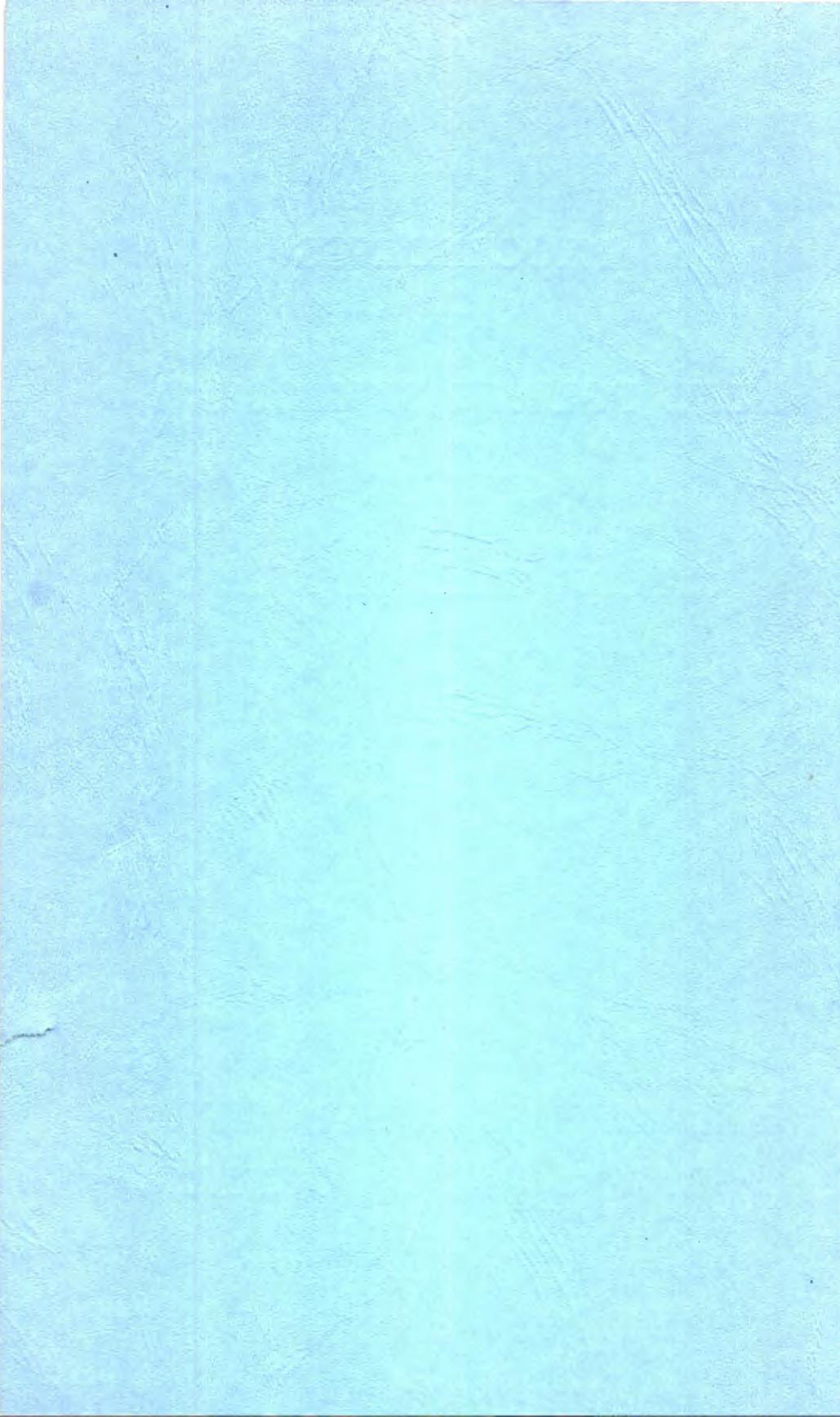


PROCEEDINGS

**THE FOURTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING**

1959

**THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY**



**THE FOURTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING**

Sponsored by
**THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY**

In Cooperation With
**CORNING GLASS WORKS
CORNING, N.Y.**

Thursday, May 21, 1959

Friday, May 22, 1959

Saturday, May 23, 1959

Published By

THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY

309 GEORGETOWN AVENUE
GWINHURST, WILMINGTON 3, DELAWARE

F O R E W O R D

For the third time in the last four years, our Society had the pleasure of holding its annual meeting in Corning, New York, where again our gracious and genial host was the Corning Glass Works.

The registration for this fourth symposium on the "Art of Glassblowing" was by far the largest of the four, by at least 150. This was, of course, most gratifying to those responsible for the symposium, as well as to your officers and directors.

In trying to establish the reason for this relatively large increase in attendance, most of our members agreed it must be the magic word "Corning". This continued growth and success of the Society is, I am sure, a matter of pride to each and every member.

We are deeply indebted to the Program Committee, which once again presented a highly interesting and informative technical program, designed to keep our members abreast of the most recent techniques and innovations in the "Art of Glassblowing".

Not only to those who registered, who will receive a copy, but to make available to those not fortunate enough to attend the symposium, we submit herewith the material that was presented.

J. Allen Alexander
President and Founder

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Welcome Address To The American Scientific Glassblowers Society

**O. M. Loytty
Corning Glass Works**

Speaking on behalf of Corning Glass Works, as well as myself, welcome to all of you. Looking around this auditorium, perhaps I should more properly say WELCOME BACK, because, for many of you, this is a return visit. We are tremendously pleased to have you with us again.

This is, you know, the third time in the last four years that Corning has been given the honor of serving as your host. Let me assure you that we do indeed consider this an honor—and we are grateful for the privilege.

I recall that on the occasion of your first symposium—held in Corning in 1956—I was amazed by the turnout. I believe I publicly expressed my surprise that SO MANY PEOPLE WERE SO INTERESTED IN THEIR WORK. But that was four years ago. The turnout this morning, I'm sure, exceeds the number we greeted in 1956—and this, of course, pleases all of us. But it is no longer cause for amazement. We have long since realized what importance you justifiably place on these sessions—and how important and worthwhile they are to all of us.

It is with this in mind that my remarks will be kept to a minimum. We are all anxious to get on with what promises to be an exceptionally fine program.

Your officers, chairmen and committees have worked long hours

in preparation for the symposium and annual meeting. I feel that they have done an outstanding job.

During the course of your sessions here, you will be touring the Glass Center and our apparatus plant. We are extremely proud of these facilities and are anxious to have you see them.

I'm sure you will find the tours interesting. May I emphasize that **WE WANT YOU TO FEEL AT HOME . . . FEEL FREE TO ASK QUESTIONS . . . AND, IF ANY TIME DURING YOUR STAY, WE CAN BE OF SERVICE, PLEASE CALL UPON US.**

Again, a hearty welcome. I know you will have a successful meeting.

Standardization Of Glassware

Elmer L. Jolley

Corning Glass Works

A native of Waterloo, N.Y., Mr. Jolley received his B. S. Degree in Chemical Engineering from the University of Alabama. After a year with Tennessee Copper Company, he joined Corning Glass Works in 1945. He has worked on various committees writing laboratory glassware specifications for the American Society for Testing Materials, the American Standards Association, the International Standards Organization, and the Micro Committee of the American Chemical Society.

In this country we have many organizations specifically engaged in the work of standardization of glassware used in the laboratory. Some of these are the U. S. Department of Commerce, American Society for Testing Material and the American Standards Association. All of these may cover the basic characteristics of a single item or of a group of items. There are also in existence federal specifications which serve as purchase specifications, prepared for the use of all federal agencies. These are detail specifications and cover items which the government buys repeatedly and in quantity. Although these are not general standards, most manufacturers follow these specifications so that only one grade of ware is manufactured rather than having items made special for the government.

I will attempt to cover the work done by the organizations which I feel will be of the most interest to you.

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards was set up in 1901 by an act of Congress. It provided for (1) the custody of the standards, (2) for the comparison of standards used in scientific investigation, engineering, manufacturing, commercial and educational institutions with the standards adopted or recognized by the government, (3) the construction, when necessary, of standards, their multiples and subdivisions, (4) for the testing and calibration of standard measuring apparatus, (5) the solution of problems arising in connection with standards, and (6) for the determination of physical constants and properties of materials, when such data are of great importance to scientific or manufacturing interests and cannot be obtained with sufficient accuracy elsewhere.

The National Bureau of Standards at this time was a part of the Treasury Department but in 1903 was transferred to the Department of Commerce of which it is a part today. The Bureau does research in many areas toward the establishment of basic standards for items such as electricity and electronics, metallurgy, and data processing as well as improving the accuracy of such items as the inch, the ounce and the second.

In 1904, the National Bureau of Standards published Circular No. 9, *The Testing of Glass Volumetric Apparatus*. This specification, outlining the requirements of volumetric apparatus, agreed, with only a few exceptions, with those recommended by a committee of the American Chemical Society. In many cases, the regulations set up by Kaiserliche Normal-Eichungs-Kommission of Germany were adopted without appreciable change. In other instances, important changes were made, based on experimental work performed by the National Bureau of Standards.

Circular C9 was revised several times between 1904 and 1941, when Circular C434 was issued. The latest of this long line of revisions, with resulting improvement in the quality and accuracy of volumetric apparatus, is N.B.S. Circular 602, *Testing of Glass Volumetric Apparatus*, issued April 1, 1959.

The requirements outlined in this specification are for precision

grade instruments and items submitted must meet these specifications before being certified by the National Bureau of Standards. Included are not only volumetric accuracy but also dimensions, graduations and inscriptions. This circular, like its predecessors, requires that each item be serially numbered and on items which deliver their contents, such as burets and pipets, the delivery time must be specified. Only a fraction of the total number of items manufactured each year to these specifications are sent to the Bureau for certification.

The requirement on serialization and outflow time increases the cost of the item to the user as a result of the additional time spent in performing these operations. Therefore, most manufacturers offer a choice to the users, i.e. with and without the added requirements. Volumetric flasks and pipets are a good example of the ware being made today by reputable manufacturers as Class "A" items which will meet the dimension, graduations and accuracy tolerances specified by the National Bureau of Standards, but not the other requirements. There are also many burets, measuring pipets and cylinders made and used today for routine test purposes which, although meeting the requirements for dimensions and graduations, are not made to the accuracy since either through use or the test method being used it has been determined that this accuracy is not required. Therefore, the payment of a premium price to obtain this precision is not justified.

As you know, prior to World War I, this country was dependent for laboratory glass upon imports from Europe. This included ground joints and stopcocks. At the outbreak of war, this source of supply was cut off and Corning Glass Works began making PYREX brand chemical glassware. During the early days of this endeavor, little or no attempt was made toward fabrication of ground joints. As the need for this convenient feature in laboratory glassware became more evident, Corning in 1918 was asked to produce ground joints. These first American-made interchangeable ground joints had a Morse taper of 1:8, the ratio commonly used in the machine industry. Following the successful progress of these early experiments, several other manufacturers undertook similar devel-

opments. However, there was no attempt at standardization of taper or size and it soon became apparent to all that some definite standards must be adopted. A group of manufacturers and distributors requested help from the National Bureau of Standards on this problem and on December 17, 1929 a general conference of users, distributors and manufacturers of interchangeable ground joints was held at the National Bureau of Standards in Washington, D.C., to consider establishment of specifications for diameter and taper of ground joints on the basis of a preliminary draft submitted by the committee. The conference adopted the preliminary draft with minor changes and recommended acceptance by the industry. The proposed standard was circulated to industry and, after formal acceptance was obtained, it was printed as Commercial Standard CS21-30, which became effective on August 1, 1930.

It soon became apparent that this standard should be extended to include other items such as straight bore stopcocks, flask stoppers, reagent bottle stoppers and additional sizes of ground joints. The standing committee, as a result of conference on May 25 and July 20, 1933, proposed a revision to Circular CS21-30 which was sent to industry on January, 1934 for written acceptance. The result was that the revised standard was accepted and authorized for publication as Commercial Standard CS21-34. This was again revised in May, 1936.

At that time, the designation on ground joints was changed from a single number, which designated the approximate diameter at the small end, to the familiar two number designation, which specifies the approximate diameter at the large end and the approximate length of the ground surface. The standard was also expanded to cover additional sizes of ground joints and became known as CS21-36.

In February, 1939, the standard was again revised to provide for a series of short length joints designed primarily for use on weighing bottles with interchangeable covers. It then became known as CS21-39. This standard was in use for several years, but during 1955 and 1956 the standing committee again recommended

a change to cover various styles of stopcocks and ball and socket joints which had become widely used. In October, 1957, the Commodity Standards Division announced acceptance of the revision to become effective July 1, 1958. There were several changes in this specification, the most important of which are :

1. Title was changed to provide for the inclusion of spherical ground joints and the omission of the word "glass". The inclusion of spherical ground joints is obvious and the word "glass" was omitted in order that the specification could be used to cover other materials, such as metals and plastics.

2. Inclusion of more styles of stopcocks which are routinely used. These are the single and double oblique bores, T bores and 120° bore.

3. Outlined the requirements for ball and socket joints and the method of testing these joints.

4. Gauge drawings to check standard taper ground joints, stopcocks, flask and bottle stoppers were included.

5. Some important dimensional changes were also made. The fit of a ground joint is now specified so that the inner joint will fit either flush with the top of the outer joint or will ride high. This was done to eliminate the difficulty encountered when the inner joints protrude through the outer on such items as distilling flasks. This change was accomplished by allowing a plus tolerance of 0.15 mm on the gauging diameter of an inner member and a minus tolerance of 0.15 on the outer member. The 12 mm diameter joint, i.e., Standard Taper 12/30, Standard Taper 12/18 and Standard Taper 12/10 had the diameter at the large end or gauging joint changed from 12.0 mm to 12.5 mm. This change was made in the interest of international standardization.

There is in existence an International Standards Organization, which is composed of the standards group of a number of countries who are working together to obtain uniform standards on certain basic items. Our contact with this group is through the American

Standards Association, located in New York City. This is advantageous to both the manufacturer and the consumer. To the manufacturer, it means that ware can be shipped to other countries and perform satisfactorily. This also holds true for the consumer and is particularly advantageous to the American consumer with holdings throughout the world since equipment and tests can be duplicated. The final international standard requires compromises by all countries since differences existed in the standards of the various countries. Although differences do exist between Commercial Standard CS21-58 and the I.S.O. requirements, these are comparatively minor and the joints when mated will still perform satisfactorily. We realize, of course, that for a time this change will be inconvenient to you but as soon as all of the old joints, 12 mm diameter at the top, are used up throughout the country, this situation will improve.

The other change in dimension was on reagent bottles and stoppers having a Standard Taper 29 stopper. This change consisted of changing the diameter at the large end from 29.0 mm to 29.2 mm to conform with the dimension of Standard Taper 29/42, Standard Taper 29/ 26 and Standard Taper 29/12 ground joint at this point. This change seemed to be very desirable since reagent bottles are being connected to items such as burets by this joint.

Commercial Standards are developed by manufacturers, distributors and users in cooperation with the Commodity Standards Division of the Office of Technical Services and with the National Bureau of Standards. The United States Department of Commerce has no regulatory power in enforcement of their provisions but they become established trade custom and the provisions contained in the standard are upheld by reliable manufacturers.

AMERICAN SOCIETY FOR TESTING MATERIAL

The American Society for Testing Material also works in standardizing items of glassware as well as other materials. The items standardized by this group, such as Engler and Saybolt flasks, are usually designed for a specific test or group of tests, although they recently issued a tentative specification for the determina-

tion of maximum pore diameter and permeability of rigid porous filter media used in the laboratory. An A.S.T.M. committee working on a definite problem is faced with a need either for a new piece of equipment or a modification of an existing item to perform the test properly. They usually work with a subcommittee, composed of manufacturers and representatives of other committees, in designing the final item so that the best and most economical item will be obtained. Tests are then run on the item to insure that it meets the requirements made of it and that the test itself is satisfactory.

THE AMERICAN CHEMICAL SOCIETY

The American Chemical Society has a committee which recommends specifications on micro chemical apparatus. This committee is also composed of manufacturers, distributors and users of the items in question. The small volumetric flasks with bases for greater stability and the companion pipets were designed by this committee as well as the microliter (λ) pipets.

AMERICAN STANDARDS ASSOCIATION

As an outgrowth of the many standards developed by various trade associations, technical societies and the military services during World War I, the need for coordination led to the establishment of the American Engineering Standards Committee in 1918 by five of the leading technical and professional trade organizations. Later the name of this group was changed to the one by which it is presently known, American Standards Association.

A.S.A. itself has never written or developed a standard but is essentially a clearing house where other groups engaged in writing standards can cooperate in reaching an agreement so that there will be no conflict or duplication of standards. All parties interested in the subject have a right to participate and work toward this agreement. The A.S.A. function is to see that procedures are followed and to judge the importance of negative votes as well as the reason behind them. By doing this, A.S.A.

guarantees a consistent standard for American standards.

There are various methods used in developing and securing approval of American standards. One of these is the balanced committee, comprised of manufacturers, distributors, users and generally interested members. This committee operates under the administrative leadership of one body, usually the one which requested that the work be performed. Another method is for a leading society or association to propose that a standard, which they have developed, be given an American Standards Association number. A third method is the general acceptance method, used in cases where the nature of the standards is such that agreement can be reached on the details in a single meeting and acceptance can be obtained by letter ballot.

INTERNATIONAL STANDARDS ORGANIZATION

The International Standards Organization is an outgrowth of the pre-war International Federation of the National Standardizing Association and the United Nations Standards Coordinating Committee and was organized in November, 1946. Its primary objective is to harmonize national standards. It is a federation comprised of the national standards bodies of 40 countries and is administered by a council elected by the members. Standardization projects can be proposed by any country and, if five or more countries agree to participate, a project can be authorized and a committee organized. Recommendations developed and approved by a majority of Technical Committee members can be distributed for vote by all member countries. If approval by a majority is obtained, the document is printed as an I.S.O. recommendation. If there are no adverse votes, the document is printed as an I.S.O. standard. Presently, in our field, we are working with I.S.O. on five different standards: Quality of Glassware, Thermometers and Hydrometers, Ground Joints, Volumetric Glassware, and Apparatus for Determining the Percentage of Fat in Milk by the Babcock Method.

Standardization, whether on a national level or on an international level, is beneficial to all of us as long as only the basic

requirements that effect the function of the item are standardized. Standardization should not be carried to the point where better methods or changes in design cannot be made to utilize advances in technology. One of the changes in laboratory glassware, which occurred as the result of advanced technology in manufacturing procedures and processes, is our Accu-Red pipet and buret line where, on a production basis, we can screen scales on such items while still maintaining the accuracy required.

Standardization allows manufacturers to produce at a lower cost, not only because of larger runs but frequently with less highly trained or specialized help thereby providing greater product reliability as well as reduced costs which benefit all of us.

Recent Innovations In Glass-to-Metal Sealing

J. E. Benbenek

R. C. A. Laboratories

Mr. Benbenek was born in New Jersey and attended Rider College and Rutgers University, in addition to armed forces schools. As an officer in the Army, he was a glassblower and consultant to the Technical Service Unit of the Corps of Engineers, War Department Technical Staff. He joined R. C. A. Laboratories Division in 1944 and became supervisor of the glassblowing section of the Materials and Devices Technology Department in 1957.

The art of sealing metals to glass is not new. Literature on this subject is full of success stories using certain sealing combinations. These stories are replete with descriptions of and the details of "how to do it". Recently, the increased demand for a wider variety of glass-to-metal seals has focused attention on the need for greater experimentation in metals, glasses and methods to fill these recent requirements.

The large scale fusion of glass to metal, such as is used in color television and power tubes, is a comparatively recent development. The primary purpose of this paper is to describe several methods which can be used to simplify the preparation of materials for use on recent versions of sealing combinations, as well as a completely new process of sealing non-magnetic stain-

less steel to a hard borosilicate glass. The processes described here are unrestricted by size or shape of metal and glass that can be used.

In sealing glass-to-metal for vacuum tight joints (seals that can be reheated), it is essential that the glass wet the metal and that the stresses which are set up in the glass or metal by thermal expansion or contraction during fabrication, or in subsequent use, must not exceed the tensile strength of the glass or metal. This paper deals with hard glass-to-metal combinations and is based on the above requirements.

TUNGSTEN

Composition of tungsten: Approximately 99.9 per cent pure tungsten, density 19.3, ground and polished. Coefficient of expansion 4.6×10^{-6} at 0-500°C.

Tungsten is substantially a pure, hard, brittle metal and one of the few capable of being sealed directly to several low expansion hard glasses. Extreme care must be taken to avoid splitting the material when cutting the short lengths usually required in seal making. Short lengths should be cut by grinding on a wet wheel rather than with mechanical cutters.

Tungsten rod can be purchased in various lengths, ground, polished, with special welded junctions to other metals such as nickel, nichrome, copper, etc., for attaching to other metal components.

Tungsten wire or rod should be cleaned before pre-oxidation by first degassing the material. This is done by heating to a bright red color in oxygen-gas flame and rubbing the seal area with a stick of potassium nitrite, KNO_2 , and then plunging into cold water. Then it should be rinsed and dried to remove the tungsten oxide.

The next step is to re-oxidize the tungsten prior to sealing to glass. This is important to effect a good seal. Re-oxidize by pass-

ing tungsten slowly through an oxygen-gas flame (bench burner) heating to a dull red. When cool, it should show a coating of oxide that is blue to blue-black in color.

The cleaned and oxidized tungsten is now ready for sealing to glass. The necessary length of hard glass sleeving is then slipped over the rod and progressively heated from one end of the sleeve to the other in an oxygen-gas flame. This serves to shrink the glass to the rod and drive out the air through the open end, preventing air occlusion and gas bubbles. The appearance of this seal should be light to dark brown in color. It may require one or two more progressive heatings to obtain the proper color providing the pre-oxidation was correct. With a little practice, one can soon determine the amount of oxidation that is necessary. When the proper color is obtained, the beaded tungsten wire or wires can be sealed into multiple button stems, presses and various other assemblies.

When a beaded tungsten rod is to be sealed as a support into a large envelope, a second sleeve of glass can be slipped over the beaded section for additional strength, and then a collar of glass can be sealed to the second sleeve. This simplifies the fusing of the beaded assembly to the envelope.

