# Optical Glass at the National Bureau of Standards

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United States Department of Commerce
National Bureau of Standards

UNITED STATES DEPARTMENT OF COMMERCE • Charles Sawyer, *Secretary*
NATIONAL BUREAU OF STANDARDS • E. U. Condon, *Director*

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*by* Francis W. Glaze and Clarence H. Hahner

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## Preface

The purpose of this Circular is to give a general description of the plant at the National Bureau of Standards for the production of optical glass. Details of production with which an experienced glass manufacturer is familiar have been largely omitted. Sufficient information is given, however, to enable one experienced in the trade to enter the optical glass field with some hope of success, especially if some of the equipment is duplicated.

E. U. Condon, *Director*.

## Contents

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**Abstract**

A description is given of the procedures used at the National Bureau of Standards for the production of optical glass. This includes compositions, melting schedules, molding procedures, and annealing schedules for many types of optical glass. During World War II, the types of optical glass regularly produced increased from 5 to 28. Also, in this period, the quality of the glass and the appearance of the molded blanks were improved.

## I. Optical Glass in the United States

The production of optical glass in the United States is a modern industry. As nearly as can be determined, the first optical glass was successfully produced about 1893 by MacBeth & Co., Pittsburgh, Pa. [1].[1] Beginning in 1896, and continuing for approximately 6 years [2], the Manhattan Optical Co. operated a small glass plant at Cresskill, N. J. This company then combined with the Gundlach Manufacturing Co., Fairport, N. Y., and ceased operation. In1912 the Bausch & Lomb Optical Co., Rochester, N. Y., started production that has continued to the present day.

The National Bureau of Standards entered the optical glass field in July 1914, at its Pittsburgh laboratory, then under the direction of P. H. Bates. To indicate its growth, table 1 gives the annual production of optical glass at the Bureau for a number of years within a 27-year period. Four additional manufacturers produced optical glass during World War II because of the unusual demand.

TABLE 1. *Production of optical glass at the National Bureau of Standards* [1]

| Fiscal year [2] | Glass delivered | Fiscal year | Glass delivered |
|---|---|---|---|
| | *lb* | | *lb* |
| 1921 | 1,145 | 1941 | 36,785 |
| 1923 | 2,200 | 1942 | 136,550 |
| 1931 | 3,001 | 1943 | 236,558 |
| 1934 | 5,552 | 1944 | 230,012 |
| 1937 | 7,530 | 1945 | 195,777 |
| 1939 | 8,422 | 1946 | 57,495 |
| 1940 | 7,297 | 1947 | 16,040 |

[1] Shipments of optical glass of good quality started in November, 1917, and continued from then on, being 3,021 lb. for October 1918 (Report of the Secretary of Commerce to the President for 1919).
[2] Selected years are given merely to indicate trend of production.

## II. Production of Optical Glass

Optical glass is divided into three general types: transfer, rolled, and ophthalmic. This paper will deal only with transfer glass as that is the type produced at the Bureau. Transfer glass is processed by three methods, namely, stick up, or punty; paddling; and slumping.

### 1. Properties of Optical Glass

The most important requirement for glass of optical quality, and one that is extremely difficult to meet, is that the glass in each lens or prism element be of uniform quality throughout, and that its optical constants agree closely with those of the standard type glass for which the element was designed. To manufacture on a large scale a series of different glasses to this degree of perfection requires close attention to detail.

Wright [3] made an excellent list of the properties of good optical glass. They are

I. Homogeneity.
  1. Uniformity of chemical composition.
    a. Freedom from striae.
    b. Freedom from bubbles.
    c. Freedom from inclusions, stones, and crystallites.
    d. Freedom from cloudiness.
  2. Uniformity in physical state.
    a. Freedom from strains.

II. Definite refractive indices for different wavelengths.
  1. Refractivity.
  2. Dispersivity and dispersion ratios.

[1] Figures in brackets indicate the literature references at the end of this paper.

III. Freedom from color.
IV. High degree of transparency.
V. High degree of chemical and physical stability.
1. Resistance to action of weather and certain chemical agents.
2. Toughness and hardness.

## 2. Pots

Optical glass is made at the National Bureau of Standards in clay pots of approximately 7-cu ft capacity, which are used only once. Pots may fail because of attack of the glass on the pot body or from cracking induced by thermal shock. One of the principal compromises in optical glass production is the selection of the most suitable pot body. The denser, or less porous, a pot is, the less it is attacked by the molten glass or batch materials, but the denser the pot, the more sensitive it is to thermal shock. Any particular body is, therefore, somewhat less than ideal both as to corrosion resistance and resistance to thermal shock. All of the optical glasses except borosilicate crown 517/645 are melted in pots containing a lining of low porosity. This lining is sprayed with an additional thin coating of slip to seal incipient and invisible cracks. In this way, a pot is obtained with an inner layer of low-porosity material in contact with the glass, but with the greater part of the pot having a relatively high porosity that will resist thermal shock. These pots and the clay thimbles used for stirring are described in a recent paper by Heindl, Massengale, and Cossette [4].

Dried pots are burned in pot arches immediately before use and are transferred while hot to the melting furnace. Figure 1 shows a pot and stirring thimble in the arch. The pot is moved from the arch to the melting furnace by a carriage equipped with a pair of tongs to hold the pot. These tongs are controlled from the rear end of the carriage as is also a counterweight that can be moved along the carriage to balance the weight of the empty pot or of one filled with glass. Each pot arch is heated by gas from the rear by three low-pressure air burners which make use of a premixture of gas and air. The flame is deflected upwards by means of a brick wall immediately in back of the pot, and the products of combustion are exhausted through flues in the floor. The burning requires from 53 to 109 hr, depending on

FIGURE 1. *Optical glass pot and stirring thimble in a pot arch preparatory to burning.*

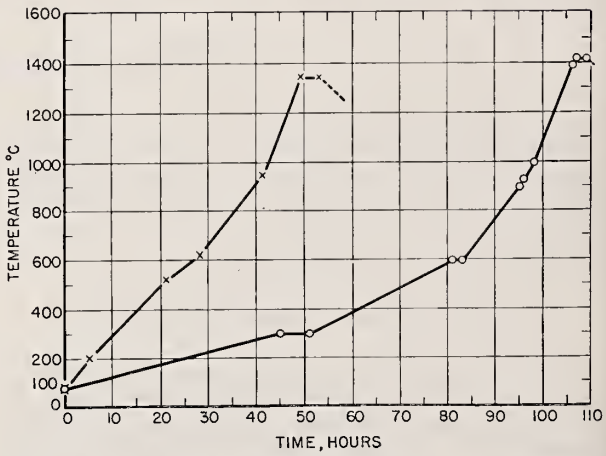


FIGURE 2. *Burning schedules for pots used at the National Bureau of Standards.*
×, High-porosity pot; ○, low-porosity pot.

the type of the pot body, the longer schedule applying to the body of lowest porosity. Burning schedules for the two types of pots are given in figure 2.

## 3. Batch materials and batch composition

Batch materials of the highest commercial purity are necessary as ingredients for the production of optical glass. Since decolorizing agents cannot be used, the amounts of coloring oxides in the materials must be limited to traces, especially in those making up the larger portions of the batch. The specifications for some of the batch materials are given in table 2. Batch materials should be of approximately uniform grain size so that they will mix easily with each other and, when incorporated in a batch, will not segregate.

