SURVEY OF FACTORS CONTRIBUTING TO VARIABLE MICA BULK DENSITY READINGS

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Background of Tests

In many cases, when the Scott Volumeter has been used in the past to measure the bulk density of minus 20 mesh mica, the observed readings have been of doubtful value because it could be proven in many cases that an aliquot of one coarse mica sample could be run through the volumeter several times in succession and could yield a quite different bulk density reading each time, regardless of efforts to employ identical technique. This limited severely the utility of the test when applied to quality control or research. The Scott Volumeter was originally designed to evaluate material of much finer mesh size, and so the technique for evaluating bulk density of fine material, as well as this instrument, might logically be expected to have shortcomings when applied to relatively coarse mica.

Object of Tests

It was hoped to find a procedure giving reproducible bulk density readings for any given sample of minus 20 mesh flotation-type mica in the general range of 30 to 40 pounds per cubic foot. A further object was to determine what procedures in testing were most important in maintaining reproducibility. A still further object was to see whether differences in handling or sample preparation would affect bulk density reading of a single sample.

Sample Used

The sample used was a green muscovite mica froth product from a plant in the Spruce Pine area, North Carolina, which produces this concentrate. It was collected in March 1966 after passing through both rougher and cleaner circuits, plus a wet-screening operation to remove some minus 60 mesh material, presumably largely non-micaceous grit. Oversize from the screening operation mentioned was collected wet, and aliquots dried in several different ways. The mica was originally one component of an alaskite ore whose mineral parts can be largely liberated at 20 mesh. The mica sample was of minus 20 mesh size. Total sample was about 20 pounds, dry weight. Being the froth product of an acid-amine-fuel oil flotation operation, this mica, wet, exhibited an oily characteristic, indicating reagent adherence (amine and/or fuel oil) to its surfaces.

Screen analysis showed 71.5 percent plus 60 mesh (almost all 20-60 mesh), 19.6 percent 60-100 mesh, and 8.9 percent minus 100 mesh. A mineralogical check of these three screen products gave the following results:

plus 60 mesh	93% mica,	7% non-micaceous grit
60-100 mesh	85% mica,	15% non-micaceous grit
minus 100 mesh	92% mica,	8% non-micaceous grit
total product	90% mica,	10% non-micaceous grit

The mica-grit percentages are assumed weight ratios derived from correction factors applied to particle count. They are cited only to show the general characteristics of the sample.

Original Test Procedure

The Scott Volumeter, designed to test fine-ground or powdered samples, is equipped with a funnel divided by a screen of about 60 mesh size. The sample aliquot is supposed to be placed upon this screen surface and brushed through the screen with a fine brush, causing it to fall, bounce, and disperse upon the glass baffle plates below, go through the bottom aperture, and fall into the one-cubic-inch container underneath. When the operator has brushed through enough sample to fill the volumetric container up to or beyond all its upper edges, the container is removed carefully, leveled with a scraper, tapped to compact the sample and avoid loss, and weighed. The net weight of the sample, in grams, can then be converted to pounds per cubic foot with a factor of 3.81.

The use of this volumeter to evaluate flotation mica of minus 20 mesh size required a change of screens. A combination 20 mesh screen and funnel was built to deliver a loose mica product to the glass baffles, and the sample brushed through this. At this point, with no additional refinements, bulk density readings on the sample varied between 40.2 and 46.4 pounds per cubic foot, which underlined the need for refined procedures.

Standardization of Procedures; Causes of Variable Results

As one variable, the mica sample was first screened through 20 mesh, then poured freely through the volumeter in an uninterrupted flow, at a fast rate. A bulk density reading following this technique gave a considerably lower figure. A standard procedure was then worked out, as follows:

> About 150 grams of mica sample was split out, rolled, and spread flat on a sheet of paper. An aliquot of 15.5 grams was dipped out upon a sensitive (± .01 g.) beam balance. This was dumped into a glass funnel with a stem one-inch long with an inside stem diameter of 11/32-inch, held shut at bottom with a finger. The sample was released over the funnel holder (minus screened funnel) of the volumeter and allowed to flow through uninterrupted. The glass funnel emptied in about two seconds. In several instances where "bridging" of sample chanced to occur in the glass funnel and interrupt flow, the test was voided.