A good rule to follow in tungsten multiple seals, button stems and pinch presses, is to use Corning Glass No. 7720 Nonex and grade the flange area to match the glass envelope or vessel that the seal will be fused to.

CLEANING TUNGSTEN GLASSED ASSEMBLIES

Assemblies are cleaned electrolytically by immersion in a bath composed of sodium carbonate solution, saturated. Apply A.C. current through pulse type variac until clean.

RECOMMENDED GLASSES FOR TUNGSTEN

(1) For rod sizes up to .080 inch diameter. Corning Glass No. 7051. Coefficient of expansion 3.4×10^{-6} . This can be sealed to a

No. 7740 Pyrex envelope. Anneal 10 minutes at 570°C.

(2) For rod sizes up to .250 inch diameter. Bead with No. 7720 glass. Coefficient of expansion 36.0×10^{-7} . Seal to No. 7720 glass. Anneal at 550°C for 10 minutes.

(3) For rod sizes over .250 inch diameter. Bead with No. 7780 glass. Coefficient of expansion 37×10^{-7} . Seal to No. 7720 glass envelope. Anneal at 550°C for 10 minutes.

(4) For rod sizes to .120 inch diameter. A thin sleeve of No. 3320 glass followed by a second sleeve of No. 7740 glass, plus a collar of No. 7740 glass can be sealed to a No. 7740 glass envelope. Coefficient of expansion 32.5×10^{-7} .

MOLYBDENUM

Composition approximately 99.9 per cent pure molybdenum of density 10.2 ground and polished. Coefficient of expansion 5.5×10^{-6} at 0-500°C.

Molybdenum is similar to tungsten with two exceptions: (1) the thermal expansion is higher and as a rule requires different hard glasses than tungsten, (2) the main exception is because of its tendency to over oxidize and form a thick, smoky, non-adherent, semi-liquid oxide.

The molybdenum should be cleaned and degassed by furnace firing to 1065°C for 20 minutes in a line hydrogen atmosphere, and then cleaned with potassium nitrite as in tungsten cleaning.

The next step is to rapidly oxidize by passing through a burner flame above 600° C. After oxidizing, a close fitting glass sleeve of a longer than necessary length is sealed to the molybdenum rod as in tungsten sealing. The finished beaded assembly should be a chocolate brown to violet in color.

CLEANING MOLYBDENUM GLASSED ASSEMBLIES

Immerse in a solution of 90 per cent ammonium hydride and 10 per cent hydrogen peroxide until clean.

RECOMMENDED GLASSES FOR MOLYBDENUM

Corning Glass No. 7050—coefficient of expansion 4.6×10^{-6}

Corning Glass No. 7040—coefficient of expansion 4.8×10^{-6}

Corning Glass No. 7052—coefficient of expansion 4.6×10^{-6}

Corning Glass No. 7060—coefficient of expansion 4.8×10^{-6}

The same glass type is used for envelopes. Anneal at temperatures recommended for the above glasses.

CHROMIUM OXIDE METHOD OF SEALING GLASS TO MOLYBDENUM ROD AND TUBING

In the beading of large diameter rods, and in the making of tubular seals, the molybdenum is first degassed by furnace firing at 1065°C for 15 minutes in a line hydrogen atmosphere.

Plate the molybdenum with chromium approximately .0002 inch thick and oxidize the chromium plate by firing in line hydrogen at 1065°C for 15 minutes. The molybdenum is now ready for the glassing operation.

In the process of making a moly-to-glass tubular seal, it is advisable to by-pass-flush the internal area of the seal with nitrogen away from the glass area to the opposite end of the moly to prevent clouding the glass with hot moly oxides.

CLEANING CHROMIUM PLATED GLASSED ASSEMBLIES

Immerse in a solution of 90 per cent ammonium hydroxide and 10 per cent hydrogen peroxide until clean.

KOVAR

Composition of kovar: 29 per cent nickel, 17 per cent cobalt, 0.3 per cent manganese, 52 per cent iron. Coefficient of expansion 4.6×10^{-6} .

Kovar is a "wonder alloy" as far as the glassblower is concerned. It is available in almost any form, shape or size. It can be electroplated, tin dipped, soldered, welded, brazed, drawn and machined. Because its thermal expansion properties are similar to a wide range of standard glasses, seals can be made with relative ease by following a few simple rules.

Prior to glass sealing, the seal area of the kovar part must be highly polished or sand blasted so that the surfaces are free of sharp corners or edges, tool imperfections and scratches. Material is then degreased and furnace fired to 1050°C for 20 minutes in a line or dry hydrogen atmosphere.

The kovar is then re-oxidized by flame heating to approximately 700°C in the oxidizing portion of the flame. The kovar is now ready for the glassing operation.

CLEANING KOVAR GLASSED ASSEMBLIES

Immerse in a bath of hot concentrated hydrochloride acid until clean.

Bright dip solution: 400 ml of acetic acid
200 ml of nitric acid
75 ml of distilled water
9 ml of hydrochloric acid

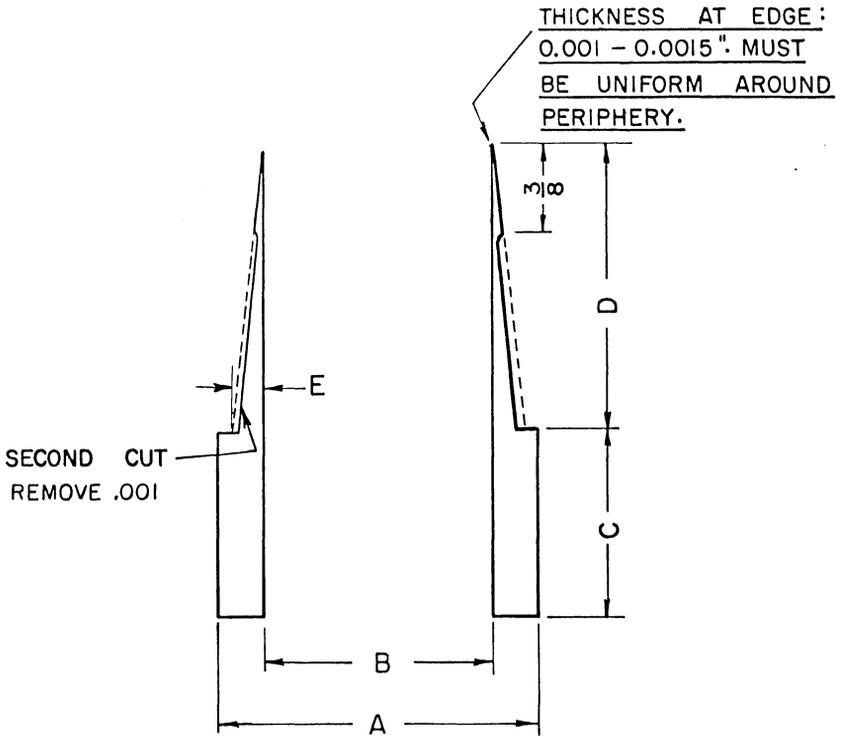
Immerse until brown coating forms and rinse in water immediately until bright.

RECOMMENDED GLASSES FOR KOVAR

Corning Glass No. 7040—coefficient of expansion 4.8×10^{-6}

Corning Glass No. 7052—coefficient of expansion 4.6×10^{-6}

Corning Glass No. 7050—coefficient of expansion 4.6×10^{-6}



DIM.	I	II
A	1.250	1.750
B	1.170	1.625
C	0.4	3.
D	0.75	1.
E	0.012	0.012

NOT DRAWN TO SCALE. ALL DIMENSIONS ARE IN INCHES.

Figure 1

Dimensions of Stainless Steel Part For Housekeeper Seal

A Method For Sealing Stainless Steel To Glass

INTRODUCTION

For some time there has been a great need for sealing nonmagnetic metals to hard glass. While copper-to-glass seals of the "Housekeeper" type have been known⁽¹⁾ and employed for many years, they have two major disadvantages:

- (a) the Cu feather edge is heavily oxidized during the sealing operation, may eventually become porous and even collapse under external pressure;
- (b) there is no convenient way of joining Cu to stainless steel, the nonmagnetic material used extensively in metal vacuum systems.

To avoid these difficulties, it would be desirable to join stainless steel (SS) directly to glass. While the expansion coefficients of these materials differ considerably (SS: $10 - 12 \times 10^{-6}$; hard glass: ca. $4 \times 10^{-6}/^{\circ}\text{C}$), this difference is actually less than for Cu (17×10^{-6}) and glass. Thus it should be possible to make SS-to-glass "Housekeeper" seals, provided the greater stiffness of SS is taken into account. This report describes in some detail how to produce successful seals of this type.

PREPARATION OF METAL PARTS

For these seals, nonmagnetic stainless steel tubing No. 304 (also known as "18-8") is used. To eliminate strains resulting from machining, the material is first fired in DRY H_2 at 1065°C for 15 minutes. The strain-free tubing is then machined, as shown in Figure 1, to the required dimensions. The most important feature is the second cut (shown as a dotted line) taken at the tapered end. This yields a smooth and, at the same time, sufficiently flexible feather edge. To obtain an edge that is uniform in thickness around the periphery, a cut should first be taken from

the inside. The tubing is then put on a mandrel for machining the outside. If the edge is less than 0.001 inch in thickness, it is likely to be rough. If it is thicker than 0.0015 inch, it will not have sufficient flexibility. After machining, the stainless steel part is degreased and then oxidized by firing in line H₂ at 1065°C for 20 minutes. If it is to be welded within 2 inches of the glass seal into another metal part, a snug fitting copper plug is inserted and the Heli-Arc welding process done before glassing. After welding, the piece may have to be remachined on the end opposite to the feather edge to true it up. Then the assembly should be degreased and finally vacuum fired with the copper plug at 1000°C for 15 minutes. The stainless steel part is now ready for glassing.

THE SEALING OPERATION

For sealing to stainless steel, borosilicate glass No. 7052 (FN) was chosen for the following reasons:

- (1) Its coefficient of expansion ($4.6 \times 10^{-6}/^{\circ}\text{C}$) is larger than that of other hard glasses, such as Pyrex, therefore matches the metal somewhat better.
- (2) It readily wets the oxidized stainless steel.
- (3) It seals well to other hard glasses, including Pyrex.
- (4) Its optical quality is high.

For the sealing operation, the stainless steel tube is chucked in a lathe and wrapped within 1/2 inch of the end to be glassed with wet asbestos paper tape to keep it cool. A piece of FN glass is shaped to fit the exterior of the feather edge to a depth of about 0.050 inch and fused to the outside of the metal. Care is taken to point the flame on the glass rather than on the metal to prevent overheating the thin metal edge. The glass is flame cut at a distance of 1/8 inch from the feather edge and then carefully rolled inside to complete the internal beading of the stainless steel tube. Finally, an FN glass tube is sealed to the beaded edge and the assembly flame-annealed.

A number of stainless steel-to-glass seals have been successfully made by this technique and have been used where nonmagnetic assemblies of this type are required.

- 1) **John Strong, "Procedures in Experimental Physics," p. 25, Prentice-Hall, N. Y. (1945)**

Technique Of Sealing Glass-to-Sapphire

G. P. Spindler

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Mr. Spindler started his career in glassblowing at the age of 16 and received his early training with Eimer & Amend Company in New York City, where he stayed for six years. Before taking his present position with Bell Telephone Laboratories in 1926, he spent four years with various chemical glass apparatus companies where he designed, as well as fabricated, apparatus for the scientific field.

INTRODUCTION

In this paper, we will describe the technique of sealing glass to sapphire and the various types of fabrication methods involved. We have sealed two types of sapphire parts and they will be discussed independently. First, we will explain the glass-to-sapphire tube sealing and, secondly, we will describe sealing glass to sapphire windows. The techniques are somewhat similar, but it is felt that we should spend a little time on each separately.

The reason for starting this seal was due to an interest of L. S. Nelson with discharge lamps which have a body of transparent sapphire tubing.¹ The initial lamp design used a glass-metal-sapphire seal whereby the sapphire parts were first sealed with hard solder to a Kovar sleeve. The Kovar could then be attached by conventional glassblowing techniques to 60 mil tungsten rod for insertion of electrodes in the lamp body. The glass seals were used so that the lamps could be opened easily for cleaning and so that the sealing off would be quite simple on glass vacuum stations. The construction of this lamp is shown in Figure 1.

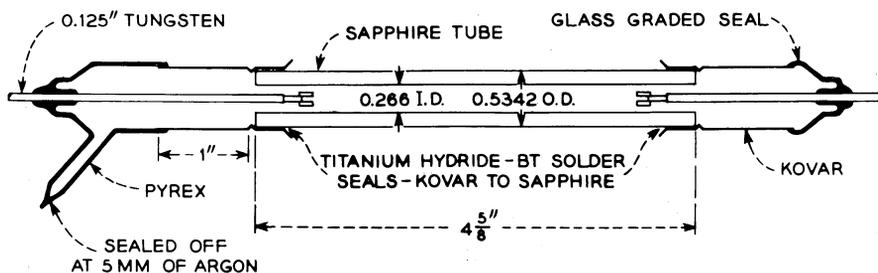


Figure 1

Sapphire discharge lamp with metal seals.

This type of lamp was quite sturdy and satisfactory in design. One drawback was that the metal sleeves could not be enclosed in an evacuated (10^{-6} mm Hg) apparatus because severe arcing would occur between the metal sleeves when the pressure outside the lamp fell below that inside the lamp.

In order to eliminate the Kovar sleeves from this type of lamp, we first tried the glass-to-sapphire seals which had been previously described,^{2,3} where relatively low melting glasses were used to seal windows onto Pyrex. With our considerably heavier sapphire parts, bad sagging occurred during the sealing process due to the low softening temperatures of the glasses used. To eliminate this sagging during the sealing, it was immediately evident that higher melting glasses would have to be used. We then decided to try a number of possible seals in the attempt to find the highest melting combination possible. With the cooperation of L. S. Nelson, we then worked out this technique of sealing to obtain the sapphire discharge lamp which was desired.

Characteristics of Sapphire

In Table 1, we have listed the prominent characteristics of sapphire which make it of interest to the scientist today.

As far as glassblowers are concerned, the most important properties are No. 1 and No. 2. That is, the material is definitely

TABLE I
Characteristics of Sapphire

- 1. Crystalline—NOT A GLASS!**
- 2. Melts sharply at 2000°C.**
- 3. Poor thermal shock resistance.**
- 4. Hard and strong.**
- 5. Transmits ultraviolet, infrared, microwaves.**
- 6. Conducts heat.**
- 7. Electrical insulator.**
- 8. It is inert to hydrofluoric acid.**

not a glass, even though it is transparent and looks somewhat glassy. Also, it melts sharply at 2000°C, without first softening. In other words, even if you could apply direct heat to the sapphire, it would melt and drip like wax rather than gradually softening as glass usually does. Therefore, the glass seal must be fastened onto the preformed sapphire part by adhesion of the glass without attempting to soften the sapphire.

Experimental

Tube Seals

The details of making the seal to sapphire tubes have been described earlier.⁴ Since that publication, we have made certain valuable changes which have simplified and also extended the process to window seals. The technique involves principally a conventional graded seal attached to the sapphire part with Corning Code No. 1826 glass in a laboratory-type resistance furnace. Figure 2 shows our furnace setup indicating the method of clamping both the graded seal above the furnace and the fused silica holder for the sapphire part below the furnace. We have found the Fisher buret clamp to be very valuable in maintaining rigid support and proper alignment.



Figure 2

Double-wound furnace for sapphire tube seals. Seal has been made and quartz wool insulation applied top and bottom.

Figure 3 is a cutaway drawing of the internal portions of the furnace. For the tube sealing, we use a double-wound furnace with the top quarter of the furnace wound for 900°C and the remainder of the furnace wound for 700°C. Two thermocouples, good to 1000°C, are placed in the furnace, one in the upper winding area and one in the lower winding area. We have found the meter-type thermocouple to be most convenient for this work. The sapphire tube is wedged tightly in a fused silica holder by a snug wrapping of asbestos paper. The silica holder should not be allowed to touch the sapphire because a chemical reaction will occur between the two at the high temperatures of the fusing operation.

Table II shows the physical properties of the glass-sapphire sealing materials used. It should be noted that the differences in thermal expansion coefficients between the glasses in the graded

TABLE II

Physical Properties of Glass-Sapphire Sealing Materials

Material	Coefficient of thermal expansion (deg C)-1×10^7	Softening point (deg C)
Sapphire	Along A axis 67 ^a	
	Along C axis 50 ^a	
No. 1826 glass powder	49 ^b	585 ^b
No. 7280 glass	64 ^b	870 ^b
No. 3320 glass	40 ^b	780 ^b
No. 7740 glass	32.5 ^b	820 ^b
Tungsten	43	

^a Supplied by Linde Air Products Company

^b Supplied by Corning Glass Works.

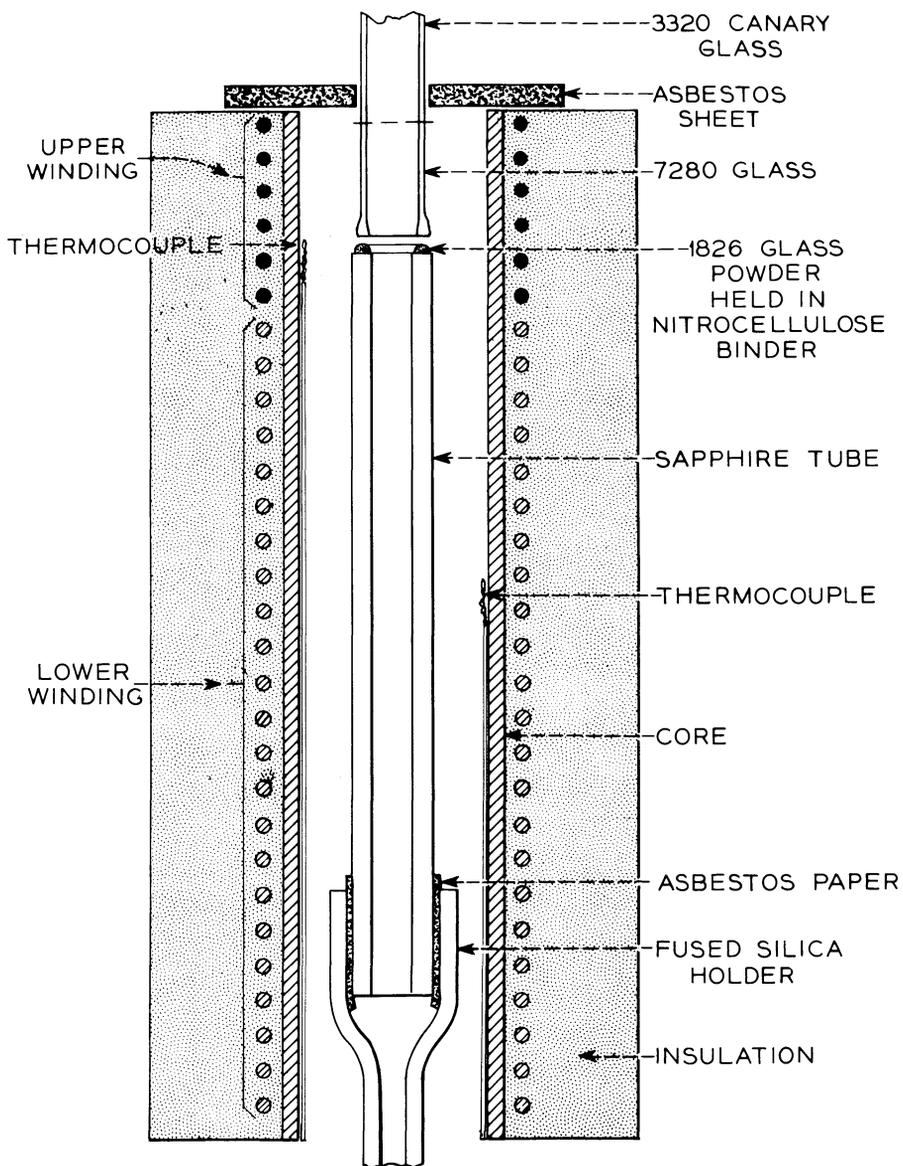


Figure 3

Diagram of furnace shown in Figure 2.

seal, namely the 7280, 3320 and 7740 glasses, are quite great. Also notice that all of the glasses except the glass powder soften above 780°C. In this way, the seal can be made with much less distortion at the sealing temperatures of 800-900°C than in the case of the lower melting sealing glasses.^{2,3}

Care must be taken when making the graded step-seal which is applied to the powdered glass seal. Figure 4 shows the important stages in making this type of graded seal. One starts with a tube of 7280 glass, rounded off with a test tube end as in (a). A 3-mm ball of 3320 glass is then put onto the test tube end using the hydrogen flame under oxidizing conditions as in (b). Then care-

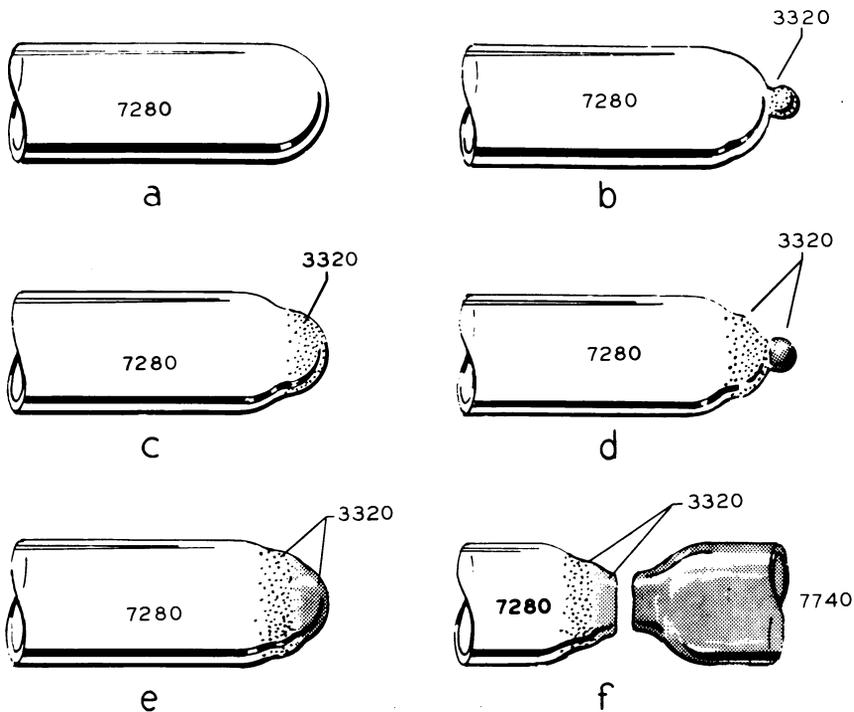


Figure 4
Stages in making 7280-3320-7740 graded glass seal.

fully, this ball of glass is melted strongly by applying the hydrogen flame only to the ball of glass so that the 3320 glass diffuses into the 7280 glass. This diffusion must be observed closely. Under this intense heating, a small ring of light-colored green will appear between the 7280 and 3320 glass. This ring must be seen to be assured that a good diffusion has taken place. This tube is then blown out slightly as is shown in (c). For strength, another 3-mm ball of 3320 glass is added to the area previously blown out, as in (d). This ball, in turn, is strongly heated with the hydrogen flame under oxidizing conditions and the tube blown out a second time to give a configuration shown in (e). Then with a sharp hydrogen flame, the tube is opened as in (f) so that it is ready to be sealed onto the 7740 chemical Pyrex glass. This seal is then made in the hydrogen flame and the tubing, plus the graded seal, are blown out to the original diameter of the two tubes. Then the 7280 portion of the glass is cut off about 3/4 inch from the graded seal, flared out slightly, and is ready for the seal after slight roughening on the carborundum wheel.

