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SOME IMPROVEMENTS IN TECHNIQUES, LIGHT REFLECTANCE EVALUATION OF COARSE FLOTATION MICA

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During the years 1963 through 1965, there have been several projects at the Minerals Research Laboratory (one sponsored, one unsponsored) wherein the light reflectance characteristics of spiral or flotation size mica products have been of importance, due to the potential use of such micas in products which must be light colored. In the evaluation of this characteristic of the micas, the instrument employed has been a Model 610 Photovolt Photoelectric Reflection Meter.

Procedure with this instrument has involved taking reflectance readings on a given mica product, using the search unit or sensor with interchangeable glass filters, known as tristimulus filters. There are three of these in the set: green, amber, and blue. In use, the sensor, with one filter at a time installed, activates a galvanometer by the response of its photoelectric cell. Light from an ordinary incandescent bulb passes through the filter, reflects on the mica, and returns to strike the photoelectric cell on the near side of the filter.

Working standard in this procedure is a white ceramic tile whose reflectance readings are in turn established against a primary standard of pure magnesium oxide powder. If the galvanometer readings of the instrument are adjusted to register 100-100-100 during use of the green, amber, and blue filters, respectively, then it has been established that the furnished secondary standard of white glazed tile registers 79 on green, 80 on amber, and 79 on blue. The tile is easier to handle than the powder in this application, and so the former has been employed throughout the tests to be discussed, with above appropriate standard settings.

Further details of theory and procedure can be read in the technical instructions furnished by the manufacturer. The particular procedure used at the MRL in evaluating coarse mica is now dealt with briefly:

Until November 1965 the bottom of a pan or dish was well-covered with coarse mica. The cylindrical sensor, with proper filter installed, was simply laid, lens down, upon the mica sample, and a galvanometer reading taken. Before and after this, standardization was made by placing the sensor on the tile. When the mica had been thus evaluated with the green filter, the amber filter was substituted and the procedure repeated, then likewise with the blue. The changing of filters and the necessary returns to standard meant that the sensor had to be lifted out of the mica sample, then replaced, at least three times per sample.

The procedure just mentioned was indicating, at best, trends or approximate results, so far as coarse mica (as specified) was concerned. Those of the MRL staff who had undertaken mica evaluation with such a product, as well as people actually in the mica business, had declared that a reproducibility of plus or minus one and one-half points on the galvanometer for any given sample with any given filter was the best that could be

expected. The mica in the test series to be mentioned gave readings approximately in the high 40's to high 50's with assorted filters. Thus, a given reading with, say, the blue filter, might be 45.5 on one try, but on subsequent tries might be anywhere from 44 to 47. Such variability could not be accepted if the reflectance tests were to be closely depended on for comparative or evaluative purposes: greater reproducibility was needed.

It was surmised that the mica, because of its characteristics, did not maintain a constant reflective surface when subjected to repeated contact and withdrawal of the sensor lens. The flaky surfaces would lie first one way and then another; darker mica (also thicker and more massive) might be slightly shaken down from the reflectance area to a greater or lesser degree; the act of placing the sensor in the mica sample could be performed with varying degrees of pressure, thus varying flake orientation; or the finer mica might shake down underneath the coarse, which, alone, gives a lower reading. The important goal seemed to be to render the tested mica immobile, and thus amenable to being evaluated by highly reproducible readings. Further, a technique was required which would immobilize a given mica sample in the same manner in identical repetitive tests so that test readings for that particular sample were close to identical.

The obvious solution was to keep the sensor from touching the mica. However, as the surface of the mica moves further from the sensor lens, readings keep getting lower. Further, a loosely-piled mica sample could be stirred or altered on its surface by breathing too close, or by an accidental bump. Thus, compression behind a thin lens or pane was indicated. The question then arose of how to secure constant compression of the sample in all instances, and this behind a piece of glass - with sample and glass easily brought up flat and close against the lens of the sensor. This was a fairly simple engineering problem involving a small glass-topped slide-type container, having the following basic components:

1. A base plate of aluminum, 1-3/4" wide, 3-3/4" long, 1/4" thick, with a hole centered on the diagonals 1-1/4" in diameter on one side, increasing to 1-9/16" diameter halfway through the plate.

2. A brass ring of 1-9/16" outside diameter, 1-7/16" inside diameter, inserted into the larger hole section and secured by epoxy, then machined off so that the overall height of the entire assembly is 0.58 inches.

3. A circular metal plate screwed over the under side (opposite from the ring), with a slightly convex metal disc attached to it on the inside and protruding up through the hole: the plate 1-1/4" diameter, thus fitting the lower side of the hole.

4. A piece of "single weight" window glass, 1-3/4" by 2-3/4".

5. A pair of brass cross-bars, each tensioned on either side by springs attached to the base plate sides.

6. A set of guides to position the glass plate over the brass circle.

See Illustration No. 1 for further clarification.

Preparation of a given mica sample involves the following procedure:

1. The slide is placed on a small elevated stand built to hold it, with the glass cover off. On top of the ring portion is placed another aluminum base plate, (item 1, components, preceding) with its ends sawed off to about 1/4 inch of the edge of the hole. The sawed-off plate is placed over the ring with the larger diameter portion of the hole over the top edge of the ring.

