LABORATORY GLASS BLOWING

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LABORATORY MANUAL OF GLASS BLOWING

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PREFACE

In the fourteen years that have passed since the first publication of my "Laboratory Manual of Glass Blowing," there have been some important changes in the problems confronting the laboratory glass worker, particularly on account of the introduction of Pyrex glass. The steady demand for the book, requiring two reprintings during these years, has made it seem worth while to amplify it and bring it up to date. As I have had little personal touch with the art of glass blowing in recent years, I have entrusted the revision and enlargement of the book to my friends and co-workers, Messrs. Junius D. Edwards and Cyril S. Taylor. Out of their practical experience they have made a few alterations and have added a considerable amount of new material which we believe will be of value to those who have occasion to work glass in the physical and chemical laboratory.

The purpose of this book is to provide a clear and detailed discussion of the elements of glass working. Many laboratories in this country, especially in the West, are located a long way from any professional glass blower, and the time and money spent in shipping broken apparatus several hundred miles to be mended could often be saved if some of the laboratory force could seal on a new stop cock, replace a broken tube, or make some temporary repairs. Many men in physical or chemical laboratories have occasion to modify some piece of apparatus, designed, perhaps, for other uses, or to design new apparatus. To such the ability to perform some of the operations herein described may also be very valuable.

No originality is claimed for the methods here described. They are those which the authors have found most suitable and convenient in their own work and most easily learned by students. The aim has been to describe each operation in such detail that a beginner can follow the process without help and, with practice, attain satisfactory results. It is, however, much easier to perform any of the operations described after seeing someone else perform it correctly, since the temperature, the exact time to begin blowing the glass, and many other details are very difficult to obtain from a description.

It has not been thought worth while to describe the process of making stop cocks, thermometers, vacuum tubes, and so forth, as such things can be purchased more cheaply and of much better quality than any amateur can make unless he is willing to spend a very large amount of time in practice. For similar reasons, the manipulation of quartz glass has been omitted, but the practical importance of Pyrex glass in the laboratory and its increasing popularity seemed to justify a description of methods for working it.

The authors will be grateful for all suggestions and criticisms tending to improve the methods presented. If some of them appear to be given in excessive detail, the reader will remember that many things which are obvious to the experienced worker are not so to the beginner and that it is the little details in the manipulation which often spell success or failure in glass blowing.

Acknowledgment is due Mr. F. Kraissl, of the Corning Glass Company, for many suggestions regarding the working of Pyrex glass and to Mr. George W. Morey, of the Geophysical Laboratory, Carnegie Institution, Washington, D. C., for the information on the composition of the various glasses as given in Table 1.

OAKMONT, PA.
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FRANCIS C. FRARY.

CONTENTS

PAGE

PREFACE vii

CHAPTER I. GLASS AND ITS WORKING CHARACTERISTICS . . . 1
- Soft Glass 3
- Devitrification 5
- Annealing 7
- Pyrex Glass 8
- Lead Glass 9
- Glass-blowing Flame 9
- Cross Fires 11
- The Work Table 12
- Arrangement of Exercises 13

CHAPTER II. GENERAL OPERATIONS 15
- Cleaning the Glass 15
- Cutting Soft Glass 16
- Cutting Pyrex Glass 17
- Bending Glass 21
- Drawing Out a Tube 21
- Constricting a Tube 22
- Flanging a Tube 23
- Special Supports 26
- Rotation of the Tube 27

CHAPTER III. ELEMENTARY EXERCISES 30
- Joining Two Pieces of Tubing End to End—First Method . 30
- Joining Two Tubes End to End—Second Method 36
- The "Tee" Tube 38
- To Join Two Tubes of Different Diameters 41
- Tube for Condensing Sulphur Dioxide 44
- Bulb at the End of a Tube 44
- Blowing a Bulb in a Tube 49

CHAPTER IV. ADVANCED EXERCISES 52
- Sealing a Tube through Another Tube 52
  - First Method—Making a Gas-washing Tube 52
  - Second Method—Making a Suction Pump 57