Each barrel of potash is checked for its alkalinity, calculated as $K_2CO_3$, prior to use. Materials such as soda ash and borax, the moisture content of which varies with storage, are mixed in 2,000-lb lots and analyzed for their $Na_2CO_3$ and borax ($Na_2B_4O_7 \cdot 10H_2O$) content shortly before use.

A scale with a printweigh attachment, which makes a permanent record of the weights of the materials added, is used for weighing. The batch is transferred from the scale to a concrete-type mixer, or to a drum-type mixer, and mixed for 15 to 20 min. From the mixer the batch is dumped into a cart for use by the melting crew.

Table 3 gives batch compositions on the basis of 100-percent purity. However, the materials used are not 100-percent pure; consequently the weights must be adjusted for variations in composition as determined by chemical analysis.

## 4. Melting

The melting furnaces are of the single-pot regenerative type. There are three burners on each side of the furnace. Bag walls, extending slightly above the top of the pot, separate the melting compartment from the regenerator ports and direct the flames toward the crown. Direction of flow of the gases is reversed at half-hour intervals. The siege on which the pot rests is leveled with anthracite breeze or ash immediately before the pot is set in the melting furnace.

The filling of the pot is accomplished by means of a long-handled scoop. When the furnace temperature reaches 1,250° C (thermocouple reading), 100 to 150 lb of cullet are charged into the pot. The furnace is brought up to the melting temperature (1,350° to 1,450° C, depending on the type of glass) within ½ to 1 hr, after which the pot is filled three-quarters full of batch. One hour later the pot is filled with batch, and additional fillings are made every half hour thereafter until the pot is full of glass. The time required to fill a pot varies from 5 to 10 hr, subject to the ease with which the batch melts.

One to three hours after the last fill, depending

TABLE 2. *Specifications for batch materials used in the production of optical glass*

| Constituent | Batch materials [1] | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Sand | Flint, powdered | Boric acid, borax, saltpeter, sodium nitrate and potash (83 to 85%) | Soda ash | Litharge | Barium carbonate and hydrate | Zinc oxide | Potash (99%) | Calcium carbonate | Barium nitrate | Lithium carbonate | Strontium carbonate | Titanium oxide | Beryllium oxide | Zirconium oxide | Arsenic and antimony oxide | Alumina hydrate |
| | % | % | % | % | % | % | % | % | % | % | % | % | % | % | % | % | % |
| $Fe_2O_3$ [2] | <0.02 | <0.025 | <0.002 | <0.003 | <0.005 | <0.005 | <0.005 | <0.0025 | <0.010 | <0.005 | <0.010 | <0.030 | <0.005 | <0.020 | <0.030 | <0.025 | <0.010 |
| Cl | | | <.10 | <.25 | <.10 | <.10 | | <.125 | <.10 | <.10 | <.10 | | | | | | |
| $SO_3$ | | | <.10 | <.10 | <.10 | <.25 | <.35 | <.125 | | <.10 | <.10 | | | | | | |
| $SiO_2$ | >99.25 | >99.0 | | | | | | | | | | | | | | | |
| $Al_2O_3$ | <0.20 | <0.25 | | | | | | | | | | | | | | | >64.3 |
| $CuO$ [2] | | | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.002 | <.003 | | |
| $Cr_2O_3$ [2] | | | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.002 | <.003 | | |
| $MnO$ [2] | | | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.002 | <.002 | <.003 | <.025 | <.005 |
| $CoO$ [2] | | | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.002 | <.003 | | |
| $NiO$ [2] | | | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.001 | <.002 | <.003 | | |
| MgO | | | | | | | | | <.50 | | | | | | | | |
| $CaCO_3$ | | | | | | | | | | | <.50 | | | | | | |
| $K_2CO_3$ / $Na_2CO_3$ | | | | | | | | | | | <2.5 | | | | | | |
| $TiO_2$ | | | | | | | | | | | | | >98.5 | | | | |
| BeO | | | | | | | | | | | | | | >99.0 | | | |
| $ZrO_2$ | | | | | | | | | | | | | | | >98.5 | | |
| BaO / CaO / MgO | | | | | | | | | | | | <1.0 | | | | | |

[1] Materials of the specified purity are readily available.
[2] Coloring oxides.

TABLE 3. *Batch compositions for optical glasses* [1]

| Batch material | Calculated as— | Types of glasses [2] (weight of ingredient in pounds required to make 1 pot of glass) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | BSC 511/635 | BSC 517/645 | BSC 536/644 | LC 512/605 | LC 523/586 | LC 528/580 | BaC 541/599 | BaC 574/577 | BaC 611/588 | BaC 617/550 | BaC 620/600 | BF 584/460 | BF 588/534 |
| Sand or powdered flint | $SiO_2$ | 670.8 | 530.4 | 760.9 | 701.0 | 744.1 | 812.8 | 602.7 | 506.2 | [3] 429.0 | 466.3 | [3] 453.6 | [3] 656.4 | [3] 581.7 |
| Litharge | PbO | | | | | | | | | 2.2 | 17.5 | | 247.5 | 127.0 |
| Barium carbonate | $BaCO_3$ | | | | | | | 262.5 | 331.1 | 272.7 | 542.8 | 320.2 | 226.9 | 423.3 |
| Boric acid | $B(OH)_3$ | | 62.1 | 277.0 | | 28.2 | 31.7 | | 60.8 | 212.9 | 97.7 | 241.9 | | 158.0 |
| Borax | $Na_2B_4O_7 \cdot 10H_2O$ | 227.0 | 178.2 | | 133.5 | | | 95.4 | 96.5 | | | | | 62.5 |
| Soda ash | $Na_2CO_3$ | 67.0 | 53.1 | | 207.9 | 232.4 | 260.9 | 29.6 | | | 4.3 | | 35.1 | |
| Saltpeter | $KNO_3$ | 65.0 | 59.0 | 69.9 | 45.4 | 56.9 | 63.9 | 50.0 | 48.7 | | 10.7 | | 173.7 | 75.0 |
| Zinc oxide | ZnO | | 9.6 | | | | | 42.0 | 84.0 | | 70.0 | | 102.4 | |
| Arsenic oxide | $As_2O_3$ | 2.0 | 4.0 | 5.0 | | | | 3.1 | 3.4 | 4.5 | 3.8 | 3.6 | 6.6 | 6.4 |
| Antimony oxide | $Sb_2O_3$ | | | | 11.7 | 12.7 | 14.3 | | 7.8 | 2.2 | 8.8 | 2.4 | | |
| Potash | $K_2CO_3$ | 165.9 | 100.6 | 61.3 | 40.5 | | | 120.7 | 81.8 | | | | 40.5 | 73.6 |
| Beryllia | BeO | | | 24.0 | | | | | | | | 27.6 | | |
| Strontium carbonate | $SrCO_3$ | | | 170.8 | | | | | | | | | | |
| Lithium carbonate | $Li_2CO_3$ | | | 148.5 | | | | | | | | | | |
| Calcium carbonate | $CaCO_3$ | | | | 38.3 | 177.8 | 240.0 | | | 89.9 | | 77.1 | | |
| Salt | NaCl | | | | | 15.8 | 17.7 | | | | | | | |
| Salt cake | $Na_2SO_4$ | | | | | 9.4 | 10.6 | | | | | | | |
| Alumina | $Al_2O_3$ | | | | | | | | 33.6 | 32.5 | 61.3 | | | |
| Barium hydrate | $Ba(OH)_2 \cdot 8H_2O$ | | | | | | | | 131.8 | 436.0 | 162.6 | 428.4 | | |
| Barium nitrate | $Ba(NO_3)_2$ | | | | | | | | | 94.7 | 100.8 | 125.0 | | |
| Zirconia | $ZrO_2$ | | | | | | | | | | | 3.0 | | 19.1 |
| Cullet [4] | | 100.0 | 300.0 | None | 100.0 | 100.0 | None | 300.0 | 300.0 | 300.0 | 300.0 | 400.0 | 200.0 | 200.0 |

