

A TORSION MICROBALANCE FOR THE DETERMINATION OF SPECIFIC GRAVITIES OF MINERALS

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INTRODUCTION

The specific gravity of a substance, especially a crystalline substance, is one of its fundamental properties. Since *x*-ray crystal studies have appeared, the property has become of considerable importance because the volumetric relations of the *x*-ray cell and the composition are tied together by the specific gravity.¹

Specific gravities of minerals have been useful in determinative schemes, but the property has not been used extensively because: (1) there is a certain amount of variation in composition within the mineral species, (2) the existing data are not too reliable, (3) reliable values could only be obtained under exceptional circumstances, or with an expenditure of considerable time.

A satisfactory and practical method for measurement of specific gravity must provide:

1. Homogeneity of sample.
2. Simplicity and rapidity of measurement.
3. Significant accuracy.
4. Large range of application.

The writer believes that the new microbalance now in use in the Harvard mineralogical laboratory meets the requirements of a satisfactory method and, because the balance can give satisfactory results with small samples, it is especially useful to mineralogists.

METHODS IN GENERAL USE

The ordinary methods for the determination of specific gravity of solids are: (1) pycnometer methods, (2) suspension methods, (3) hydrostatic methods.

Pycnometer methods. The principal advantage of the pycnometer method lies in its use of powdered material. Under favorable circumstances, i.e., homogeneity and abundance of sample, no other method can yield such accuracy. The chief disadvantage of the method lies in the excessive care necessary and consequent time required to secure reliable results.

Recently Bannister and Hey (1938) and Winchell (1938) have described micropycnometer methods. As little as 5 mg. of solid can be used,

¹ $W = dVA$, where W = molecular weight of unit cell, V = volume of the unit, A = Avogadro number, d = specific gravity.

according to Bannister and Hey, with a computed accuracy of about 0.5 per cent, using a microbalance for weighings. Winchell used about 100 mg. of material with a comparable accuracy, using an ordinary balance.²

The Bannister and Hey method is apparently of some complexity, since it involves either a centrifuge process or special evacuation methods to free residual air bubbles without ejection of the solid. The Winchell method cannot conveniently handle less than the specified amount of material (100 mg.). The least time required for a determination (after considerable experience and using a previously calibrated pycnometer) is about an hour.

Suspension methods. The so-called suspension method has as its only serious limitation the restricted range over which suitable liquids are available. Clerici solution and a number of other heavy liquids³ are suitable, but such minerals as the sulphides and oxides fall out of the range of the liquids. The liquids of highest specific gravity sometimes react with the sample and thereby further restrict the usefulness of the method.

Minerals of high specific gravity can be brought into the range of the liquids by attaching a glass float of known weight and density to the mineral sample. The combined mineral and float are then measured, and the specific gravity of the mineral can be readily computed. The accuracy obtainable is about that of the micropycnometer methods, i.e., about half a per cent.

In general, a good determination with the suspension method requires about an hour, or perhaps somewhat less, under favorable conditions.

Hydrostatic methods. The method of hydrostatic weighing is the simplest of the three principal methods here discussed. Only two weighings are necessary, in air and in the liquid. The only limitations to accuracy are weighing difficulties and surface tension effects. The former may be

² The evaluation of errors, in the pycnometer method, due to weighing and temperature uncertainties, is given by the expression (Winchell, *Am. Mineral*, **23**, 805-810, 1938):

$$\frac{dS}{S} < \frac{dW_1}{W_2 - W_1} + \frac{dW_2 + dW_3}{W_2 + sv - W_3} + \frac{(W_2 - W_1)ds}{(W_2 + sv - W_3)s}$$

where

- S = specific gravity of powder
- s = specific gravity of the liquid used
- W₁ = weight of pycnometer empty
- W₂ = weight of pycnometer with powder
- W₃ = weight of pycnometer with powder and liquid
- v = volume of pycnometer.

³ See Rosenbusch-Wülfing, *Microskopische Physiographie*, v. 1, pt. 1, 678, Stuttgart, 1921-24, for a general description of various liquids.

overcome by using a microbalance for small samples, and the latter by utilizing a liquid of low surface tension together with a suitably designed set of pans for weighing both in and out of the liquid.

The crudest of hydrostatic weighing devices can yield fairly good results on large enough samples, and is therefore suitable for the determination of the specific gravity of rocks. Many minerals are, however, unattainable in homogeneous fragments larger than 20 to 50 mgs. Experience in this laboratory has shown that reliable samples greater than 50 mgs. are rare.