PAGE

CHAPTER V. MODIFIED METHODS AND SPECIAL OPERATIONS . . . 62
Capillary Tubing . . . 62
Glass Rod . . . 63
Temporary Handle for Short Pieces . . . 64
Mending Stop Cocks . . . 66
Closed Circuits of Tubing . . . 69
Making Joints and Repairs on Apparatus . . . 71
Spirals . . . 73
Cone-and-socket Joint . . . 75
Ground Joints . . . 79
Smoothing and Squaring the End of Glass Tubing . . . 79
Sealing Vacuum Tubes . . . 80
Closed Tubes for Heating under Pressure . . . 81
Special Glass-sealing Method . . . 83
Pyrex-to-soft-glass Joints . . . 84
Sealing Glass to Porcelain . . . 90
Etching of Glass . . . 92

CHAPTER VI. GLASS-TO-METAL SEALS AND JOINTS . . . 94
Sealing in Platinum Wire . . . 94
Glass to Metal Joints with Solder . . . 97
Sealing in Base Metals . . . 99

APPENDIX . . . 109
Platinizing Solutions . . . 109
Stop-cock Lubricants . . . 109
Vacuum Wax . . . 110
De Khotinsky Cement . . . 111
Cement . . . 111

INDEX . . . 113

LABORATORY GLASS BLOWING

CHAPTER I

GLASS AND ITS WORKING CHARACTERISTICS

Some knowledge of glass is fundamental to a study of glass working. A tender consideration must be shown for its mechanical and thermal properties, if glass is to be successfully fashioned into the many forms in which it is used in the laboratory. Glass is an amorphous material which is hard and rigid at room temperatures, but when it is heated it gradually softens and becomes less viscous. The change on heating is continuous from the temperature at which it is a rigid solid to temperatures at which it becomes a thin liquid; glass has no melting point or melting range. Heating glass, even to a low temperature, produces some softening, even though it may not be ordinarily appreciable. At temperatures of several hundred degrees Centigrade, however, the degree of softening is quite important. The temperature at which glass softens enough to work varies appreciably with the composition of the glass.

One of the most important factors in the success of any piece of glass blowing is the glass employed. There are many varieties of glass available for glass-blowing operations. The two most generally useful

TABLE 1.—ANALYSES OF A NUMBER OF TYPES OF GLASS TUBING
(Table Prepared by George W. Morey, Geophysical Laboratory, Carnegie Institution, Washington)

	1	2	3	4	5	6	7	8	9	10	11	12
SiO_2	80.75	72.86	64.7	79.57	66.90	66.58	69.30	69.10	71.88	61.26	68.69	61.5
B_2O_3	12.00	10.43	10.9	.....	7.22	0.91	.....	.....	.....	.....	.....	.....
Na_2O	4.10	9.82	7.5	0.66	1.25	14.80	12.80	14.50	18.57	13.08	15.87	8.8
K_2O	0.10	0.10	0.37	11.60	2.40	tr	4.96	6.38	1.98	3.98	7.32	5.3
CaO	0.30	0.35	0.63	7.80	7.94	7.18	8.28	6.89	4.90	9.25	5.66	.....
ZnO	.....	.....	10.9	.....	7.27	6.24	.....	.....	.....	.....	.....	.....
MgO	.....	0.20	0.21	0.11	0.61	0.17	0.07	0.20	0.20	0.20	0.24	.....
Al_2O_3	2.20	6.24	4.2	0.32	6.38	3.84	4.05	3.10	2.75	7.92	2.11	0.8
Fe_2O_3		tr	0.25	0.04	0.22	tr	0.18	.....	.....	0.30		
Mn_2O_3	.....	tr	0.01	.....	.....	0.28	0.20	0.10	0.10	0.30	.....	.....
Sb_2O_3	.....	.....	.....	.....	.....	.....	.....	.....	.....	3.45	.....	.....
PbO	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	23.6
As_2O_5	0.40	.....	0.14	.....	.....	.....	tr	0.10	0.10	.....	.....	.....