The powdered glass is painted in place as a mixture of about 300 mesh powder suspended in a 2-1/2 per cent solution of nitrocellulose in amyl acetate. The amyl acetate solution is added dropwise with stirring to the powdered glass material until a thick, creamy consistency is reached. With an artist's brush, a small portion of the powdered glass mixture is painted on the end of the sapphire tube. The solvent is allowed to evaporate, and then another coat is placed on top of the first coat. By repeating this successively, a layer of 0.5 to 1.0 mm in thickness is built up. When it is dry, the sapphire tube is clamped in the furnace, as shown in Figure 3, and heating is started.

The lower winding is brought to about 700°C over a period of two hours. At the end of this time, the upper winding is brought to a temperature of about 900°C and held there for approximately 1/2 hour until the 1826 glass has fused into a transparent mass. The graded seal made earlier is now suspended above the furnace so that it can preheat from the heat radiated upward from the furnace. When the 1826 glass is fused properly (it must not have a milky appearance), the covering at the top of

the furnace is removed and the graded seal lowered into the upper winding area and allowed to preheat still further. This is quite important since attaching a cold piece of glass to the sapphire tube will cause it to shatter from the thermal shock. The graded seal is then lowered onto the powdered glass by loosening the buret clamps slightly and sliding the graded seal downward until it touches and is wetted thoroughly by the molten 1826 glass. The alignment is checked and the upper and lower parts of the furnace are lagged with wads of quartz wool, as shown in Figure 2, to minimize loss of heat. The current on both windings is reduced to a low value and the furnace allowed to cool to room temperature overnight. The next morning, the tube can be removed easily from the fused silica holder and the seal leak tested with a Tesla coil and a simple vacuum pump.

Window Seals

We have varied this technique somewhat to seal glass tubes to sapphire windows. A photograph of the furnace used is shown in Figure 5. The window seal is made possible by heating the window on a disc of Al₂O₃ high-alumina ceramic supported in the center of the furnace as shown in Figure 6. The disc is about 1/2 inch thick with a diameter a few millimeters less than the inside of the furnace. This ceramic disc is supported on top of a fused silica tube which is again held in a Fisher buret clamp beneath the furnace. The furnace has a single winding, is about 4 inches high and has an inside diameter of approximately 1-1/2 inches. This was found ideal for sealing windows up to 1 inch in diameter.

In this case a 1 to 2 mm ring of the powdered glass is applied to the outside edge of the sapphire window. A disc of masking tape, cut with a cork borer, was found to be an excellent mask when applying the mixture of 1826 glass in the nitrocellulose solution. Again the mixture is applied with an artist's brush. A ring of about 1/2 mm thick is applied to the window and, just before completely dry, the masking tape is removed with fine tweezers. The window is laid on the alumina block and, by lowering the fused silica tube, the block and window are placed in

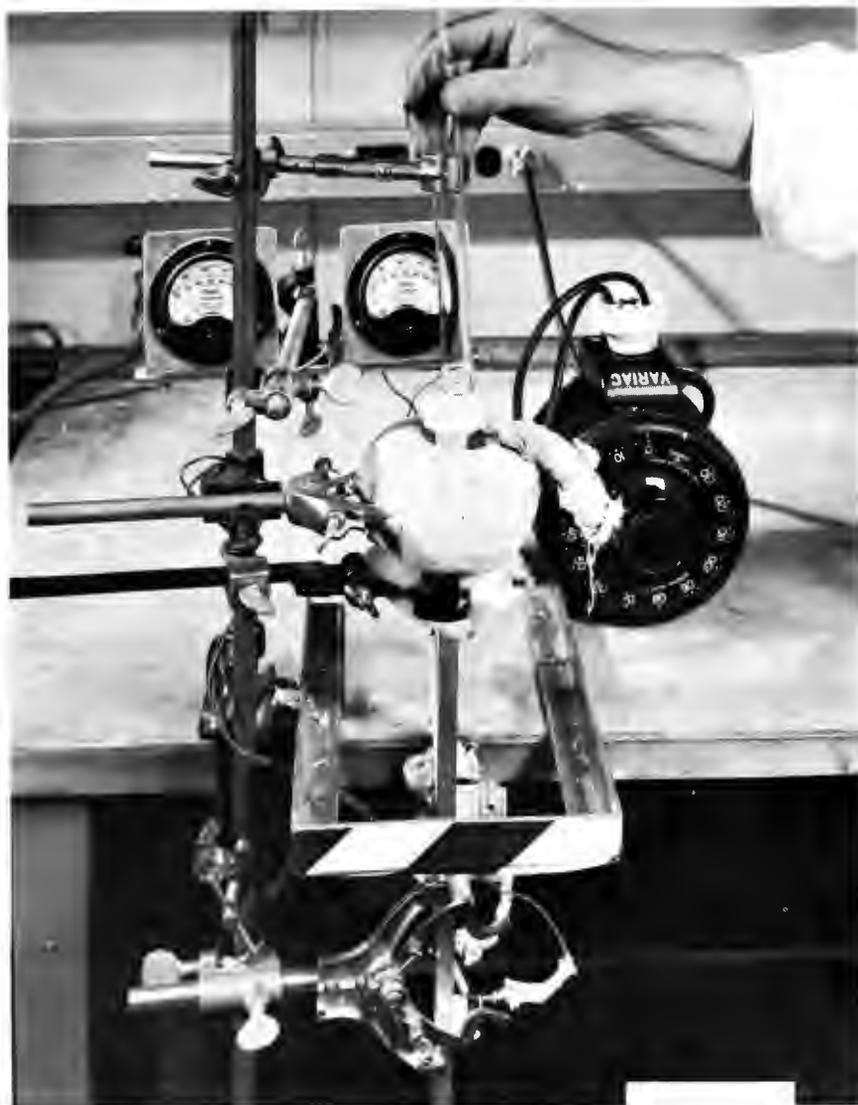


Figure 5

Furnace for making sapphire window seals. Graded seal is being applied by hand.

the center of the furnace. The top of the furnace is then covered with asbestos and the temperature of the furnace brought to 900° over a period of about three hours. Again the 1826 powdered glass must be fused until completely transparent.

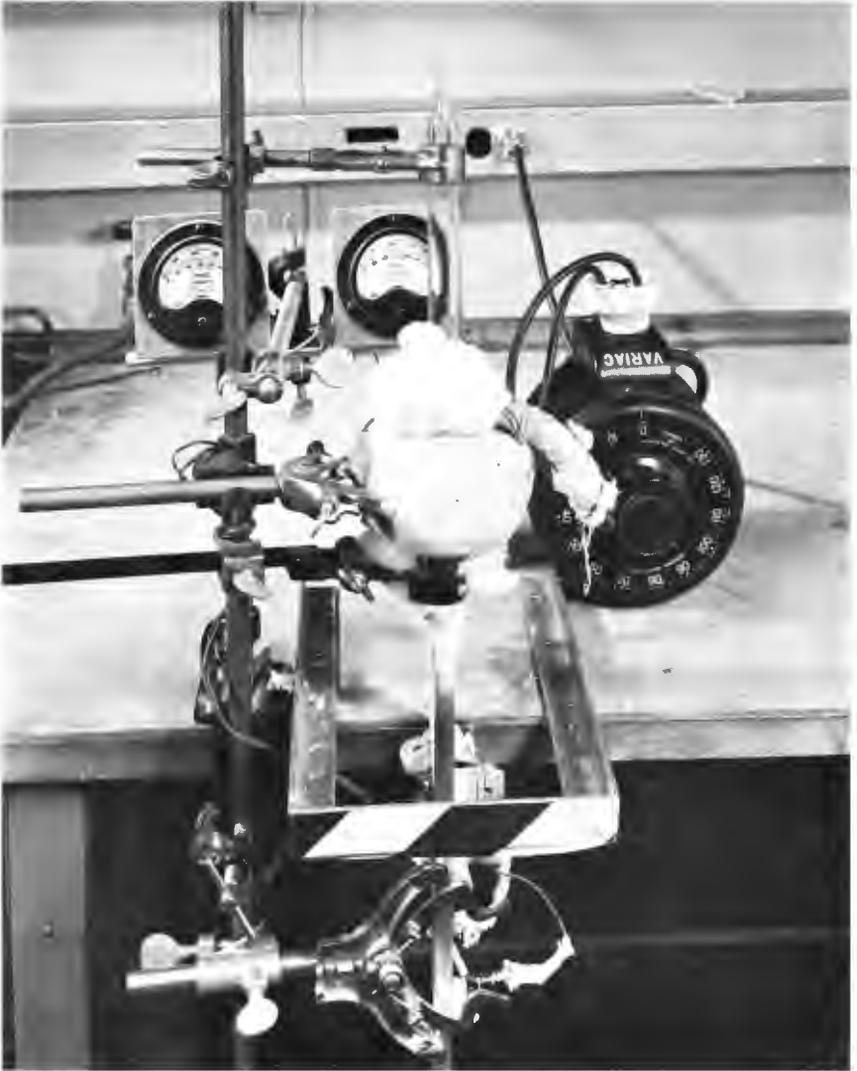


Figure 6

Furnace for making sapphire window seals. Seal has been made and quartz wool insulation has been applied top and bottom.

When the 1826 glass powder is ready for sealing, the graded seal is lowered down into the furnace area for preheating and supported with an ordinary clamp. Just before the seal is made, the alumina block is raised just above the top of the furnace as

shown in Figure 5. The graded seal is applied by hand, first to one edge of the window and then with a flip-over motion raised to a vertical position so that the tube will cover the window. We found that this permits much more accurate centering than if the graded seal were lowered in clamps as was done with the tube seal. Finally, the fused silica holder, ceramic block and the completed seal are now lowered into the center of the furnace again and the top and bottom of the furnace are lagged with quartz

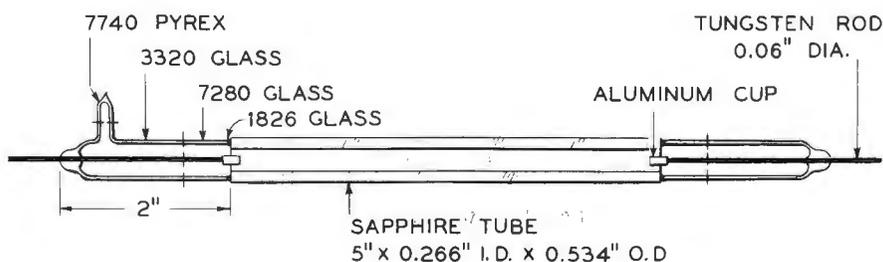


Figure 7

Schematic drawing of glass-sealed sapphire discharge lamp.

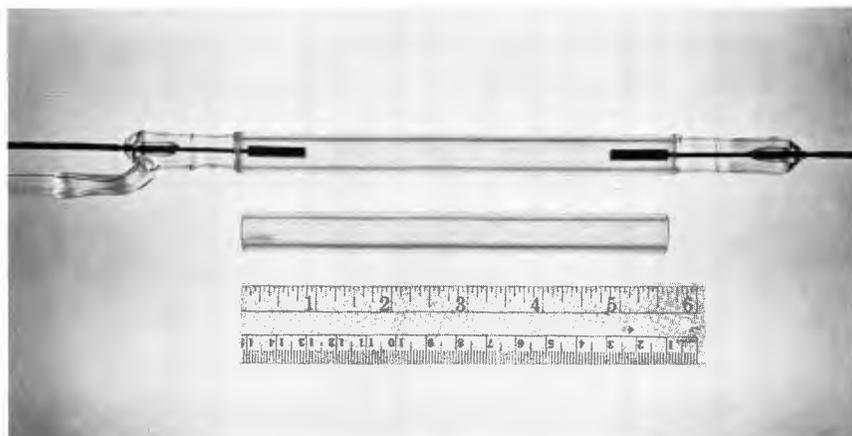


Figure 8

Sapphire tube and glass-sealed sapphire discharge lamp.

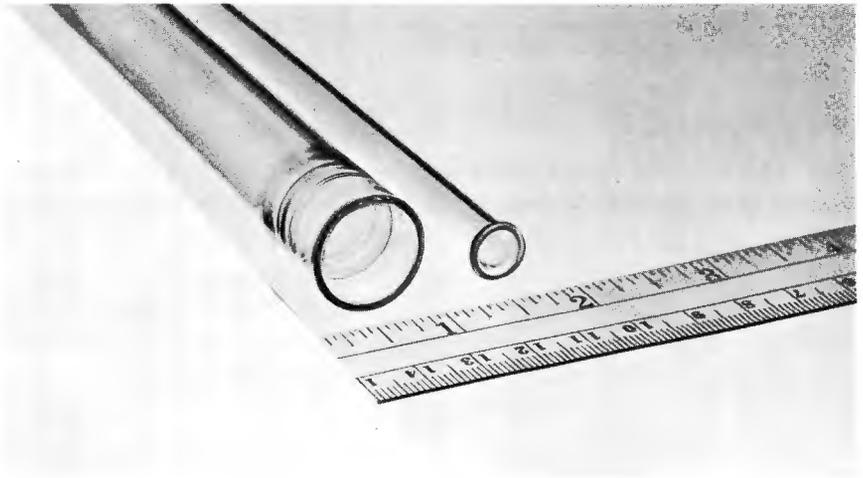


Figure 9
Saphire window seals.

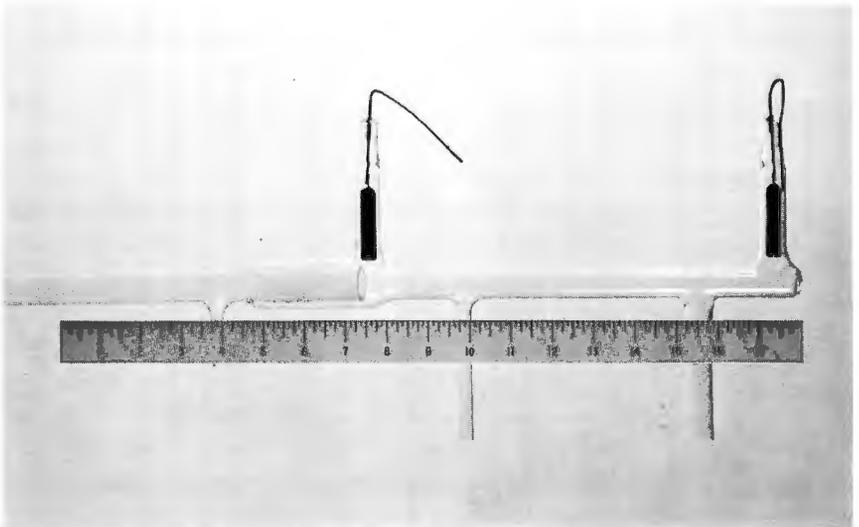


Figure 10
Discharge lamp with internal saphire window.

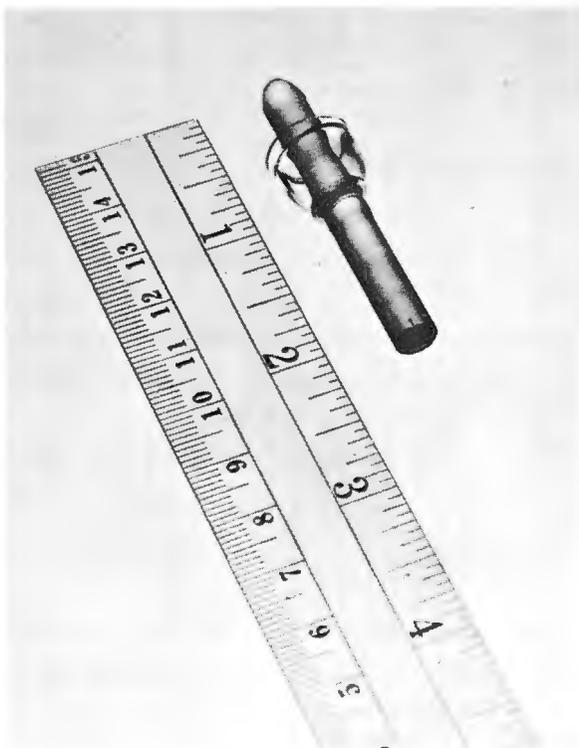


Figure 11

Experimental ring seal to sapphire rod.



Figure 12

No. 7280 glass sealed to high alumina ceramic tube with No. 1826 glass powder.

wool as shown in Figure 6. The current is turned down and the furnace is allowed to cool to room temperature overnight.

Leak testing is done as described above.

Applications

In this section, we will describe some completed devices using this type of sapphire seal. In Figure 7 there is a drawing of the completed sapphire discharge lamp which is shown in Figure 8 as a photograph. Notice that in these cases sapphire tubes approximately 5 to 6 inches long and 1/2 inch outside diameter are fabricated into discharge lamps. Figure 9 shows some successful window seals that were made. The large window is 3/4 inch outside diameter and 0.4 mm thick. The small window is 1/2 inch in diameter, approximately.

In Figure 10, we have sealed the large window shown in Figure 9 into a Pyrex discharge lamp with a Dewar seal. Notice that the graded seal is completely enclosed in Pyrex for mechanical strength. Figure 11 shows a ring seal onto a sapphire rod which is currently being investigated in our laboratories. This is still very much in the experimental stage and we have not had as great success with this as we might have wished.

Figure 12 illustrates a possible use of this technique for sealing glass to high alumina ceramic. This figure shows a piece of 1/2 inch, 99 per cent alumina ceramic tube sealed to a ring of 7280 glass by the 1826 powder technique described here. This seal is a very sturdy one and can be used for high vacuum applications of the high alumina ceramic.

Acknowledgments

The author wishes to express his thanks to Drs. R. H. Dalton and F. W. Martin of the Corning Glass Works for continuing advice which made this seal possible.

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Designing Glass Laboratory Fractionating Columns

E. L. Wheeler

University of California

Mr. Wheeler was a musician until 1932 when he joined Shell Development Research Laboratories, where he learned the art of glassblowing. In 1941, he went with Johns Hopkins University and three years later joined the Percleve Corporation in Oak Ridge, Tenn., to set up a glassblowing shop. He returned to California in 1945, to take charge of the glassblowing department at the University of California at Los Angeles. He is the founder and head of the Precision Apparatus Company of Santa Monica, Calif., which specializes in laboratory fractionating equipment. He is the author of two books.

Fractional distillation is one of the more important and widely used techniques in chemical research, resulting in many glassblowers having more requests to build distillation equipment than requests for equipment of any other process.

There are two methods of distillation to be considered:

Method 1—Continuous Distillation in which a continuous feed of pre-heated material of a constant composition is admitted into the column at a point near the middle, while at the same time the simultaneous removal of several fractions from different sections of the column is permitted. Each fraction, although different from the others, is of fairly constant composition. The lower

boiling fractions are removed at the head and the higher boiling fractions are removed from the bottom of the column.

Method 2—Batch Distillation in which a charge is placed in the still pot and product is removed only at the head. This method produces a progressive series of fractions, each having a different composition. Batch distillation, therefore, is more satisfactory for analytical distillations and for purifying material preparatory to its use.

In fractional distillation, there are many standard terms used in describing distillation equipment and in discussing the process itself. For the sake of clarity, I will briefly review a few of these terms.

THE COLUMN: The column is the portion of the fractionating assembly in which rectification takes place (Figure 1).

RECTIFYING SECTION: The section of the column occupied by the packing or rectifying devices. In Figure 1, this is the section extending from just above the indented packing supports to just below the mercury seal joint.

STILL HEAD: The portion of the fractionating assembly above the rectifying section, usually consisting of one or more condensers and a reflux-dividing device.

PACKING: The packing is composed of particles of material placed in the rectifying section to increase the opportunity of liquid vapor contact.

THEORETICAL PLATE: A mathematical unit of efficiency in the operation of a fractionating column.

H.E.T.P.: This is the designation for "height equivalent to a theoretical plate". That is the length of a column necessary to make the same separation as is made by one perfect plate.

PERFECT PLATE: A section of column which effects com-

plete equilibrium between the vapor and the liquid.

STILL POT: The flask or container in which the material to be distilled is heated (Figures 2, 3 and 4).

In designing any fractional distillation equipment, there are several factors which must be considered, or facts which must be known.