The one-cubic-inch container underneath, resting on a small tray with raised corners, was gently removed avoiding jarring or tilting, by grasping the tray corners. The contents of the container were then leveled with a spatula edge, the blade being held as close as possible to exactly vertical and moved in gentle contact with the top edge, in a steady sweep between two opposite sides, and parallel to them (not diagonally). The container was then tapped to sink the leveled sample, and weighed to determine net sample weight and bulk density.

Using the procedure just described, a high degree of reproducibility was obtained (see Table 1, following).

Certain tests which involved slow or interrupted flow of sample ("bridged" sample in funnel, or brushing through a screen directly into the volumeter), gave a higher bulk density(Table 2, following). Also, results were quite variable.

Using the preceding rapid free flow technique, the variation between bulk density readings using 14.5 and 20 grams of sample did not vary appreciably. Standard tests used 15.5 grams. See Table 3.

The same sample (No. 3056), when dried at different temperatures, gave decidedly different readings (Table 4).

The standard technique described gave reproducible readings on the following screen fractions of the sample: plus 60, 60-100, and minus 100 mesh (Table 5).

The presence of reagent on the sample (specifically, oil and/or amine) caused a lower bulk density reading. Portions of the sample which had been (1) air-dried and (2) dried at 200^{o}F were agitated, washed, and rinsed with ether, using filter paper to drain. Bulk density tests run after drying gave higher readings (Table 6).

Tables and Further Observations

Table 1

Reproducibility of Results, Standard Procedure
Sample No. 3056-B (Dried at 200^{o} F)

No. of Tests	Bulk Density Rdg.	
		Total No. of Tests
7	39.6	22
4	39.8	
7	39.9	Average Bulk Density
3	40.1	
1	40.2	39.9

Comments, Table 1

The preceding 22 standard tests were run by two different persons over a period of three days. The sample was left open in a large room at about 70°F. There was little or no wet weather during this period. Indoor heat was on.

The variations in bulk density shown had no correlation with the chronology of testing, the person carrying out the test, or any other recognizable factor.

After about 20 bulk density tests of one sort or another had been carried out on one particular 150-gram portion of sample No. 3056-B, a slight upward trend (to 40.4) in bulk density developed. When a new portion was used, this immediately returned all readings to previous limits. It is surmised that, after a great deal of handling, some fine-mesh light mica escapes, and this loss is shown in the heavier reading.

Table 2

Reproducibility of Results, Sample Brushed Through Screen

No. of Tests	Bulk Density Reading
1	40.2*
1	43.6
1	43.8*
1	44.4
1	44.7*
1	44.8
1	45.0
1	45.4*
1	45.6*
1	45.7*
1	46.4
1	46.6*

*Indicates test where exactly 15.5 gram sample was put through. Other tests were with estimated weight sample.

Comments, Table 2

When the mica sample was brushed through a screen down into the volumeter, a widely variant factor was introduced, causing a range of readings differing by over six pounds per cubic foot, and averaging 44.7 pounds per cubic foot as opposed to 39.9 (as Table 1). The varying factor is apparently the interrupted or fluctuating fall of material down through the volumeter, which is not controllable as a constant. Interrupted fall creates a series of impacts upon the portion of sample already in the container, compressing it tighter and giving a higher bulk density reading. The total energy of these impacts will vary from one test to another and cause variable results.

Table 3

Effect of Varying Weight of Sample

No. of Tests	Weight, Sample (grams)	Avg. Bulk D., lbs/ft^3
4	14.0	40.2
22	15.5	39.9
4	16.5	40.1
4	20	40.2(2), 40.5(2)
1	30	40.1

Comments, Table 3

A substantial increase in weight of sample may raise bulk density reading slightly, but it appears to be a minor factor. The same high reproducibility was obtained in all groups.