1. Pyrex laboratory glass.
2. Schott, borosilicate thermometer 59^{IV}.
3. Schott, Jena *geräte*.
4. Kavalier combustion tube.
5. Jena combustion tube.
6. Schott, normal thermometer 16^{III}.
7. A good German lamp-working tubing.
8. Similar to 7.
9. A good French lamp-working tubing.
10. Good German tubing with tendency to discolor.
11. Inferior Thuringian glass.
12. Lead glass.

kinds for laboratory work are lime-soda glass and the low-expansion borosilicate glass known as Pyrex laboratory glass. A lead glass is used but to a lesser extent. Other glasses are available for special purposes. The composition of a variety of types of glass tubing is shown in Table 1.

Soft Glass.

Glass-blowing tubing of the kind rather vaguely known as *soft glass* is usually a lime-soda glass, although potash constitutes part of the alkali content. A small percentage of potash seems to decrease the devitrifying tendency of the glass. Some of the *hard* glasses carry more potash than soda in their makeup. The soft glass from different makers will vary in composition, and, if not carefully controlled, glass from different batches of the same maker may vary considerably in composition and properties. Uniformity of properties is necessary where more or less complicated pieces of apparatus are to be put together from different pieces of glass.

When working with unknown lots of glass which are to be joined together, it is advisable to make a small test joint first. If the glass fuses together nicely and if the joint does not crack upon cooling and upon tapping it rather vigorously upon a wooden block, it may be assumed that the two lots of glass are near enough alike to be joined together.

The following qualities are desirable in a soft glass for ordinary working:

1. Moderately low working temperature.
2. Freedom from air bubbles, striations, and irregularities.

3. Proper composition so that the glass will not devitrify or crystallize while being handled at its working temperature.

4. Ability to withstand fairly rapid heating without cracking.

The working temperature of different samples of so-called *soft glass* varies a good deal and is best determined by trial. The glass should become almost soft enough for blowing in a flame which still shows a little yellow near the tip, so that at the highest temperature of the flame it may flow fairly freely and, thus, easily eliminate irregularities in thickness. If the glass is too hard, the shrinking of the glass, collection of material for a bulb, and, in fact, most of the working processes will be slower, and the glass will not stay at its working temperature long enough after its removal from the flame to permit it to be blown readily.

Air bubbles in the original batch of glass are drawn out into long, hairlike tubes during the process of manufacture. When such tubing is worked, the walls of these microscopic tubes may collapse in spots, and the air thus enclosed will often collect as a small bubble in the wall, thus weakening it. Occasionally, one end of one of these microscopic tubes will connect with the interior of the tube, and the other end will be open on the outside of the tube. Such a piece of glass, if used in an apparatus to hold high vacuum, will be a constant source of leakage. This condition can usually be detected by determining whether a colored liquid, such as dyed alcohol, will be drawn up by capillarity into the suspected opening.

Irregularities are of various kinds. Some of the larger sizes of thin-walled tubing often have one half of their walls much thicker than the other, and such tubing should be used only for the simplest work. Some tubing has occasional knots or lumps of unfused material. The rest of the tube is usually all right, but often the defective part must be cut out. The presence of striations running along the tube is, generally, an indication of hard, inferior glass. Crookedness and non-uniformity of diameter are troublesome only when long pieces must be used.

Devitrification.