1—EFFICIENCY OF SEPARATION DESIRED

In choosing a column to perform a given separation, it would be advantageous to know the approximate number of plates required to produce the desired separation. Carney¹ gives the for-

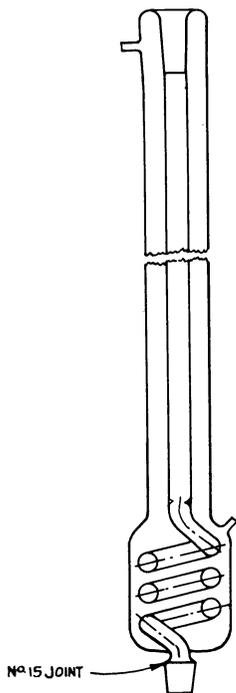


Figure 1

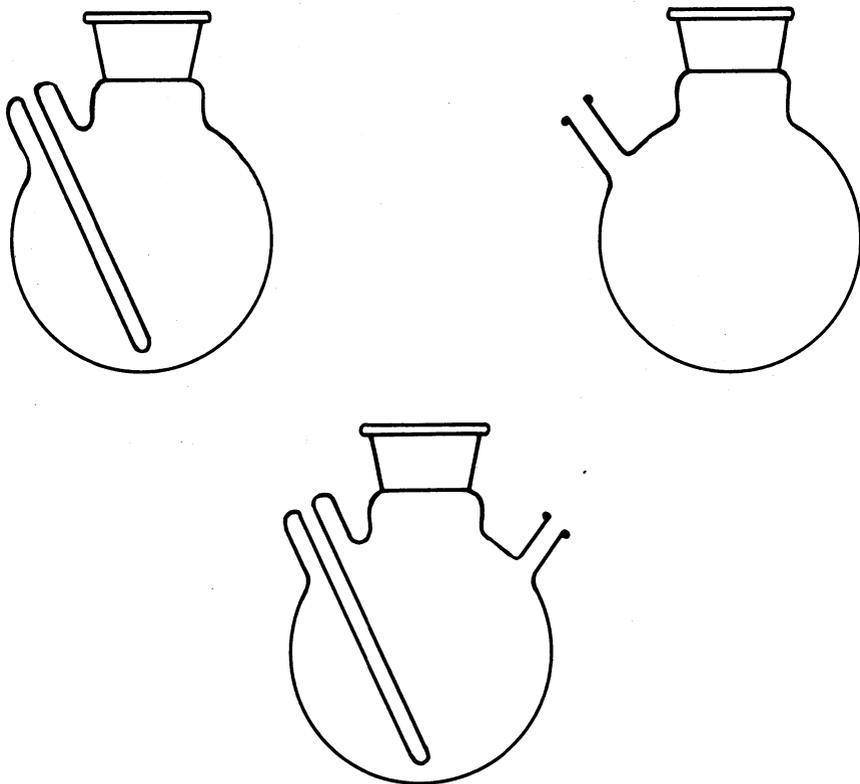


Figure 2

mula for calculating this in his book on fractional distillation.

The efficiency of separation is determined by the number of plates a column passes at an infinite reflux ratio. This is merely a comparison between stills and does not necessarily represent the column efficiency at actual operating conditions. It is safe to assume, however, that with two columns, the one showing the highest theoretical plate value at total reflux will probably be the most efficient under operating conditions. Other conditions being equal, the column showing the greatest efficiency per unit length is the most desirable.

2—RATE OF DISTILLATION DESIRED

With any column, there is an optimum rate above which there

will be poor separation and below which the increased efficiency will not warrant the increased time required. The ideal column would be one with a low operating holdup, high through-put and a high theoretical plate efficiency. Present laboratory fractional distillation equipment, as designed, is a compromise between these ideal conditions. The distillation rate is a function of the vapor velocity and the reflux rate. With a high vapor velocity, the possible distillation rate increases, but at the same time the time of contact between the liquid and the vapor is decreased. Therefore, a high reflux ratio increases the length of time necessary for a given distillation. However, it also increases the degree of fractionation which may be obtained from some columns.

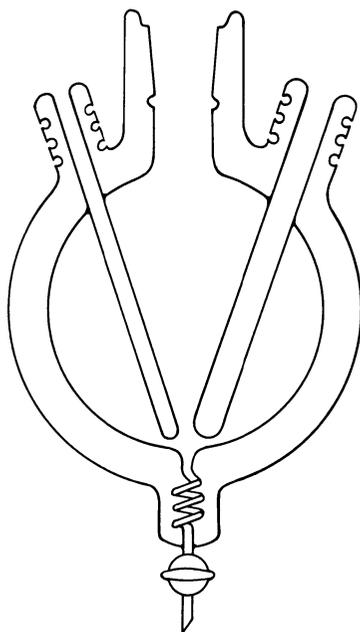


Figure 3

3—TEMPERATURE RANGE TO BE COVERED

The temperature range over which the still is to be operated strongly influences the design of the still. With stills which are

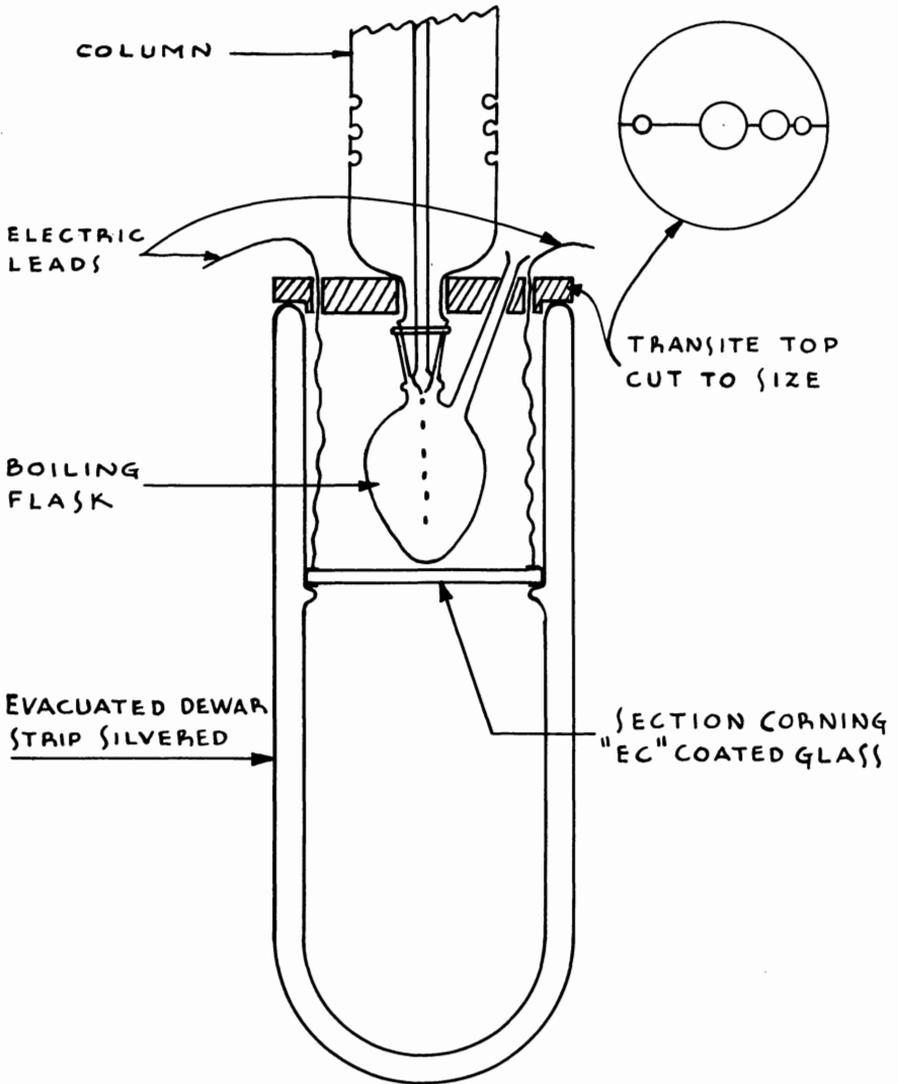


Figure 4

to be operated at high temperatures, care must be taken to prevent or compensate for excessive heat loss. If such care is not taken, the column will probably not operate smoothly. Reflux in the column may fluctuate, showing up as a flooding condition in the column, or possibly as a lack of reflux at the still head. It has been the author's experience that a silvered, vacuum jacketed column up to the reflux condenser is the best possible arrangement for controlling the problem of heat loss.

4—MATERIAL TO BE FRACTIONATED

Knowledge of the material to be fractionated in a column is important because of such physical and chemical properties as viscosity, surface tension, latent heat, tendency to foam and corrosiveness. These same factors determine, in part, the type of packing to be used in the finished column.

In addition to these four factors influencing the design of fractional columns, there are several variable conditions which also effect the operating characteristics of a column.

VARIABLE 1: THE DIAMETER OF THE COLUMN.

Increasing the diameter of the column increases the throughput. Ward² states, "The capacity of a column is proportional to the square of the diameter and the holdup increases in somewhat the same ratio resulting in a loss in fractionating efficiency." An increase in the diameter of a column decreases the vapor velocity for the same through-put.

VARIABLE 2: COLUMN HEIGHT

An increase in the height of a column increases the number of theoretical plates separation which may be obtained from the column. However, this increase follows the law of diminishing returns, i.e. each additional section of length is less efficient than the preceding length. This increase in H.E.T.P. is smaller than the gain in efficiency due to the increased length. Therefore, the

long column with increased holdup is still preferable to a short column, since at total reflux the degree of efficiency is increased logarithmically, not linearly, with the number of theoretical perfect plates. Since the number of perfect plates is a function of its length, the best results will be obtained with as long a column as possible, taking into account the amount of material to be distilled.

VARIABLE 3: OPERATING HOLDUP

The amount of operating holdup in any column is important since this effects the sharpness of separation. Carney³ says, "The maximum allowable holdup should not exceed 10 per cent of either component being separated, since the column of the intermediate cut between two pure components will be equal to the volume of the operating holdup."

VARIABLE 4: PRESSURE DROP

At atmospheric pressure, a slight difference in pressure between the still head and the boiling kettle is of small concern. An increase in the pressure difference will increase the pressure in the boiling kettle, which will, in turn, increase the temperature necessary to make the material boil. The pressure difference between the head and the still pot may be put to use as a means of regulating or controlling the heat input to the kettle. Glasgow and Schecktzanz⁴ describe such a procedure.

VARIABLE 5: VAPOR VELOCITY

An increase in the vapor velocity or through-put in the column results in a less efficient separation and higher H.E.T.P. The loss of efficiency may be attributed to the shorter and, therefore, less efficient contact between the liquid and vapor. This may be due either to channeling of the reflux in the packing or to the fact that the velocity is so great that the time of contact between the liquid and vapor is too short for efficient heat transfer.

This loss of efficiency of separation, with the increased vapor velocity, shows the need for accurate control of heat input into

the boiling flask. This is especially true of the high efficiency, low holdup columns being operated for maximum efficiency, and is also true with columns being operated under reduced pressures.

VARIABLE 6: WATER IN THE MATERIAL BEING DISTILLED

Small amounts of water in organic materials should be thoroughly removed before any precise fractionating is attempted. Water not removed will steam distill during the first part of the distillation and give erroneous still head temperature readings. In many cases, the water will separate from organic distillates and cling to the walls of the still head. Frequently, the water will redistill over and over again in the still head before passing into the takeoff line or condenser where it will again cling to the side walls.

VARIABLE 7: PRE-FLOODING THE COLUMN AT THE START OF THE DISTILLATION

For random packed columns, many micro columns, and for all concentric tube columns, pre-flooding at the beginning of each distillation is necessary to obtain the maximum efficiency from the column. Pre-flooding consists of super-heating the still pot to the extent that the ascending vapor prevents the reflux from returning to the still pot. This results in a column of liquid completely filling the column and thoroughly wetting the packing.

VARIABLE 8: PACKING

The last, but certainly not the least important, is the packing itself.

The choice of packing depends greatly on the intended use of the column. The ideal column would be one which has a large surface for contact between the liquid and the vapor. It should be corrosive resistant and pack evenly into the column, thus assuring

uniform distribution of the free spaces throughout the packing. Fenske, Quiggle and Tongsburg⁵ noted that the surface area per cubic inch of packing, multiplied by the percentage of free space, gave numerical ratings which parallel usefulness of various packings.

The following is a brief description of a few commonly used types of packings.

GLASS CYLINDERS: For viscous materials, those which entrain badly or for those which foam, glass cylinders perform well. Glass cylinders may be made from 5 mm O.D. tubing cut in 3 mm lengths. Tests with a mixture of carbon tetrachloride and benzene show a one-foot long column, 22 mm O.D., packed with 5 mm x 3 mm cylinders, to have a H.E.T.P. of about 3½ inches

Glass cylinders have a good through-put, since in a column distilling liquids boiling at 200° C. while maintaining a 10-1 reflux rate, the maximum takeoff is about 3 ml per minute. When greater through-put is desired, a one-inch column packed with cylinders 10 mm O.D. x 4 mm long may be used.

RASCHIG RINGS: Raschig rings are small, thin-walled cylinders, usually of porcelain, made with equal length and diameter. Because of their uniform shape, Raschig rings pack evenly into a column and present a reasonably large surface area and free space.

SPIRAL PACKING: A spiral coil of wire, which fits closely to the inner walls of the rectifying section, makes an efficient type of packing. Glass spirals may be substituted for the metal with the same efficiency. However, the fragility of the glass coil and the difficulty of making the glass fit closely to the walls of the tube make the glass spirals much less widely used than the metal spirals.

GROUND GLASS FUSED TO THE RECTIFYING SECTION: A ground glass surface produces a column with about the same

separating efficiency as the glass spiral without the danger of frequent breakage as is experienced with the glass coil (Figure 5).

However, in the case of the ground glass surface, the operating holdup is greater. The coated glass section may be made free hand by half filling a Pyrex tube of the desired size (8-9 mm) with ground glass (50 to 60 mesh). The tube is then rotated a

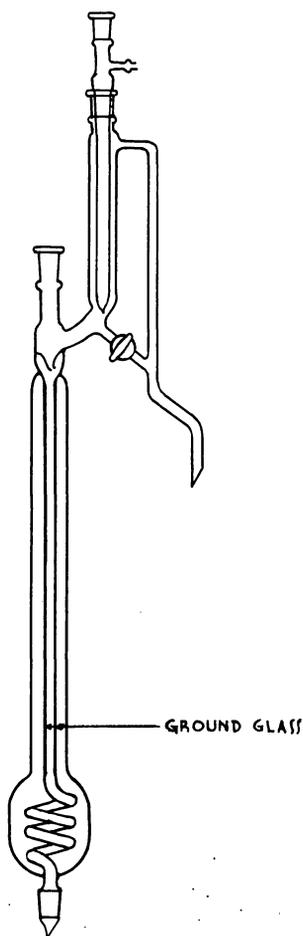


Figure 5

section at a time in a gas-oxygen flame until a layer of ground glass is fused to the side wall. When the proper length has been prepared, the tube is lifted vertically and the excess glass is removed by tapping on the side of the tube.

A more precise method of making these ground glass coated rectifying sections is to support a metal rod in the center of the tube, pack ground glass around the metal rod and bake the entire assembly in an oven for about five minutes at 900-1000° C. When the tube is cool, the metal core may be removed by soaking the assembly in acid until the metal becomes loose.

OTHER PACKING SINGLE TURN HELICES (METAL):

Metal helices give good results for atmospheric distillations and some reduced pressure distillations with many materials. Metal helices were developed by Fenske, Tongburg and Lawraski⁶. These are available commercially in many sizes, allowing both large and small columns to be packed with the proper size for the ratio between the column and the packing.

GLASS HELICES: Glass helices were developed as a substitute for metal helices when corrosive mixtures were to be distilled. Glass helices may also be obtained commercially, but not in as many sizes as the metal helices. Considerable care must be exercised in packing a column with glass helices so as not to break them.

Figure 6 shows an apparatus for packing glass helices in a column. This was developed by Allenby and L'Heureux⁷. With this apparatus, it is possible to pack a large column quickly and evenly.

COMMERCIAL METAL PACKINGS: While not commonly used now, *metal chains* have had great popularity for column packings, especially for micro columns where gold and platinum jewelers' chains were used to obtain a packing of inert material. The popularity of chain packing was no doubt due in part to their availability and to the ease with which a column could be packed.

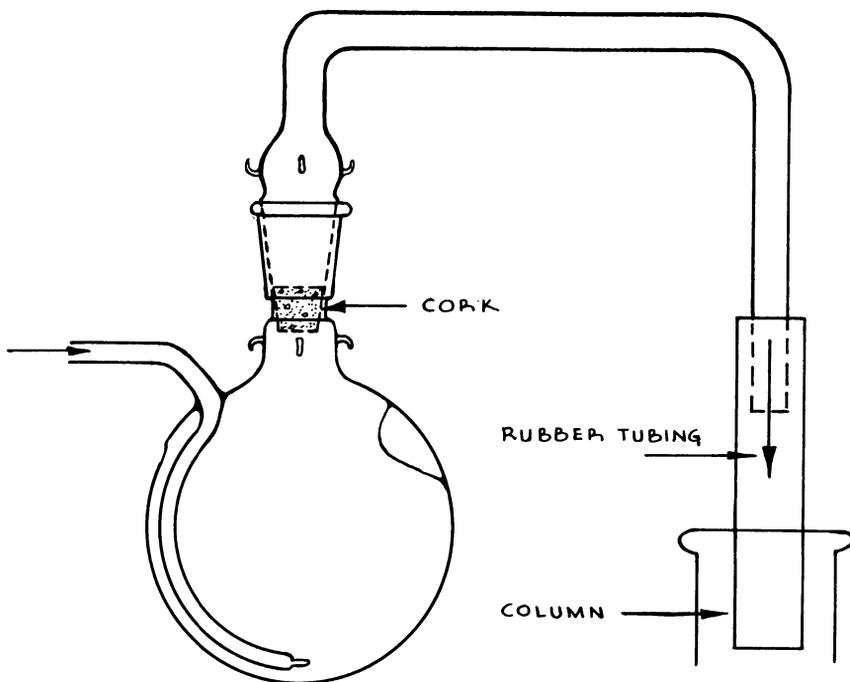


Figure 6

STEDMAN CONE SHAPED SCREEN PACKING: This is widely used and is supplied commercially by H. S. Martin Company, as well as other scientific laboratory suppliers.

PODBIELNIAK HELIPAK AND OCTA-PAK: Both of these packings are a metal wire-coil type with the wire coil cut into many identical small sections. These packings are reported to have a very high efficiency of separation. Not being of a single turn construction, they are free from becoming entangled. Therefore, this packing may be dumped into the column without any special precautions, such as are required when packing single turn helices.

PACKING SUPPORTS AT BOTTOM OF THE RECTIFYING SECTION: While considering the random or dump-in type of

packing, it is appropriate to give some consideration to the method of supporting this packing in the rectifying section.

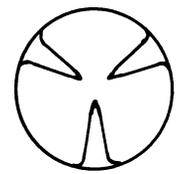
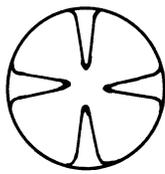
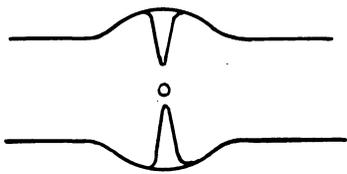
In Figure 7, "A" is an enlarged section at the bottom of the rectifying section in which three or four indentations have been pushed. "B" is an enlarged section in which two rods have been sealed at a 90° angle from each other to form the packing support. With either of these methods of support, it is necessary to first insert a few larger glass cylinders to rest on the indents or rods and form the actual support for the smaller main packing. It is desirable to enlarge the rectifying section where the indents are to be pushed, so as to prevent possible flooding in the column at this point. "C" and "D" show metal screens, either cylinder or cone shaped, which are held in place by means of glass indentations. If platinum is to be used for making the screen supports, hydrogen sulphide should be flushed through the column to poison the platinum and eliminate the catalytic action of the platinum. "E" represents a glass cone, which is ring sealed to the bottom of the rectifying section, and in which a number of small holes has been made. Each hole must be small enough to hold the packing, but the total area of the holes must be considerably more than equal to the free spaces in the packing.

So much for the packings and variables which must be considered in the design of the fractionating columns. Now for the construction features of fractionating equipment.

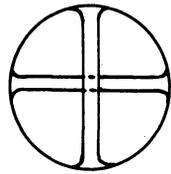
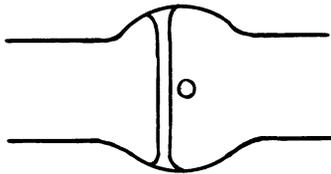
There are three main parts to any fractionating assembly, the *still pot* or boiling kettle, the *column* and the *still head*. All three must be considered together when designing a useful and efficient distillation unit.

THE STILL POT.

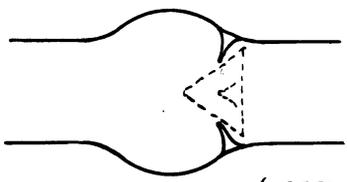
Several designs and shapes are shown in Figure 2. The still pot must be sufficiently large to care for the thermal expansion of the material to be distilled, as well as sufficiently large to handle any possible foaming action of the material. However, it should not be so large that the evaporative surface of the liquid is so great that flooding is caused in the column before a brisk boiling begins in the pot.



A

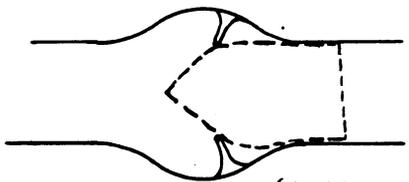


B



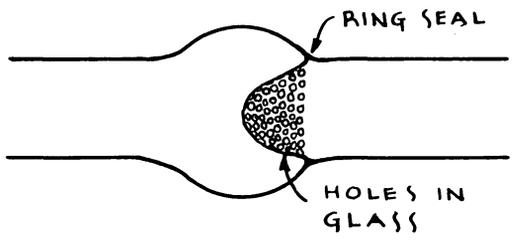
SCREEN
CONE

C



SCREEN
CYLINDER

D



RING SEAL

HOLES IN
GLASS

E

Figure 7

COLUMN.

In addition to the diameter, as was mentioned earlier, the length of the column is also important. According to Perry⁸, the ratio of the height of the column to the diameter should not be less than 15-1. Should the ratio be less than 15-1, the reflux will have a tendency to channel in the packing during operation and this will greatly decrease the operating efficiency of the column. A column should be well insulated and, in some cases, there should be a closely regulated source of external heat available to compensate for radiation or heat loss.

In view of the present knowledge of fractionation and the development of highly efficient fractionating columns, the usefulness of unpacked or deformed hollow tube columns is somewhat limited. However, these columns cannot be completely bypassed in a discussion of distillation equipment.

Probably the most widely used of this type of empty tube column today is the Vigreux and the Snyder floating ball columns. Others in use to some extent would be the Widmer, the Young and possibly the Wurtz bulb columns.

