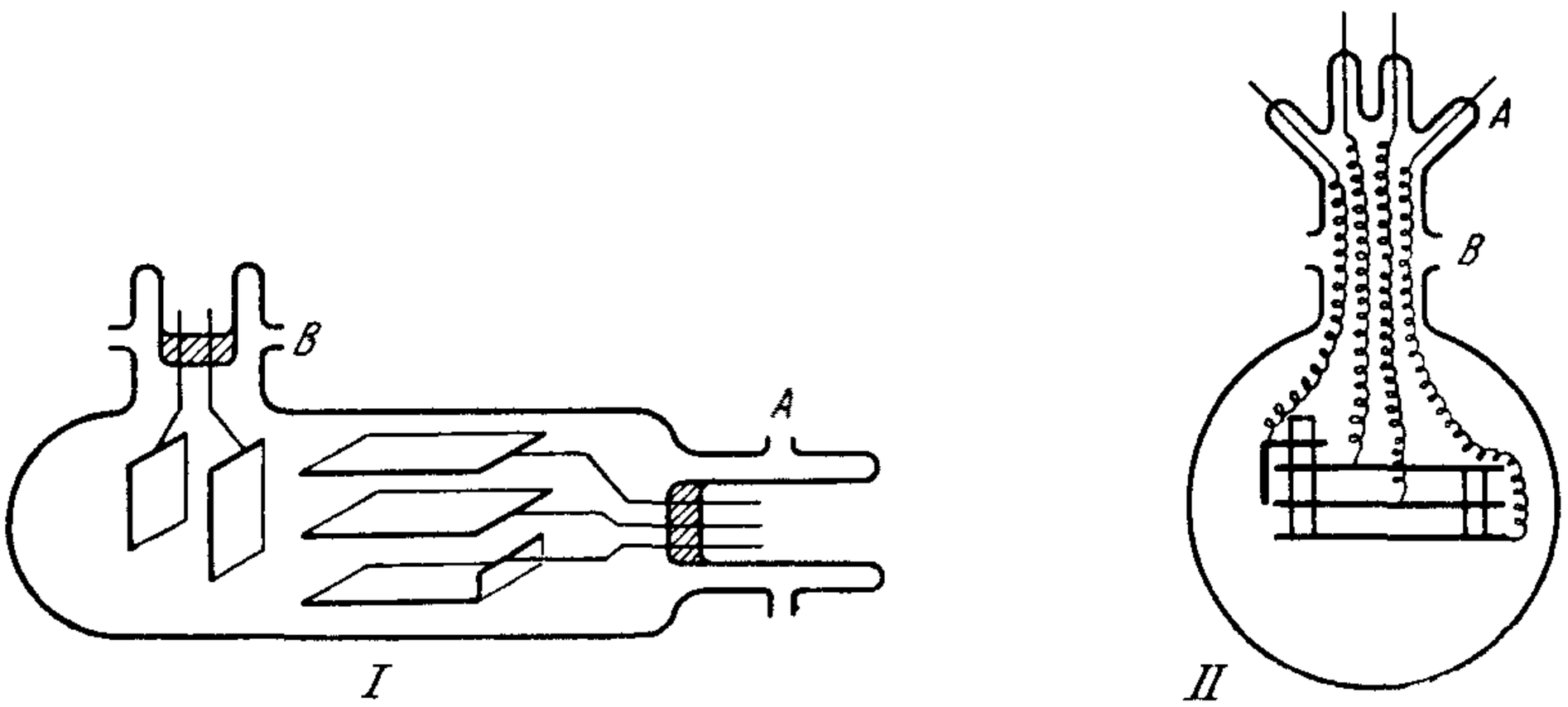

FIGURE 78. Apparatus with many electrodes

through the tube *A*. We have found that fine wires are best (provided low currents are to be passed) because these tend to work-harden, with resultant fracture, less easily than thicker wires. Pieces of thin-walled glass capillary, drawn from 1 cm tubing, make very good insulating sleeves where these are necessary. The wires can be connected with small connectors to the tungsten rods, or welded with a pin-point flame to intermediate nickel wires. This is done as near as is practicable to where the final joint *B* is to be made.

In high vacuum work all metal parts to be mounted inside the glass envelope should be thoroughly cleaned by immersion in appropriate cleaning and passivating dips. Noble metals can be polished with 3/0 or 4/0 (000 or 0000) emery paper. Thorough cleaning of the metals lessens outgassing effects. Any fluxes used in hard or soft soldering must be thoroughly washed away.

To prevent oxidation of electrodes in an envelope which is being glass-worked, a current of carbon dioxide can be passed through the apparatus. This is conveniently obtained from a filter flask containing

solid carbon dioxide. A side arm is provided to allow the gas to escape when the final joint has been made in the glass envelope. The carbon dioxide can also be used to blow the final joint; to do this a rubber tube fixed to the side arm is squeezed when blowing is desired.

Leaks for Molecular Flow of Gases

Molecular flow occurs when a gas passes through a tube or a hole with cross-sectional dimensions which are smaller than the mean free path of the gas molecules. The molecules then flow through independently of each other. A leak consisting of a hole in a very thin diaphragm is often useful. For most purposes this can be made of metal foil, but sometimes a glass diaphragm with a hole in it is useful—for example, when free radicals are to be passed through the hole and the diaphragm material must be chemically inactive. We find that microscope cover glasses can be used to make a suitable leak. The cover glasses, about 0·1 mm thick, are first individually measured with a micrometer and sorted out so that about twenty are obtained with very nearly equal thickness. These are covered with paraffin wax on one side, and on each one an area of about 1 mm^2 of wax is scraped away. They are put in a basin, and the exposed 1 mm^2 area is etched by placing on it a drop of dilute hydrofluoric acid (20 per cent). All are etched simultaneously and the etching is started as nearly as possible at the same moment for each one. After a time, two or three of the cover glasses will be completely penetrated by the acid, the drop of which will be seen to pass suddenly through the glass. The cover glasses are then all swamped in water, and those which have not been penetrated will have very thin portions of glass about 1 mm square; a thin part can be punctured by a spark so that a smooth circular hole of diameter around 0·01–0·05 mm is formed. A single spark is obtained from an induction coil by giving one light tap to the trembler. Pointed electrodes are used one of which is placed very near the thin part of glass. The cover glass is held in a slot cut in a solid piece of rubber. A spark length of 1·5 cm is suitable, but this can be varied depending on the size of hole required.

Precautions to be taken when using Hydrofluoric Acid

Precautions are necessary when using hydrofluoric acid. It must not come into contact with the skin; the acid can cause severe burns which heal extremely slowly. A large beaker of water should always be handy so that any acid on the skin can be washed off immediately. Running water should also be available. Rubber gloves can be used,

and they must be free from pin-holes. Barrier creams of the anti-acid type can be used to coat the hands. A paste of magnesium oxide in glycerine is an antidote for hydrofluoric burns.

Bourdon Gauges and Glass Spiral Gauges

Among the methods for measuring gas pressures down to 10^{-1} or 10^{-2} mm of mercury, one of the most convenient is the use of a gauge which depends upon the distortion of a thin glass envelope under varying pressures. These gauges can be used above room temperature, and for corrosive gases which would attack metals.

The simplest form is shown in FIGURE 79, *I*: *T* is a thin-walled glass bulb with one side collapsed inwards. The shape alters with pressure difference across the glass, and the pointer *P* moves. The gauge needs calibration.

