THE AMERICAN MINERALOGIST, VOL. 51, MAY-JUNE, 1966

A MICROPYCNOMETER FOR THE DETERMINATION OF THE SPECIFIC GRAVITY OF MINERALS[1]

IRVING MAY AND JOHN MARINENKO, *U. S. Geological Survey, Washington, D. C.*

INTRODUCTION

The specific gravity of fine-grained or powdered minerals is commonly determined with a pycnometer. This technique is quite satisfactory on the macroscale. However, if a determination must be made on a sample as small as 10 mg, errors associated with filling the pycnometer to a reproducible volume usually limit the accuracy possible.

To make a determination on a 10-mg sample of specific gravity 3, by use of a liquid of specific gravity 1.6, the pycnometer must be filled with a reproducibility of about 0.1 lambda (0.0001 ml), in order not to introduce an error of 0.1 in the specific gravity. Such precision cannot be obtained with ordinary pycnometers. In practice, errors substantially greater than 0.1 lambda result from irreproducible stopper closures and filling of the pycnometer, and sometimes also result from excessive evaporation during manipulation.

Several micropycnometers have been described. Syromyatnikov (1935) used a small tube having a volume of 0.07 ml and a glass plate as a closure. He obtained the weight of the filled tube by extrapolating the weight losses on standing. Using 40-mg samples of specific gravity 4, he claimed an accuracy of 0.3 per cent, taking into account only the errors of weighing. Bannister and Hey (1938) used a 0.5-mm capillary tube, closed at one end, as a pycnometer for 5 to 15-mg samples. The height of the liquid was measured under a microscope, and the specific gravity calculated with the aid of calibration data for various levels of filling. An accuracy of ± 0.5 per cent was attained. Ksanda and Merwin (1939) also used capillary tubing (1.6-mm bore) with a flat polished top. They filled the pycnometer with water and observed the illuminated meniscus as the water evaporated.

The pycnometer described here can be filled with a reproducibility much better than 0.1 lambda. The novel features are the reproducible closure by means of a metal ball, and the delicate method of detecting the end of the evaporation of excess liquid. The desirable rate of evaporation of tetrachloroethylene used as the displacement liquid allows excellent

[1] Published with permission of the Director, U. S. Geological Survey.

control of the evaporation step; its high specific gravity of 1.6 reduces the effect of small weighing errors.

Construction

Figure 1 shows the construction of the pycnometer. It is made from heavy-walled capillary tubing having a bore of 2 mm. The base is formed by sealing one end in a hot torch and then pressing the softened glass on a

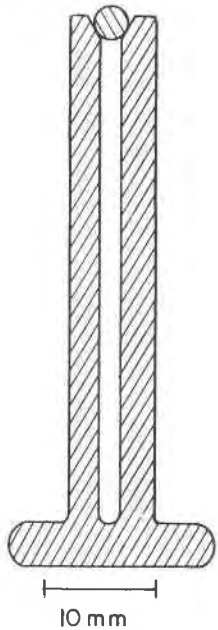

Fig. 1. Cross-section of micropycnometer.

graphite block. The open end is ground flat with a fine carborundum abrasive on a polishing table, and the outer edge then gently bevelled. The capillary is tapered by grinding with fine carborundum, using a 60° tapered copper tool on a lathe, and finishing with a polishing abrasive. The ball used for closure is a $\frac{1}{8}$-inch precision monel ball bearing.

When the ball is seated after filling the pycnometer, it displaces excess fluid without trapping any air. The precision fit of the ball and the tube results in a highly reproducible closure. If a liquid of moderate volatility is used, the liquid trapped above the contact circle between the ball and glass evaporates reasonably rapidly. Thereafter, the evaporation is sufficiently slow to allow time for accurate weighing on a projection balance.

The pycnometer shown in Figure 1 has a capillary height of 45 mm and

a volume of 144 lambda. This pycnometer was used in most of the experimental work described here. A shorter pycnometer having a capillary height of 25 mm and a volume of 81 lambda was also tested for reproducibility of filling.

Procedure

Weigh the pycnometer empty and with sample on a semimicrobalance (preferably one with a projected scale). Place the pycnometer in a small bell jar fitted with a Teflon stopcock. Evacuate the jar with a vacuum pump, and then deliver enough tetrachloroethylene through the stopcock to cover the sample. Place the pycnometer on a filter paper, and fill it completely with tetrachloroethylene. Use metal tipped tweezers to seat the ball on the tube. Wipe any overflow and remove most of the excess liquid on the lip of the pycnometer by gently dabbing with hardened filter paper. Place the pycnometer on the balance and observe through a magnifier the liquid-ball interface against a white background. Upon the appearance of a very small bubble just below the contact between the ball and the glass, record the weight. Empty the pycnometer, and fill with tetrachloroethylene; fill and weigh as described above.

Using the specific gravity of tetrachloroethylene at the appropriate temperature, calculate the specific gravity of the sample. A calibration curve should be prepared by measuring the specific gravity of the tetrachloroethylene at various temperatures, using a 10-ml-specific-gravity bottle.

We generally place the pycnometer slightly off center on the balance pan so that the bubble will tend to form on the front side. A bubble forming behind the ball will not be detected until it grows sufficiently to drop to the bottom of the ball.

Precision

The precision of the measurement of the volume of two pycnometers was estimated from repeated alternate weighings of the pycnometers empty and then filled by syringe with tetrachloroethylene. Nine such measurements with each pycnometer gave a mean volume of 143.57 lambda with a standard deviation of 0.044 lambda for the larger pycnometer and a volume of 80.82 lambda with a standard deviation of 0.016 lambda for the smaller one. The somewhat greater precision in filling the small pycnometer is in accordance with the experience of other investigators and is due to the smaller effect of temperature changes.

The specific gravities of six mineral samples were determined by the microtechnique described above on 9- to 12-mg samples and by a standard macrotechnique on 1-g samples. In the latter case, toluene was used

TABLE 1. COMPARISON OF MICRO- WITH MACROPYCNOMETRIC DETERMINATIONS OF SPECIFIC GRAVITY OF SELECTED MINERALS

Mineral	Determinations with micropycnometer		Determinations with macropycnometer (1-g samples)
	Wt taken (mg)	Sp gr	5 Sp gr
Mullite (fused)	11	3.27	3.249*
	12	3.20	
Zircon (−100+200 mesh)	11	4.60	4.64
	12	4.69	4.65
Garnet (−100+200 mesh)	12	4.19	4.21
	11	4.12	
Rhodolite (crushed)	12	3.83	3.85
	9	3.87	3.85
Quartz (−60+80 mesh)	11	2.69	2.64
	11	2.64	
Synthetic quartz (free from inclusions; 56%—97+200 mesh, 44%—200 mesh)	11	2.66	2.6510*
	11	2.61	

* Labeled value accepted.

as the displacement liquid. The results given in Table 1 show very satisfactory agreement. The standard deviation for the microdeterminations is 0.045.

REFERENCES

BANNISTER, F. A. AND M. H. HEY (1938) A new micro-pyknometric method for the specific gravity of heavy solids; with a note on the accuracy of specific gravity determinations. *Mineral. Mag.* **25,** 30–34.

KSANDA, C. J. AND H. E. MERWIN (1939) Improved micropycnometric density determinations. *Am. Mineral.* **24,** 482–484.

SYROMYATNIKOV, F. V. (1935) The micropycnometric method for the determination of specific gravities of minerals. *Am. Mineral.* **20,** 364–370.