The most simple type of rectifying section to make is the empty tube. This consists of a straight, vertical, empty tube, circular in cross-section. In spite of their simplicity, empty tube columns have been reported as highly efficient when vacuum jacketed and properly operated. In the empty tube column, the downward flow of reflux covers the inner surface of the rectifying section with a thin film of liquid. The ascending vapor, on coming into contact with this liquid film, effects the heat transfer necessary for the separation of the components of the mixture.

In most unpacked or wetted-surface fractionating columns, the vapor flow is streamlined, resulting in a vapor diffusion process which is very slow. This makes a low through-put essential when the column is being operated for high efficiency of separation. With the low through-put, a low takeoff is required, sometimes as low as 0.1 ml per hour, to obtain the maximum efficiency

from the column. A vacuum jacketed, empty or hollow tube column, under ideal conditions, has been reported to have an H.E.P.T. of 3.19 cm.

*Sargent*⁹ developed an improved empty tube or deformed tube (Figure 8).

In this column, the rectifying section is formed in a series of sharp 90° bends. In assembling the section, each angle is formed by sealing a separate section of tubing to the preceding section. It is of prime importance that each bend be *sharp*, so as to permit a complete reversal of the surface of the liquid at each bend. In this manner, a fresh surface of liquid is exposed to the vapor for a short period of time. Sargent reported a column of 7 mm O.D. tubing, having an electric heating jacket, to have an H.E.T.P. of 3 - 4 inches. The author found that a similar column, vacuum jacketed and with a built-in electronic head, to have an H.E.P.T. of 1.5 cm.

*Warren*¹⁰ reported on a spiral empty tube column in which a glass spiral formed the rectifying section. In this column, the vapor and liquid path are of the same length. However, both are considerably longer than they would be for a straight, empty tube column of the same height.

Probably the most efficient of all wetted-surface type columns is the *concentric tube* column (Figure 9).

These columns depend on the liquid-vapor contact taking place in the small, annular space. Over the entire length of the rectifying section, it is essential that both the rod and the tube be of uniform diameter. For this reason, precision bore tubing and precision ground rods should be used. It is also important that the rod be sealed vertically in the tube and that the entire rectifying assembly be sealed vertically in the column jacket.

Plate columns, such as the *Bruun bubble plate* and the *Oldershaw perforated plate* columns, are a challenge to the experienced glassblower. Collins and Lantz¹¹ found that the efficiency of an

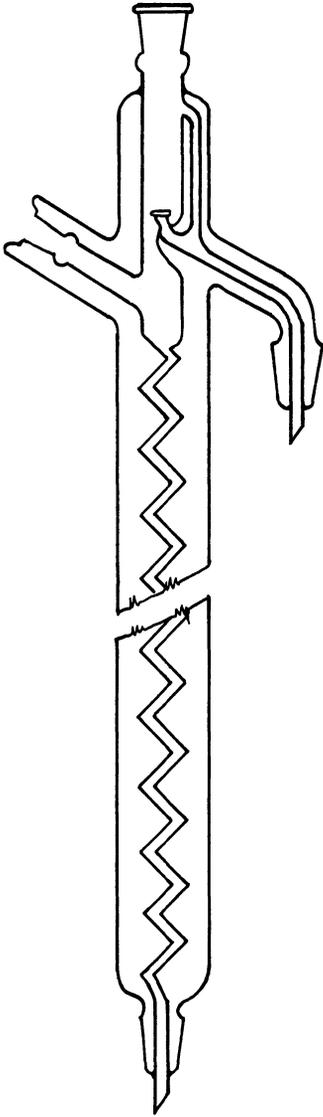


Figure 8

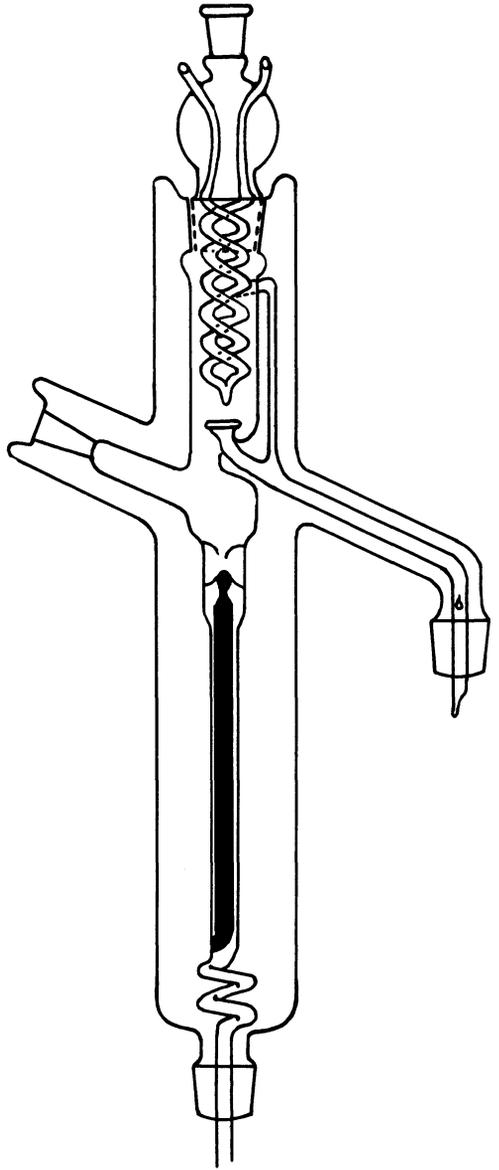


Figure 9

Oldershaw column was nearly independent of the through-put rate and showed no significant variation from column to column, making these columns especially important in laboratories where reproducibility of results is of primary importance. Bubble plate and perforated plate columns are difficult to make as well as time consuming. They can be obtained commercially and so many busy glassblowers prefer to buy these columns.

RANDOM PACKED COLUMNS. The work horse among laboratory fractionating columns is the empty tube column filled with random packing, such as glass helices.

A column for random packing, with an electronic reflux control, is shown in Figure 10.

This type column may be made in many diameters and lengths. Random packed columns may be used to purify material prepara-

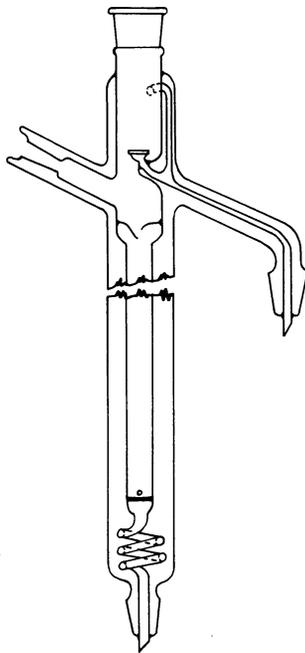


Figure 10

tory to its use, or as a rough purification prior to distillation in one of the more efficient, concentric tube columns when material of the highest purity is required.

According to Feldman, Myles, Wender and Orchin¹², most columns may be used at reduced pressures down to a few centimeters of mercury. Below this pressure, special columns, such as the *empty tube* design, *Sargent columns* and some larger concentric tube column or spinning band columns, should be used. A spinning band column manufactured by the Precision Distillation Apparatus Company is shown in Figure 11.

Spinning band columns are relatively simple to build, have a very low pressure drop between the still pot and the still head, as low as 0.05-0.06 mm of Hg and a low operating holdup. For reduced pressure, the magnetic stirring assembly shown in Figure 11 is very satisfactory. The magnet inside the plastic cap is drawn by the magnet on the motor, resulting in a continued smooth operation with no stops or jerks to effect the equilibrium of the column.

While we are considering columns for reduced pressure distillations, I might state here that for ordinary distillations, the preferred pressure is atmospheric, provided the distillation can be carried out without the sample decomposing, or without the distillation having to be carried out at excessively high temperatures.

The principle advantage of distilling under reduced pressures is the lowering of the boiling temperature required. The chief disadvantage of distilling under reduced pressure is the fact that the through-put or capacity of any column is reduced and the lower the pressure the lower the through-put. Thus, more time is required to fractionate at a desired reflux rate since the sharpness of a separation in any given distillation depends on the same factors, regardless of the pressure used. The transition between plateaus of pure components is not usually as sharp or easy to follow as during a distillation run at atmospheric pressures. An added disadvantage of running distillations under reduced pressure is the additional equipment necessary.

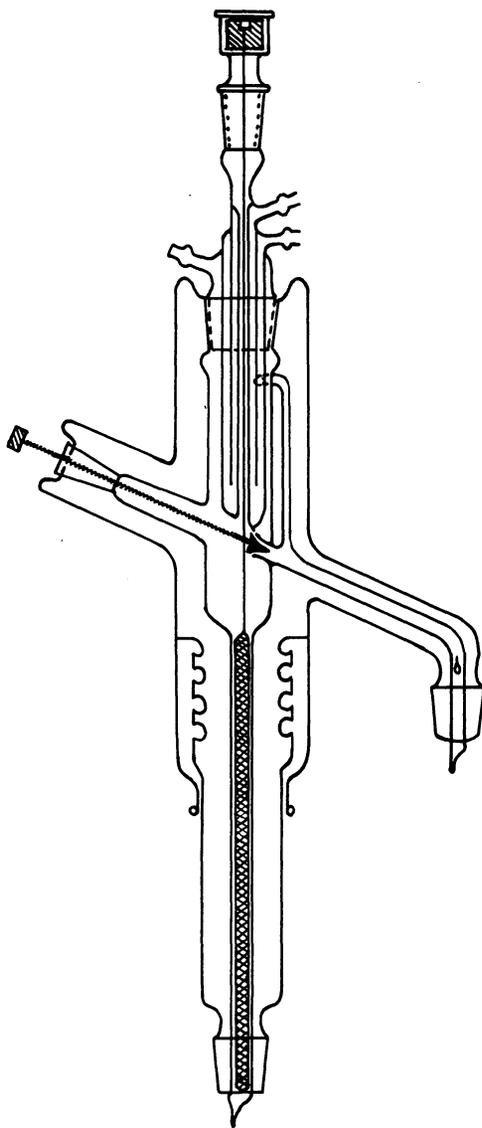


Figure 11

Figure 12 shows auxiliary equipment for reduced pressure distillation.

STILL HEAD

Now let us consider the construction of a *still head*.

The function of a still head is to receive vapors from the top of the rectifying section, condense part or all of the vapor, and return reflux to the column. Generally speaking, no fractionation occurs in the still head unless the head contains a partial condenser in which case some fractionation does take place.

A still head, like the column, must be properly insulated. When an immersion thermometer is to be used, the thermowell must be located so as to be connected to or be a part of the vapor path

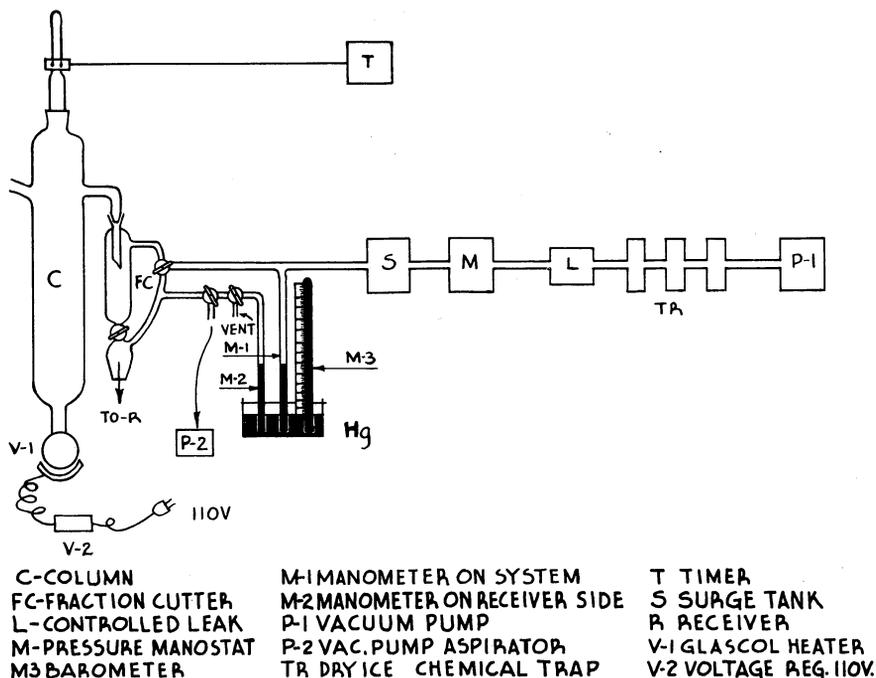


Figure 12

below the reflux condenser. If possible, it is highly desirable that the thermowell also be vacuum jacketed. For high temperature distillations, it will be necessary to have the complete vapor path vacuum jacketed up to and above the takeoff line to assure no heat loss taking place between the top of the rectifying section and the takeoff line. Still heads, which are built into the vacuum jacket directly above the rectifying section, such as in the column shown in Figure 9, are especially serviceable for high temperature distillations.

While discussing still heads, it is appropriate that consideration be given the type of takeoff control to be used. The most common method of controlling the product takeoff is by means of a stopcock. By simply turning the stopcock, any variation between total reflux and total takeoff may be obtained. To facilitate adjustment, one side of the stopcock may be notched with a triangular file. This "V" shaped notch will make fine adjustment of the takeoff easier and more accurate. A second method of controlling the takeoff is by simply turning the cold finger condenser. By turning the drip tip directly into the takeoff line, or by moving it away from the takeoff line, any variation from total reflux to total takeoff may again be achieved. When operating the still head for this type of takeoff control, it should be operated with the stopcock wide open. A third method of controlling takeoff is that in which control is maintained by a combination of the stopcock and moving the cold finger.

Another adaptation of the revolving cold finger control is to have the distillate drop on a rounded ball through which the takeoff line is sealed. Another variation of the cold finger falling drop method is the use of a takeoff cup in which one side of the cup is ground to a sharp knife edge. The drops, when directed onto this sharp edge, will be accurately split into product takeoff and reflux.

Tilting funnels in which movement of the funnel is controlled by means of an electronic repeat timer and a solenoid coil is an often used method of takeoff control (Figure 13).

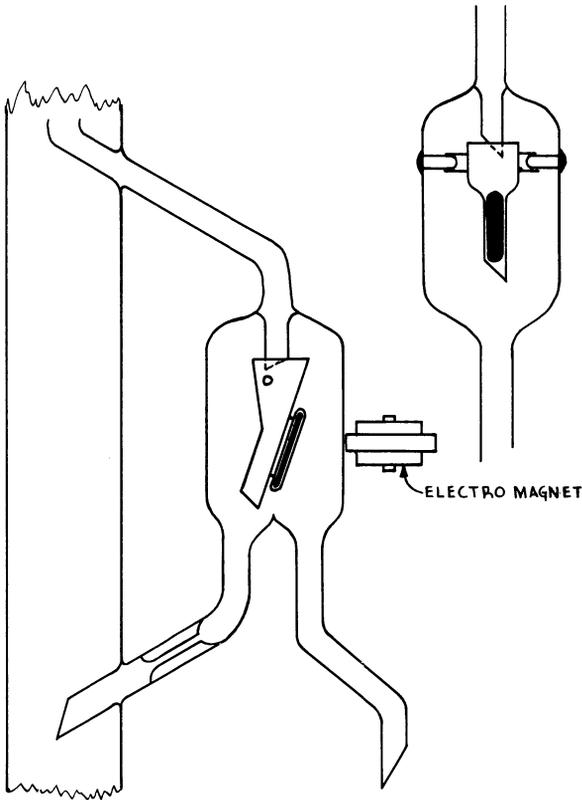


Figure 13

Tilting funnels may be fastened to the outside of the vacuum jacket, as shown in Figure 13, or they may be placed inside the vacuum jacket, as is the case of many Shell-designed still heads. In tilting funnel devices, when the funnel is in a neutral position, the flow of condensate is back into the column as reflux. When the solenoid coil is energized, the flow of condensate is directed into the takeoff line from which it is removed as product.

The most satisfactory type of takeoff control, in the author's opinion, is that in which an electronically controlled and solenoid operated glass check valve is opened and closed at prescribed times by an electronic repeat timer (Figure 14). Such a takeoff control can be built directly above the rectifying section inside the

vacuum jacket and, as such, is especially suitable for high temperature and reduced pressure distillations.

When electronic timers are not available, a column with a still head of basically the same design may be made, but with a manually operated takeoff. In this head, the takeoff cup need not be made from a ground spherical joint and the plunger should be

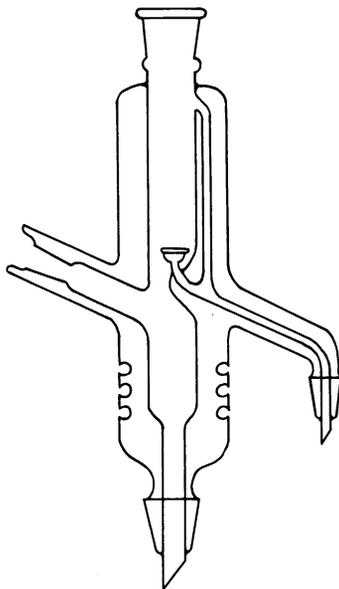


Figure 14

made of Teflon. The Teflon plug is raised or lowered by means of a stainless steel threaded screw. In this type of manual control, the takeoff is slow but constant instead of intermittent as is the case with electronic control. However, careful adjustment of the threaded screw will produce any desired ratio between total reflux and total takeoff.

There are, of course, other designs for product takeoff control. Each in some way is especially suitable for the particular distillation for which it was designed. Basically, however, the stop-

cock control, rotating cold finger control, the swinging funnel, electronic, and manual Teflon plunger control are suitable for a wide variety of still heads and columns.

Before leaving the subject of still heads, let's consider still head condensers briefly. The condensers discussed previously have been total condensers. Now let us discuss a column with a partial condenser.

Partial condensers (Figure 15) may be operated so as to condense all the vapor and return it to the rectifying section. Or, by careful control of the partial condenser temperature, only a desired percentage of the reflux is condensed, allowing the balance

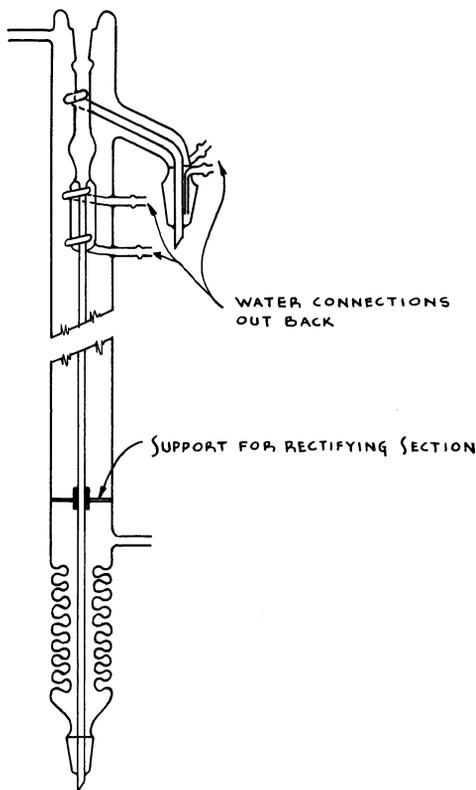


Figure 15

of the vapor to pass through the partial condenser and be taken off as product. Partial condensers can best be regulated if the coolant being used is first passed through a pre-heater or pre-cooler to bring it to the desired temperature before entering the partial condenser.

Partial condensers are especially suitable for micro and semi-micro columns because of their low operating holdup. They are more difficult to operate, however, because of the extreme care which must be taken in regulating the reflux ratio and the takeoff.

COLUMN LAGGING OR INSULATION

There are many different types of insulating jackets for a distillation column. Some of the most common are asbestos, magnesia, air jacket, electric heating jackets and vacuum jackets. Marshall and Southerland¹³ found that a properly lagged Hemple column was from 2.4 to 41.5 times as efficient as an unlagged column, both operating at the same distillation rate.

When magnesia lagging is used, it should be of the same thickness as the diameter of the column. Hence a 25 mm O.D. column should have 25 mm of lagging and a 100 mm O.D. column should have 100 mm of lagging. Assuming heat conductivity to be a fixed constant at a given temperature, a 25 mm column with 25 mm of lagging should have exactly the same heat loss as a 100 mm column covered with 100 mm of lagging, since heat transmission through cylindrical lagging on a cylindrical pipe varies inversely as the logarithm of the ratio between the external and internal diameters of the lagging.

Electric Heat Compensating Jackets. The construction of columns with electric heat compensating jackets is relatively simple. Columns with this type of jacket are particularly adaptable to laboratories where the facilities and equipment for evacuating columns are not available.

Figure 16 shows a column with such a jacket. The tube around which the resistance wire is to be wrapped should be about 3/4 inch

larger than the outside diameter of the column and also slightly longer than the section to be packed. The spacing of the coils should be from 1/2 to 3/4 inch. One thermometer or several thermometers may be supported inside the jacket, or thermocouples may be used to follow the temperature inside the jacket.

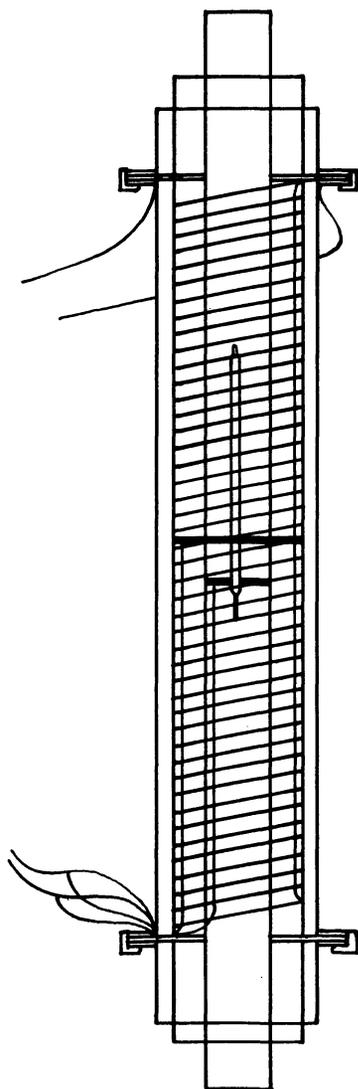


Figure 16

Silvered Vacuum Jackets. Silvered vacuum jackets provide the most efficient insulation for columns, provided the silver plating is done so as to produce a highly reflective surface inside the jacket. Baking improves the reflective surface of the silver, but it can only improve what is already there and not make a reflective surface of a surface coated with a powder deposit. Obtaining this highly reflective surface is properly the subject for a paper in itself; therefore, I will not try to cover it at this time.