The Bourdon tube (as this bulb-shaped part is called) is made by putting a spindle in tubing about 10 mm diameter and blowing a thin-walled bulb just below the shoulder. The bulb is made slightly elliptical by pulling whilst being blown into. The thinner the bulb the more sensitive is the gauge. One side of the bulb is heated by a soft flame until it collapses; the radius of the collapsed side should be slightly less than that of the other side. On gently blowing into the tube the pointer should move several millimetres. The spindle can be left on as a pointer, or be replaced by a thin glass rod. The tip of the pointer should be drawn out to a fine point. The lower internal seal of the gauge can be made with a hand torch, the internal tube being first bulged. A bench torch can also be used. The glass must be blown into from both ends, since the end of the pointer *P* is closed. If the method used for this internal seal involves first rounding off the wide tube and then dropping in the Bourdon tube (Method 2, p. 75), the final hole must be made by pulling away the very hot molten glass with a glass rod, since it is not possible to blow out a bubble of glass.

The movement of the pointer may be observed with respect to a reference pointer which is sealed into the jacket from above. The pointer *P* can also be made to move a glass lever system which magnifies the movement (S. G. FOORD, 1934).

Another type of gauge depends upon the movement of a thin-walled glass spiral—S. G. YORKE (1945, 1948) has given constructional details. The spiral is wound on a carbon mandrel (FIGURE 79, *II*), which is tapered and threaded, and has a hole in it at the top of the taper. The flame is directed against a metal shield just below the mandrel; this gives uniform heating. The thin tubing collapses if heated directly. The thin tubing for the coil is made by drawing out, with blowing, 6-mm diameter tubing to 1·25 mm in a ribbon flame.

The thin tube is put through the hole in the mandrel, and 7 or 8 turns are wound. The spiral is cut where it goes into the hole in the mandrel. The gauge can be used with the spiral horizontal (FIGURE 79, *III*) or vertical (FIGURE 79, *IV*). In the vertical form shown a mirror *M* is fixed to the pointer with *Picien*. A piece of capillary tube *C* is also sealed into the jacket to protect the spiral against excessive movement. An optical window *W* is sealed in or cemented in. A gauge of this type is more sensitive than that shown in FIGURE 79, *III*.

Both the Bourdon and spiral gauge can be used with a compensation method or with direct observation of the movement of the

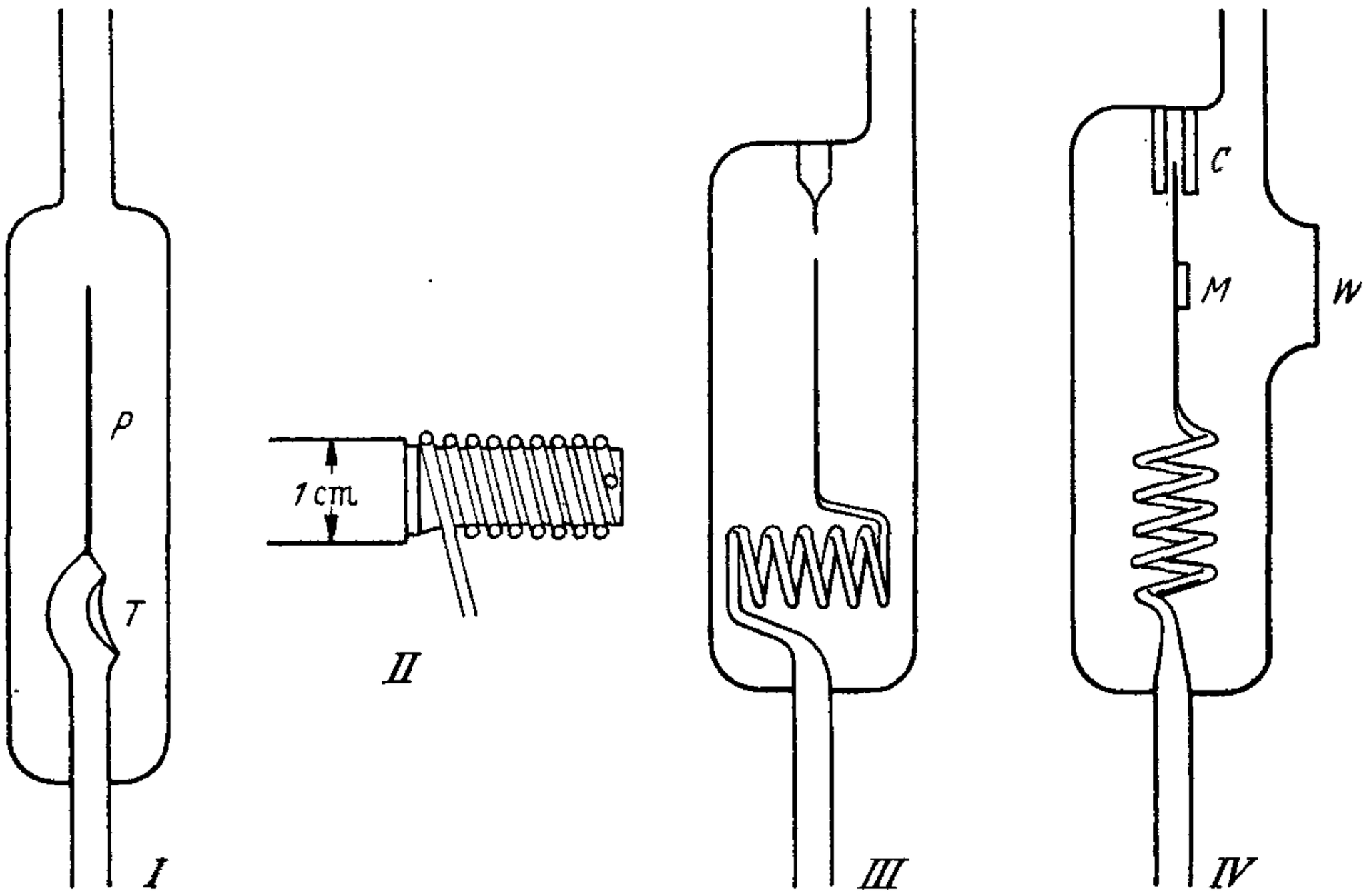

FIGURE 79. Some glass gauges for pressure measurement

pointer. In the compensation method a pressure is applied to the outside of the bulb or spiral until the pointer deflexion becomes zero. For maximum sensitivity it is best to control the gauge temperature by adding an outer jacket through which a liquid at constant temperature is passed.