Devitrification is one of the worst faults glass can possibly have. It is especially common in old glass and in glass which has contained acids. It seems to be of two sorts. One variety manifests itself on the surface of the glass before it reaches its working temperature, but if the glass be heated to the highest temperature of the flame it will disappear except in the portion at the edge of the heated part. The glass seems to work all right, but an ugly, crystallized ring is left at the edge of the portion heated. This kind appears most frequently in old glass which was originally of good quality but has, in time, been superficially altered, probably by the loss of alkalies. The other variety of devitrification does not appear when the glass is first heated; but after it has been maintained at or above its working temperature for a longer or shorter time, it will be noticed that the outer surface has lost its smoothness and appears to be covered with minute wrinkles. It will, also, be found that the glass has become harder, so that it becomes impossible to work it easily. Further heating only makes the matter worse, as does the use of a higher temperature from the start. In fact, it will often be found that a piece of comparatively soft glass which devitrifies almost at once in a "hissing" flame can be worked without serious difficulty if care be taken to use a flame still decidedly tinged with yellow. Even good glass will begin to devitrify in this way if heated too long at the highest temperature of the flame, so care should always be taken (1) to reduce the time of heating of any spot of glass to a minimum, *i.e.*, get the desired result at the first attempt, if possible, or at least with the minimum of reheating and "doctoring," and (2) to avoid keeping the glass at the highest temperature of the flame any longer than necessary. This may be accomplished by doing all heating, shrinking, etc., of the glass in a flame more or less tinged with yellow and raising the temperature to the highest point only when ready to blow the glass. This kind of devitrification is apparently due to volatilization of the alkalies from the glass in the flame.

Devitrification is fundamentally the result of surface alteration of the glass. Old glass apparently undergoes a kind of weathering as a result of the action of moisture and carbon dioxide upon the oxides of the glass. Under certain conditions, it has been noted that old glass will show an efflorescence of sodium bicarbonate upon the surface, and such a glass shows a marked tendency to devitrify upon heating. Germann[1] believes that heating drives off water, leaving dehydrated or separated silica on the surface, which

[1] Germann, A. F. O.: *J. Am. Chem. Soc.*, **43**, 11 (1921).

is responsible for the devitrified appearance after heating. In some cases, this excess silica will redissolve in the underlying silicates upon heating and the glass partially clears up as previously noted. Germann found, however, that if the glass were washed first with dilute hydrofluoric acid to remove the silica, it would then work in the flame just as if it were new glass. Glass which has lost alkali by volatilization as the result of too long heating in the flame may also be washed with hydrofluoric acid and reheated without devitrification. This treatment should be very useful in the repair of old glass apparatus.

It is said that devitrification caused by volatilization of the alkalies from glass can be partly remedied or prevented by heating the glass in a strong "sodium" flame. This is accomplished by wrapping asbestos paper around a piece of tubing, saturating it with a strong salt solution, and holding it in the flame as the glass is heated.

In order to avoid, as far as possible, this weathering or aging, glass should be stored in a *dry* place free from acid vapor and laboratory fumes. In handling, care should also be taken to avoid scratching of tubing by drawing one piece over another. Scratched tubing is especially susceptible to breakage. In fact, it is common practice to scratch a tube preparatory to breaking it in two.

Annealing.

The toughness of glass, *i.e.*, its ability to withstand variations of temperature, depends on its composition and the care taken in its annealing. In general, large pieces of glass should be heated very slowly in the smoky flame, and the larger the diameter of the tube the greater the length which must be kept warm to prevent cracking. All large pieces should be carefully heated over their whole circumference to the point where the soot deposit burns off, before being finally cooled. After being thus heated, they are cooled in a large, smoky flame until well coated with soot; then the flame is gradually reduced in size, and the object finally cooled in the hot air above it until it will not set fire to cotton. If thought necessary, it may then be well wrapped in cotton and allowed to cool in the air. If not properly annealed, the place heated may crack spontaneously when cold, and it is quite certain to crack if it is reheated later.

Pyrex[1] Laboratory Glass.

Not all of the above remarks are applicable in the case of Pyrex glass. Pyrex glass softens at a considerably higher temperature than soft glass, so that the flame tinged with yellow will not furnish a temperature hot enough to soften the glass properly. In fact, a special burner or special means must usually be employed to produce the necessary temperature. As Pyrex glass will become rigid more quickly than lime-soda or lead glass when removed from the flame, the removal and blowing should be carried out more rapidly than is necessary with soft glass. Pyrex glass is a high-silica glass and has a very low temperature coefficient of expansion. It can, therefore,

[1] Pyrex is a trade mark owned and used by the Corning Glass Works, Corning, N. Y., for a group of its special low-expansion glasses. The glass referred to as Pyrex, throughout this book, is Pyrex laboratory glass.

be introduced into the flame and heated up at a much greater rate than is permissible in the case of soft glass. Also, the precaution of allowing the article to cool slowly in a smoky flame is not usually necessary. If Pyrex glass is reheated too often or at a too-high temperature, it tends to become opaque. This is not detrimental to its usefulness but may spoil its appearance.