The Jolly balance can conveniently handle a sample from 1 to 45 gms. and yield an accuracy of about 0.2 per cent with a 1 gm. sample of specific gravity 5.⁴

A microbalance with a sensitivity of 0.01 mg. handling 25 mg. of material, where the density of the liquid is correctly known to 0.001 and the sample has a specific gravity of 5, will yield an accuracy of about 0.2 per cent. This does not take into consideration a surface tension effect, but experience with such a balance in the Harvard laboratory has shown that this effect can be minimized by using a suitable pan and liquid, as described below.

THE TORSION MICROBALANCE

Description. The instrument shown in Fig. 1 is a modification of the Roller-Smith type "C" microbalance.⁵ The range chosen for specific gravity measurements is to 25 mgs., with a vernier scale reading to 0.01 mg. The specified accuracy is 0.01 mg. The range of the balance may conveniently be increased to about 75 mg. by hanging suitable counterweights on the arm (*A* of Fig. 1). At *B* is hung a double weighing pan, made of .005" platinum wire, coiled to make a basket at the bottom (Fig. 2) and holding an aluminum pan in its middle. The total weight is around 15 mg., and a tare hung at *A* is used. For weighing many small fragments together a basket is provided (Fig. 3). This is made of

⁴ In the equation for hydrostatic weighing methods

$$S = \frac{WD}{W - W_1}$$

where *S* = specific gravity of mineral, *W* = weight of mineral in air and *W*₁ = weight of mineral in liquid of density *D*, the limitations of accuracy due to weighing and liquid density effects may be expressed approximately as follows:

$$dS = \frac{WdD + DdW}{W - W_1}$$

⁵ Made by the Roller-Smith Company, Bethlehem, Pennsylvania, and distributed by Baird Associates, Cambridge, Mass.

220-mesh screening and weighs about 10 mg. The basket is hung on the top and bottom hook successively for weighings in and out of the liquid. The liquid is held in a glass dish which may be lowered and elevated by

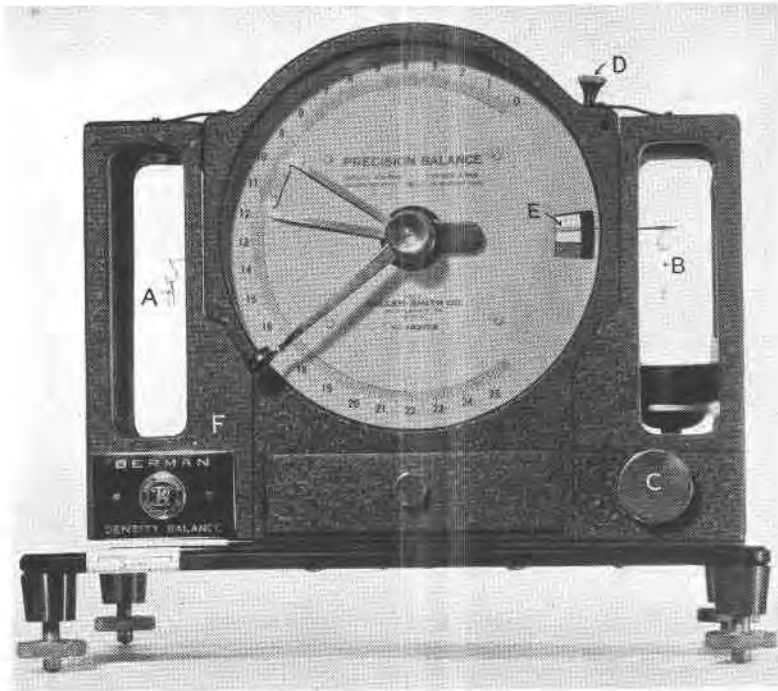


FIG. 1. Torsion Microbalance.



FIG. 2. Double weighing pan for single fragments.

FIG. 3. Basket and wire hook for coarse powder weighings.

the knob, *C*, in order to facilitate loading the bottom pan. A conveniently located knob (*D*) enables the operator to adjust to a proper zero point (*E*) of the scale. At *F* a release (not seen in the figure) for the balance beam is provided.

After considering possible mechanical devices for loading single specimens on the upper and lower pans, all were rejected in favor of a small chute and a needle. The specimen is rolled from the chute (made of a folded piece of paper) onto the upper pan for the first weighing, and is pushed back on the chute with a needle after the weighing. On lowering the vessel carrying the liquid, the specimen is again rolled into the lower basket. From the latter the specimen cannot easily be pushed out but the weighings are completed, and the whole pan may then be lifted from the beam and inverted to expel the specimen.

A mark scratched on the vessel containing the liquid can be brought to the same place with respect to the pans for every reading. This cancels surface tension effects, and the weight of the immersed wire.