| Batch material | Calculated as— | Types of glasses [2] (weight of ingredient in pounds required to make 1 pot of glass) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | BF 604/435 | CF 529/516 | F 572/425 | F 579/410 | F 605/381 | F 617/366 | F 620/362 | F 649/338 | F 666/324 | F 672/322 | F 689/309 | F 720/293 | F 754/277 |
| Sand or powdered flint | $SiO_2$ | [3] 603.2 | [3] 717.3 | [3] 754.9 | [3] 663.8 | [3] 725.9 | [3] 570.0 | [3] 595.1 | [3] 615.9 | [3] 609.2 | [3] 713.9 | [3] 625.3 | [3] 607.0 | [3] 561.6 |
| Litharge | PbO | 307.6 | 109.7 | 434.3 | 443.8 | 628.3 | 540.0 | 589.9 | 763.9 | 843.2 | 1019.4 | 981.9 | 1110.7 | 1191.6 |
| Barium carbonate | $BaCO_3$ | 231.0 | 2.7 | 17.6 | 9.7 | | | | | | | | | |
| Boric acid | $B(OH)_3$ | | | | | | | | | | | | | |
| Borax | $Na_2B_4O_7 \cdot 10H_2O$ | | | | | | | | | | | | | |
| Soda ash | $Na_2CO_3$ | | 246.6 | 117.1 | 8.6 | 49.5 | 98.3 | 66.9 | 17.9 | | | | | |
| Saltpeter | $KNO_3$ | 252.2 | 50.0 | 75.0 | 72.1 | 70.0 | 61.0 | 63.0 | 140.0 | 125.7 | 217.3 | 166.9 | 122.3 | 88.9 |
| Zinc oxide | ZnO | 106.9 | 39.1 | | | | | | | | | | | |
| Arsenic oxide | $As_2O_3$ | 5.3 | 2.0 | 4.1 | 3.8 | 7.6 | 6.3 | 6.5 | 7.5 | 4.7 | 5.5 | 5.1 | 5.3 | 5.4 |
| Antimony oxide | $Sb_2O_3$ | | 20.0 | | 6.3 | | | | | | | | | |
| Potash | $K_2CO_3$ | | 56.2 | 87.4 | 126.8 | 149.1 | 70.2 | 66.1 | 46.9 | 50.6 | | | | |
| Beryllia | BeO | | | | | | | | | | | | | |
| Strontium carbonate | $SrCO_3$ | | | | | | | | | | | | | |
| Lithium carbonate | $Li_2CO_3$ | | | | | | | | | | | | | |
| Calcium carbonate | $CaCO_3$ | | | | | | | | | | | | | |
| Salt | NaCl | | | | | | | | | | | | | |
| Salt cake | $Na_2SO_4$ | | | | | | | | | | | | | |
| Alumina | $Al_2O_3$ | | | | | | | | | | | | | |
| Barium hydrate | $Ba(OH)_2 \cdot 8H_2O$ | | | | | | | | | | | | | |
| Barium nitrate | $Ba(NO_3)_2$ | | | | | | | | | | | | | |
| Zirconia | $ZrO_2$ | | | | | | | | | | | | | |
| Cullet [4] | | 200.0 | 165.0 | None | 300.0 | 100.0 | 300.0 | 300.0 | 300.0 | 300.0 | 100.0 | 300.0 | 300.0 | 300.0 |

[1] Compositions for BaC 5725/574 and BaC 6109/572 are omitted; BaC 574/577 and BaC 611/588 have practically replaced them.

[2] The letter symbols for the various types of optical glass are BSC, borosilicate crown; LC, light crown; BaC, barium crown; BF, barium flint; CF, crown flint; and F, flint. The number designations following the letter symbols, taking BSC 511/635 as an example, mean a glass of index of refraction of 1.511 and a nu-value of 63.5.

[3] Powdered flint is the source of silica; for all other glasses sand is used.

[4] The amount of cullet may be varied; "none" meaning no cullet of this glass available.

on how badly the glass foams, the stirring of the glass by machine can begin. This is accomplished by means of a refractory thimble, made of the same materials as the pot. After the stirring machine is set in place and the water-cooled rod with stirring thimble connected to the machine, the thimble is centered in the pot. The machine is raised until the end of the thimble is ½ in. above the inside bottom of the pot. The rod is then set to the diameter of stir desired, the motor started, and the speed adjusted by means of a rheostat. The water-cooled rod is connected to a horizontal, motor-driven drum and works over a pulley as a fulcrum (fig. 3). Such a device does not give a true circle as the length of the arm is not constant even when using the so-called circular stir. The type of stir used is one of continually increasing and then decreasing diameter resulting in a curtate epitrochoid type of motion as shown in figure 4. The stirring machine has a cam that, when engaged, will automatically raise and lower the pulley which acts as a fulcrum for the stirring rod. By this means, a vertical motion can be superimposed on the horizontal motion. This vertical motion is used in the melting of dense flint glasses to replace "blocking." "Blocking" is the process

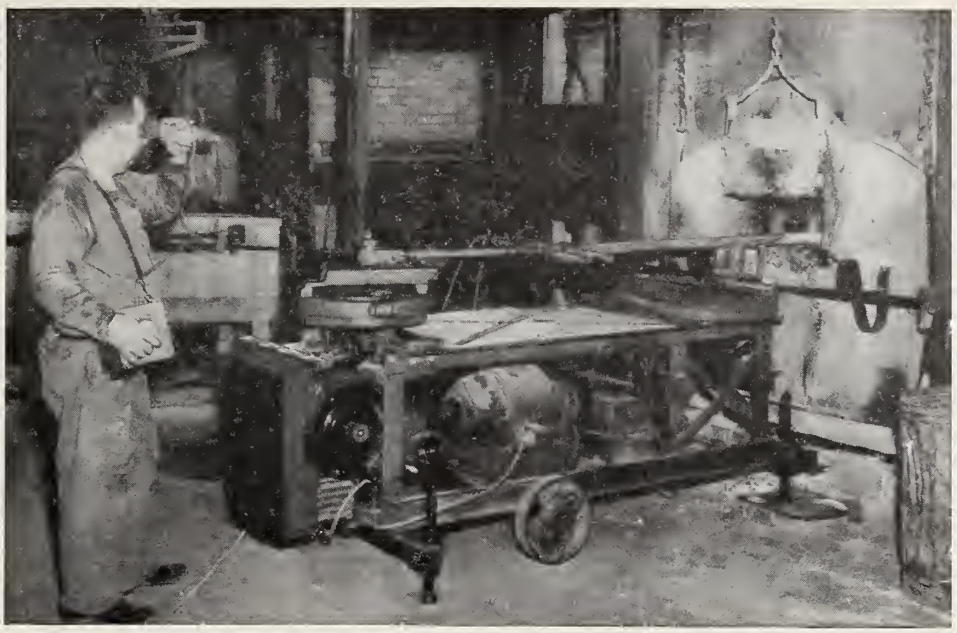

FIGURE 3. *Pot of optical glass is being stirred; at the same time, the furnaceman is determining the temperature of the glass with an optical pyrometer.*

of stirring and fining a melt by the introduction of a highly volatile substance, such as arsenious oxide or ammonium nitrate, which produces a sudden evolution of gas.

