Preparation of Sintered Pyrex Glass Filters

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The advantages of filters made from sintered glass disks are well known, largely because of the wide use of such filters constructed from Jena glass. The practical difficulties of incorporating Jena filters into general apparatus, which in this country is rarely made from Jena glass, have long indicated the desirability of using Pyrex brand glass. Methods for constructing such filters have been very briefly described by Bruce and Bent (*1*) and by Shatenshtein (*4*), and other workers (*5*) have recorded the construction of filters from Pyrex glass but have not given their methods. Thomas (*5*) and Prausnitz (*3*) have detailed various special uses for sintered glass filters. However, no careful study of the method of making the sintered disk is given, nor are methods recorded in sufficient detail to enable the investigator to prepare at will any desired size or porosity of filter. On this account, this study has been made and it has been found possible to prepare sizes from about 0.375 to 2 inches (0.94 to 5 cm.) diameter or larger, in any desired porosity and without undue chance of failure.

Method

Ground glass of uniform sized particles and of graded sizes is necessary if reproducibility of porosity is to be obtained. Moreover, it is essential that the glass used be reasonably uniform in quality, preferably of the same lot as the tubing into which the sintered disks are to be sealed, in order to have a minimum of breakage. While it is probably not necessary to use glass of the same lot for the disk and the tube into which it is sealed, scrap Pyrex glass from broken apparatus, even though it be thoroughly cleaned, does not seal well into new tubing. This may be a phenomenon of aging of the glass, such as devitrification.

The glass must not be ground in a porcelain or glass mortar, unless the mortar is of Pyrex glass. An iron mortar and pestle were used in this study. The glass is ground and sifted rapidly through a 100-mesh sieve, the coarse particles being reground until a considerable amount of sifted glass is available. This contains considerable iron and some material insoluble in strong acids, presumably iron carbide. The glass is treated with strong acid and warmed to dissolve the iron. The insoluble material will usually float or at least settle more slowly than the main portion of the glass and can be removed by decantation. When the glass cannot be completely cleaned by this treatment, caustic solution instead of acid is applied, and in the rather strong caustic solution the insoluble material can be suspended sufficiently to make decantation possible. The clean glass powder must be graded rather accurately, preferably by use of differential settling rate in an elutriator such as is commonly employed in the separation of different constituents of soils. Grading may also be accomplished by means of a series of sieves of graded fineness. The glass was arbitrarily divided into four grades, as shown in Table I.

Table I. Grading of Glass

Grade	Average Particle Diameter	Approximate Screen Size
	Mm.	*Meshes/inch*
1	0.16	100–150
2	0.08	150–200
3	0.04	200–275
4	0.02	275–325

The glass must now be placed in a mold for sintering. For this purpose the authors have used a mold constructed from brass, because of the ease of obtaining and working this metal, but other metals may be more suitable, since brass invariably discolors the surface of the disk. This discoloration is easily removed by use of carborundum paper after sintering. Bruce and Bent used nickel molds and record no difficulties, though their design is inconvenient and allows only one disk to be made in one mold. The authors' molds were constructed of two brass plates each 3 by 6 inches (7.5 by 15 cm.) The base plates were 0.3125 inch (0.8 cm.) thick and had two upright pins, one at either end, for holding the upper plate which varied in thickness depending on the size of disk being made. The upper plate should be a little thicker than the desired thickness of the disk and should contain a convenient number of holes a little larger than the final size of the disk desired, since the disk shrinks a trifle while being sintered. The upper plate should also have holes corresponding to the pins in the base plate over which it slipped, so that, when assembled the plates are held rigidly together. When paper or Cellophane inserts were placed between the plates to prevent discoloration of the disk by the brass, the carbonaceous material from the paper was mixed with the glass powder and entirely prevented the particles from sintering.

The holes were completely filled with the glass powder, packed as tightly as possible to minimize shrinkage. With grades 1 and 2, simply tapping the mold sharply produced satisfactory settling. With the finer grades, it was necessary to moisten the powder with a few drops of water and pack the wet glass. When this was done the shrinkage was very slight. The water was dried out before placing at sintering temperature to prevent formation of vapor bubbles. While filling the mold, an electric muffle furnace equipped with a pyrometer accurate to $\pm 3°$ C. was heated. The pyrometer junction was some 2 inches (5 cm.) above the mold in the furnace, thereby registering the temperature of the furnace interior rather than that of the mold. When the temperature reached 800° C., the molds were quickly inserted. The temperature fell off rapidly in all cases until the molds became heated. When the temperature had risen to the correct value for sintering, the rheostat controlling the furnace temperature was manually operated to maintain the sintering temperature as accurately as possible.

Experiments were run to test the effect of time and temperature on the sintering process, and it was found in all cases that a change of a few degrees in temperature had much more effect than even doubling the time. As a result, the standard time chosen for all grades of glass was 15 minutes, since this length of time was not tiresome, and it allowed well for inequalities of temperature, either locally in the furnace or due to lag in the heating elements, both being factors of great importance when temperature control is critical.

The optimum temperatures for the grades of glass used are given in Table II.

At the expiration of 15 minutes at the full sintering temperature, the mold was immediately withdrawn from the furnace and the sintered disks extracted as soon as the mold had cooled sufficiently.