Toluene has been adopted as the displacement liquid for several reasons, most important of which is its low surface tension ($29 \pm .01$; water = 73). The temperature coefficient⁶ is such that little effect in the specific gravity can take place over the time of the experiment. Toluene of high grade may easily be obtained, and variations in specific gravities of different batches are small in terms of the accuracy obtainable with the balance. Carbon tetrachloride has also been found to be satisfactory.⁷

Advantages of the method. The method here described has as its chief advantages the following: (1) a small sample may be used; (2) a fair degree of accuracy is attainable; (3) the technique is simple and rapid; (4) it can be applied over the whole range of specific gravities of solids.

The use of a small sample is a great advantage because few minerals are homogeneous in bulk. Further, when single crystals are used for crystallographic, optical and x-ray work, the specific gravity of the same crystal can be determined. If a number of crystals are available, the method is still of advantage since a dozen measurements on separate samples can be made in the time usually given to making one measurement by other methods, and the averaged accuracy is high.

As before stated, the obtainable accuracy is 0.2 per cent with a 25 mg. specimen of specific gravity 5. Using a large number of fragments of total weight of 25 mg., specific gravity 5, and weighing in a basket, the accuracy is reduced to about 1 per cent. A complete determination takes about five minutes, and the technique is so simple that a student without experience can get good results at the first attempt. The instrument

⁶ The following expresses the variation of toluene with temperature: $d_t = d_s + 10^{-3} \alpha(t - t_s) + 10^{-6} \beta(t - t_s)^2$ where $d_s = 0.8845$, $\alpha = -0.9159$, $\beta = +0.368$, $t_s = 0^\circ\text{C}$. From International Critical Tables, 3, 29 (1928).

⁷ For carbon tetrachloride, $d_s = 1.63255$, $\alpha = -1.9110$, $\beta = -0.690$.

itself is rugged and apparently is not easily thrown out of adjustment.

Examples and new data. In the following table are listed a portion of the routine measurements made in the Harvard laboratory. Where the calculated value from *x*-ray measurements is known, that value is given by way of comparison. Some of the listed minerals have been first measured, with some degree of accuracy, by this method. Most of the minerals, with the exception of the test samples, were measured in order to improve existing data which appeared imperfect as given in the literature.

<i>Mineral</i>	<i>Measured</i>	<i>Calculated (X-ray)</i>	<i>Per cent of error</i>	<i>Wgt. of sample in mg.</i>	<i>Remarks</i>
Albite	2.59			20	Ontario. Nearly pure. Coarse powder.
Antlerite	3.88 ± .005	3.93	1.29	31-6	
Baumhauerite	5.24			6	
Brochantite	3.97 ± .01	3.98	0.25	15-20	4 determinations.
Calcite	2.72	2.73	0.47	24	
Cobaltite	6.33	6.30	0.48	25	
Colusite	4.50	4.43	1.58	10	
Diamond	3.52	3.511	0.40	8	
Dyscrasite	9.82	9.85		70	Andreasberg.
Galena	7.58 ± .01	7.57	0.13	33-19	2 determinations.
Glaucodot	6.16 ± .02			4.56	Hakensbö, Sweden.
Gratonite	6.24 ± .01	6.17	1.12	14-8	2 determinations. Type.
Hopeite	3.04	3.08	1.32	12	Pycnometer measurement on 1.035 gms. gave 3.04 (Spencer).
Kallilite	6.66				
Kermesite	4.68	4.69	0.21	8	
Kröhnkite	2.90 ± .02	2.95	1.70	14-10-9	3 determinations.
Loellingite	7.40				3 determinations (29.40% Fe).
Loellingite	7.48				4 determinations (27.89% Fe).
Magnetite	5.00			29	Coarse powder.
Marshite					
Muscovite	2.84			13	Ontario. Coarse powder.
Nagyagite	7.40			15	
Natrochalcite	3.49 ± .01	3.54	1.43	20	3.476 from 2½ gms. by pycnometer.
Nepheline	2.62			18	Ontario. Coarse powder.
Pearceite	6.10			24	
Penroseite	7.05 ± .05			38-34-68	Hey's average 6.86.
Pyrostilpnite	5.94			5	
Quartz	2.65			21	Coarse powder.

<i>Mineral</i>	<i>Measured</i>	<i>Calculated (X-ray)</i>	<i>Per cent of error</i>	<i>Wgt. of sample in mg.</i>	<i>Remarks</i>
Salesite	4.77 ± .05	4.89	2.51	9	3 grains weighed together. Type.
Sphalerite	4.10	4.083	0.42	20	Santander.
Ullmanite	6.65	6.79	2.06	20	4 determinations.
Wagnerite	3.153 ± .003	3.158	0.16	15-23	4 determinations.

REFERENCES

- BANNISTER, F. A., and HEY, MAX H.: *Mineral Mag.* **25**, 30 (1938).
 WINCHELL, HORACE: *Am. Mineral.*, **23**, 805 (1938).