Previous to this stage of the operation, all temperatures are regulated from thermocouple readings; but, with the beginning of the stirring operation, temperatures are regulated from observations taken with the optical pyrometer sighted on the surface of the glass.

Some typical melting schedules are shown in figure 5. The viscosity of those glasses for which measurements are available ranges between $10^{1.65}$ and $10^{2.01}$ poises at the melting temperature. The pot of glass is maintained at the melting temperature until proofs indicate that no further improvement in the seed quality can be expected. The temperature is then reduced at the rate of approximately 50 deg C/hr to the fining temperature with or without a "no stir" period. The fining temperature is somewhat empirical, but is defined as that temperature at which "the molten glass approaches freedom from undissolved gases."[2] The glass remains at the fining temperature until proofs indicate freedom from seeds. The rate of fining of a glass is influenced by both viscosity and surface tension [5], those of high-surface tension being more difficult to fine than those of low-surface tension. The viscosity at the fining temperature ranges between $10^{1.87}$ and $10^{2.18}$ poises.

After fining the pot of glass it is cooled at a rate of 50° C/hr to a specified temperature (average viscosity $10^{3.3}$ poises) preparatory to removing it from the furnace. During cooling, the diameter and rate of stirring are gradually decereased Various methods have been tried to cool the pot of glass from the bottom while it is in the furnace. Generally, the furnace door is raised about 4 in. and plugs in the two openings in the rear of the furnace[3] on a level with the floor are removed to cool the lower part of the pot more rapidly than it

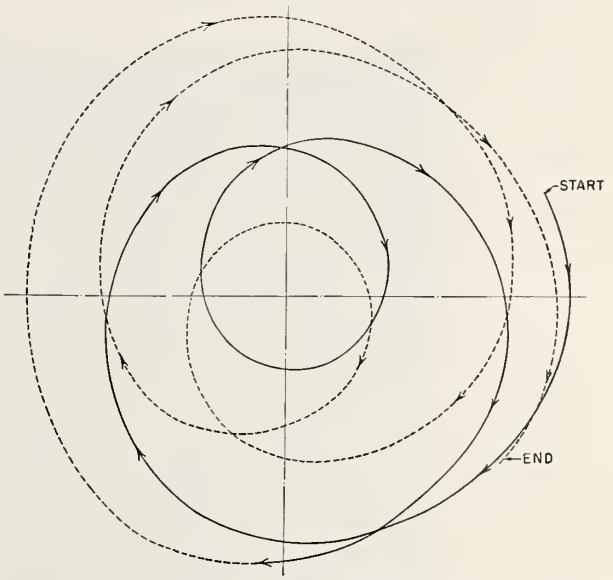


FIGURE 4. *Path of the refractory thimble during stirring.*

This represents only a part of the complete cycle that repeats itself every 60 to 65 revolutions.

[2] Glass Division Standard Definitions, Bul. Am. Ceram. Soc. **26**, 239 (1947).

[3] The use of two openings in the rear of the furnace was suggested in a private communication from J. C. Young.



793853°—48——2

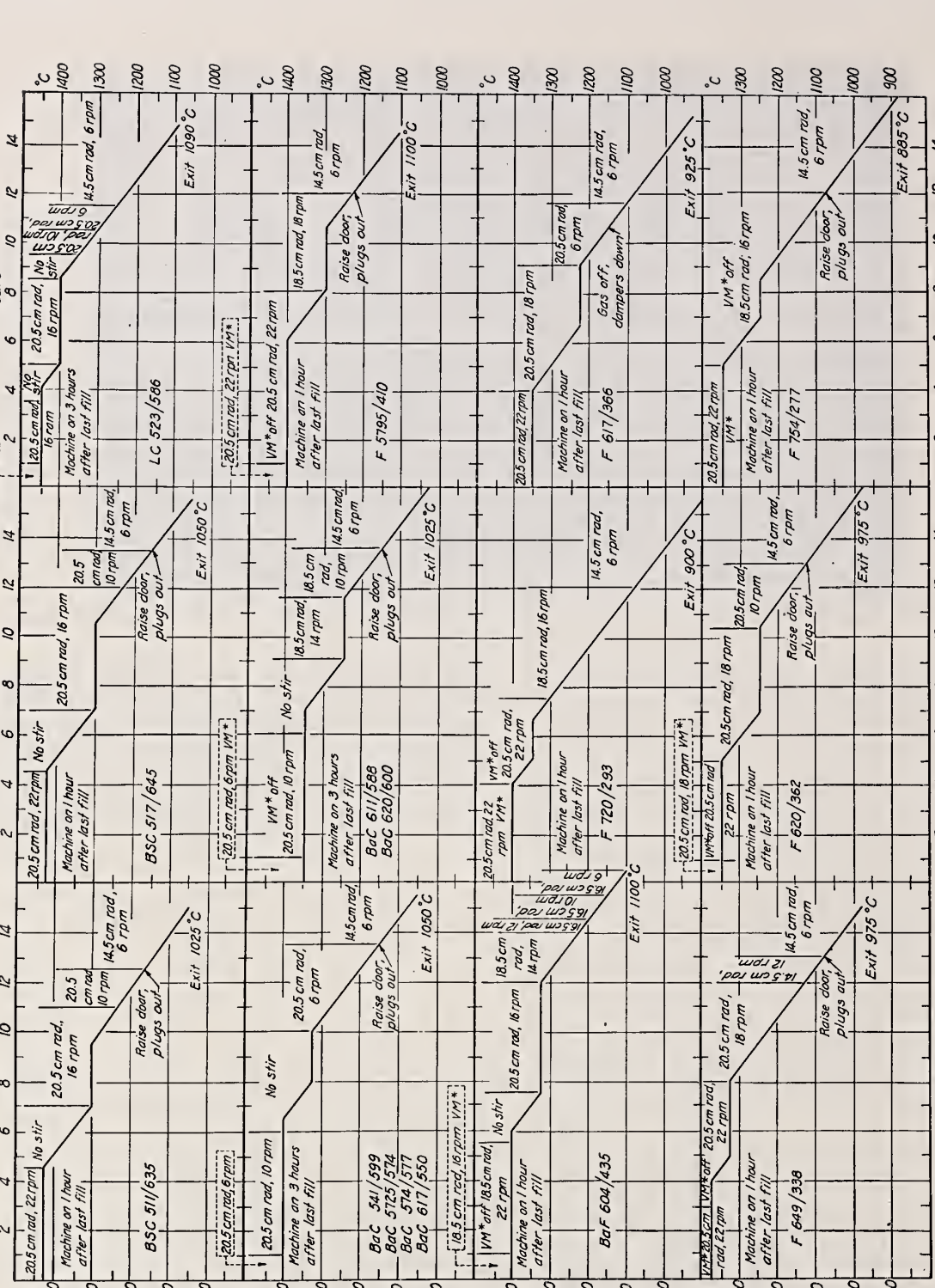


FIGURE 5. *Melting schedules for various optical glasses.*

*VM, vertical motion. The stirring thimble has an up-and-down motion superimposed on the regular horizontal motion of the stirring rod.

