A simple and accurate constant-volume pyknometer for specific gravity determinations.

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EXPERIENCE with the usual types of glass pyknometers long ago convinced the writer that these instruments as ordinarily used are subject to considerable errors and may be quite unreliable when dealing with small quantities of material. It is true there are forms[1] intended to be used in conjunction with a very precisely regulated water-bath which are capable of giving results of high accuracy, but this method of determination at constant temperature involves too much time and trouble for routine mineralogical work. But no matter what the design of glass pyknometers, and their variety is legion, in actual practice they suffer in common from the defects of the material of which they are constructed.

The heavy-solution method is inconvenient; it cannot be used for the heavier materials and there is often danger of contaminating the material which may be intended for analysis. There is a real need for a simple method capable of giving consistently accurate results—particularly on small specimens and powdered material—with a minimum expenditure of time and attention.

To fill this need the silica-glass pyknometer here described was designed by the writer some eight years ago, and has since been in constant use with very gratifying results. The design was carefully worked out in an endeavour to secure the following characteristics:

1. Not more than 0·0001 c.c. change in volume of the bottle for ordinary variations of room temperature (in Canada). This called for fused silica construction and a volume not exceeding 10 c.c.

2. Constant depth of seating of stopper. This again called for silica-

[1] A. E. H. Tutton, Crystallography and practical crystal measurement, 1911, p. 519, et seq. J. Johnston and L. H. Adams. Journ. Amer. Chem. Soc., 1912, vol. 34, p. 566.

glass, with its low coefficient of expansion, fine grinding and accurate fit of stopper, and a stopper having not too acute an angle.

3. Stopper with capillary side tube graduated at intervals of one-half centimetre and of uniform bore such that the capillary column of water weighs approximately one milligram per centimetre at 20°, so that fine weighing can be done entirely by the balance-rider with the unaided eye. The meniscus of such a column can be seen readily through the balance-case at a distance of 12 to 18 inches. The graduations on the side tube

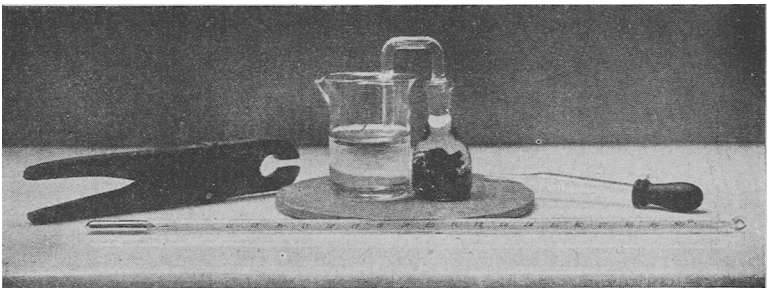

FIG. 1. Photograph showing pyknometer and accessories.
(About one-third actual size.

extend completely around the tube to avoid parallax in reading the meniscus.

4. Perfect over-all contact between stopper and bottle with no groove at the top or other pockets or depressions to retain water and produce irregularities. This is achieved by grinding and subsequently fire polishing the top of bottle and stopper, and by special attention to the grinding of stopper.

5. Stability, with strength and rigidity, to withstand vacuum and heating.

6. Suitable ring on neck so that it may be handled conveniently by means of tongs.

The result of these considerations is the instrument shown in fig. 1. It is made of transparent fused silica, has a volume of almost exactly 10 c.c., and weighs about 10 grams. Approximate dimensions in millimetres are indicated on the sketch (fig. 2).

The anticipation of superior performance has been fully realized. The pyknometer can be heated to 100°, subjected to vacuum, and cooled without changing in apparent capacity more than 0·0002 c.c. at temperatures between 18° and 25°, the apparent change including also all errors

of filling, weighing, and temperature determination, and these results can be repeated year after year.

The method of specific gravity determination using this pyknometer is based on the assumption that minerals will suffer only a negligible change in volume between, say, 18° to 22°, as compared to their volume at 20°, the standard temperature; so that sufficiently accurate results may be obtained by using a pyknometer of constant volume at these temperatures.

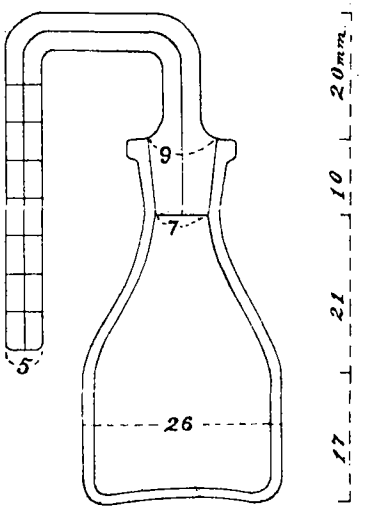

FIG. 2. Sketch showing dimensions of pyknometer. (Actual size.)