Circulating Pumps

Simple pumps for circulating gas or liquid can be made using two valves with a chamber between them. One ingenious pump uses a heat engine (A. R. PEARSON and J. S. G. THOMAS, 1925). The hard glass or silica bulb *A* (FIGURE 80, *I*) contains air initially at atmospheric pressure. This is heated with tap *T* closed; and when the

pressure is 5–8 cm above atmospheric, T is quickly opened and closed. The pressure in the bulb suddenly drops and the mercury in the U-shaped tube is set into oscillation and continues to oscillate as long as the bulb is heated. To ensure regular motion the dimensions of the bulb must be properly chosen. Another way of starting the pump is to heat the bulb (to about incipient redness), then to open T and to fix on to the outlet from T a rubber bulb. By squeezing and

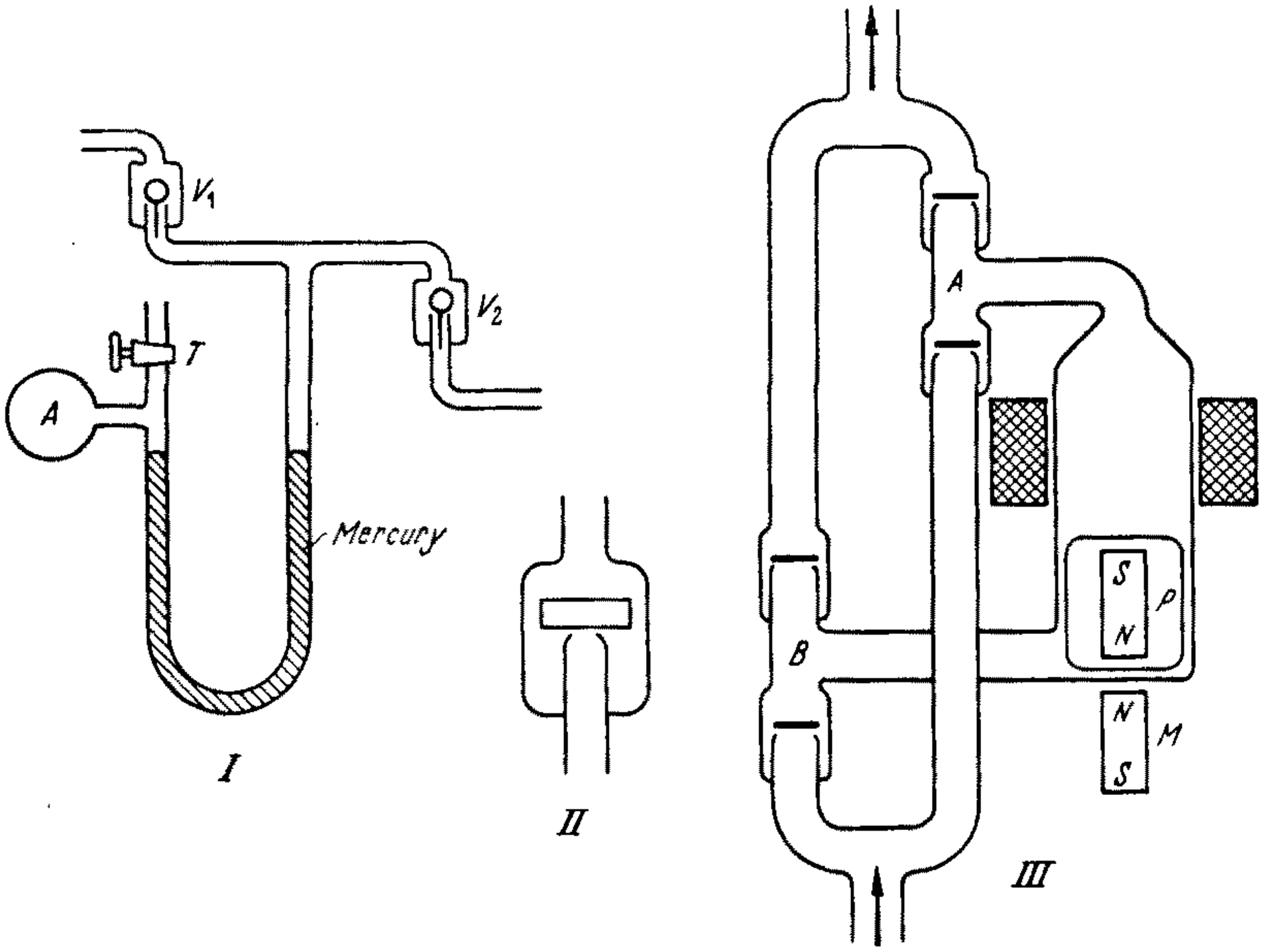

FIGURE 80. Circulating pumps

releasing this bulb the mercury is set into oscillation. The tap T is then closed, when the oscillations should continue. The valves V_1 and V_2 can be of the type shown in FIGURE 67, *VI*. V_1 opens and V_2 closes as the mercury rises. Another kind of valve is shown in FIGURE 80, *II*. The U-tube can be made of 5 mm tubing and A can be a 250 ml flask. A liquid or gas can be pumped.

The pump in FIGURE 80, *I*, pumps at every other stroke, because one stroke is required to fill the chamber between the valves. A. H. COCKETT (1955) describes the pump shown in FIGURE 80, *III*, which pumps at every stroke. The piston P consists of a magnet encased in polythene—this avoids trouble which can arise from a glass piston in a glass tube forming powdered glass so that the piston sticks. It is

made by pouring molten polythene into a mould containing the magnet—the mould can be a piece of glass tube similar to that of the piston chamber. The piston is finally rubbed with emery paper till a good fit is obtained; it should not be too tight, nor too loose. The piston is lifted with a solenoid of 1000 turns of 22 gauge wire (about 7 Ω resistance). When the current is cut off the piston falls—the magnet *M* prevents it from breaking the apparatus. As the piston moves up, chamber *A* is emptied and *B* is filled; and as it moves down, *B* is emptied and *A* filled, so there is continuous pumping.

Apparatus for Semi-Micro Qualitative Analysis

Most workers develop their own particular methods of performing semi-micro qualitative analyses, and the number of special appara-