The degree of evacuation is also of the highest importance in producing a jacket of efficient and enduring qualities. Even with good vacuum jackets, however, it is often desirable for micro columns, columns used for reduced pressure distillations, and columns to be used at elevated temperatures (up to 400° C) to have a glass fabric electric heating mantle over the vacuum jacket.

When silvered vacuum jackets are used, some means of compensating for the temperature difference and resulting expansion differential between the inner rectifying section and the outer vacuum jacket must be used.

Expansion Members For Vacuum Jacketed Columns. Figure 17 illustrates two different designs for compensating for the difference in expansion between the inner and outer members of the column.

“A” shows the widely used internal expansion members formed on the outer wall of the vacuum jacket. These, when properly formed, will have an expansion of approximately 0.1 mm per bellow and be able to withstand a pressure of 50 pounds. This type of expansion bellow was developed by Pompeo and Meyers¹⁴ of the Shell Development Research Laboratories. They are reproduceable and the number of bellows required to protect the column per degree difference in temperature may be calculated.

In “B” is shown a double spiral at the bottom of the rectifying section. These produce an extremely strong column because the

expansion member is inside the vacuum jacket. When a double coil is used, it may be made from 11-12 mm O.D. tubing while in a column of 18 mm I.D. and under, single coils of 11-12 mm tubing may be used.

Figure 18 shows a variation of the double coil expansion member. With this design, the small diameter coil drains off much of

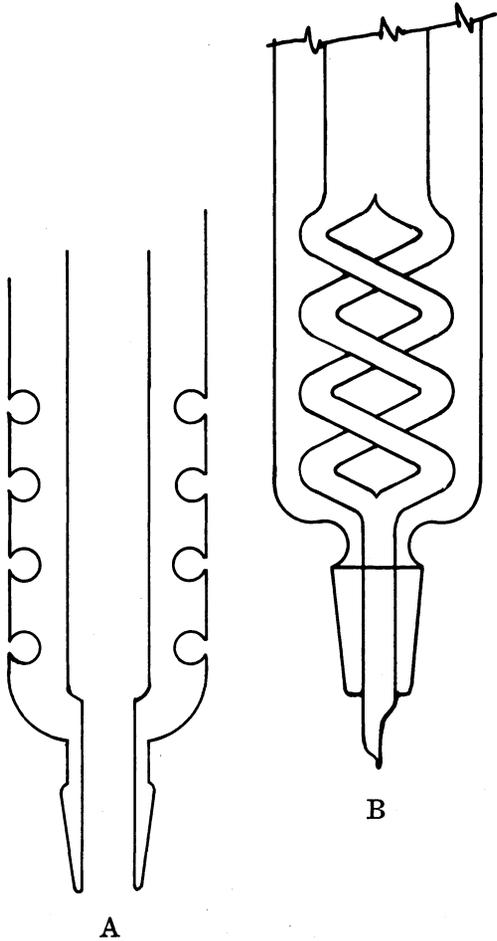


Figure 17

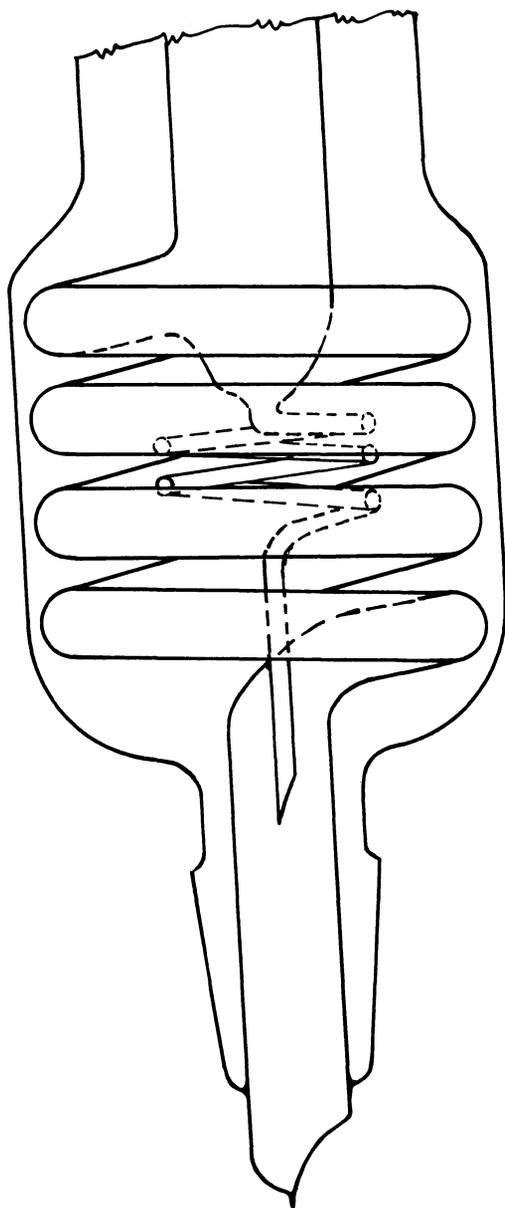


Figure 18

the reflux passing into the still pot, thereby greatly reducing the danger of the column flooding in the single large coil, even under conditions of high through-put.

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Panel Discussion

Stenographic notes were made of the question and answer session held Saturday, May 23.

These notes have been edited to delete repetitive material. However, wherever possible, the exact wording of the speaker has been used.

J. Allen Alexander, president of the American Scientific Glassblowers Society, presided over the panel discussion. Members of the panel were:

Elmer L. Jolley, a product development engineer in the Product Development Section, Laboratory Glassware Sales Department, Corning Glass Works.

Jules E. Benbenek, supervisor of the Glass Blowing Section in Materials and Devices Technology, R.C.A. Laboratories.

George P. Spindler, senior glass apparatus technician, Bell Telephone Laboratories.

Dr R. H. Dalton, Research Chemist, Corning Glass Works.

E. L. Wheeler, University of California.

HELMUT DRECHSEL: Is Corning going to manufacture Vycor stopcocks and is it true that Corning is coming out with a new boron-free glass?

MR. JOLLEY: We now manufacture some Vycor stopcocks on a special order basis. At the present time, to the best of my knowledge, there is no intention of making any in our regular line.

Now, the second question was on the boron-free glass. We have made experimentally a pot melt of a new glass and samples were submitted to the field, certain individuals and companies that have specific needs for this. We don't know yet about the future

plans of this glass and what we are going to do with it because we don't have the results of the test.

CLAIR CAMPBELL: Regarding the Vycor stopcocks, is that Vycor plug to a barrel, or just a barrel like a Teflon plug? What is the stopcock itself and in what types?

MR. JOLLEY: I am sorry, Mr. Campbell, I can't give you a definite answer on that. Now, the plug and barrel are both made of Vycor glass. As I remember, it's a hollow barrel. It's sold by our special apparatus section and I'm not familiar with the sizes they have been making. I'll be glad to have them furnish you specific details.

KARL WALTHER: Will the Vycor be vacuum fired, the 7913 or the 7900 on the stock card?

MR. JOLLEY: It would be the 7910, I believe, Karl, which is the vacuum fired. It would be the vacuum type.

ALLEN CARDINAL: In relation to this boron-free glass, is it available to almost anyone who wants to buy it and what is the code number?

MR. JOLLEY: The answer to that is no. It's an experimental glass which was made in the laboratory. We made a pot melt of it. What the future of the glass is at this time, I can't tell you.

HARRY WEIR: Relating to the boron-free glass, what are the advantages of having the glass boron-free, or aren't you at liberty at this time to divulge these things?

MR. JOLLEY: The new glass, and this I'm drawing from my memory now, has a lower coefficient of expansion and is slightly harder. It's just an experimental glass, I said. We've got a lot of these in the laboratory and so on over a period of years and lots of them don't get into commercial production.

MR. CARDINAL: What specific assistance can we as glass-

blowers receive from the N.B.S.—that's the National Bureau of Standards—in relation to general glass working problems?

MR. JOLLEY: To the best of my knowledge, the National Bureau of Standards does not have any program of assistance to glassblowers. Now, they do, and they have in the past, set up control checks on glasses, methods of analysis and so on, but for specific problems like the sapphire seals or something like that—I don't know that they have any program under way on things like that. The National Bureau of Standards essentially does basic research and one of the prime requirements of this basic research, as I understand it, is that it must not be in competition with industry.

QUESTION FROM UNIDENTIFIED PERSON: Does the annealing oven have any damaging effect on the finished ground stopcocks?

MR. JOLLEY: The answer to that is no if the oven is below 550° C. Most of our items—I won't say most, but an awful lot—having stopcocks on them are annealed as a final operation.

QUESTION FROM UNIDENTIFIED PERSON: How and where may one procure heat-resistant color glass rod and tubing?

MR. JOLLEY: I would say the best answer is to write to Mr. E. L. Jacobs of Corning Glass Works.

QUESTION FROM UNIDENTIFIED PERSON: Is there more than one composition of sapphire glass used in these seals? What was the type used on your experiment?

MR. SPINDLER: No, there is not. It is just a glass, powdered glass seal.

MR. WALTHER: On tube seals, do you concern yourself with crystal orientation?

MR. SPINDLER: No, I do not.

J. ALLEN ALEXANDER: In use, does the sapphire tube haze due to heat?

MR. SPINDLER: No, it does not.

MR. ALEXANDER: It doesn't haze similar to what Vycor would?

MR. SPINDLER: No.

MR. ALEXANDER: What mesh 1826 is best?

MR. SPINDLER: Well, we found about 300 mesh.

MR. WALTHER: What washing techniques are safe for your finished seal, and does this dissolve 1826 in nitric acid? Can you wash these in several acids? How about the frits if they are dissolved in nitric acid? Can they be washed?

MR. SPINDLER: Oh, yes. Definitely. Definitely. The only thing is, HF, of course, will attack the glass bond but it will not attack the sapphire part.

MR. WALTHER: On concentric tube columns, what ratio of liquid do you find advisable between the outer wall and the center rod?

MR. WHEELER: I'm not real sure what you mean. Are you asking about the ratio of the reflux to the takeoff?

MR. WALTHER: No. I'm not. When you run that column, you have a separator on top of the center piece. Some of your liquid has to be run down the outside wall; some on the center wall.

MR. WHEELER: Oh, I see. It's split 50-50 and it's done by way of the divider.

MR. WALTHER: Even though the center one is larger than the outer one, giving the outer one more than the inner one?

MR. WHEELER: If you will check your cup so that you can split that ball in four actual sections, then you'll have half on the outer wall and half on the inner wall.

MR. ALEXANDER: Does copper wire used in the spacer between the tube and rod during annealing and manufacture cause surface difficulties in the column?

MR. WHEELER: Well, I used copper wire for a number of years. Actually, I did get no reaction from it. It marks the glass. I think that's why you raised the question. So, I've gotten away from that by not using copper wire. I use the German silver. It's an annealed silver and it pulls very nicely to give me the accurate dimensions of the wire and it doesn't mark the glass as much as the copper.

MR. WALTHER: If you test the concentricity of it to get the true tolerance, which really makes it a true tolerance, I wanted to know how much tolerance you may have in that, and also what you use to make the test.

MR. WHEELER: I don't test the concentricity of them. I use the true ground rod when I use the true bore tubing. The rod is ground to the same precision as the tubing. For that reason, the only thing that you have to be careful about is getting it centered properly and it's centered properly by the copper wire—excuse me—by the silver wire. In a sense, the silver is soft enough that I can pull it to the exact clearance between the two, the tubes are concentric.

LEO F. DUSEK: What would the distance between the rod and the tube be? What is that consisting of—a very fine tube involving a few thousandths?

MR. WHEELER: Actually, it's three-quarters of a millimeter.

MR. DUSEK: That would be the difference between the inner and outer walls?

MR. WHEELER: Yes, that's right. The difference between the

O.D. of the rod and the I.D. of the tube is one-and-a-half millimeters, which gives us then three-quarters on each side.

HARRY LIPSON: I would appreciate all data on Corning's new glass, 1720 alumina silicate glass, the workability and what metal seals to it and what type of fires are to be used for the same.

DR. DALTON: I am not sure what data you are most interested in. The glass, in general, is an alumina silicate type with quite a high softening point and a high annealing point; expansion of around 40 or so. It can be sealed to tungsten and also molybdenum. It lies intermediate between the two in its expansion behavior. It has a very high resistivity, has fairly good electrical properties, fairly good durability. I think perhaps the outstanding characteristic is the very high softening temperature and the high annealing—particularly the high annealing point. There are actually two glasses that we make which are almost identical—the 1710, which you perhaps are all familiar with in our flame-ware, and the 1720, which is made for laboratory purposes. The principal difference is that the glass that's made for flame-ware, the 1710, has a little color added so that you can easily distinguish it from the ordinary chemical Pyrex, the 7740.

The properties of the 1720 are softening point, 915 degrees C.; annealing point, 715 degrees C.; strain point, 668 degrees C. Expansion is 42, density, 2.53 and index of refraction, 1.53.

You mentioned the flame workability. The glass is very hard, of course, therefore, it requires a fairly high temperature flame.

It has another characteristic which causes considerable annoyance in flame working. It tends to reboil rather badly as in the handling in the flame; it tends to bubble. That can best be avoided by working it at as low a temperature as you can. That is, don't heat it excessively. Don't use a sharp oxidizing flame. That type of flame is bad. It's better to use a softer reducing type flame.

There is a third step which we have done considerable work on

here in Corning in the last year or two; not only in connection with this glass but with other glasses which show a tendency to reboil on flame working. We have found that by adding vital compounds of either boron or silicon to the flame in which we're working the glass, we get a very dramatic improvement, a very dramatic suppression of this tendency of the glass to reboil.

Now the simplest, in most cases, the simplest and easiest thing to use is merely to take some ordinary boric acid that you can get at any drug store and dissolve it in methyl alcohol, or ethyl alcohol would also do, and then bubble all or part of your gas which you are using in the flame through this solution of boric acid in the alcohol. You can tell right away when you're getting a good effect because the flame will be colored a bright green, as a result of the boron that's feeding into it.

MR. LIPSON: I have tried that and it's too complicated. I was told I could use hydrogen and I did.

DR. DALTON: Well, I think perhaps a hydrogen flame may be a little better than an ordinary gas flame, although we have never found a very dramatic difference there in general. The only thing we have found is that the sharp oxidizing type of flame gives you the most trouble. The softer, more reducing you can make the flame, the better off you are. We have a little leaflet describing recommended procedure for handling this boric oxide solution. If you like, I would be happy to send you one. Maybe with the help of that, you wouldn't find it so complicated. It is being used industrially. It is being used by ourselves, also, in cases not only of that glass but in other glasses where reboil has been a serious problem.

I mentioned also that the silicon compounds would work and the best thing to use there, I think, is ethyl silicate which is a liquid. You can bubble your gas through that and pick up a certain amount of oxy-silicon into the flame and that is just as effective or maybe more so. We favor the boric oxide ourselves because it's a comparatively innocuous material. It doesn't do

any harm if fumes get around in the room. It's very readily available and, also, you can tell right away whether or not you have it in the flame by this color effect.

PAUL ROMAN: I might add, we had a little difficulty with that glass and I injected a little air into our gas and oxygen. I don't know what diameter Harry Lipson is interested in, but I know they work fairly well in the smaller diameters and we don't get the bubbling. I think the diameter I used was 22 millimeters.

HOWARD A. SCHAEFER, Dr. Dalton, Mr. McLellan mentioned yesterday in passing that Pyroceram was treated as a two-phase heat treatment. Could you elaborate on that?

DR. DALTON: The Pyroceram, of course, consists of a material which is made first in the form of a glass and then is converted into a body which is largely crystalline. That conversion must be carried on in a controlled manner so as to produce the right type of crystal, the right size of crystal, the right quantity of crystal, the right number of crystals per unit in volume in order to get the advantageous properties which they have been able to achieve in these materials.

Now that heat treatment, that crystallization process, you might say, consists of two steps. First of all, in the creation of nuclei which form the initial starting point of a crystal; if you take water, for example, and purify it very carefully and have it very free from any particles, anything of that kind or any foreign materials, you can cool it way below its normal crystallization temperature and still keep it in the liquid condition. Now, that's true of most liquids and that's true also of these glasses. So, your first step in the treatment is the production of these nuclei and that takes place at a temperature range considerably lower than the range at which you get maximum crystal growth.

This general theory of this type of phenomenon goes back to the work of Gustav Tamann in Germany around the turn of the

century. He studied it in the case of organic liquids and found—he was the first to point it out—that before you get these crystals formed, you must have these nuclei formed and that they form at a much lower temperature than the temperature at which you get maximum crystal growth. So, you pause first in this range where the nuclei will form and you heat treat in that range. But, at that temperature, crystal growth is extremely slow and it would take a very, very long time to grow these and to develop any appreciable percentage of the crystallization in the composition as a whole. So, after creating the nuclei at this lower temperature, you then go up to a higher temperature where crystal growth is rapid and convert the complete body over largely into the crystalline form.

MR. ALEXANDER: Is there any standard set up for porosity? What one company designates as a medium, another company may call fine. Now, if we're going to work with and go through the trouble of having standardization and this is not standardized, I think that probably there should be something done about it.

MR. JOLLEY: There is an A.S.T.M. standard published as a tentative a year ago which gives the method of determining maximum pore diameter and permeability. There has been no effort to standardize the pore diameter between companies. We were one of the original manufacturers—original in this country—of the fritted disc and we have shifted our pore sizes over a period of years based on requirements from the field. It used to be that our fine porosity disc had a pore diameter of 4.5 to about 7; now it's 4 to 5.5. We shifted them as the requirements—as the industry required, let's say. So, there has been no definite standardization that a medium porosity disc manufactured by all companies will have the same pore diameter. There has been a method published so that you can determine the pore diameter of the filter that you're using which was giving you good results.

MR. ALEXANDER: Do you think that something should be done? What do you recommend? To me, it seems that the companies should be able to get together the same as they did in

other standardization. Or, would you want us to recommend to the people that they do that?

MR. JOLLEY: Well, this one is a little ticklish, frankly. I think, actually, in a sense, we are serving two different fields. The different pore sizes are available. Naturally, both companies, or the two prime people that manufacture them in the country, think that their pore size is the one that should be standard. So, there has been actually no attempt to standardize pore sizes. Stuff that comes from abroad, the English stuff which a lot of people have, has an entirely different pore diameter. They go on the average maximum pore diameter. In this country, we specify that it is the absolute maximum instead of the average.

Frankly, as far as standardization on fritted ware goes, I don't know where we're going at this stage.

MR. WEIR: I actually have three questions. First, when are you going to start fire polishing the tops of the grinds of the resin kettles?

MR. JOLLEY: Frankly, this is the first time I've heard of a need for fire polished top on a resin kettle.

MR. WEIR: I am referring to the grinds. The grinds are cut off. When you cut them off at the top, then you grind your taper and you do not come back and fire polish the material.

MR. JOLLEY: Oh, the top. You're talking about the opening on the resin—the top, the ground joint surface.

MR. WEIR: That's right. That's your one, two, three and four liter sizes.

MR. JOLLEY: Yes, but you're talking about the cover only?

MR. WEIR: Yes, the cover.

MR. JOLLEY: Where the ground joints fit in?

MR. WEIR: Yes.

MR. JOLLEY: It has never been brought up before. Fire polished top? I don't see why it couldn't be done. I think right now it's cut and ground.

MR. WEIR: It is. I looked at it yesterday out at the plant.

MR. JOLLEY: They could be fire polished, I assume. It is put in the mold and the cover comes out the reverse of what is normally used. At the place where the ground joints go in, there is a big knob of glass, like a desiccator knob, on the top of that and that has to be cut off. When they cut it now, they just grind it. I see no reason, unless it would upset the grinding in some way, why that couldn't be flame polished after it's cut. Instead of doing a cut and grind, they probably could do a cut and fire polish.

MR. WEIR: It is rumored that you are making compact Vycor graded seals. If this is true, when are we going to have them made available to us?

MR. JOLLEY: They are listed in the new supplemental catalog.

MR. WEIR: Why then are the supply houses still giving us the long monstrosities?

MR. JOLLEY: We list both sizes, actually. We list both the ones with the extensions on the end and this year, in our new supplement, we included the short length ones.

MR. WEIR: This one is directed to Mr. Wheeler. You say that the ratio of diameter to the height column you should carry is 15-1. How much is the efficiency of a column reduced? We'll take, for example, a 25 mm column that's one inch I.D. You're saying that should be no more than 15 inches tall. Suppose you go to 20 inches tall. How much would the efficiency of the extra plates available be diminished?

MR. WHEELER: I did not state that was the maximum. That was the minimum ratio of 15-1. If your inside diameter is 25 mm,

then you should have 15 inches of length. You can go any length you want, but you shouldn't go shorter than that 15-1.

MR. ALEXANDER: What is the recommended ratio of the cross section to the diameter of the column? From what I can understand, it is supposed to be 8-1.

MR. WHEELER: The diameter of the packing compared to the diameter of the column should be in that ratio of 8 to 1. If you have too large a packing, you distinctly have channeling of the reflux in the column. If you have a larger ratio there, of course, you're still all right. This, again, is a minimum. This was, yes, the minimum that you shouldn't go below 8 to 1.

MR. DUSEK: This is directed to Mr. Wheeler. In sealing a magnet in glass, what is the requirement, without a hit and miss system, after you have one end sealed? How do you close the other end without having to blow it out and also make the uniform wall thickness?

MR. WHEELER: Are you thinking distinctly of magnets or just the material that is magnetic?

MR. DUSEK: Well, it can be any kind of metallic material.

MR. WHEELER: If you're referring to a metal that you want to activate by a solenoid coil, and it doesn't have to be magnetic, the easiest way to put it in is to put it in red hot and drop the metal bar in the tube while it's red hot. Then seal it off on one end and, of course, as the metal cools, the air contracts and it will pull your glass in instead of blowing out.

MR. DUSEK: You can't do it with a magnet. You do that just with a metal.

MR. WHEELER: That's right.

MR. DUSEK: How do you do it with a magnet?

MR. WHEELER: I don't know of any sure-fire method.

MR. DUSEK: Just hit and miss; is that correct?