FIGURE 6. *Removing a finished pot of optical glass from the melting furnace.*

would normally cool during the last several hours of the melt. This procedure apparently reduces the convection currents, thereby improving the quality of the glass for striae.

The temperature at which a pot is removed from the furnace is very critical. If the pot of glass is too hot when removed, striae will be formed due to convection currents; if stirring is continued at too low a temperature (too high a viscosity), seeds will be formed in the glass. In fact, for most glasses the stirring schedule is a compromise. Seeds will be formed if the stirring is too fast; yet for the best striae quality, stirring should be as fast as possible without causing the glass to slop over. The stirring schedule finally selected is usually too fast for the best seed quality and too slow for the best striae quality.

The thimble is removed and the pot transferred from the furnace to four brick piers for cooling (fig. 6). An insulated can[4] is lowered to cover the upper third of the pot, and the bottom of the pot is cooled by means of a blower (fig. 7).

The insulated can is lowered to cover the pot

[4] These insulated cans consist of shells (46 in. in diameter by 42½ in. tall) made of 20-gage black sheet iron and lined with 4½ in. of insulating brick 2,000° F series, set on end. The bricks are held in place in the shell and top by means of draw bar bolts.

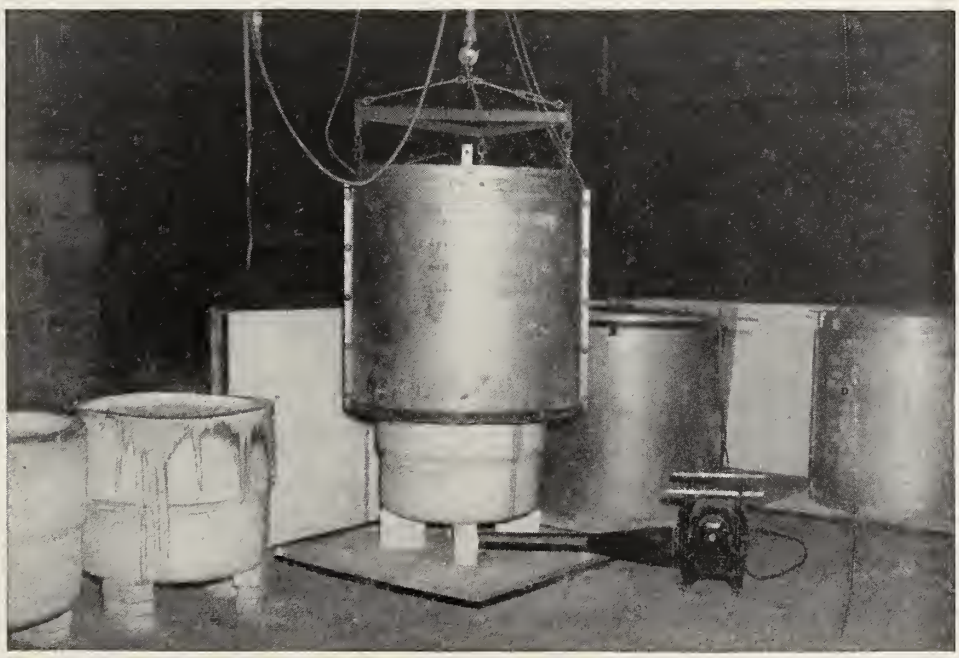

FIGURE 7. *Initial cooling of a pot of glass (center); completely covered pots in the right background.*

FIGURE 8. *Pot of glass after being broken open.*

completely when the temperature indicated by a thermocouple in the top of the can is within the 400° to 650° C range. For flint glasses this is done in the lower end of the range and for crowns in the upper end. The covering temperature is reached in from 3 to 7½ hr after the pot is set under the can. In the case of flints 5795/410, 617/366, and 620/362, the can is raised, and the glass is inspected at 250° C for fracture; if none is present, the glass is left uncovered until it cracks and then covered again. The insulated can is removed at 150° C, an insulated lid placed over the top of the pot to prevent shattering of the surface of the glass, and the pot left to cool to room temperature. The time necessary to cool a pot of glass to its uncovering temperature is approximately 3 days.

Convection currents are minimized by keeping the top hot, and any convection currents starting from the center stay on the surface instead of being forced down into the glass as they would be if the surface chilled. Cooling the bottom of the pot fixes the layer of striated glass due to pot solution.

The procedure for cooling partially anneals the glass and causes it to break into a relatively few large pieces instead of many small pieces. The partial annealing is necessary to prevent spontaneous breaking or shattering of the glass when trimmed.

## 5. Breaking Open the Pot of Glass

The cool pot is broken open (fig. 8) [6] and the moldable chunks of glass are boxed and weighed. At this time, samples are taken from critical locations in the pot for grading with respect to striae and seeds. Samples for index of refraction and transmission measurements also are taken.

Routine index measurements are made on the glass from each pot by the immersion method of Faick and Fonoroff [7]. Periodically, precision index and dispersion measurements are made on fine-annealed, ground, and polished prisms.

The pot shell is reclaimed and is used as grog in making new pots.

## 6. Preparing the Rough Glass for Molding

For medium- and small-sized blanks, the chunks of glass are broken into pieces of approximately the desired weight by means of hammers and, at the same time, striae and other imperfections are trimmed out. The hammers have square heads of case-hardened steel, and the face must be ground at least once during each shift to maintain sharp edges. Otherwise, marks may be left on the glass and it may not break properly. The glass for large blanks is trimmed to the proper weight by means of a diamond saw, which is also used for removing striae and other imperfections marked by the inspector. The waste glass from the trimming operations is used as cullet for succeeding melts.

## 7. Molding

### (a) Blanks up to 0.25 Pound—Paddling

Small blanks can be molded by procedure (b) which follows. However, it is generally more economical to mold them by the paddling process. The rough glass is molded into a flat of appropriate thickness, which, after scoring with a diamond, is then broken into cubes of a weight somewhat more than that desired in the finished blank. The cubes are then ground down to weight, the corners and sharp edges being removed in the process. The pieces are placed in the cooler section of a small gas-fired furnace, the floor of which is heated by radiation from the crown. The slabs used for the bottom of the furnace are Alundum with a veneer on top that gradually dusts, thereby keeping the glass from sticking (graphite slabs can also be used). As the cubes become heated they are gradually moved into hotter zones and, finally, to the paddling zone. Here they are paddled into shape by means of iron rods flattened at the ends.

Care must be exercised not to work into the glass the material that dusts off the floor slabs. Folds may be introduced during paddling unless extreme care is taken. When the glass has attained the shape to fit in the mold and the proper consistency, it is pulled into the preheated mold and pressed. The blank is removed from the mold and placed in an oven on top of the furnace where it slowly cools. For the larger blanks in

this category, a separate preheating furnace and cooling oven may be necessary.