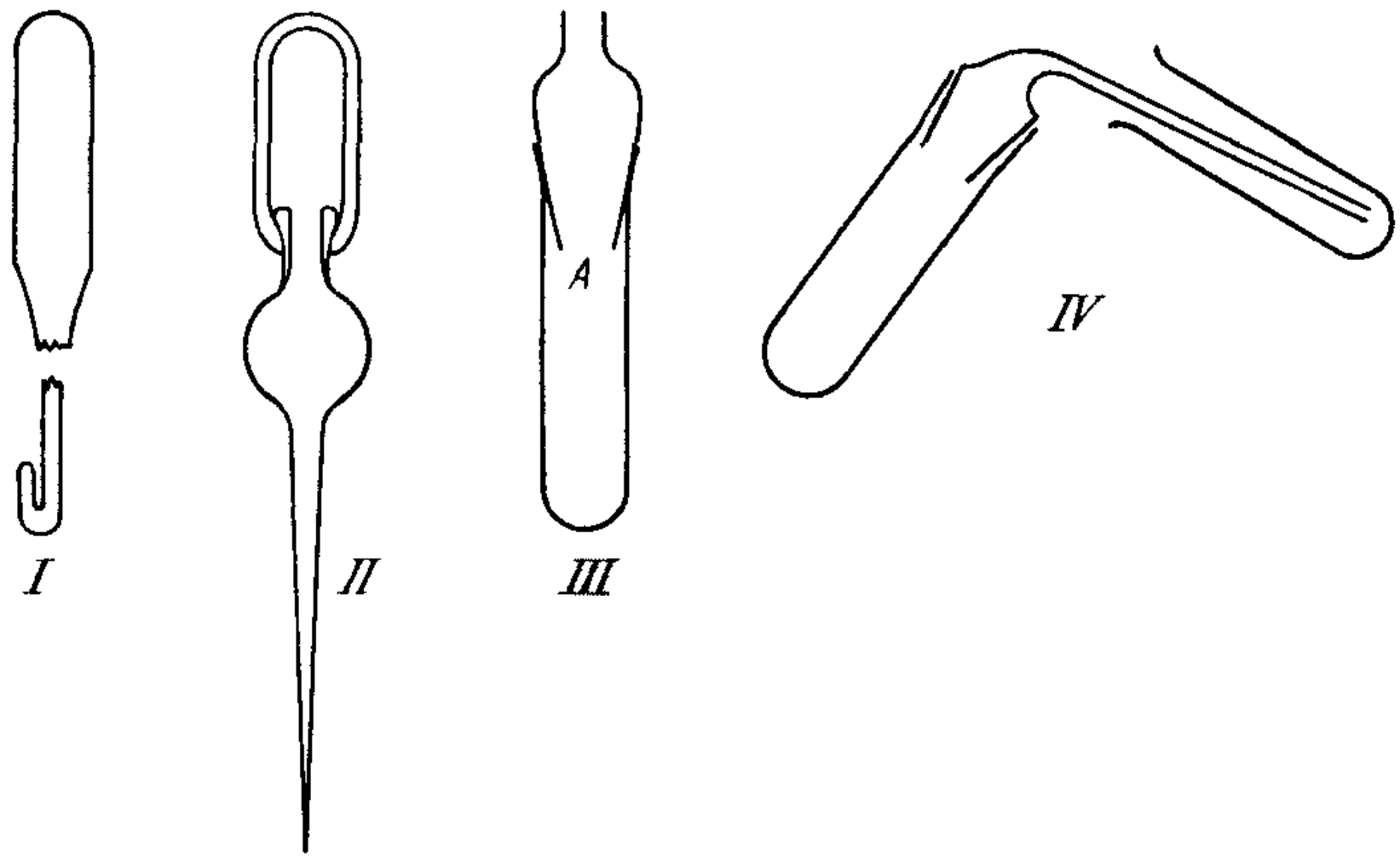

FIGURE 81. Some devices for semi-micro qualitative analysis

tuses is considerable. Usually, however, certain simple items such as stirrers, spatulas, droppers etc. are used for most of the work. These can be made with great ease. Stirrers and spatulas are made from glass rod: thin rod about 10 cm long and 1·5 mm diameter is drawn out from ordinary rod, or from tubing (p. 58). For a stirrer, about 5 mm of the rod is bent through 180° at the end (FIGURE 81, *I*). Some thick rod (or tube) can be left on as a handle, as shown. A spatula is made by drawing a spindle in rod, putting a blob of glass on the end and flattening the blob with tweezers. The edges can be bent up with tweezers to form a spoon if desired.

The dropper shown in FIGURE 81, *II*, has a small bulb so that for maximum uptake liquid does not enter the rubber teat; reagent solutions are then less likely to be contaminated. The capillary tube

can be drawn after the bulb is blown, and it should not have too thin walls. Some glass should be accumulated before the tube is drawn down.

For detecting gases a tapered tube is made which fits snugly into the top of a small test tube, as in FIGURE 81, *III*. The hole *A* should be about 2 mm diameter, and the top about 5 mm diameter, so that the reagent can be put in with a dropper. This apparatus is not reliable because the gas usually escapes up the side of the tube instead of through the reagent. The two parts can be ground together to stop this; care is then necessary, if the test tube is heated and cooled, to stop the reagent entering it. A thin tube can be prepared, as in FIGURE 81, *IV*, for bubbling gas through a reagent: this is again tapered to fit the test tube, and is preferably ground in; alternatively a rubber ring can be used as a gasket. The volume of the gas delivery tube should be small, so the glass tube is first worked till the walls are quite thick by pushing it together while it is rotated in the flame.

Gas Analysis Apparatus

The art of analyzing gases by chemical means has made vast progress since the early researches of R. W. Bunsen. Most gas analysis apparatus, however, still contains a reservoir, a combustion chamber, and an absorption vessel, and formidable-looking apparatus is found on inspection to be capable of assembly by a series of moderately

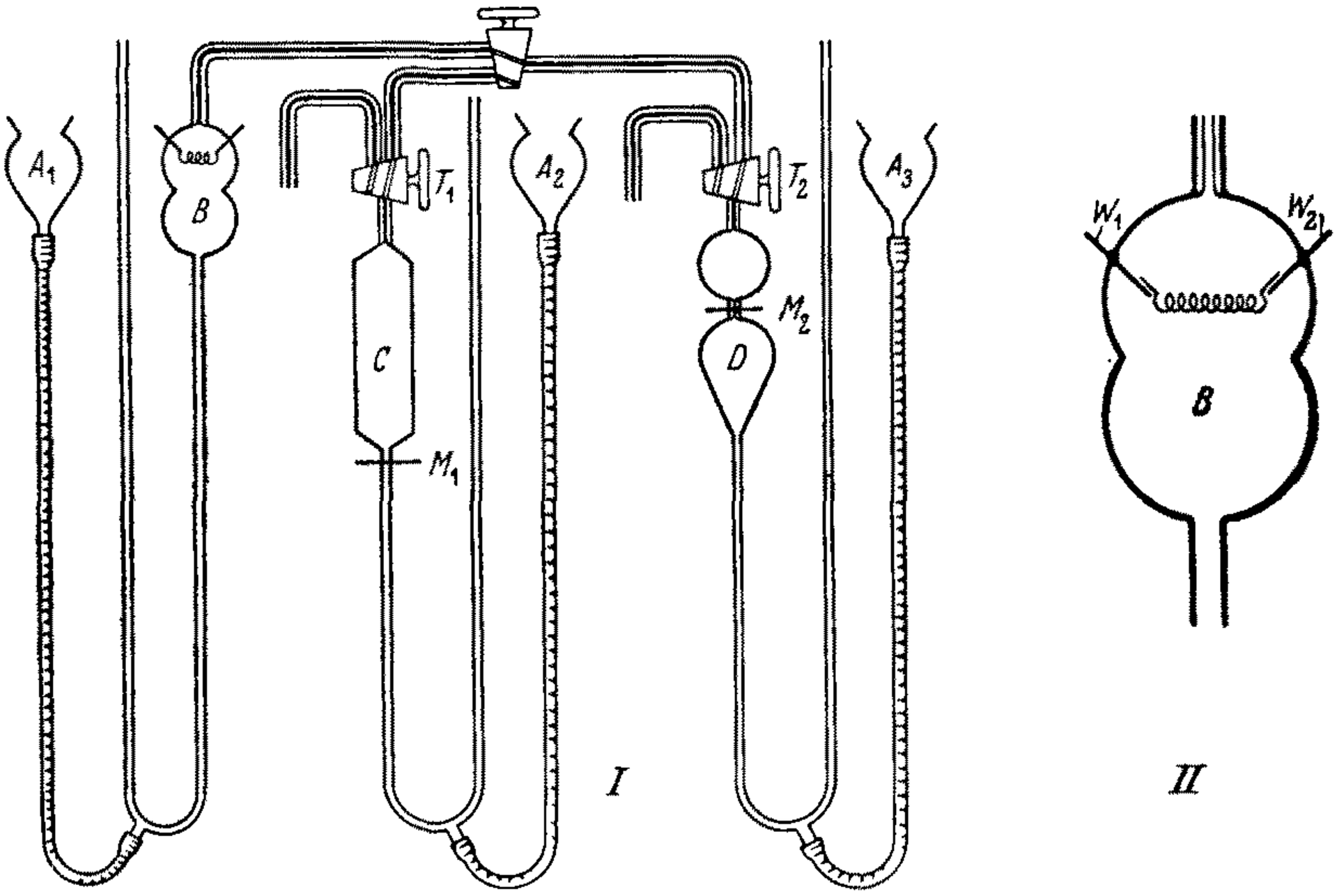

FIGURE 82. A simple form of gas analysis apparatus

straightforward glass-working operations. A simple form of apparatus is shown in FIGURE 82; *B* is the combustion chamber. The apparatus can be used in different ways: for example, both *C* and *D* can be used as reservoirs or absorption vessels—*C* is usually the reservoir and *D* the absorption vessel.