MR. WHEELER: It is pretty much hit and miss. Maybe somebody here could answer that a little better.

MR. ALEXANDER: I think I can. We encase a lot of these $\frac{1}{4}$ inch x $1\frac{1}{2}$ inch magnetic rods in borosilica glass and here is how we do it.

First, we grind off the sharp corners of the magnet and also the sides so the whole metal piece is bright, not tarnished. Then a 6-inch piece of standard wall tubing is selected to make a fairly close fit over the metal. The glass tube is then closed at one end and thickened to about 4 mm wall; to which a 6-inch piece of 3 mm rod is sealed as a handle. Into the glass tube a small wad of glass about $\frac{1}{8}$ inch thick is placed against the sealed end. It should be stated here that the glass wool had to be previously heated in the annealing oven to remove any organic matter from the fibers. The polished metal magnet is then inserted into the glass tube with the glass acting as a cushion. The glass tube is then heated about one inch from the magnet, and drawn to a point or stem. A second piece of tubing is then drawn to a finer stem so as to fit into the one previously drawn and inserted until it touches the magnet. A small stream of hydrogen gas is then passed through the inner tube to flush out any air around the magnet or in the glass wool, and to keep the metal from oxidizing when heated. With the hydrogen flowing through the inner tube, the glass around the metal magnet is heated considerably. Then in rather rapid succession, the small hydrogen tube is removed, the glass tube heated directly above the metal with a hot fast flame, sealed off by sucking the glass together and causing a slight vacuum inside the tube. This end is reheated and rounded off by adding glass cane. When the glass encased magnet cools, the 3 mm rod used as a handle is broken off and the end ground smooth.

If this procedure is done correctly, the metal magnet will remain bright, and will remain so until the glass cover is cracked or broken.

GEORGE SITES: I think that if you're not quite so particular in wanting a bright piece of material what I have accidentally discovered has been very helpful to me. It is that even on magnetic

material, I haven't ruined the magnetism by heating the metal. It hasn't hurt it at all. All that I do is seal off one end, pack it with a little piece of chemically pure asbestos. You can burn off your powdered asbestos, if you wish. Put that down about a sixteenth of an inch. Put your piece of magnet in—I am speaking of a cylindrical rod now—and use any type of holder on the one end. I sometimes use a standard taper joint and stick it in and warm the whole thing up in the flame. You don't have to get it glowing hot, but practically glowing—oh, say, eight or nine hundred degrees Fahrenheit—and pack another sixteenth of an inch of asbestos in on top of it. You can do this before you heat it. Then you pull the tip seal very close and, at this point, you heat it up to your—let's call it dull red heat, and seal it off quickly right at that point. Let it cool and then heat right on the surface, right on the end of the tube and it will suck itself in tight against this asbestos.

I didn't think that this would work but I happened to accidentally be going through a handbook one day and, I found that asbestos has an expansion of 32 so I tried it. And I have yet to have one break. It actually makes a surface seal against the asbestos. But I have never had one come back broken yet.

FRANK ARRISON: Alex, would you use nitrogen rather than hydrogen in sealing your magnet in?

MR. ALEXANDER: No. We use hydrogen.

MR. ARRISON: You do the same thing with nitrogen?

MR. ALEXANDER: We use hydrogen. We don't use nitrogen. You see, the hydrogen keeps it from oxidizing.

SAMUEL KNISELY: Mr. Jolley, I would like to get the handbook that you mentioned on that standardization of frits. How can I secure it?

MR. JOLLEY: We don't have any of those pamphlets. Actually, it is an A.S.T.M. book. The number, it seems to me, is E-1-26-58-T. It's in the E series of your A.S.T.M. book. It's a D-2 petroturi. In the back section, there is an "E" series that apply to that group of products.

MR. WALTHER: Perhaps Mr. Jolley would like to comment on this, too. He has been very helpful in the past year on the subject. All the materials that are building up in the background count radiation-wise. They're stripping lead off the bottom of old ships and making protection for Geiger counters because it is more radioactive. Likewise, glass has gone up in radiation. We have found that any glass manufactured after 1955 is more radioactive in background than the older glass.

I would like to suggest that any glassblower who has any real old Pyrex or glass on the shelf coated with dirt, see if you can trade with the fellow who is interested in making counters with a low background in nature. We're especially interested in seeing more team work to trade this old Pyrex and stuff older than 1955. It has a low background of 65 counts per minute. New Pyrex has about 100 to 115.

Mr. Jolley also helped us get some glass from overseas. We also found that the English equivalent of Pyrex was high in radiation. There is only one thing we still have to check; whether the glass, if there is any left, manufactured below—that is, in the lower segment of our globe, whether that's as radioactive as in the northern sections of our globe.

Mr. Jolley, do you have anything you can say?

MR. JOLLEY: I think the only thing we've pretty much decided so far, Karl, is that it's the raw materials which have been contaminated—the borax and so on. And it all comes from essentially one place. It's just a problem that's going to be here until either we get another source of supply or raw materials. It's radioactive fallout. That's all we know at this time and the only thing we can think of.

WILLIAM A. GILHOOLEY, JR.: Are the specifications for machining stainless in the paper presented and when would copies be available?

MR. BENBENEK: The answer is yes. The sketches are in the publication and if I could get your address and name, I'll gladly mail you the duplicate of this paper that I have presented.

QUESTION FROM UNIDENTIFIED PERSON: This is also directed to Mr. Benbenek: What is the color of 304-7052 seal?

MR. BENBENEK: Chromium green.

JOHN DALY: I would like to know the material that is required in the heating element of a sapphire oven.

MR. SPINDLER: It is nichrome wire. It comes in different qualities but you use the best type.

MR. DALY: Could that be de-blackened or oxidized to prevent breaking?

MR. SPINDLER: No, because you've got it coated over. If you don't go over 1000 or 1100 degrees Centigrade, you're safe. But, don't go over that. It will burn out.

MR. ARRISON: Where is chrome plating of moly being done? Is it available commercially?

MR. BENBENEK: Commercially, I'm not certain because we have just developed this process about two months ago. But, we have chrome plating facilities in our laboratory and I suppose that almost any plating shop would be glad to do this for you. It's not difficult in any way and the amount of plating is also in the publication which is two-tenths of a mil. This is not very critical, so far as I found. I have had less on and I have had more on and they both work equally well.

MR. ALEXANDER: I guess this written question is directed also to you, Mr. Benbenek. What is the final color of the glass to stainless steel seal?

MR. BENBENEK: Green, wherever chrome is used.

BRUCE GRAVES: How is stainless oxidized in a hydrogen atmosphere as stated? Hydrogen is a reducing gas.

MR. BENBENEK: It's oxidized in lime hydrogen, wet hydrogen. Usually bubbling through water, this does oxidize chromium but not the stainless steel itself. No, that's a mistake. The dry hydrogen will keep the stainless steel clean. In fact, it is a cleaning method. But, using wet or lime hydrogen, this oxidizes the chromium. If it is placed in a temperature of 1,065 degrees Centigrade, this is adequate and it does the job.

HARRY HUTH: Are metals oxidized in the hydrogen atmosphere? What is meant by limed hydrogen?

MR. BENBENEK: There are, of course, metals like Kovar which can be oxidized in lime or dry, but stainless steel is one that will oxidize in lime and stay clean and dry.

MR. ALEXANDER: Can tungsten beaded with 3320 canary be pinched into 772 tube?

MR. BENBENEK: Yes. I have found that if you bead with 3320 canary glass and follow it by adding an additional sleeve of 772 nonex and, by progressively heating this sleeve and attaching it to the canary, then it can be inserted into a larger nonex member and pressed or pinched.

MR. CAMPBELL: I would like to direct this to Dr. Dalton. On the Vycor—metal, glass metal seals, is there anything new that can be published at this time, or anything on real fine wires in the neighborhood of 12 wires into approximately an inch or an inch-and-a-half or two or in that range, that could be put through without going into moly discs and stuff like that?

DR. DALTON: I am sorry to say, Clair, there isn't anything new. As far as I know, there is no magic way of solving the problem yet of sealing metals into these very low expansion materials like Vycor or fused silica. The metallurgists have let us down there. They haven't got any metals that are anywhere near low enough to go through and so you have to resort to one

of two dodges. You have got to thin out the metal so that it doesn't stretch the glass excessively, as was done in the mercury lamps, for example, where they use a thin moly strip which is about a half a thousandths thick. That's sucked down in that vacuum to collapse the glass onto it and you've got to, of course, protect it from oxidization.

They also have a technique in which they get away from the vacuum by flushing dry nitrogen through to make a pressed stem that way. So, they've made a little progress, I think, in getting around to a more practical, commercial process, but it still depends on the same old thing of making the metal so thin that it can't stretch the glass.

Now, the other approach, of course, is to use some kind of a graded seal and you're probably familiar with the various things available there. There are some low expansion glasses that can be used for the joint. There is the tier type graded seal that our laboratories supply department manufactures. There is a multi-form type of graded seal, such as is used, for example, in the germicidal lamps, which enables them to seal Kovar wire to the end of Vycor tubes. But, that is not news. You are probably familiar with all of them. I guess the answer to your question is that, as far as I know, there is nothing. There are no radical new developments.

W. L. JOHNSON: A university medical school asked my advice in solving one of its problems. They use a lot of these micro-pipettes, micro-connectors, half a micron in diameter, 200 microns long. They insert these in living cells for the purpose of taking PH measurements, current measurements and introducing drugs within the cell. The idea is that they are so small and they don't want any damage done to the cell as a result of sticking the electrode in there.

Their problem is that they want to know how far they can stick this electrode in the cell. They use a micro-manipulator to do it, but the cell has a certain amount of give on the surface. So, they really want to know if they push this electrode down, say 20 microns, if it goes in the cell 20 microns or not.

Their idea was to be able to place some kind of mark on this tube so they can measure it under the microscope and stick it into the cell so that it would be the proper distance in the cell.

How in the world do you mark something half a micron in diameter?

DR. DALTON: I might suggest that possibly they might use a photosensitive process. Mark it photographically.

ROBERT MILLER: In tissue culture work, is most sensitive glass inert enough?

DR. DALTON: I am sorry, I really can't answer that because I am not at all familiar with tissue culture work and what the requirements are there. I don't know how durable a glass you have to have to handle it successfully.

As a sort of an approximate answer, I could say that the photosensitive glass, the types that are most common, at least, have a durability similar to that of ordinary lime glass. So, I think if an ordinary lime glass—an ordinary bottle or window glass or lamp bulb glass—was satisfactory, this probably would also. But, beyond that, I can't answer the question.

ERNEST HAMPEL: Mr. Jolley, can you tell me why the old type diameter tube was changed to a heavy shank on their 10/30, 12/30 joint?

MR. JOLLEY: I assume that what you're referring to is the outer ground joint, the large diameter tubing at the end. Well, we changed over to that about a year and a half ago, after a survey of the glassblowers here in this country. On the small-sized joints, actually, I think they find it's easier to work the larger diameter tubing—the ground joint with the larger diameter tubing on it—than the old, small diameter tubing.

MR. ALEXANDER: I think I can add to that, too, that on the old type 10/30s, you can't get through the thermometers that have the 10/30s ground on them. Therefore, most of the outlets you put on a flask for that size are usually for the thermometers.

With the old type, you had to ream it out and, therefore, took a chance of hurting the grind by working close to it. However, I do think that the Corning people should make the old type available for this. It might add to their inventory, but there are some cases where the specifications have been written up with the old type diameter tube.

MARVIN SCHURMAN: When Corning changed to the larger diameter, at the same time they also went to a heavy wall thickness which does not match up with the standard wall tube of the same diameter. I wonder why the heavier wall does not go as well with the larger diameter?

MR. ALEXANDER: I think I can answer that. The recommendation of the glassblowers was that that should be of a heavier wall. I don't think it's heavy wall. It's probably medium wall. Am I right?

MR. JOLLEY: It is not heavy wall. It's medium wall.

MR. ALEXANDER: From the glassblowers' standpoint, we think that's pretty good because most of the time when we make a seal on the ordinary type of glass, we make a gather on the bottom of it anyway. We thicken it up, so that when you make your seal you've got the thick glass there. You don't have to thicken it up when you're making the seal. It's available to you and it makes it easier glassblowing. It therefore makes it more uniform. You can heat it up or make your seal and then blow it and pull it and you've got a beautiful joint.

PAUL V. YEAGER: I need glass that will hold liquid helium for 48 hours. I came out to break the seal in my pots and my four wall Duvar flask didn't stand up. Do you have any suggestion of glass that will?

DR. DALTON: Perhaps the best glass readily available for containing helium as having the lowest diffusion rate—in other words, the lowest loss rate for helium—is the 1720 glass.

I am not quite clear, though, on whether there is something else about your requirement that interferes with the use of that glass. The glass we talked about earlier is an alumina silicate

type of glass. At room temperature, the diffusion rate for helium is about 1/10,000 of that for the ordinary chemical Pyrex, the 7740. In other words, you have a ratio there of about 10,000 to 1 in diffusion rate of helium to the glass.

The glass has an expansion, as I mentioned before, I think the figure was 42. It has a high annealing point—a rather hard glass. It has been used for duraglass for containing liquid helium.

MR. YEAGER: What is the annealing point?

DR. DALTON: It's a little over 700; 715 degrees Centigrade.

MR. YEAGER: We are putting this in big amounts also. We want to take it up to at least 1000 degrees C. Where can we get that?

DR. DALTON: Well, of course, the softening point of the 1720 glass is 915 degrees C., so you couldn't possibly take it to 1000. I don't know of any glass that would stand 1000 degrees C., as it does have a low permeability. The glasses, in general, that will stand those high temperatures—that would stand that high a temperature—are, in general, rather permeable to helium.

We are working experimentally on some glasses that will stand higher temperatures. I doubt that even those would be satisfactory to 1000. But, we are planning to develop glasses of the general type of the 1720 and pushing to as high a temperature as we can with them. That's all experimental, though. There is nothing commercially available and even there, I would be surprised if we succeed in going as far as you are seeking.

MR. YEAGER: A thousand degrees is about the lowest we want to go. We want to go higher. How about this Pyroceram?

DR. DALTON: That might be a possibility. I don't have any data on the helium permeability. I don't know whether any exists or not. If it does, I am not aware of it, but that would be worth taking a look at. It would stand those temperatures and it being largely crystalline, rather than vitreous, it might have a favorable permeability.

Another possibility might be sapphire. There again, I don't know of any data on helium diffusion through sapphire.

CARL NYMAN: Is there any way that we can spot weld iridium to tungsten without a metal of nickel in between?

DR. DALTON: I'm afraid I can't answer that. We're not very familiar with the welding and joining possibilities in metals. If I were going to do it myself, I would put on the nickel. I know that will work and can be done.

MR. NYMAN: I don't think this can be tolerated enough.

DR. DALTON: But, whether it is possible to do it without nickel or not, I can't say.

MR. BENBENEK: I can't help you as to how to do it, but I know that Fan Steel Metallurgical can do it. Maybe if you will check with them, they will give you the process. I have seen some of their samples. In fact, they're welding and brazing in just about all the metals that I've ever heard of.

WALTER SCHAEFER: I have a question with reference to your article on your Teflon needle valve, the 4.7 millimeter bore. It's the manostat stopcock. Could they, the Teflon, be available in a larger size, a larger bore?

MR. JOLLEY: I don't know any reason why they couldn't be. Actually, Walt, it would be a question of demand for the item.

MR. SCHAEFER: Do you make it yourself?

MR. JOLLEY: No, we do not.

MR. SCHAEFER: On the ground boron-free glass, is that available?

MR. JOLLEY: You're talking about ground and screened cullet? It's available commercially.

MR. SCHAEFER: Could someone tell us some information about the didymium eyeglasses, 520 glass didymium eyeglasses,

used by the glassblowers. I am sure there are developments made from time to time. We have just recently had a new optician in our company and he has had trouble getting used to the glassblowers' requirement. Didymium comes in several numbers. Which ones are more suitable for glassblowers and what do they recommend?

MR. ALEXANDER: Your question then, if I get it correctly, is that didymium comes in two or three different grades and you want to know what one is more suitable for the glassblower? In other words, the one that cuts out the sodium part of the spectrum rather than some of the others.

LEROY BRYANT: I just had contact with the American Optical Company and they do put out the blanks. They are used by all of us fellows here in the glassblowing and they are available through American Optical. If you'll contact them, they'll grind them. They'll do anything that you want them to do if you tell them. They'll even grind them to your prescription.

MR. SCHAEFER: Bifocals?

MR. BRYANT: No bifocals.

MR. ALEXANDER: I do wish that there was something done or this Society could go on record and try to get didymium with the bifocals in them.

FRANK CROTIN: I have just ordered a pair of bifocals, filled to my prescription and I will get them in a few weeks from American Optical.

OWEN KINGSBURY: When I worked for General Electric, Bausch and Lomb had a glassblowing plant dealing in the G-20s. That's the type I use. I still have my didymiums and I have my 20s and I work prescriptions in those too.

MR. ALEXANDER: Bifocals?

MR. KINGSBURY: I am pretty sure, because we had some discussion on that with the Bausch and Lomb representative in Schenectady. I know they grind prescriptions there.

MR. ALEXANDER: Oh, yes, you can grind a prescription, but we're talking about bifocals.

MR. KINGSBURY: I am not too sure, but I do know that G-20s seem to be used more in grinding prescriptions, more so than the didymium.

MR. ALEXANDER: In other words, you like the G-20 better than the didymium.

MR. KINGSBURY: That's right

ANTHONY J. HAWKINS: As long as the story is being told, Vancouver has the same type of problem. We have the machines based upon the socket joints which were double ended. By that, I mean, you had your length of tubing and you had your socket formed on either end. This struck me that from the average apparatus point of view, something like this would effect a great saving in space because, of course, you can put double the number of joints in the same floor space and you cut down a large amount of glass wastage if something like this would, of course, be available.

I would like to direct the question to the Corning members because, as far as I remember, I don't see a stock item of this nature in the catalog.

MR. JOLLEY: We don't have a stock item of that nature in our catalog. We actually use that as a part in the manufacture of our own ware. There is absolutely no reason, except for convenience, that a ground joint is six inches overall length now, except that a lot of times, apparently, you use the six inches overall length. This has been established. It's in the federal specification that it should be six inches. Where it originated, I don't know, because I know a lot of these ground joints are cut off. Theoretically, a 24/40 could be sold as a three inch length instead of double end. I mean, it could be either double end, six inch, or three inch length. We're pretty much stuck with the standards that have been established.

MR. WEIR: A couple of years ago when we were up here, we managed to get a couple of 21-inch television face plates. We have used them to extreme advantage in handling films. Since Corning does not manufacture a dish of this size, how are these items available from Corning? Tell us how we can get them.

MR. JOLLEY: Frankly, we don't know.

MR. WEIR: I would like to see a representative of your Optical Department to find out where I could find optical filters. I would like to know what bulletins are available and, if they are available, what is the cost of these filters. I am not referring to all optical filters, but to a particular one. Actually, we're interested in 4200 angstroms wave length. How will I find the prices of these filters?

MR. JOLLEY: We have an optical filter catalog available.

DROHAN: In this relation of grinding glass to prescription, perhaps we can have some of our optical people contact American Optical and check on this question you brought up about bifocals, et cetera.

F. J. MALLOY: On two different occasions in the past, I have had bifocals ground by Bausch and Lomb.

FRED SHEDD: It is true that Bausch and Lomb did make glassblowers' glass into bifocals, but they have discontinued that. The reason is that the demand was not large enough to continue the bifocals.

MR. ALEXANDER: Would they consider a special run now that we know and can contact in the neighborhood of 600 to 1000 glassblowers. I expect 50 per cent or better are over 40 and need them. Do you think they would?

MR. SHEDD: I just go from our own experience in attempting to get the bifocals.

AL GREINER: I would like to direct a question to Mr. Spindler. I wonder if he would elaborate a little more on the electric oven as to the temperatures that there should be.

MR. SPINDLER: In which way?

MR. GREINER: I would like to know some rough dimensions that I know we have. I am told you have two points with a temperature around 900° Centigrade, and the lower temperature which I haven't got.

MR. SPINDLER: Seven hundred Centigrade.

MR. GREINER: I know you use 1826 powder fritted glass. When that seal cools off, that's 7280 glass; is that right?

MR. SPINDLER: That's right.

MR. GREINER: Why do you use a thread? Why can't you take that 1826 in a round form and wrap it on the 7280 directly beforehand?

MR. SPINDLER: I haven't tried that. That's a new one.

MR. GREINER: Run a little bit around it and possibly put it in the oven and let the thing drop down on a sapphire wire.

MR. SPINDLER: Oh, no. Then, you're probably getting away from your powdered glass.

MR. GREINER: You feel it is absolutely necessary to have the powdered glass?

MR. SPINDLER: Well, yes. Your powdered glass just wets the top of the sapphire. And the tube is coming down. You pre-heat that tube and by having that 900° temperature it seals right on to it. It's almost like a DuPont seal, if you can visualize that.

MR. GREINER: And what is the overall length of the element?

MR. SPINDLER: On the sapphire lamps, that was 10 inches. And the core inside was about an inch and a half. The bottom

winding—to overcome that, you just take about a roll and a quarter from the top—that's a 900 Centigrade, and from there on down, it's a 700. You see, the more winding you put on it, your resistance comes higher.

WILLIAM LEONARD: Mr. Jolley, I am interested in changing those joints to heavy tubing on your new joints. I am with Ohio State University and it is all right for the glassblowers to use those joints, but they have ovens available. But, at the University, the students do a lot of their own work in which they use hand torches. They do not have the ovens available. Therefore, the Ace Company has come out and said they will stay with the old type. I am afraid you are going to lose a lot of business because you have haircracks, which you do not have in annealing when they have hand torches.

MR. JOLLEY: I think that probably the next supplement to our catalog will have the old type available on a special order basis like our special apparatus. After we had made this change, we have received quite a few orders from companies or individuals that prefer the lighter wall tubing and with the bulge. It was one of the things we thought was going over a hundred per cent and it hasn't. So, I think we will have to go back in and make those available on a special basis. I do not think we'll have two types, one of the light and one of heavy wall, but we will go back and make them available.

ARTHUR D. MACK: I want to find out about EC coating. We have a problem I would like to get straightened out on EC coating for Vycor. I would like to know when it will be available.

