

FIG. 1. Theoretical quartz percent (by volume) associated with plagioclase of varying An content. The curve is based on calculations involving the following unmixing relationships (Phillips, 1964):  $\text{NaAlSi}_3\text{O}_8 \rightarrow \text{NaAlSi}_3\text{O}_8 + \text{OSiO}_2$ ,  $\text{Ca}(\text{AlSi}_2\text{O}_8)_2 \rightarrow \text{CaAl}_2\text{Si}_2\text{O}_8 + 4\text{SiO}_2$ .

kites from other areas may further substantiate this relationship. The project noted here forms part of a wider structural investigation at Broken Hill and this will be presented at a later date, Ranson (1968).

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#### POWDER DENSITY MEASUREMENT BY HYDROSTATIC WEIGHING

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#### INTRODUCTION

The hydrostatic method is usually used in the laboratories for the determination of solid and liquid densities (Hidnert and Pepper, 1950; Fahey, 1961; Thewlis, 1961; Guillemin, 1962), but it is not often used for

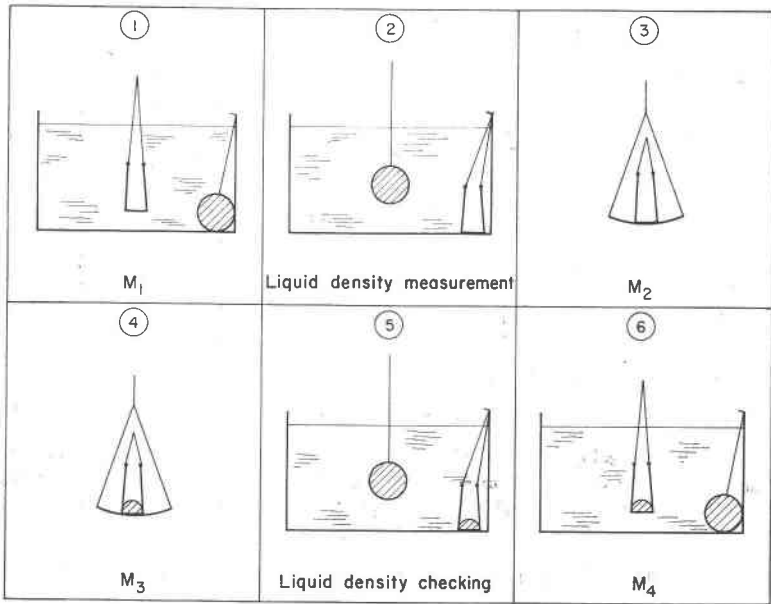


FIG. 1. Principle of measurement.

powders. With some simple technical adaptations, this method can give a more accurate value of density and, at the same time, be easier and faster than the pycnometric method usually used. It is well known that the pycnometric method can only give accurate results after very long and delicate manipulations (Guillemin, 1952). There are a number of errors involved in this method; namely, the determination of the liquid level at the mark, and the effect of temperature on the density of the measurement liquid. These errors are eliminated in the hydrostatic method due to the replacement of volume measurement by weighing, and the stabilization and the simple measurement of the liquid density. The accuracy of the method will then depend on the precision of the balance.

#### PRINCIPLE OF MEASUREMENT

The determination of the density of a powder by the hydrostatic method is schematically shown in Figure 1 (Fahey, 1961).

$M = M_3 - M_2$  represents the weight of the powder,  $m = M_4 - M_1$  the apparent weight of the immersed powder,  $\rho_1$  the density of the measurement liquid and  $a$  the air density in the experimental condition. After air buoyancy correction, the powder density  $\rho$  is given by

$$\rho = \frac{M\rho_1 - ma}{M - m}$$

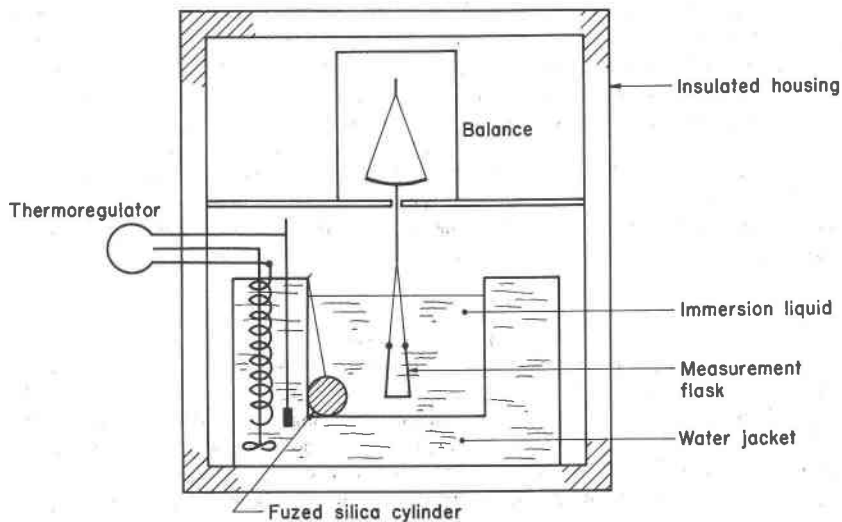


FIG. 2. Schematic of experiment apparatus.

Thus the only delicate parts of the method are the weighings of  $M_1$  and  $M_4$  and the measurement of liquid density  $\rho_1$ .