In use, the pyknometer is first calibrated once for all by determining its volume at room temperature with the meniscus of the capillary column of liquid at the various graduations on the side tube. This is done as follows. After weighing clean and dry, it is about half filled with water and the water boiled. It is then completely filled with boiled water and the stopper inserted quickly so that no air is entrapped and water rises through and passes out of the capillary side tube. The free end of the side tube with a drop of water adhering is inserted to a depth of $\frac{1}{8}$ inch in water contained in a small beaker and allowed to stand near the balance until cooled to room temperature. The pyknometer thus will automatically fill with water at room temperature. It is then lifted from the beaker by means of wooden tongs, the wet end of the side tube allowed to dry, and weighing begins. The meniscus in the capillary retreats at a regular rate as water evaporates from the capillary and from around the stopper; so that by using the balance-rider it is very easy to catch the exact weights for the second or third divisions and for each successively to the last of the graduations. Having completed weighing to the last mark, the pyknometer is taken off the balance at once with the pair of wooden tongs at room temperature, the capillary stopper is disconnected, and with a small medicine dropper also at room temperature about 1 c.c. of water is quickly removed. A suitable thermometer is then inserted and the temperature read to at least $\frac{1}{20}°$. Knowing the weight of contained water at the various graduation marks

and the temperature, the volume is calculated from the tables of the Reichsanstalt [1] or of Chappuis.[2] Since the volume is thus derived from the International Tables, it is necessary that the weights used be calibrated against standard weights and that the thermometer should be similarly corrected by reference to the international hydrogen thermometer scale.

Using weights and thermometers corrected by the U.S. Bureau of Standards, the writer has obtained the following values for the capacity of one of these pyknometers :

Date.	Temperature.	Capacity.
1922	19·28	10·1500 c.c.
1922	20·77	10·1497
1922	22·77	10·1497
1923	18·83	10·1500
1923	21·92	10·1501
1924	24·22	10·1498
1924	22·47	10·1498
	Average	10·14987

These are not selected results, but include every determination made from the time the procedure was first perfected. Since an error of 0·0001 gram in weighing or an error of $\frac{1}{20}^\circ$ in reading the temperature will cause an error in apparent capacity of 0·0001 c.c., it is evident that the results prove the remarkable certainty and uniform performance of the instrument itself. It may be seen that no single result varies more than 0·0002 c.c. from the average and that the 1924 results appear to be practically the true value.

The procedure for a specific gravity determination scarcely needs detailed description, being similar to that used in calibrating for capacity. Knowing the capacity of the pyknometer and the temperature of the water, the weight of water displaced is found from the International Tables. It is always advisable to heat the mineral powder and water in the pyknometer under vacuum. One or two precautions should be noted. In seating the capillary stopper it should be slightly rotated once or twice and finally brought always to the same definitely fixed position of rotation by reference to a permanent mark on the rim of the bottle. This is necessary because the volume may vary slightly for different positions of rotation of the stopper. Such variations may

[1] M. Thiesen, K. Scheel, and H. Diesselhorst, Wiss. Abh. Phys. Techn. Reichsanstalt, Berlin, 1900, vol. 3, p. 69.

[2] P. Chappuis, Travaux et Mémoires du Bureau International des Poids et Mesures, Paris, 1907, vol. 13.

be rendered visible by noting the advance and retreat of the meniscus in the capillary side tube as the stopper is turned.

The medicine dropper is used to remove the amount of water that would be displaced by insertion of the thermometer, so that the water does not overflow the bottle and cause lowering of temperature by evaporation. t can be adjusted in such a way as to remove automatically exactly the right amount of water.

The whole equipment of pyknometer, thermometer, medicine dropper, and wooden tongs should be at room temperature near the balance-case when in use. The temperature of the water in the pyknometer after weighing will ordinarily be a fraction of a degree higher than room temperature, due to radiation from the body of the operator during weighing.

Pyknometers of this design were made of transparent silica-glass by the Silica Syndicate,[1] London, at a moderate cost and satisfactorily fulfilled the specifications in every respect. The fit of the stoppers, however, was improved by grinding with the finest grade of alundum and finally with optical alundum.

Special thermometers were designed to use with the pyknometers. They are 5 mm. diameter, 260 mm. long, and are graduated from 12° to 30° C., each degree occupying a length of 1 cm. on the stem and divided in twentieths, though it is easy to read to the fortieths by the aid of a hand-glass. The thermometers in use by the writer were made by the Precision Instrument Company of Philadelphia and were very satisfactory, tests by the U.S. Bureau of Standards showing almost negligible uniform corrections of one to three hundredths of a degree. These thermometers should be calibrated and tested under the same conditions as when in actual use, i.e. with 1-inch immersion and the remainder of stem in air at room temperature.

Although the method as outlined may seem rather complicated it is really very simple, requires but little actual working time, and for exactness leaves nothing to be desired.

[1] Now the Thermal Syndicate, Limited, Neptune Bank, Wallsend-on-Tyne.