Table 4

Effect of Variable Drying Conditions, Sample No. 3056

Sample No.	Drying Conditions	No. of Tests	lbs/ft^3 Avg. Bulk D.
2056-A	Air-dried for 6 days, 72°F.	6	36.3
3056-B	Dried overnight, 200-210°F.	22	39.9
3056-C	Dried for 4 hrs., 550°F.	5	36.9

Comments, Table 4

All test groups had the same degree of reproducibility: $\pm$ 0.3 pounds per cubic foot. The lower bulk density of the air-dried sample is probably due to the presence of a larger amount of residual oily reagent. The action of this reagent might be explained in terms of physical swelling of the mica from reagent absorbtion, or on the basis of electrostatic repulsion, or by some other theory. But when drying in the 200°F range occurs, the reagent seems altered or removed to some degree: the physical appearance and feel of the sample are changed. Verification of this would depend on careful chemical tests.

The "C" portion of the sample, dried more at intense heat, had a charred appearance, and had lost any physical feel of oiliness. Its lowered bulk density was considered to be due to expansion caused by escape of water of hydration. Reagent removal by solvation raises measured bulk density (Table 6), so some such factor must apply here. A mineralogical microscopic examination of the A, B, and C sample portions indicated that laminar expansion had definitely occurred in the case of the "C" sample.

Table 5

Standard Technique on Separate Screen Fractions, Sample 3056-B

Screen Fraction	No. of Tests	Avg. Bulk Density
Total	22	39.9
Plus 60	4	38.8(2), 38.9(2)
60-100 mesh	4	38.1(1), 38.4(3)
Minus 100	4	37.1(36.9-37.4)

Comments, Table 5

Reproducibility remained high for the screen fractions shown. An interesting phenomenon is the lower measured bulk density of all three fractions compared to that of the total. It seems likely that, in the case of some coarse mica samples, more space is taken up in the container by solid mica when a wide size range is tested: i.e., the finer mica can work into smaller spaces between the coarser mica if the fine fraction is part of the sample. This situation may not necessarily always be true: for example, if a large amount of grit is present in finer fractions, their bulk densities will be highest to start with; or else a coarse fraction which is very booky can have a higher bulk density than the total sample.

Table 6

Samples 3056-A and 3056-B, Washed With Ether and Dried (Standard Procedure)

Sample No.	Treatment	No. of Tests	lbs/ft^3 Avg. Bulk D.
3056-A	Air-dried	6	36.3
3056-A	Air-dried, ether wash	2	42.6 (both)
3056-B	Dried, 200°F	22	39.9
3056-B	Dried, 200°F, ether wash	3	42.2

Comments, Table 6

When reagent is removed, the bulk density reading goes up. Ether seems to bring about the same reading whether the sample is dried at room temperature or in the neighborhood of 200°F. Comments made following Table 4, regarding possible reasons for correlation of bulk density and reagent coating, apply here also.

Subsequent De-Oiling Research

Because the process of washing with ether is bothersome and hazardous, a technique of light scrubbing with anionic surfactant was tried. One-hundred-fifty to two-hundred grams of wet mica concentrate, as received from the plant, was scrubbed in a 600 ml. glass beaker at about 40 percent solids with one gram of 85 percent active sodium dodecylbenzene sulfonate for two minutes, using a laboratory conditioner with four approximately 2½-inch blades at 45° pitch turning at 700 rpm. Following this, the sample was placed in a ten-quart bucket, which was filled with water and agitated, then settled for one-half minute and poured across a 325 mesh screen. These steps of dilution through dewatering were then repeated, following which the sample(dewatered as much as possible)was placed in an aluminum pan in a layer one-quarter-inch to one-half-inch thick and dried at 200°-210°F for approximately 15 hours. After cooling and standing for two hours, the sample gave an average bulk density reading of 42.8 pounds per cubic foot, which compares favorably with the ether wash tests shown on Table 6.

Scrubbing for longer periods (5-7 minutes) or at thicker pulp (60 percent solids) resulted in lighter bulk density readings, indicating the probability of delamination, which must be avoided.