Table II. Optimum Temperatures

Grade	Temperature
	° C.
1	810
2	800
3	790
4	780

Small variations in porosity inevitably occur between individual disks of the same lot, but properly made disks of any one grade invariably differ in porosity from all disks of another grade. Grade 1 is suitable for a rapid filter for coarse precipitates, and is especially adapted to use as a gas bubbler where gases must be scrubbed rapidly. It has been found useful in constructing extractors to retain the solid nonextractives. Grade 4 retained the finest analytical precipitates. Grades 2 and 3 are intermediate in porosity, and are to be recommended for separating analytical precipitates which are not of the very finest varieties, since their speed of filtration is decidedly greater than that of grade 4. Likewise they are suitable for gas bubblers where the volume of absorbent is not large and the gas must be very thoroughly scrubbed in one vessel, as in the quantitative absorption of carbon dioxide in a small volume of alkaline solution. No difficulties have been met in duplicating porosities at various times when the same glass and sintering procedure are used.

All sizes of disks which the authors have made have been successfully sealed into Pyrex tubing. No difficulty has been encountered in the seal itself, but an annular crack in the tube frequently formed at the edge of the smaller sizes of disk on cooling. This difficulty was in no case avoided by annealing with a large flame of any temperature, but could be avoided by sealing the disk into a tapered rather than a straight tube, or better, by using a very fine hot flame to anneal the edge of the seal after partial cooling. The strain is localized at the edge of the seal by contraction of the disk on cooling and the only satisfactory form of annealing is a local application of heat at the point of strain without a reheating of the disk itself. Turning the tube in a lathe and applying pressure at the seal with a carbon rod produced the best seals with a minimum of fusion of the disk. By this procedure the micro external filters described by Kirk (*2*) have been made satisfactorily, though no filters of this small size have been available hitherto.

The authors have used the method of Shatenshtein (*4*) in a few instances. It is successful in sintering Pyrex glass when the size of filter made is small—not over 1 cm. in diameter—and when the glass used is relatively coarse—grades 1 or 2—but the method has very limited application and lacks accurate control and reproducibility.

Literature Cited

(1) Bruce, W. F., and Bent, H. E., *J. Am. Chem. Soc.*, 53, 990 (1931).
(2) Kirk, P. L., *Mikrochem.*, 14, 1 (1933).
(3) Prausnitz, P. H., Ind. Eng. Chem., Anal. Ed., 4, 430 (1932).
(4) Shatenshtein, A. E., *J. Chem. Ind.* (*Moscow*), 6, 1800 (1929).
(5) Thomas, M. D., Ind. Eng. Chem., Anal. Ed., 5, 193 (1933).

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A Simple Radio Relay Circuit

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Radio circuits are used in controlling the temperature of both high- and low-temperature thermostats, in the intermittent starting and stopping of motor sets, and in amplifying the current from a photoelectric cell to accomplish these or similar purposes. The advantage of a radio type of relay circuit over that ordinarily used is that, because of the small current in the grid circuit no arcing can take place to foul or disintegrate the contacts. Furthermore, the circuit may be made and broken in an atmosphere containing inflammable vapors (*1–5*).

The principle involved is, that the application of an appropriate voltage (very low amperage is required) to the grid of a suitable radio tube will cause a large decrease in the current flowing in the plate circuit. By having the current in the plate circuit operate a relay, very large currents can be made or broken by appropriate contacts.

The circuits ordinarily described require direct current, and in some cases both direct and alternating current. Since direct current is rarely available, storage or dry cells (B or C batteries) must be used. These are an additional expense and frequently become discharged at a most inopportune time. The writers have adapted a standard single amplifying circuit, which requires only alternating current, for general use in the laboratory. The simplicity of the circuit is immediately apparent from Figure 1.

A is a 171-A power tube which will furnish a plate circuit of approximately 10 milliamperes. *B* is a device for closing the grid circuit. It may be a thermal regulator of the usual mercury type or one made from a bimetallic strip, or it may be a photoelectric cell. *C* is a fixed condenser used to smooth out the intermittent direct plate current and *L* is the source of 110 volts alternating current (60 cycles). M_1 and M_2 are 1- and 2-megohm grid leaks such as are ordinarily found in radios. If the resistor, M_1, is placed between R_1 and *B* the possibility of grounding the source of alternating current is avoided. It also eliminates being shocked while making adjustments. R_1 and R_2 are resistors whose resistances are shown in Table I. If lamps are not used, the resistors should be capable of handling about 50 watts. *T* is a suitable relay. The relays described are manufactured by the Western Electric Company. B59 and 178BY are used by telephone companies on their switchboards and sell for about $4. Relay B59 has only one set of platinum contacts, while 178BY has several. Relay 22A is a large instrument used in communication lines, and has but one contact. The current consumption using relay B59 and two 50-watt carbon lamps in the filament circuit is about 20 watts.

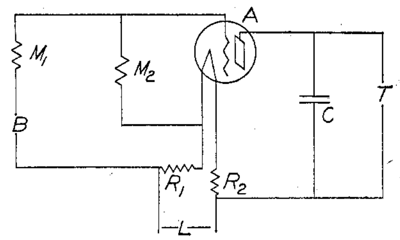

Figure 1

The heavier circuit for operating the apparatus may be closed directly through the operation of the relay contacts, or indirectly by means of a vacuum contact such as those made by the Burgess Battery Company, Chicago, Ill., which will handle 1300 watts at 220 volts, or by means of a mercury switch which may be operated by means of a solenoid con-