**(b) Blanks Approximately 0.25 to 5 Pounds—Punty Rod**

For this method of molding, the glass must be heated until it will adhere to the punty head. Experiments indicated that a rate up to 6 deg C/min was safe for heating glass; therefore, a rate of 4 deg C/min was selected as having an ample factor of safety in designing preheating furnaces. These furnaces are built of low-temperature insulating brick, except the trough in which the pans for holding the glass slide and the walls adjacent to the burners, which are built of refractory brick. They are heated by two refractory screen burners, one on either side near the exit, operating on gas from a zero governor and low-pressure air. A thermocouple for regulating each furnace projects through the crown at the hot end. Table 4 gives the preheating temperatures used for typical glasses.

TABLE 4. *Temperatures of the hot zones of preheating furnaces and cooling lehrs used for molding optical glass by the punty method*

| Glasses | Hot-zone temperatures | |
|---|---|---|
| | Preheating furnace | Cooling lehr |
| | °C | °C |
| F 5795/410 | 610 | 500 |
| F 605/381 | 550 | 470 |
| F 617/366 | 540 | 440 |
| F 620/362 | 530 | 450 |
| F 649/338 | 550 | 475 |
| F 720/293 | 575 | 485 |
| BSC 517/645 | 690 | 575 |
| BaC 5725/574 | 700 | 575 |
| BaC 611/588 | 760 | 625 |
| LC 523/586 | 650 | 550 |
| BF 604/435 | 700 | 575 |

The molding furnace (or glory hole) is of the drum type with the upper portion lined with 4½ in. of insulating firebrick and the bottom lined with a preshaped, prefired plastic fire-clay refractory. This bottom tapers up from the center toward each end and has a drain hole at the low point. The temperature ranges from 1,375° to 1,400° C and is obtained by means of a refractory-screen burner entering the furnace tangentially at the top. This arrangement gives a swirling flame and holds it until combustion is complete. Provisions are made to vary the furnace atmosphere from oxidizing to reducing.

The rough pieces of glass are cleaned and placed on shallow iron pans for introduction into the preheating furnace. When the glass has reached the working end of the preheating furnace, it will adhere to a punty.[5] A piece of glass is picked up on the end of a punty and transferred to the molding furnace. The glass is alternately heated and marvered to shape. During this operation sharp edges are trimmed and imperfections, such as seeds, folds, feathers, etc., that appear near the surface are cut out with shears. When the glass reaches the consistency and shape for pressing, it is suspended over the heated mold (400° to 500° C), and the amount necessary to make the blank is cut off with shears. The surface of the glass is heated under a burner to remove the shear marks caused by chilling and the gob is pressed into shape (fig. 9).

Surface defects attributable to operating conditions during molding are of at least four types: 1, Devitrification (barium crowns); 2, surface reduction (flints); 3, surface seeds (barium flints); 4, a "web" defect that is normally visible only when the glass is immersed in a liquid of equal index of refraction (flints 617/366, 620/362, and 649/338 and others; also, at times, borosilicate and barium crown glasses). These defects are objectionable mainly because they interfere with the inspection of the blanks.

The molds are made in three parts—the plunger, the frame, and the base. The thickness of the blank can be changed by the use of washers between the frame and the base. The molds are rather massive so that they will have sufficient heat capacity to maintain an even temperature while in use. Cold-rolled steel and cast iron are satisfactory for molds.

The pressed blank is transferred to the cooling lehr where it is placed on a pan lined with sheet asbestos. The electrically heated lehr is 30 ft long, with the hottest zone in the first 4 ft. The

FIGURE 9. *"Cutting off" a gob of hot, semifluid glass into a steel mold preparatory to pressing into shape.*

[5] At the start of the war the punty heads were made of fired sections, cut from an extruded clay cylinder, cemented to the punty rod. Four to six of these heads might be broken per 8-hr shift. L. H. Maxwell suggested welded steel heads. This was tried and has been used with success ever since. Before use the head is given a coating of glass.

rate of travel through the lehr can be varied from 3¾ ft/hr to 5 ft/hr (6 to 8 hr in the lehr). Table 4 gives the hot-zone temperatures used for some of the glasses in the cooling lehrs.

#### (c) Blanks Over 5 Pounds—Slumping

Large blanks are slumped in ceramic molds that are prepared for use by dusting or spraying, with a mixture of 95 percent of powdered flint and 5 percent of clay, which prevents the glass from sticking. The rough glass is trimmed to the proper wieght with a diamond saw (fig. 10) and placed in the mold. The preheating end of a combination slumping-preheating furnace is filled with the molds and then gradually heated up to that temperature at which the glass flows freely and accurately assumes the contour of the mold. The molds are then moved into the cooler end of the furnace where the glass is annealed.

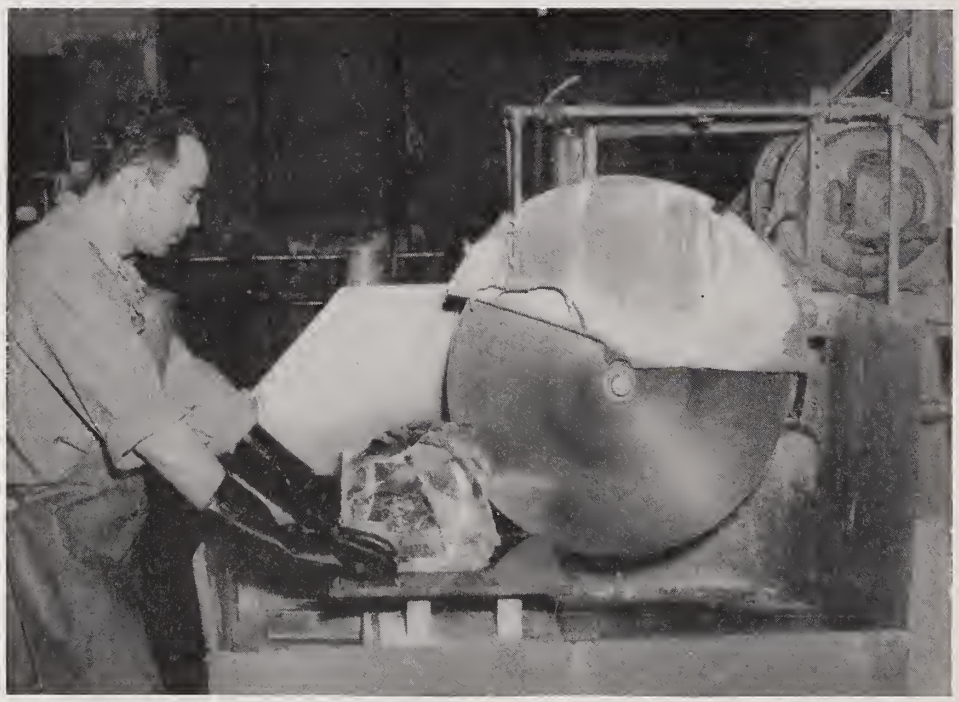

FIGURE 10. *Trimming a chunk of optical glass, with a 24-in. diamond saw, prior to slumping.*

## 8. Inspection of Molded Blanks

Blanks are first inspected for seed and those containing too many or too large seeds are rejected; the remainder are inspected for striae. This inspection for striae is made in a tank having glass windows in the ends. The tank is filled with a liquid having an index of refraction similar to that of the glass to be inspected. The blanks are immersed in the liquid and, under proper illumination, imperfections in the glass become visible. A liquid that evaporates from the glass without leaving a residue is desirable, since it makes unnecessary any cleaning operations. The optical system is essentially the Bureau of Standards tank immersion method as reported by Dodd [8]. The immersion liquids must be clear and practically colorless. Three such liquids are used at this Bureau: kerosine ($n$=1.45), monochlorbenzene ($n$=1.52), and carbon disulfide ($n$=1.62+). These liquids are mixed in the proportions required to give the desired index. Caution must be exercised in the handling of carbon disulfide because of the extreme fire hazard. Alpha-monochlornaphthalene ($n$=1.633+) can replace it and has the advantage of reducing the fire hazard. However, it presents a health hazard-inflicting some workers with a very painful rash, For indices above 1.62+, flowers of sulfur dis. solved in carbon disulfide can be used. Inspection tanks are placed in hoods exhausted from the bottom. The exhaust fan is of sufficient capacity to maintain the concentration of vapors in the inspection room below that which would be injurious to health.