MR. JOLLEY: At the present time, EC coating is not available. The coating and Vycor is such that all you can put it on as coating cracks. We are trying to develop an EC coating, which has a lower coefficient of expansion. When that will be, I have no idea.

MR. YEAGER: We are having trouble on having standardized vacuum systems on the McLeod gauges. We're also having a little trouble in some of our McLeod gauges and diffusion pumps.

They just don't seem to be the same calibration. We would like to find out if the Bureau of Standards or Corning is working on it or would start to work on it. You pick up several books to read and everybody has them a little bit different as far as the actual reading on the McLeod gauges. We would like to start putting out a paper on this if nobody else is doing it. We don't want to throw money into it if somebody has started it.

MR. JOLLEY: I don't know of anybody that's working on a standard like that. What I would suggest is that you work it out with the American Standards Association and ask the Bureau of American Standards. I would suggest that you contact the manufacturers of McLeod gauges. They will certainly know of any standardization program that is being done or is under way. That will give you a little idea as to whether any work is being done on it.

In Attendance

The following are on record as having attended the Symposium on the Art of Glassblowing held at Corning, New York on Thursday, Friday and Saturday, May 21, 22 and 23, 1959. The address following the name is the mailing address as listed on the Society files.

- Airey, AndrewSmith, Kline & French Labs., Philadelphia, Pa.
Alexander, J. AllenThe Atlantic Refining Co., Philadelphia, Pa.
Altosaar, H.Howard Smith Paper Co., Cornwall, Ont.,
Canada
Anderson, Elmer F.Ryan, Velluto & Anderson, Cambridge, Mass.
Anderson, Frederick F.Ciba Pharmaceutical Co., Summit, N. J.
Arrison, Frank C.Fischer & Porter Co., Hatboro, Pa.
Asmanes, CharlesUnion Carbide Nuclear Co., Oak Ridge, Tenn.
- Ball, William B.DuPont Electro Chemical Co., Niagara Falls,
N. Y.
Baranovic, MichaelGeneral Electric Co., Cleveland, Ohio
Barr, William E.Gulf Res. & Dev. Co., Pittsburgh, Pa.
Bate, WilliamIBM Corp., Kingston, N. Y.
Baum, JosephSterling Winthrop Research Inst., Rensselaer,
N. Y.
Bayard, Joseph F.The Fredericks Co., Bethayres, Pa.
Beck, Harry N.Beck Glassblowing, Malden, Mass.
Beck, HelmarAmersil Quartz Division, Hillside, N. J.
Bell, PhilipWright Patterson A.F.B., Dayton, Ohio
Belz, John F. Jr.Smith, Kline & French Labs., Philadelphia, Pa.
Bennett, Charles1026 N. Carey St., Baltimore 17, Md.
Betterly, DonFischer & Porter Co.
Bierman, DanPetrometer Corp., Long Island City, N. Y.
Blankenship, Homer Jr.Houston Glass Fabricating Co., Houston, Texas
Blaessig, HansBlaessig Glass Specialties, Rochester, N. Y.
Blasi, James T.M.I.T. Lincoln Lab., Lexington, Mass.
Bleeker, Albert J.General Electric Co.
Blomquist, Theodore V.Diamond Ord. Fuze Lab., Washington, D. C.
Bolan, Theodore W.R.D. 3, Mt. Horeb Rd., Warren Township, N. J.

- Boorn, E. L.Union Carbide Nuclear Co., Oak Ridge, Tenn.
- Bowen, Woodrow L.University of Michigan, Ann Arbor, Mich.
- Brandler, Frank J.Hoffman LaRoche, Nutley, N. J.
- Brangaccio, D. J.Bell Telephone Labs, Murray Hill, N. J.
- Brewin, Thomas A., Jr.Ryan, Velluto & Anderson, Cambridge, Mass.
- Brookover, George B.Kimble Glass Co., Toledo, Ohio
- Brown, FredWilliam Muller, Palisades Park, N. J.
- Bryant, Leroy M.U.S. Naval Rad. Def. Lab., San Francisco, Calif.
- Bullard, R. K.Corning Glass Works, Corning, N. Y.
- Burcar, Thomas H.U.S. Industrial Chem. Res. Labs., Cincinnati, Ohio
- Burt, Stewart W.The Dow Chemical Co., Williamsburg, Va.
-
- Campbell, C. E.Battelle Memorial Inst., Columbus, Ohio
- Campbell, William A.Warner Lambert Pharmaceutical Co., Morristown, N. J.
- Campbell, William W., Jr. ...Naval Research Lab., Washington, D. C.
- Cardinal, Allen H.General Electric Co., Syracuse, N. Y.
- Carter, Frank B.M.I.T Lincoln Lab., Lexington, Mass.
- Cassidy, C. J.Westinghouse Res. Labs., Pittsburgh, Pa.
- Castle, F. J.Department of Agriculture, Peoria, Ill.
- Clark, Wellman L.U.S. Naval Ord. Lab., Silver Spring, Md.
- Clements, Edwin H.Wyandotte Chem. Co., Wyandotte, Mich.
- Chappell, R. HaroldUniversity of Toronto, Toronto, Canada
- Christie, H. L.Atomic Energy of Canada, Ottawa, Ont., Canada
- Collins, RonaldPrecision Glass Co., Toronto, Canada
- Colson, August F.Ohio State University, Columbus, Ohio
- Copeland, Walter P.Gulf. Res. & Dev. Co., Philadelphia, Pa.
- Cosgrove, George D.Sandia Corp., Albuquerque, N. M.
- Cragg, RichardNational Aniline, Buffalo, N. Y.
-
- Daly, John M.Schutte & Koerting Co.
- Danko, R. L.Columbia Southern Chem. Corp., Barberton, Ohio
- Dawes, E. F.28 Mathew St., Sunshine, Victoria, Australia

Day, GailI.T.T. Laba., Ft. Wayne, Ind.
 DeCesare, James V.Shell Development Co., Houston, Texas
 Deery, ArthurColumbia University, New York City
 Deery, Edward D.Heights Lab. Glass, New York City
 DeFlorio, William J.Schlumberger Well Surveying Corp., Ridge-
 field, Conn.
 DeLeonibus, EnricoBureau of Standards, Washington, D. C.
 DeMaria, Vincent C.Glass Prods. Dev. Lab., Seaford, L. I., N. Y.
 DePhillip, Lewis J.IBM Corp., Endicott, N. Y.
 DeWolff, WilliamThe Upjohn Co., Kalamazoo, Mich.
 Dixon, Rufus T.Purdue University, W. Lafayette, Ind.
 Dolenga, ArthurGeneral Motors Research Labs., Detroit, Mich.
 Domizio, ArthurGeigy Chemical Co., Yonkers, N. Y.
 Donahue, John T.United Aircraft Corp., East Hartford, Conn.
 Doody, Thomas J.Argonne National Lab., Lemont, Ill.
 Doucette, Wilfred F.M.I.T., Cambridge, Mass.
 Dreschel, Helmut E.National Scientific Co., Churchville, Pa.
 Dronard, George F.U.S.A.S.R.D.L., Ft. Monmouth, N. J.
 DuPont, FosterR.D. 3, Amsterdam, N. Y.
 Dusek, Leo F.B. F. Goodrich Co., Brecksville, Ohio
 Dymock, GeorgeEmerald Glass Co., Toronto, Canada

Earley, K. D.Sylvania Elec. Prods. Co., Bayside, L. I., N. Y.
 Evans, B.Nat'l. Res. Council, Ottawa, Canada

Faust, GeraldBartol Research Found., Swarthmore, Pa.
 Fuller, John H.Amherst College, Amherst, Mass.

Garbutt, H.Westinghouse Electric Corp., Baltimore, Md.
 Gilhooley, William A., Jr. ..General Electric Res. Labs., Schenectady, N. Y.
 Glover, John A.Sinclair Res. Labs., Inc., Harvey, Ill.
 Glover, Robert G.National Lead of Ohio, Cincinnati, Ohio
 Good, GordonMonsanto Chemical Co., Dayton, Ohio
 Goodman, W. D.Olin Mathieson Chemical Corp., Niagara Falls,
 N. Y.
 Graves, Bruce B.Purdue University, W. Lafayette, Ind.
 Greeley, Edward J.Union Carbide Nuclear Co., Oak Ridge, Tenn.

- Green, Harry F.University of California Radiation Lab., Livermore, Calif.
- Greene, Walter A.General Electric Res. Labs., Schenectady, N. Y.
- Gregory, StephenThe Linde Co., Niagara Falls, N. Y.
- Greiner, AlGeneral Electric Co., E. Cleveland, Ohio
- Griffith, Thomas, Jr.P. R. Mallory & Co., Inc., Indianapolis, Ind.
- Gross, RussellVacuum Ceramics, Inc., Cary, Ill.
- Gutilla, Sam J.Delmar Scientific Labs., Chicago, Ill.
-
- Haak, Werner H.Purdue University, W. Lafayette, Ind.
- Hagedorn, James A.Podbielniak, Inc., Chicago, Ill.
- Hapstack, Richard J.Allied Chemical Corp., Glenolden, Pa.
- Harris, George E.Washington State College, Pullman, Wash.
- Hause, CorneliusFischer & Porter Co., Hatboro, Pa.
- Hawkins, Anthony J.University of British Columbia, Vancouver 8, B.C., Canada
- Hayes, Frank L.Universal Oil Prods. Co., Des Plaines, Ill.
- Heine, Alfred J., Jr.Naval Res. Lab., Washington, D. C.
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- Hensler, N. A.Westinghouse Res. Labs., Pittsburgh, Pa.
- Henson, T. A.Duke University, Durham, N. C.
- Hepler, Carl F.Gulf Res. & Dev. Co., Pittsburgh, Pa.
- Hernandez, PeterDept. of Mines & Tech. Surveys, Ottawa, Ont., Canada
- Hewitt, John M.General Electric Co., Syracuse, N. Y.
- Heyn, H. A.Eck & Krebs, Inc., Long Island City, N. Y.
- Heyn, Hilmar M.Westinghouse Electronics Div., Elmira, N. Y.
- Highhouse, Elmer A.U.S. Army, Fort Detrick, Frederick, Md.
- Hill, PaulFischer & Porter Co, Hatboro, Pa.
- Hines, OlinI. T. & T. Labs., Ft. Wayne, Ind.
- Hinman, Leon H.Cornell University, Ithaca, N. Y.
- Horn, FrancisAtlantic Refining Co., Philadelphia, Pa.
- Hunt, A. S.University of Toronto, Toronto, Ont., Canada
- Hunter, Harry, Jr.Wyeth Labs., Radnor, Pa.
- Huth, Harry J.Washington University, School of Medicine, St. Louis, Mo.

Ildsgaard, Arne, Jr.Kontes Glass Co., Vineland, N. J.

Johnson, W. L.W. L. Johnson Co., Louisville, Ky.

Johnson, Walter R., Jr.Franklin Institute, Philadelphia, Pa.

Jones, AlfredStandard Oil Co., Whiting, Ind.

Jones, Charles H.Walter Reed Army Med. Center, Washington,
D. C.

Jones, WayneIowa State College, Ames, Iowa

Subera, Andre M.Mellon Institute, Pittsburgh, Pa.

Kay, PeterBorg-Warner Corp., Des Plaines, Ill.

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Killich, George A.Superior Glass Apparatus, Ann Arbor, Mich.

Kingsbury, Owen J., Jr.Union Carbide Nuclear Co., Oak Ridge, Tenn.

Klebanoff, JosephNaval Res. Lab., Washington, D. C.

Klein, Edwin I.Nat'l Bureau of Standards, Washington, D. C.

Kleinert, RichardBendix Aviation Corp. Res. Lab., Detroit,
Mich.

Klott, LewisLewis Klott Co., Millville, N. J.

Knisely, SamuelSocony Mobil Oil Co., Paulsboro, N. J.

Koch, Edward W.Bell Tel. Labs., Murray Hill, N. J.

Kocsi, AndrewAvco Corp., Wilmington, Mass.

Kozimor, FrancisG. E. Co., K.A.P.L., Schenectady, N. Y.

Kosloff, Peter J.General Foods Res. Lab., Tarrytown, N. Y.

Krotine, Frank J.General Electric Co., Cleveland, Ohio

Kushner, JackHellige, Inc., Long Island, N. Y.

Lange, HelmutPhilips Electronics, Toronto, Ont., Canada

Lawrence, James B.Bethlehem Apparatus Co., Hellertown, Pa.

Lechner, Alois A.Ace Glass, Inc., Vineland, N. J.

Lenzi, David J.U.S. Army Med. Res. Lab., Ft. Knox, Ky.

Leonard, William E.Ohio State University, Columbus, Ohio

Lewis, Chester J.Texaco, Inc., Beacon, N. Y.

Lillie, DonGeorgia Inst. of Tech., Atlanta, Ga.

Lipson, HarryEvans Signal Lab., Ft. Monmouth, N. J.

Lister, EdwardCanadian Industries, Ltd., McMasterville, Que.,
Canada

Logsdon, MaxonUnited Aircraft Corp. Res. Dept., East Hart-
ford, Conn.

Love, R. W.General Electric Co., Cleveland, Ohio
Lynsky, Francis M.Macalaster Bicknell of New Haven, New
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McKinney, Robert W.Olin Mathieson Co., Niagara Falls, N. Y.
Mack, Arthur D.Navy Med. Center, Bethesda, Md.
Maiolatesi, ElmoU.S. Army Chem. Center, Edgewood, Md.
Maiolatesi, F.Nat'l Bur. of Standards, Washington, D. C.
Malcolm, JohnSpace Tech. Labs.
Malisjewski, ArnoldGeneral Electric Co.
Malloy, F. J.U.S. Steel Corp., Monroeville, Pa.
Marx, Leonard M.M. W. Kellogg Co., Jersey City, N. J.
Mason, AustinU. S. D. A., New Orleans, La.
Mathieu, Robert J.Corning Glass Works, Corning, N. Y.
Meldrum, W. H.Eldorado Mining & Refining Co., Port Hope,
Ont., Canada
Merriam, Donald R.Proctor & Gamble Co., Cincinnati, Ohio
Messick, G. E.Hercules Powder Co., Wilmington, Del.
Meyer, FrankGarretson Ave., Islip, N. Y.
Meyer, IrvinBrookhaven Nat'l. Labs., Upton, N. Y.
Mezynski, StanleyNew York Naval Shipyard, Brooklyn, N. Y.
Miller, Robert F.Eli Lilly Co., Indianapolis, Ind.
Miller, TonyEastman Kodak, Rochester, N. Y.
Monostori, Laszlo J.National Bur. of Standards, Washington, D. C.
Morris, James F.Northwestern University, Evanston, Ill.
Mugge, RobertPurdue University, W. Lafayette, Ind.
Muller, WilliamWilliam Muller, Palisades Park, N. J.
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Nelson, James, Jr.General Electric Res. Lab., Schenectady, N. Y.
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Nutt, Howard M.The Linde Co., Niagara Falls, N. Y.
Nyman, Carl L.General Electric Co., Schenectady, N. Y.

- Omiecinski, SigmundArgonne National Lab., Lemont, Ill.
 Ortegren, Victor H.U. S. D. A. Western Reg. Lab., Albany, Calif.
 Osty, Sigmund J.Radiation Counter Lab., Skokie, Ill.
- Pachuki, Bruno S.Argonne National Lab., Lemont, Ill.
 Pahl, Billie E.Dow Chemical Co., Midland, Mich.
 Palmer, RobertHooker Chem. Co., Niagara Falls, N. Y.
 Panczner, W. J.G. E. Xray, Clearwater, Fla.
 Pararas, John L.U.S. Nat'l. Bur. of Standards, Washington,
 D. C.
- Phillips, Leyland P.Texaco Res. Center, Beacon, N. Y.
 Phillips, Russell C.Stanford Res. Inst., Palo Alto, Calif.
 Poole, Richard W.Union Carbide Nuclear Co., Oak Ridge, Tenn.
 Putnam, CarlUnited Aircraft Corp., E. Hartford, Conn
 Pye, A. W.National Res. Council, Ottawa, Ont., Canada
- Reeves, Arthur G.Macalaster Bicknell Co., Fitzwilliam, N. H.
 Reif, Joseph J.W. R. & W. Scientific Glass Co., Inc., Port
 Washington, N. Y.
- Reinker, G. E.General Electric Co, Cleveland, Ohio
 Reese, F. J.Hercules Powder Co., Wilmington, Del.
 Ret, GeneAce Glass, Inc., Vineland, N. J.
 Reynolds, Kevin W.IBM Corp.
- Richards, HarryMonsanto Chem. Co., St. Louis, Mo.
 Ritzer, J. E., Sr.Dow Chem. Co., Midland, Mich.
 Robinson, M. H.Thermal American Fused Quartz Co., Dover,
 N. J.
- Roensch, Arno P.Los Alamos Scientific Lab., Los Alamos, N. M.
 Roman, Paul W.Brookhaven National Labs., Upton, N. Y.
 Roth, Francis M.U.S. Steel Applied Res. Labs., Monroeville, Pa.
 Russell, Robert L.Proctor & Gamble Co., Cincinnati, Ohio
 Russler, Ralph H.Westinghouse Elec. Corp., Baltimore, Md.
- Sales, William A.H. S. Martin & Co., Evanston, Ill.
 Sampson, D. E.University of North Carolina, Chapel Hill,
 N. C.

Schaefer, Howard A.Anchor Hocking Glass Co., Lancaster, Ohio
 Schaefer, Walter A.Esso Res. & Eng. Co., Linden, N. J.
 Schelhammer, H.Machlett Labs., Inc., Springdale, Conn.
 Schilling, William F.Kimble Glass Co., Vineland, N. J.
 Schipmann, Robert H.Food Machinery & Chem. Corp., Princeton,
 N. J.
 Schneider, W. OttoBrookhaven National Labs., Upton, N. Y.
 Schultz, ElwoodUniversity of North Carolina, Chapel Hill,
 N. C.
 Schumann, KarlColumbia University, New York, N. Y.
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 Searle, R. H.E. I. duPont Co. SRP, Aiken, S. C.
 Serianni, Louis J.Precision Glass Prods. Co., Philadelphia, Pa.
 Shearer, G. PaulUnion Carbide Metals Co., Niagara Falls, N. Y.
 Shedd, Fred J.Eastman Kodak, Rochester, N. Y.
 Shipley, DonNat'l. Carbon Res. Lab. Parma, Ohio
 Simpson, John C.Glass Instruments, Inc., Pasadena, Calif.
 Sites, George A.Houdry Process Corp., Linwood, Pa.
 Slominski, Harry J.Union Carbide Chem. Co., Niagara Falls, N. Y.
 Smith, Herbert G.California Res. Corp., Richmond, Calif.
 Smith, HoweFischer & Porter Co., Hatboro, Pa.
 Smith, Thomas L.Union Carbide Chem. Co., Niagara Falls, N. Y.
 Smith, W. C.Corning Glass Works, Corning, N. Y.
 Snyder, DaleDow Chemical Co., Midland, Mich.
 Souza, Raymond L.Watertown Arsenal Lab., Watertown, Mass.
 Spessard, Lewis C.Martin of Baltimore, Baltimore, Md.

 Spindler, George P.Bell Tel. Res. Lab., Murray Hill, N. J.
 Squeo, Guy J.Standard Oil Co., Whiting, Ind.
 Staiger, WilliamStaiger Inst. Co., Westbury, L. I., N. Y.
 Stanley, RussellStanley Scientific Glass Co., Hatboro, Pa.
 Steelman, Joseph A.Fischer & Porter Co., Hatboro, Pa.
 Strnad, A. R.Bell Tel. Labs., Murray Hill, N. J.
 Sullivan, FrankM.I.T. Lincoln Lab., Lexington, Mass.
 Summeril, FrankFischer & Porter, Hatboro, Pa.
 Suprenard, Arthur T.Geophysics Corp. of Amer., Boston, Mass.
 Sylvester, W. J.Washington University, St. Louis, Mo.

 Taylor, Roger K.W. R. Grace & Co., Baltimore, Md.
 Teske, C. W.Naval Ord. Lab., Silver Spring, Md.
 Thornton, Helen (Mrs.)Atlantic Refining Co., Philadelphia, Pa.

Tobin, Robert B.Mellon Institute, Pittsburgh, Pa.
 Townsley, Kenneth N.General Motors Corp., Detroit, Mich.
 Tozer, William H.Allied Chemical Corp., Glenolden, Pa.
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 Trudell, JohnBrooklyn Poyltech. Inst., Brooklyn, N. Y.
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 Voyer, Lucian A.L. A. Voyer Co., Boston, Mass.
 Voyzey, Charles A.Olin Mathieson Corp., Niagara Falls, N. Y.
 Wagner, MichaelW. R. & W. Scientific Glass Co., Port Wash-
 ington, N. Y.
 Walrod, Alfred H.Varian Assoc., Palo Alto, Calif.
 Walther, Karl H.Brookhaven National Lab., Upton, N. Y.
 Watt, G. G.General Electric Co., Cleveland, Ohio
 Weir, H. E.Atlas Powder Co., Wilmington 3, Del.
 Weiss, GuntherPrecision Glass Co., Ltd., Toronto, Ont.,
 Canada
 Wells, TheodoreCorning Glass Works, Corning, N. Y.
 West, JosephWest Scientific Glass Co., Niagara Falls, N. Y.
 Wheeler, Edgar L.University of California, Los Angeles, Calif.
 Wildvank, ArthurSpace Tech. Labs.
 Willey, MartinGeneral Electric Res. Lab., Schenectady, N. Y.
 Wisniewski, TheodoreHeights Lab. Glass, Inc., New York, N. Y.
 Wolfe, Alfred M.Monsanto Chemical Co., Indian Orchard, Mass.
 Woltz, Charles R.Westinghouse Elec. Corp., Baltimore, Md.
 Woodall, E. L.General Electric Co., Cleveland, Ohio
 Wrbican, Sam.Alcoa, New Kensington, Pa.
 Wurster, KurtK. W. Thermometer Co., Whippany, N. J.
 Yeager, Paul V.Convair, San Diego, Calif.
 Zelt, Raymond J.Lansdale Tube Co., Lansdale, Pa.
 Zurek, Tony A.Chemstrand Corp., Decatur, Ala.
 Zwirblis, HenryHellige Corp., Garden City, N. Y.
 Zwirblis, StanleyHellige Corp., Garden City, N. Y.