The best size for the capillary tubing is 1·5 mm bore; the rest of the tubing in the apparatus is 8 mm diameter. In making the part *B*, two tungsten rods W_1 and W_2 (FIGURE 82, *II*) are spot-welded or hand-soldered to a platinum wire coil—if spot-welding is used it is much easier if an intermediate piece of nickel is introduced. The tungsten is beaded, with a sealing glass if desired; two holes are blown in *B* for the tungsten, which is put in, with the platinum, through the lower tube (FIGURE 82, *II*). It is convenient if the beaded tungsten just passes through the holes. The volume of the reservoir *C* from the tap T_1 to the mark M_1 must be measured (if it is needed) before this part is joined to the apparatus. Similarly, the volume between the tap T_2 and the mark M_2 should be found before assembly. A rigid frame is needed for the apparatus because of the great weight of mercury in it.

The mercury reservoirs A_1, A_2 and A_3 are standard equipment; they can be made by joining at each end about 8 cm of 4-cm diameter tubing to 8 mm tubing. The large portion of tubing is then blown to a bulb of about 7 cm diameter, which is worked by pulling and blowing to a pear-shape. The 8 mm tubing attached to the pointed end of the bulb is corrugated to take rubber tube, and at the other end the bulb is blown out and flanged.

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Chapter 9

THE ASSEMBLY OF COMPLEX APPARATUS

General Observations

THE assembly of complex glass apparatus is comparatively easy provided it is done in a systematic manner. It is most important to work out the general arrangement of the various parts of the system before starting to assemble them, and the sequence of operations should be planned in detail.

The arrangement of apparatus is usually dictated to some extent by the laboratory space available; but within this limitation careful preliminary planning can make the construction, use and maintenance of the apparatus easier. The apparatus should be designed so that all parts are easily accessible and so that its operation does not impose too great a physical strain on the worker due to awkwardly placed components. An apparatus, badly designed in this respect, will probably be broken much more often than a more carefully planned one.

Assuming that the apparatus has been carefully planned and that it is to be a vacuum apparatus, it is generally more convenient to start building it at the pump end of the vacuum line. This allows the joints to be tested before the complete system is finished, which is an advantage if some of the joints are screened by other parts of the apparatus constructed later. Probably the most convenient method of working is to join on each section with its far end finishing in a sealed-off spindle which can easily be blown out preparatory to the joining of the next section. This technique also excludes unnecessary dust and contaminants from the system during building. If some other method of rendering the system air-tight for blowing is used, care should be taken to see that it does not contaminate the glass. Care should also be exercised to avoid introducing flakes of glass into the apparatus when holes are blown in the glass. If such flakes are carried in the air stream during evacuation and lodge in the diffusion pump, its efficiency may be seriously impaired.

The cleanliness of the inside of a vacuum system is of the utmost importance, and therefore care should be taken at all stages to see

that the glass is thoroughly cleaned before assembly and that no unnecessary dirt is introduced during assembly. The introduction of a single length of improperly cleaned tubing can render useless all the effort spent on cleaning the remainder of the apparatus. Open tubes on the apparatus should be blocked in some way, even if not required to be air-tight, to prevent dust from entering them. Sometimes it may be desirable to prevent excessive amounts of moisture from entering the apparatus. For this purpose a tube of drying agent can be included in the blowing tube, although very often large amounts of water vapour enter the apparatus as combustion products of the flame. When glass-blowing is carried out on a system containing greased stopcocks or ground joints they should not be overheated; if they are, grease is spread over the inside of the system. *Silicone* grease on ignition forms a solid mass which cannot be burnt off.

Whatever the problems and however complex the apparatus to be constructed, the sequence of operations should be carried out mentally before any practical work is started. The time spent in planning any piece of practical work is well spent and will probably save time eventually.

Joining Closed Systems

The need for joining closed systems arises quite frequently, and various methods may be used to make the final joint. Difficulty usually arises out of the necessity of blowing two holes in the system in close proximity to one another.

One method of doing this has been described already in the section on McLeod gauges; it involves blocking the first hole with a stopper, blowing the second hole and then bending the glass until the holes are in a suitable position for joining. This method may be varied so that it is unnecessary to allow the glass to cool after blowing the first hole. If care is taken in blowing the first hole, a bubble of glass, sufficiently thin to be knocked off but strong enough to withstand the blowing pressure, can be produced. It is then possible to blow the second hole and remove the thin glass bubbles.

The most difficult part of joining a closed system is making the two prepared tubes meet correctly. This can usually be accomplished by bending either or both of the tubes, and perhaps in addition drawing the tubes out until the desired fit is obtained. Once the joint has been made by pushing the glass together or by filling the gap with rod, the irregularities produced by bending and pulling can be evened out by judicious heating and blowing, although for many purposes they will not have any effect on the efficiency of the apparatus.

Another method which may be useful in joining closed systems is

described more fully in the next section. This involves making the two final joints simultaneously, at either end of a reasonably long length of tubing, inserted between prepared sites.

Making more than one Joint at once

When several tubes in a complicated piece of apparatus have been cut in order to remove a large or intricate portion for repair or alteration, difficulty is encountered, during reassembly, in obtaining a closed system for blowing. It becomes necessary to make several joints at once and we have found various techniques useful in these circumstances.

Closing Tubes with Ragged Ends— Use of Rubber Caps

The cutting operation when the tubes are not easily accessible usually results in slightly ragged edges. However, the two halves will fit perfectly, and it would be convenient not to have to fire-polish either edge, so that when the reassembly is undertaken the two will still be a perfect fit. If a rubber stopper is used for blocking a cut tube, fire-polishing is necessary in order to make an air-tight seal and in order to reduce the somewhat large danger of splitting the ragged end when the stopper is forced in. Rubber caps are therefore very useful here and, with large tubing, thin pieces of sheet rubber (see p. 35).

Glass-Blowing Two Joints Simultaneously

It is occasionally convenient, both when dealing with complex apparatus and when replacing a short section of a fixed glass tube, to be able to glass-blow two joints at once. This can successfully be undertaken when the two joints are reasonably close together and accessible, and when the tubes involved are not larger than 15 mm diameter.

The interposing portion of glass is first shaped and its ends worked until it fits as closely as possible. It is then clamped in position. By using the hand torch for a few seconds at a time on each joint they can be worked simultaneously, care being taken to heat each evenly all round so that one does not crack in the few seconds that the other is being worked. By using the spike and welding rod where necessary, the two joints can be first sealed and then blown to satisfactory shape. Each joint must be separately and carefully annealed (unless they are so close in the same tube that they can be heated together to the annealing point).

Glass-Blowing more than Two Joints at once

The following method also applies to two joints that cannot be treated by the procedure just described: Each joint is worked in turn

without blowing and sealed up, using the hand torch, welding rod and the spike. Blowing is not possible until the last joint is reached, and so care must be taken not to collapse the tubes too far nor to apply too much rod and produce large blobs which will sag. Too hot a flame should be avoided so that the portions to be sealed do not shrink away from each other. Pin-holes will not matter in this part of the operation as long as a pressure sufficient for blowing can be built up. After each joint has been worked in this way, it must be very carefully annealed since the half-finished seal is normally very uneven in thickness and resulting strains can easily cause cracking while the next joint is worked. The joint that will give the most difficulty, either because the tubing is large or because it is inaccessible, is left to last since blowing will then be possible. Finally each joint is carefully warmed up, blown to shape, finished and annealed.