## 9. Annealing

Schedules for annealing, as given in table 5, were devised with reasonable accuracy from the information obtained by the interferometer method of measuring thermal expansion [9] and from the work of Adams and Williamson [10]. Typical expansion curves for optical glasses are given in figure 11. The annealing range lies between the

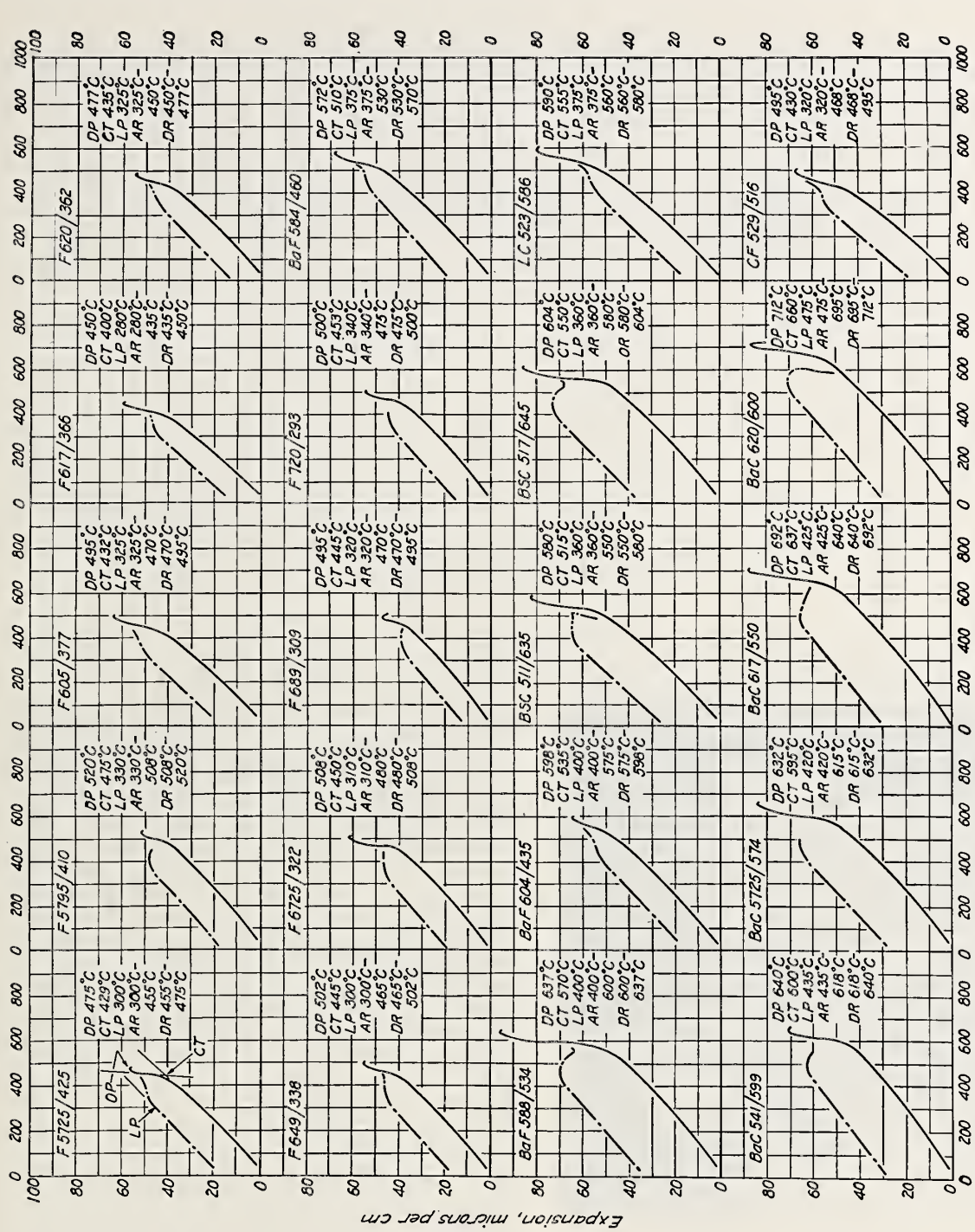


FIGURE 11. *Expansion curves for some optical glasses as determined with the interferometer.*

DP, deformation point; CT, critical temperature; LP, low point; AR, annealing range; DR, deformation range; ——·——·——, chilled sample; ——, annealed sample. Determinations made by J. B. Saunders; J. Research NBS **23**, 179 (1939) RP1227; **35**, 157 (1945) RP1668.

TABLE 5. *Annealing furnace schedules for optical-glass blanks*

[Heating the annealing furnace up to temperature for blanks weighing 5 lb or under, takes 10 to 12 hr. For blanks over 5 lb, the heating-up, holding, and cooling-down schedules would have to be proportionately lengthened]