The foregoing procedure, then, can presumably be used to easily reduce a mica flotation concentrate to a condition whereby it can be evaluated without the influence of reagent coatings, extreme drying conditions, etc.

Summary and Conclusions

In obtaining reproducible bulk density test readings on a single given sample of minus 20 mesh mica flotation concentrate, the following controls appear important, in the order given:

1) Constant and uninterrupted fall of the sample through the volumeter

2) Constant quantity (or relative absence) of residual reagent on the mica particles

3) Constant drying temperature

4) Fairly constant sample weight delivered through the volumeter.

If mica bulk density measurements are to be used as a criterion in research or quality control on coarse flotation mica, factors controlling the readings must be fixed at some solid points of reference during the running of the test series.

Variables possibly affecting bulk density which still need investigation at this writing are:

1) Differences in amount of oily reagent remaining on the mica concentrate

2) Elapsed drying time at a given temperature.

It was assumed that the anionic scrub or the ether wash brought the mica sample (as received) close to its maximum possible bulk density reading. Whether other techniques would also do so is not known for certain.

With elimination of the variables dealt with, it should be reasonable to place considerably more confidence in the evaluation of relatively coarse mica by bulk density measurement, provided screen analysis of two or more comparable samples is also close to the same.

As an addendum to this technical report, a condensed procedure is stated (following), whereby reproducible bulk density readings on a coarse mica sample should be obtainable.

PROCEDURE FOR REPRODUCIBLE BULK DENSITY MEASUREMENTS, MINUS 20 MESH MICA FLOTATION CONCENTRATE

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1. Take 150-200 grams of mica concentrate sample from float circuit, agitate in lab conditioner (using 600 cc beaker) at 700 rpm, 40 percent solids, for 2 minutes with one gram of sodium dodecylbenzene sulfonate, 85 percent or more active.

2. Wash into 10-12 quart bucket, add water in strong flow to agitate. Let settle one-half minute, pour across 325 mesh screen, return material on screen to bucket. Repeat dilution, agitation, and dewatering.

3. Place sample (dewatered to maximum possible) in non-corroding pan in a layer one-half-inch thick or less. Dry at 200°-210°F for 8 to 15 hours.

4. Allow to cool for 2 hours before running a bulk density test.

5. Run a preliminary bulk density test to establish the minimum amount of sample needed to assure complete filling of all corners of the bulk density container. Add 15 percent of this weight to the weight sample to be used in the group of accurate determinations following. Re-combine the portion thus used with the balance of the sample.

6. Spread mica sample on a flexible sheet or cloth, roll it in two directions to mix. Level it out in a uniform layer about one-half-inch thick. Dip out a weighed aliquot: weight established as mentioned in 5.

7. Dump aliquot into a glass funnel having a short stem (1½-2 in.) with an inside diameter close to 11/32 inch. Hold finger at bottom of stem to stop flow.

8. Hold funnel, with sample, directly over volumeter with its screened funnel removed. Cubic container should rest level, directly underneath aperture, on a thin tray with four raised corners which can be easily grasped.

9. Release flow of mica down through volumeter. Funnel may be tapped during flow. If sample bridges and flow stops, put mica back on pile; re-mix, re-level, and repeat.

10. Carefully move container out by lifting tray. Do not shake, tilt, or jar.

11. Level the container load by gently moving a small straight-edged spatula, with blade vertical, across the top edges of the container. Keep contact with container constant but light. Make one full sweep without stopping.

12 Lift container, tap it to compact remaining sample. Brush off outside. Weigh on a balance accurate to 0.01 gram. Multiply net mica weight, in grams, by 3.81 to obtain pounds per cubic foot.

13. Run three or four separate aliquots from the pile, each time recombining, mixing, and leveling. If total variation exceeds 1.5 percent of the minimum reading on a sample, tests should be repeated or checks made against some introduced variable.

<u>NOTE:</u> This is a part of the technical paper entitled "Survey of Factors Contributing to Variable Mica Bulk Density Readings"; September 1966, MRL(Mica, General file). Full paper available on request.