Clearly, when a portion of apparatus has been removed by cutting several tubes, each of the tubes can be drawn off before reassembly and, in the reconstruction, short pieces of new tubing can be used for joining the various pairs of cut tubes in turn. However, when the removed portion can be re-clamped in position with the cut tubes meeting well, it has been found that the above procedure is quicker and more satisfactory, particularly with tubing larger than 15 mm diameter.

Use of Thin Rubber Sheet in making Several Joints at once

With extra large tubing, greater than 35 or 40 mm diameter, a joint sealed with welding rod but not blown out to even thickness is unlikely not to crack on cooling, however well annealed. The procedure in the preceding paragraphs cannot therefore be used when more than one such joint is involved. Thin rubber sheet has been found very useful on such occasions. Small pieces, used to close the ragged end of a tube (see p. 35), allow the two halves of a joint, that fit perfectly, to be brought together with the rubber still between them. Then as each joint is made, the thin sheets of rubber can be removed one by one and the minute gaps can easily be closed with the welding rod.

Removal of Strain and Clamping of Apparatus

Strain in the glass, created during the construction of some piece of apparatus, is usually removed by annealing; however, further strain is sometimes set up when a piece of apparatus is clamped to a rigid framework at more than one point. Annealing alone may not remove strain produced in this way—the glass must be softened so that it will flow. With some pieces of equipment it is undesirable to heat anywhere after the whole has been annealed—as, for example, with a

diffusion pump—and if clamping at more than one point is unavoidable then this clamping must be performed with extreme care. The claws of the clamp should be well padded with cork, asbestos rope or layers of asbestos paper. If rubber is used it should be dusted with french chalk to prevent sticking which can cause fracture when unclamping at some later date. See p. 39 for remarks concerning types of clamps.

Wherever possible, when a glass tube has been clamped rigidly at two points, the tube between these points should be heated to the softening temperature—so that the glass will flow and take up any inaccuracy in the clamping—and then annealed. In this way an almost completely strain-free system can be obtained. Clearly a rigid framework is necessary if this strain-free condition is to be maintained.

Annealing by Flame

When an annealing oven is not available, even quite large pieces of equipment can be annealed by flame. Apparatus assembled on the bench can often only be annealed by a flame. As a general principle each joint should be annealed as it is completed, unless a further join is to be made, or another operation is to be performed immediately in the same region or so near to it that the whole of the first joint can be kept hot—then the whole is annealed together.

As large a flame as possible is used, with the oxygen adjusted so that the inner blue cone is long and a very pale transparent blue (for *Pyrex* and similar glass). The glass is warmed slowly—with large equipment, made with tubing greater than 40 mm diameter, warming should be begun with a luminous flame or even with hot air—until the whole has been raised to a dull red heat and the glass is distilling sodium (colouring the flame yellow) freely. As much of the glass as possible is kept at this temperature for a few minutes, and then, by slowly reducing the oxygen content and, finally, the size of the flame, the glass is allowed gradually to cool. The flaming is continued until an even, black deposit of carbon is formed.

Use of a Bent Blowpipe and Double Tipping Device

For getting at tubes placed awkwardly in complicated apparatus a bent tubular blowpipe is very convenient. A useful design is shown in FIGURE 83, *I*.

We think that a long thin blowpipe, as in FIGURE 83, *I*, but bent at the end through a greater angle—about 150°—would be very useful for working on complex apparatus. It would enable the back of tubing to be worked from the front.

USE OF BENT BLOWPIPE AND DOUBLE TIPPING DEVICE

A double tipping device, which forms two fine flames directed towards one another from nozzles about 2 cm apart, is shown in FIGURE 83, *II* (see p. 29). This blowpipe head is very handy for working small tubing in awkward positions. When joins have to be made in such tubes it is desirable to arrange things so that the ends can be pushed together whenever possible. The use of welding rod at long range, or when the operator has to assume a tedious position, requires a very steady hand.

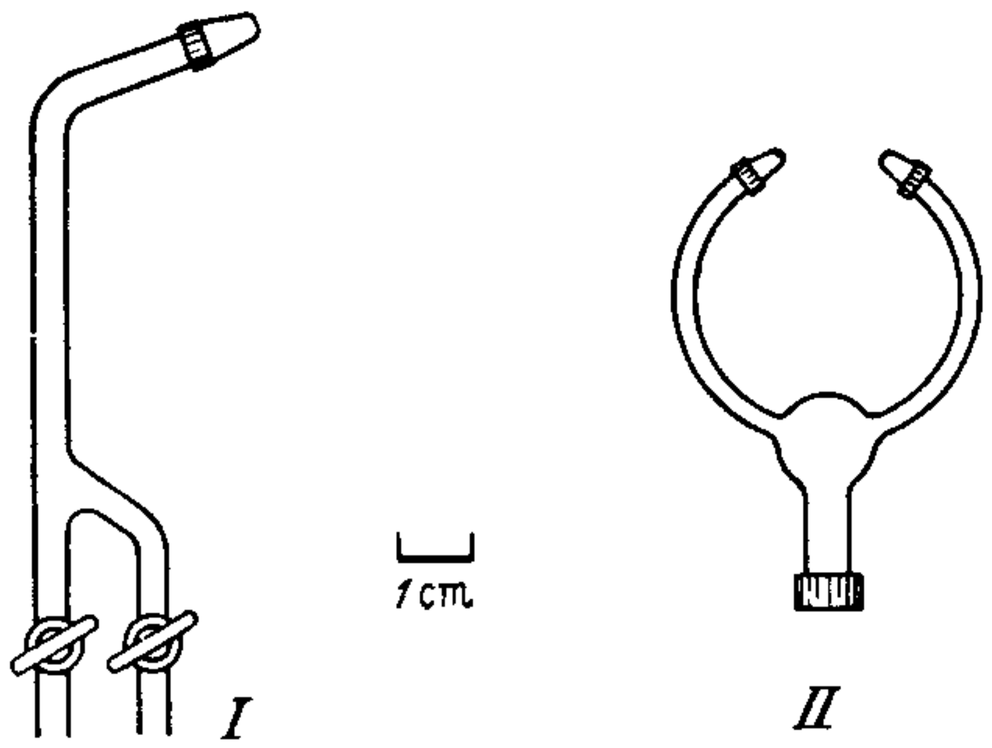

FIGURE 83. Specially shaped blowpipe heads

Danger of Flame Cracks

Recognition of Flame Cracks

Care must be taken, when working on complicated apparatus, to avoid producing flame cracks. These are caused by momentary playing of a hot flame on cool glass. Cracks formed in this way may not appear for several hours, and in some cases even days, after being caused. They generally appear in the surface or bulk of the glass in one of the two forms shown in FIGURE 84. The first is a small curling crack, usually between 5 and 10 mm diameter if circular, and is very characteristically a flame crack. The second general type has a starred appearance with small cracks radiating from a tiny curl or point. The exact mode of formation of these patterns is uncertain, but it is clear that some form of differential strain is the cause. Sudden heating of the surface layer makes it strain-free, and, on cooling, tensile stress develops in the surface layer because the inner layers of the glass are rigid. Glass in tension can undergo a delayed fracture (p. 10).