| Schedule | Glasses | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Flints 605/381, 617/366, 620/362, 720/293, and 754/277 | | Flints 649/338, 666/324, and 689/309. (Can also be used for flints 620/362, 720/293, and 754/277) | | Flints 6725/322 and 5795/410 | | Borosilicate crown 511/635, light crowns 512/605 and 523/586, and barium flints 584/460 and 604/435 | | Borosilicate crown 517/645 | | Barium crowns 541/599, 5725/574, and 574/577 and barium flint 588/534 | | Barium crowns 611/588 and 617/550 | | Barium crown 620/600 | |
| | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate | Temperature | Cooling rate |
| Hold 2 hr after[1] temperature reaches | °C<br>410 | °C/hr | °C<br>420 | °C/hr | °C<br>465 | °C/hr | °C<br>510 | °C/hr | °C<br>550 | °C/hr | °C<br>570 | °C/hr | °C<br>605 | °C/hr | °C<br>655 | °C/hr |
| Cool | | | | | 465 to 450 | 3.0 | 510 to 490 | 3.0 | 550 to 515 | 3.0 | 570 to 530 | 3.0 | 605 to 550 | 3.0 | | |
| Hold 12 hr at | | | | | 450 | | 490 | | 515 | | 530 | | 550 | | | |
| Cool as follows | 410 to 390 | 0.75 | 420 to 410 | 0.75 | 450 to 430 | 0.75 | 490 to 465 | 1.0 | 515 to 500 | 1.0 | 530 to 510 | 1.0 | 550 to 525 | 1.0 | 655 to 620 | 1.0 |
| | 390 to 370 | 1.25 | 410 to 390 | 1.25 | 430 to 410 | 1.25 | 465 to 440 | 1.5 | 500 to 475 | 1.5 | 510 to 490 | 1.5 | 525 to 500 | 1.5 | 620 to 580 | 1.5 |
| | 370 to 350 | 1.75 | 390 to 370 | 1.75 | 410 to 390 | 1.75 | 440 to 420 | 2.0 | 475 to 450 | 2.0 | 490 to 470 | 2.0 | 500 to 475 | 2.0 | 580 to 520 | 2.5 |
| | 350 to 330 | 2.25 | 370 to 350 | 2.25 | 390 to 380 | 2.5 | 420 to 400 | 2.5 | 450 to 425 | 3.0 | 470 to 450 | 2.5 | 475 to 450 | 2.5 | 520 to 400 | 3.5 |
| | 330 to 300 | 3.0 | 350 to 325 | 3.0 | 380 to 340 | 4.0 | 400 to 360 | 4.0 | 425 to 400 | 4.0 | 450 to 410 | 4.0 | 450 to 410 | 4.0 | | |
| Current off at | 300 | | 325 | | 340 | | 360 | | 400 | | 410 | | 410 | | 400 | |
| Raise top ¼ in. at | 275 | | 300 | | 300 | | 340 | | 350 | | 375 | | 375 | | 375 | |
| Remove top at | 225 | | 250 | | 275 | | 300 | | 300 | | 325 | | 350 | | 325 | |
| Steel box removed at | 225 | | 250 | | 275 | | 300 | | 200 | | 325 | | 350 | | 325 | |

[1] Top temperature in no case should exceed the maximum holding temperature by more than 5 deg C as the glass may slump.

FIGURE 12. *Loading the steel box with molded blanks in preparation for annealing.*

low point (the temperature at which the release of strain in a chilled specimen starts) and the deformation point. Mechanical strain is released almost instantaneously in the rapid expansion range that lies between the critical temperature and the deformation point. Glass annealed in the rapid-expansion range and cooled at a moderate rate will approximate the index of refraction obtained at the critical temperature.

In the annealing of glass another compromise must be made. The higher the temperature of annealing, especially for large blanks, the more slowly the furnace must be cooled at the start of the cooling cycle so as not to introduce strain in the blanks because of temperature gradients. Consequently, the glass is generally maintained at a fairly high temperature to release strain rather quickly and then held at a lower temperature within the annealing range to obtain the maximum index of refraction practicable (equilibrium at this lower temperature) [11]. During this period any stresses introduced during cooling from the higher t mperature are also released. The index of refraction obtained is somewhat higher than it would be had the glass been annealed at the higher temperature.

The glass blanks for annealing are placed on perforated steel trays. These trays are placed in a steel box (fig. 12) and separated from each other by spacers. The filled and covered box is placed in an electrically heated annealing furnace built of insulating firebrick. The top of the furnace is removable as a unit. The top is sealed to the body of the furnace by strands of asbestos tape.

These box-type annealing furnaces are heated by 19 resistance plates, three each on the sides and top, and four on the bottom. These plates are 14 by 9 in., 220 v., and are connected in series-

parallel for uniform heating. The total current is controlled by variable autotransformers. A portion of the total current is bypassed through an external resistance and controller-operated relay. This differential current is set so that the current applied to the furnace with the relay open is approximately 3 to 5 amp. less than the total current. Thus the furnace is manually regulated but automatically controlled at any chosen temperature.

Small blanks are annealed in a furnace equipped with a program controller. Temperatures throughout the furnace are equalized by circulating the hot air with a motor-driven fan. A potentiometer recorder furnishes a visual record of the furnace operation. The annealing cycle with this equipment is somewhat shorter than that of the box-type furnace because of the equalized temperatures.

There is considerable latitude in the exact annealing schedule that may be used with satisfactory results. Uniformity of temperature within the furnace, however, is very important and is sometimes difficult to obtain. A low value of birefringence does not assure satisfactory annealing because index gradients, arising from differences in effective annealing temperatures within the glass, may be present.

Finally, the blanks are examined for strain under polarized light while immersed in a liquid of index of refraction similar to the glass. If the glass is perfectly annealed, the area of the glass appears the same as the adjoining background; if not, an interference figure is obtained. This figure should show uniform distribution of residual strain and, when compared with standard strain samples, should indicate a relative retardation of 5 m$\mu$ or less per centimeter of transmitted path, although the Joint Army-Navy Specifications allow a relative retardation of 10 m$\mu$ [12]. Blanks failing to pass the inspection for strain must be reannealed.

## III. General Remarks

Previous to 1939, the Bureau supplied the Navy with five types of optical glass: BSC 517/645, BaC 574/577, LC 523/586, F 620/362, and F 649/338. During World War II the Bureau supplied to the Armed services 28 different types of optical glass. Also, the specifications for the blanks became increasingly more stringent. At present there are a number of additional types under laboratory experimentation. But taking a glass composition out of the laboratory and putting it into regular production is fraught with many difficulties.

The optical glass, to be acceptable, must at least pass the Joint Army-Navy Specifications [12]; the Navy at times requires glass of even higher quality. The index of refraction tolerance for glass of an index less than 1.56 is $\pm 0.001$; for those above 1.56 it is $\pm 0.0015$. The nu-value tolerance for those above 58.0 is $\pm 0.5$, except for LC 523/586, where it is $\pm 0.4$. Between 50.0 and 58.0, the nu-value tolerance is $\pm 0.4$; below 50.0 it is $\pm 0.3$. Also, the blanks from which reticles and elements in the focal plane are made must be free from bubbles or seeds over 0.0004 in. (0.01 mm) in diameter. For other purposes, a few larger seeds are permissible. Simply because they spoil the appearance of the polished element, the seed limits are often more stringent than is really necessary for optical purposes.

The present trend in optical glass seems to be toward crowns of ever higher index of refraction and lower dispersion. And, as a corollary, optical designers would like to have high-index flints with both higher and lower dispersions than can be obtained with the present flint compositions.

Filters of better quality than those now available are needed. And, as an outgrowth of World War II, optical glasses transmitting the ultraviolet and the infrared are much desired. So it seems safe to say that many of the chemical elements not formerly used commercially in optical glass will become important constituents of it in the future. Especially will this be true as far as infrared-transmitting glasses are concerned. To produce glasses having satisfactory transmissions beyond about 4.5 $\mu$, glass-formers other than silica, boric oxide, and phosphoric anhydride should be investigated. Hence, there remains much research work to satisfy the enthusiastic investigator for years.

Grateful acknowledgment is hereby made to the many past and present members of the Glass Section, Mineral Products Division, National Bureau of Standards, whose collaboration in the development of control and production methods was responsible for the success of the work on optical glass. This work was under the direction of A. N. Finn, Chief of the Glass Section, until the time of his death in 1942. The contributions of W. W. Coffeen, C. C. Diller, C. A. Faick, A. C. Francisco, R. A. Heindl, D. Hubbard, F. R. Matson, L. H. Maxwell, C. B. Riecks, L. Shartsis, A. Q. Tool, O. F. Valentine, A. E. Williams, and J. C. Young also deserve special mention.

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