

ART. XLV.—*A Method of Determining the Density of Minerals by means of Rohrbach's Solution having a Standard Refractive Index*; by H. E. MERWIN.

Two methods of finding the density of minerals or glasses which are in granular form, or which require to be granulated in order to be made homogeneous, are in general use—the pycnometer method and the suspension method.¹ Fine powders (grains less than about $\cdot 1$ or $\cdot 2^{\text{mm}}$ in diameter) cannot be used successfully with the suspension method on account of the disturbing effects of convection currents and viscosity in the suspending liquid, and of flocculation of the solid particles. The pycnometer method is a universal method if sufficient material (a few grams) is available. The method is laborious, and requires much skill and standardized apparatus. The suspension method, with the same skill and care as to the adjustment of the instrument used, should yield equally accurate results for much less labor, provided the material may be obtained in sufficiently large homogeneous grains.

The difficulty of convection currents in the liquid can be largely overcome by preventing evaporation and by surrounding the cylinder in which the liquid is held with a closed vessel of air or water. The proper adjustment and standardization of the specific gravity balance by means of which the density of the liquid is obtained are, of course, essential. Some of the more sensitive balances cannot be adjusted and tested without the use of special devices not furnished with the balances. If properly adjusted, the best of these balances are sensitive to $\cdot 0003$, but errors in the graduation of the beam often amount to $\cdot 001$. The density of a liquid may be correctly determined within $\pm \cdot 001$ with such a balance. If the grains of material suspended in the liquid are not moved about by convection currents, then the density of the grains may be matched by the liquid much closer than $\pm \cdot 001$. A disadvantage of the specific gravity balance is that the column of liquid must be deep enough to receive the sinker; this depth increases the possibility of convection currents.

An alternative method of finding the density of the liquid in which the grains are suspended, which depends upon the relation of refractive index to density, has been found to be expeditious and accurate. The liquid is contained in the small glass cell accompanying the standard refractometers, and its

¹ Recently J. L. Andreae has described a method of finding the density of suitable solids correctly within $\pm \cdot 0001$ by suspending a fragment of the solid in a heavy solution in a dilatometer. *Zeitschr. phys. Chem.*, lxxvi, 491-496, 1911.

refractive index is determined while the grains are suspended in it.

A liquid having as great a range of refractive index as possible for a given difference of density is most advantageous for accuracy. The liquid should have also a wide range of density. Further requirements are convenience in attaining a standard condition, and permanency. Rohrbach's solution of barium-mercuric iodide meets these requirements.

Rohrbach's solution as ordinarily prepared² and as sold by the dealers has a maximum density of about 3.5. To standardize the liquid it is diluted, at a temperature of 19° to 21°, with water, to the density of clear crystals of sulphur (about 2.07), or until the refractive index for sodium light is 1.510 to 1.520. The solution is cleared of the precipitated mercuric iodide, a part is left dilute and the remainder concentrated by evaporation. But if crystals are present in the cold concentrated solution, enough water should be added to dissolve them, for they differ in composition from the liquid above them. Mixtures of these solutions thus prepared have a fixed density (correct to ± 0.001) for a given index of refraction. If desired, the concentrated solution may be diluted with water if it is stirred rapidly to prevent precipitation of mercuric iodide; it is to be noted, however, that a very slight precipitate on the bottom of the cell of the refractometer greatly obscures the signal.

The prepared Rohrbach's solution or the materials for making it may be obtained from the dealers sufficiently pure³ for the preparation of the standard solution. As a check the purity of the solution should be tested by bringing it to the density of clear quartz, 2.6495, and determining its index of refraction ω for sodium light at 20°. This index should be 1.6208.

The relation of density to refractive index of this solution at 20° was found by means of a standard refractometer giving results correct to ± 0.0015 , and indicators of standard density, correct to ± 0.0015 .

The results are contained in Table I.

These results may be plotted on coördinate paper and connected by a curve; or their relations may be stated in a simple

² By rapidly mixing 100 g. BaI₂ and 130 g. HgI₂, adding 20 cc of water, then placing immediately in an oil bath at 150° to 160°, and agitating till boiling and solution take place. The solution may be prepared equally well by rubbing the constituents together in an evaporating dish over a hot water-bath.

³ Barium iodide may be strongly colored by iodine. Washing with benzene, xylol or ether will remove the iodine.

If the heavy liquid becomes discolored it may be cleared by shaking with mercury. Restandardizing is not necessary unless the solution was deeply colored.

empirical formula which gives the density,⁴ d , between 2.25 and 3.40 at 20° with a maximum probable error of ± 0.002 .

$$d = 5.39(n - 1.5467) + 2.25$$

which in a simpler form is $d = 5.39n - 6.0865$. For accurate work, if the temperature is more than 3° from 20° a correction for d of -0.001 for each 2° below 20° and of $+0.001$ for each 2° above 20° should be made.

TABLE I.

Density at 20° C.	Refractive index.
3.449	1.7686
3.396	1.7590
3.246	1.7312
3.180	1.7195
3.046	1.6944
2.980	1.6823
2.748	1.6391
2.649	1.6207
2.648	1.6205
2.367	1.5685
2.163	1.5320
2.067	1.5148

V. Goldschmidt⁵ constructed a similar table for a solution of potassium-mercuric iodide. The standard solution is difficult to prepare and maintain.

At given densities the observed refractive index varied with the temperature as follows:

Density.	Refractive index.	Temperature.
2.067	{ 1.5147	23
	{ 1.5150	20
2.367	{ 1.5695	14
	{ 1.5685	20
2.748	{ 1.6398	13
	{ 1.6391	20
3.449	{ 1.7690	16
	{ 1.7686	20

In using the cell with the refractometer it is more convenient to use only one part of the vertical circle scale. If accuracy within ± 0.002 is desired, it is then necessary that the

⁴ The formula between 2.0 and 2.25 is $d = 5.7n - 6.567$, and between 3.4 and 3.5 is $d = 5.52n - 6.313$.

⁵ N. Jahrb. f. Min., Beil.-Bd., i, 234, 1881.

accuracy of the refractometer be tested, for errors in the adjustment of the cross-hairs and of the vernier are not compensated as they are when both parts of the circle are used. The testing may be done by observing the error of the instrument for ωNa of quartz, 1.54425. A drop of the concentrated solution may be used between the cell and the hemisphere. The trouble from diffraction bands may be largely avoided by placing a triangle of thread between the cell and the hemisphere.

Before finding the density of grains of a material the grains should be examined under the microscope in a watch glass of xylol or alcohol to make sure of freedom from foreign matter. So little attention has been given to such an examination that many of the recorded densities of minerals which have been carefully analyzed are in error .5 per cent or more. Greater error is apt to come from lack of care in selecting material than from other causes.

If only the approximate density of a substance is desired, it may be found quickly by matching its density approximately with that of the heavy solution, placing a drop of the solution on the refractometer, covering with a glass slip (to prevent evaporation) and determining the index of refraction.

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