Precautions for avoiding Flame Cracks

With intricate apparatus, particularly when a hand torch is being

used, it is sometimes impossible to avoid playing the flame on to some portion of the assembly that is not actually being worked. Such portions must be protected by wrapping with asbestos paper; this is particularly important when a joint such as an internal seal is in danger. When a hot flame has been inadvertently played on a piece of cool glass, the latter should be annealed, after the work has been completed, to avoid subsequent formation of a flame crack.

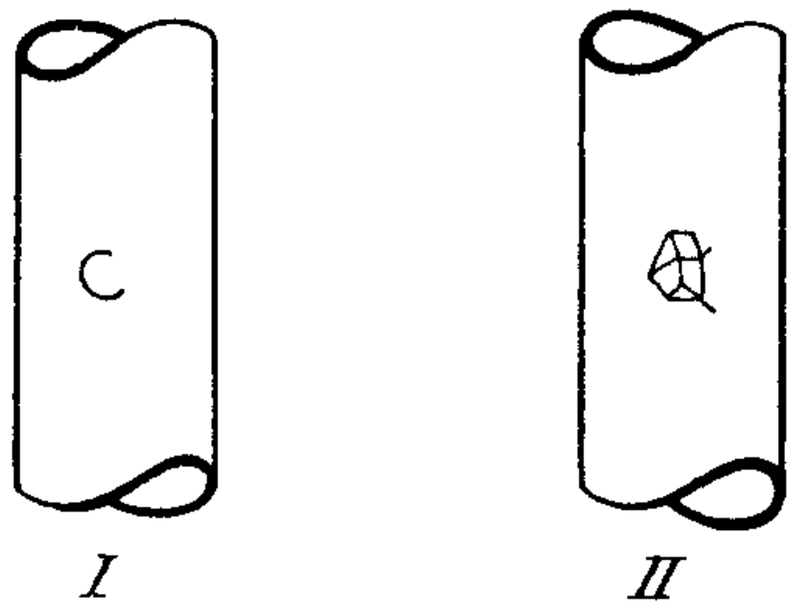

FIGURE 84. Flame cracks

Removal of Flame Cracks

Flame cracks are easily removed by warming the glass, gently at first to avoid spreading the crack, and raising it to the annealing temperature. When joints are in the vicinity care must be taken to heat everything uniformly and to anneal as a whole.

Chapter 10

THE MANIPULATION OF SILICA

The Properties of Fused Silica and General Remarks

VITREOUS silica, commonly known as *Vitreosil*, is pure homogeneous fused silica containing over 99·8 per cent SiO_2. This glass has many extremely useful properties—such as chemical resistance, extremely low thermal expansion with resultant constancy of volume and resistance to thermal shock, resistance to heat and transparency to ultra-violet and infra-red radiations—making it a valuable material for many scientific purposes. These properties are dealt with in varying detail in Chapter 2. Resistance to heat is often of considerable importance since *Vitreosil* has an extremely high softening range, in the region of 1500°C, and can, under normal working conditions, be safely kept at temperatures of 1100°C and as high as 1300°C for short periods. In vacuum work, for evacuated furnaces for instance, fused silica can be used at temperatures up to 1100°C; and provided no devitrification has occurred the glass remains practically impermeable to gases at these high temperatures, with the possible exception of helium.

Fused silica is manufactured in both translucent and clear, transparent forms; the particular requirements govern the form to be used. The translucent variety can be obtained with a sandy, satin or glazed finish. An interesting historical account of the early work on the practical manipulation of fused silica has been given by W. A. SHENSTONE (1902).

Devitrification is a factor requiring particular attention. Impurities on the surface of *Vitreosil* will readily cause crystallization of the surface glass when it is worked. Salts of sodium, potassium and lithium, salts of vanadic acids, fluorides, phosphates and radioactive substances, are especially active in this respect. It is therefore very important, particularly when it is required that the silica remains clear and transparent, that the glass be washed carefully, inside and out, before working. If it has been necessary, during preparation, to grind the fused silica, then all traces of abrasive must be removed by dipping in dilute hydrofluoric acid followed by careful rinsing. This treatment, with subsequent heating to just below the softening point

with a flame, is also effective in removing any condensed surface deposits of silica due to volatilization during working. Precautions advisable when using hydrofluoric acid are mentioned on p. 158.

Any cleaned portion of the *Vitreosil* must not be touched with the fingers before being worked. When using a bench flame, if the part being handled is subsequently to be worked, the piece of equipment must be re-cleaned before this further work is carried out. For this, cotton-wool dipped in alcohol is very useful—after the piece has been allowed to cool.

General Technique for Working Fused Silica

Cutting Tubing

Fused silica is slightly more brittle than borosilicate glasses and is therefore more easily fractured. Tubes up to just over 1 cm in diameter can be snapped with the fingers after scratching with a file or glass knife. Any ragged end so formed can be cleaned up with an abrasive wheel.

With larger tubing a cutting wheel is necessary since the low thermal expansion renders local heating entirely ineffective in starting a crack. A silicon carbide cutting wheel, $\frac{1}{16}$ to $\frac{3}{32}$ inch thick and running with a peripheral speed of 6000 feet per minute, is very suitable and quite adequate. A copious supply of water directed on the cutting edge is necessary and should be directed from either side of the cutting wheel. Copper or phosphor-bronze, bonded, diamond, cutting wheels can also be used but are much more expensive items.

Fusing and Working Operations

As a medium for glass-working, fused silica has the considerable advantage over the softer glasses that careful and uniform heating and annealing are unnecessary. Slightly offsetting this advantage is the necessity for careful cleaning, as described above, and for stronger heating.

An ordinary oxy-coal-gas torch is only adequate for small size equipment; when working with tubing of greater diameter than 15 mm, oxy-hydrogen or other very hot flames must be used. Special burners are obtainable however—as, for example, the *No. 8 Thermal Syndicate* torch—with which oxy-coal-gas, or, better, oxy-propane mixtures, will give a sufficiently hot flame for this larger tubing, so avoiding oxy-hydrogen mixtures which are less safe in operation unless anti-burn-back valves are employed.

In all fusing operations with fused silica, protective spectacles must be worn—a glass whch absorbs at least 96 per cent of the incident

light is necessary. The recommended glass for best protection is green *Protex* grade C, $\frac{1}{8}$ inch thick (Chance Bros. Ltd).

For hand work with a bench torch, tubing is rotated in the flame to secure even heating and hence even shaping; while when clamping of the various tubes is employed and the hand torch is used, all the dangers of cracking, which are present when borosilicate or soft glasses are being worked, are negligible. The *Vitreosil* must be blown while still in the flame; and therefore, when a bench torch is used and the apparatus is manipulated by hand, a blowing tube must be attached at one end. It is preferable then to use a glass-blower's swivel (see p. 42).

For tooling of molten *Vitreosil* $\frac{3}{16}$ inch *Nichrome* wire may be employed; though if much tooling is required, then carbon rods or plates are necessary. In general, most of the operations described in earlier chapters can be modified for working with *Vitreosil* and, if a sufficiently hot flame is available, less manipulative skill is usually necessary because the material stays fairly viscous.

When operating with tubes it is well to take care to prevent the flame licking the insides; the vapours can cause contamination which results in devitrification when more work is done.