*Weighing of  $M_1$  and  $M_4$ .* The measurement flask is suspended twice, once without powder (1-Fig. 1), and once with (6-Fig. 1), by means of two thin (1/10 mm diameter or less) metallic wires of tungsten (Guillemin, 1962), or of platinum alloy with 20 percent of iridium (Thewlis, 1961).

*Measurement of the liquid density.* The measurement of the liquid density is easily found in the following manner. A vitreous fused silica cylinder,<sup>1</sup> the volume of which is well known (by hydrostatic weighing in pure water), is constantly immersed in the measurement liquid. Before and after each hydrostatic weighing (2 and 5-Fig. 1), this cylinder is suspended under the pan of the balance in order to measure and verify the liquid density at the same time.

#### APPARATUS

A schematic drawing of the experimental apparatus is shown in Figure 2. A Mettler balance (Model H 15) was used with its special equipment for weighing under the weighing compartment.

Two types of flasks may be used for 5- to 10-g samples of powder: glass flask and aluminum tubes. Glass flasks have the advantage that degassing and immersion of the powder can be observed, but unfortunately they are heavy and brittle. For coarse powders, an aluminum tube is recommended, whereas for fine powders, it is better to use a glass flask in order to observe the degassing process.

Carbon tetrachloride and xylene are often used in the hydrostatic method because they prevent any chemical or dissolving action of most of the mineral powders and have a good

<sup>1</sup> Vitreous fused silica has one of the lowest linear thermal coefficient of dilatation  $\alpha = 0.42 \cdot 10^{-6} \text{ C}^{-1}$ . For Invar  $\alpha, \alpha = 0.9 \cdot 10^{-6} \text{ C}^{-1}$  (Strong, 1963).

wetting power. The high density of carbon tetrachloride suggests its use (the higher the density of the liquid measurement the lower the relative error  $\Delta\rho/\rho$ ) but, extreme volatility and toxicity render it unsuitable for permanent installation. It is better to use xylene since it is less dense, but has, on the other hand, a very small viscosity coefficient  $\eta=0.6$  cp that permits a relatively quicker settling for very thin powders.

Usually a suitable stabilization of the temperature is obtained by the use of a large volume of liquid (4 to 5 liters) surrounded by a water-jacket, the temperature of which is maintained within  $\pm 0.04^\circ\text{C}$ . All the equipment mentioned above must be kept in an insulated housing. The use of a thermoregulator is necessary when one seeks an accuracy better than  $\pm 0.001$  in density measurement.

### EXPERIMENTAL PROCEDURE

It is necessary to have a good wetting of the powder with no loss during the immersion process. Wetting of powder under vacuum is not desirable because there are too many experimental complexities. There are also risks of violent boiling that produce unmeasurable losses of powder. Better results may be obtained by pouring xylene to the half mark of the flask containing the powder and then shaking it; mechanically for the coarse powders, and ultrasonically for the very fine ones. Before the immersion of the flask containing fine powders, it is preferable to fill the bottle with xylene and let the powder settle completely.

### RESULTS

Table 1 shows three series of results for different powders at  $20^\circ\text{C}$ . The weighings have been done on 5- to 10-g samples, the measurement liquid was xylene and the powders were degassed by ultrasonics. The duration of each series was about three hours.

The precision of the results can surely be improved by the use of a more accurate balance and by changing xylene by carbon tetrachloride (with the inconveniences noted above).

### CONCLUSION

The main aspect of the hydrostatic method consists of obtaining accurate value of the liquid density under the same temperature conditions

TABLE 1

Trials	Calcite $d < 50 \mu$		Cement <sup>a</sup>
1	2.7147	2.7142	3.0789
2	2.7144	2.7146	3.0791
3	2.7147	2.7142	3.0791
4	2.7138	2.7143	3.0792
5	2.7140	2.7144	3.0792
6	2.7140	2.7147	3.0796
7	2.7144	2.7146	
8	2.7141	2.7144	
Average	2.7143	2.7144	3.0792
Absolute dispersion	$9 \cdot 10^{-4}$	$5 \cdot 10^{-4}$	$7 \cdot 10^{-4}$

<sup>a</sup> Standard CPAC 325 NF P 15-302, Papadakis and Venaut (1966).

as the powder, and the use of ultrasonics for degassing. Consequently the hydrostatic method has a substantial advantage when a series of accurate determination of powder density is desired. This method leads to simple and rapid manipulations that reduce human errors considerably in contrast to the pycnometric method.

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## ANTIMONIAN GROUTITE: A CORRECTION

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Dr. L. G. Berry has drawn my attention to several errors and omissions in the indexing of the X-ray diffraction pattern of antimonian groutite (Klein and Frondel, 1967). Corrected indices for several measured interplanar spacings are as follows:  $200+140-2.2881$ ;  $230+150-1.9199$ ;  $240-1.7271$ ;  $231+151-1.5992$ . Using the measured interplanar spacings of 021, 111, 140, 200, 230, 211, 240, 221, 151, 310, and 330 in a unit-cell refinement program, the unit-cell becomes:  $a = 4.568 \pm .002 \text{ \AA}$ ;  $b = 10.581 \pm .011 \text{ \AA}$ ;  $c = 2.885 \pm .003 \text{ \AA}$ .

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