Lead Glass.

Lead glass carries a substantial percentage of lead oxide and is softer and more readily fusible than lime-soda glass. It is used in the manufacture of lamp bulbs, vacuum tubes, etc., and by professional glass blowers. It has the disagreeable property of turning black in a few seconds unless worked in a strongly oxidizing flame. This may be prevented by using a "hissing" flame, with a large excess of air, and working in the extreme end of the flame; or the black lead may thus be reoxidized and the glass restored to its original clearness.

Glass-blowing Flame.

Next in importance to the glass is the flame in which the glass is to be worked. Any good blast lamp, such as is ordinarily used in a chemical laboratory for the ignition of precipitates, will be satisfactory for soft glass, provided it gives a smooth, regular flame of sufficient size for the work in hand, and when turned down will give a sharp-pointed flame of well-defined parts. Manufactured gas as supplied for domestic use makes an ideal fuel for such a lamp, but, where it is not available, some laboratories use "gasoline gas" made by saturating air with gasoline vapor in special apparatus. Most laboratories are now supplied with low-pressure air, which can be used for operating the lamp. There is also available on the market a number of small-capacity compressors large enough to operate a blast lamp and which can be conveniently used for this purpose where compressed air is not continually "on tap." A Bunsen burner should also be close at hand on the work table, as it is a valuable adjunct to the blast lamp in many operations.

The laboratory type of bellows with rubber diaphragm can be used if it gives a steady supply of air under sufficient pressure for the maximum size of flame to be used. A bellows with a leaky valve will give a pulsating flame which is very annoying and makes good work very difficult. Where much glass blowing is being done, the operation of the foot bellows is tedious, to say the least.

It is very difficult to operate the ordinary glass-blowing lamp with natural gas and compressed air and secure a flame which can be varied in size as needed and not be continually blowing out. A lamp can be operated satisfactorily, however, with natural gas and air enriched with oxygen. The flame is very hot, naturally, and only the simplest operation can be performed in it with soft glass. Pyrex glass, however, can be handled very satisfactorily with it. Special lamps are now available for use with natural gas and compressed air.

Pyrex glass tubing requires a higher temperature than soft glass for easy working, and, therefore, it is very essential to have a blowpipe or blast lamp so equipped that it will produce the higher temperature required. While the smaller sizes of tubing up to about 1/2 inch in diameter will soften sufficiently in the ordinary gas-air blast lamp and still better in the so-called *cross fires,* which will soften Pyrex glass tubing up to 1 inch in diameter, the addition of oxygen is necessary when manipulating various sizes in order to get the proper softening so that a uniform joint may be obtained upon blowing. There are now several types of burners on the market which are quite efficient in handling Pyrex glass. One, a regular blast lamp with a third inlet for oxygen, is preferable, as it enables the worker to heat the part which is to be worked more uniformly by the air blast before adding the oxygen for the final softening. If acetylene is available, it can be used with compressed air in an ordinary glass-blowing lamp for working Pyrex glass. If a gas-oxygen or oxyacetylene flame is used alone, it will quickly soften this tubing, but the glass is liable to be strained, in which case the object has to be re-annealed. In using a burner of the inlet type, it is very desirable that the cylinder containing the compressed oxygen be supplied with a pressure regulator, so that the amount of oxygen introduced into the burner can be properly controlled. Warning! No oil or grease should be used for the lubrication of the stop cock or regulator.

Cross Fires.

Another type of burner used by glass blowers is known as the *cross fire.* Cross fires are particularly useful in heating tubing of large diameter and are standard equipment with the professional worker.