Large Diameter and Thick-Walled Tubing

With very large silica tubing. over 4 cm bore, the walls are usually very thick. Special methods are required when the walls are 5 mm thick or more. The wall edges, at the ends to be joined, are cut or ground to sharp knife-edges, so that a V-shaped notch is left all round when the ends are butted together. The ends are fused together, using a hand torch and 3–5 mm welding rod to fill in the V-shaped notch. With very large tubes the flame must be manipulated to keep the tubes hot all round, maintaining an even heat, and the apparatus must be annealed—preferably in a furnace for one hour at 1050°C.

Translucent and transparent *Vitreosil* rods of the proper size for the above operations are obtainable.

Silica Torsion Fibres

These fibres can be made by drawing by hand or by simple mechanical means, and by blowing them out with a flame. Here we describe only simple methods for making fibres by drawing them out with a weight falling under gravity. With these methods fibres with a range of sizes can be made quite quickly. T. C. KEELEY (1924) fixed a small weight to a silica rod and heated this at a point until the silica softened sufficiently for the weight to fall and draw out a fibre. V. J. CLANCEY (1950), during an investigation of the evaporation and

surface mobility of naphthalene molecules, used a delicate torsion apparatus, the fibre for which was made by the gravity method. He fused a silica hook to a 1-mm diameter silica rod. The rod was clamped vertically and a weight of about 10 gm hung on the hook. The rod was heated with a flame just above the hook, and just as the weight started to fall the flame was whisked away. The weight fell on to a soft material, such as a rubber sheet, to stop it bouncing. By varying the weight and the moment the flame is taken away, fibres with different sizes can be made. Some preliminary practice is re-

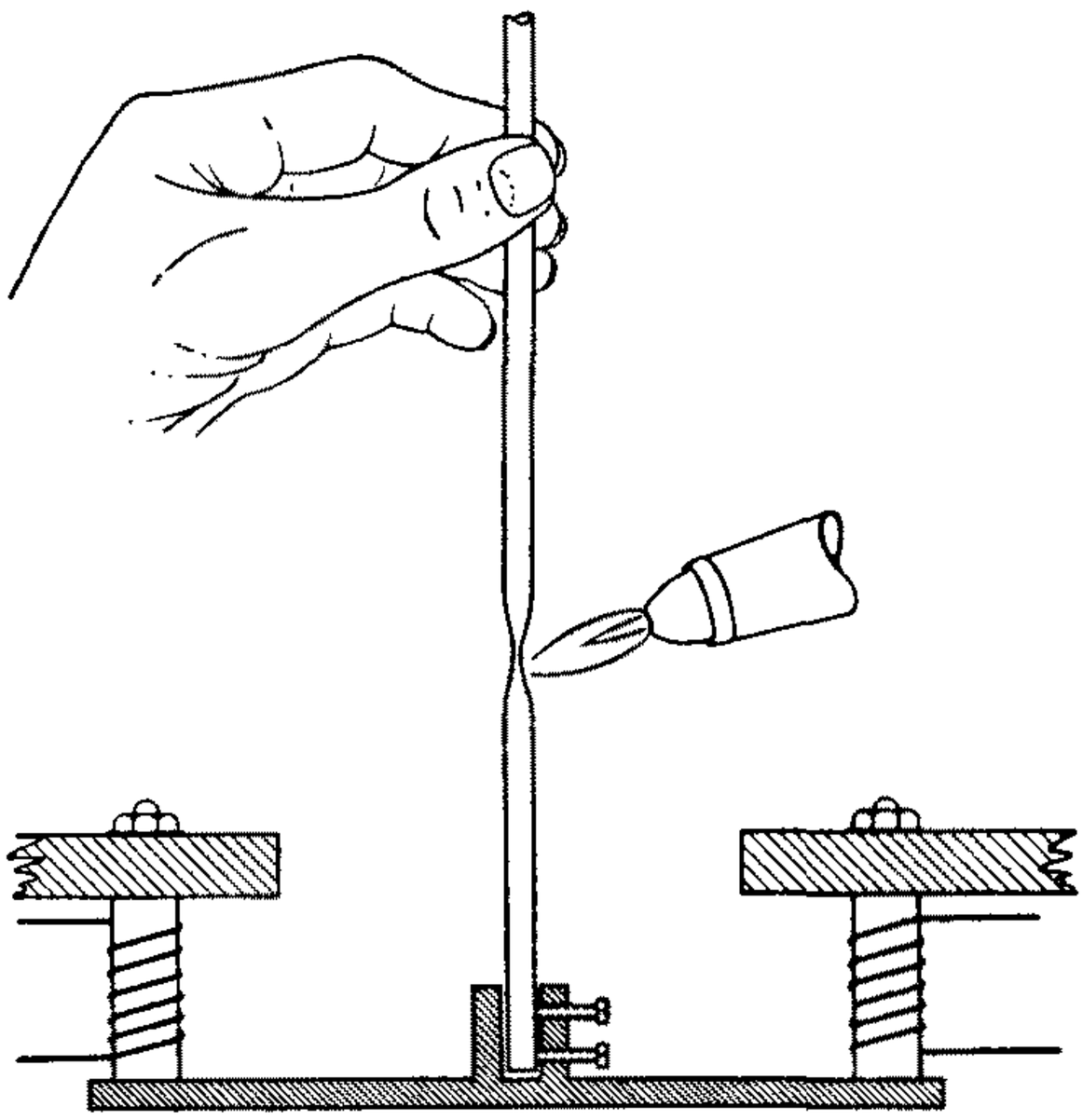

FIGURE 85. The production of a silica fibre by drawing out a fused rod with a weight falling under gravity

quired. If the flame is kept on too long the fibre will fuse and break, and if the flame is kept on for too short a time the weight will not fall the full length.

We find it more convenient to draw out fibres by the gravity method with a weight of about 500 gm which is released from a pair of electromagnets. The weight consists of an iron bar about 25 cm long, to the centre of which the silica rod of 2–5 mm diameter is attached with a socket and bolt arrangement. The bar is held at each end by a small magnet, and the two magnets are connected in series. A portion of the rod is heated with a hand torch and the top of the rod is held in one hand (FIGURE 85). A semi-molten portion of

desired thickness is obtained, the magnet current is turned off with a foot-operated switch, the flame is whisked away, and the weight falls into a box of sand. If all goes well a long fibre is produced. The semi-molten part of the silica rod, when initially thin, gives a thin fibre, and when thick, a thick fibre; the fibre thickness is also determined by the precise moment the flame is whisked away—for a thin fibre this should be just after the weight starts to fall. Fibres down to 5–10 microns diameter can be made in this way.

N. J. TIGHE (1956) has given a comprehensive survey of the production, properties and applications of silica fibres; G. A. DOWNSBROUGH (1937) has discussed the damping of fibres. With a rubber catapult method, introduced by R. THRELFALL (1898), fibres in the range of 2–30 microns diameter can be made. Smaller fibres can be made by the drag of the flame gases in an oxy-hydrogen flame on a tiny globule of molten silica, a method which Threlfall describes. This author also gives an account of the bow-and-arrow method of C. V. Boys.

Small fibres should be manipulated against a dark—preferably black—background, and small camel's-hair brushes moistened with water are very convenient implements. Fibres, when examined under a microscope, become more easily visible if they are mounted under a cover slip and a drop of glycerine, coloured by a dye, is allowed to run in.

P. L. KIRK and R. CRAIG (1948) have discussed the construction of fibre devices; L. WALDEN (1937) has given a practical account of several uses of fibres as instrument suspensions; D. R. BARBER (1930) has described a device for mounting fibres with a degree of tension; and H. V. NEHER (1940) has given a very practical account of the manipulation of silica fibres.

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