2. A piece of stainless steel tubing 13/16 " inside diameter and 2-1/4" long, with ends cut off square and smoothed, is laid upright in the center of the slide cavity, bottoming on the under-cover. The mica sample, after being carefully rolled on a sheet of paper, is poured from the troughed paper into the top of the upright tube until the fill appears equivalent to level-full (no attempt to level is made).

3. The tube is slowly withdrawn straight upward, thus finally allowing some mica sample to pile up and spread out over the top of the leveler (the inverted, cut-off base plate).

4. The glass plate is used as a scraper and is gently dragged in a vertical position across the top surface of the leveler until the heaped mica is pushed off, leaving the hole in view with the remaining mica level in it.

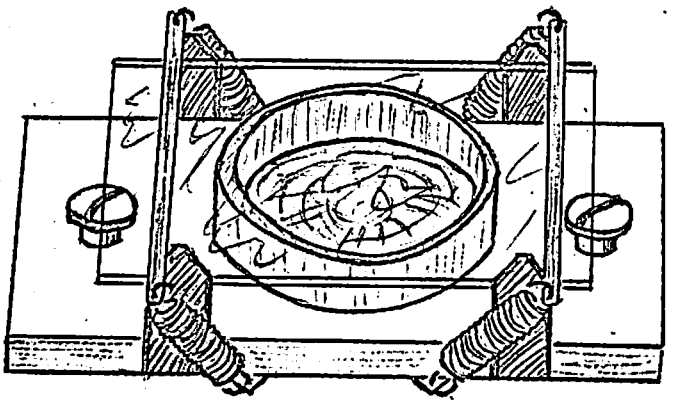
5. The leveler is gently lifted straight off, leaving the mica sample in the slide slightly heaped above the top surface, but level at the periphery.

6. The glass cover is lowered, taking care to hold it as horizontal as possible, and pressed downward on the mica sample; pressure is continued, with a little oscillating rotation of perhaps ± 2 degrees, until the glass cover bottoms on the top of the ring. The spring-tensioned cross-bars are then drawn over the cover to hold it.

7. The entire slide is gently turned over and exterior mica brushed off. It is now kept inverted for the duration of the sample evaluation, taking care not to bump or jar it (although its degree of compression insures considerable stability).

ILLUSTRATION NO. 1

Glass-Covered Sample Container For Mica Reflectance Evaluation



8. The reflection meter is equipped with a 6" diameter magnifier tilted over the galvanometer scale, with convex face up and flat face down. Across the top surface is taped a scale corresponding to 5-point intervals on the galvanometer scale such that, when the operator sights his reading, he usually lines up the magnifier scale with the galvanometer scale, using the 5-point mark nearest the needle reading. He then takes the reading. This insures some constancy of reading technique. Variation in this area may account for a points' difference.

9. After activation and warmup of 30 minutes or more, the instrument is operated with the sensor anchored beam upward. Samples and white tile standard are laid face downward on the sensor beam with continual repeated standardization against both the tile standard and a relatively good-quality mica standard (compared to the mica being tested) which gives readings within, say, 6 points of the general run of mica samples. Both tile and mica samples have over them a pane of glass, in order to create a more identical situation as between tile standard and mica samples.

10. Each mica sample in its slide is given four exposures to the sensor in four rotational positions, 90° apart. The reading for each position is recorded, and the four readings for each filter averaged. The white tile standard is used to calibrate the instrument before, then after, each group of four readings. If the instrument drifts, the four readings are repeated after re-calibration.

The mica slides are always placed in one certain position to start, then rotated at 90° intervals, counterclockwise. Thus, a repeat on a reading series can be exactly checked against the original.

The refinements in equipment and technique here mentioned have resulted in considerably higher reproducibility on any given mica sample, and in more reliable comparative ratings of assorted mica samples. The following difficulties, encountered in the earlier procedure, are now reduced closer to a minimum:

1) The stirring-up, and thus variation of, the sample surface

2) Variations in compression of the sample surface

3) Variations in small regional orientations of the mica flakes, producing varied reflectance

4) Variations in readings from the stance of the operator

5) Variations due to difficulty in reading the small markings of the scale.

6) Inadvertant variations in technique by the operator as work progresses.

The procedure discussed involves a time expenditure of about 45 minutes to run complete readings in six mica samples, plus an accompanying mica standard, and the tile standard. It has been customary to reconstitute the mica standard and run it after every six unknowns in order to assure the operator that he is not allowing some variable to creep in.

To illustrate the change accomplished, some figures are offered. Table No. 1 (following) illustrates the typical variation in readings obtained from several successive runs on the same group of samples.

Table 1

Sample Comparison, Original Technique

Test No.	Green	Amber	Blue
R310N - 1st run	53	54	45
R310N - 2nd run	51	53	42
311N - 1st run	52	54	45
311N - 2nd run	51	53	42
312N - 1st run	53	54	44
312N - 2nd run	54	56	46
302N - 1st run	55	57	47
302N - 2nd run	57	58	45

Compared to the foregoing, a series of readings by the new system on the same sample (a mica standard) is tabulated (Table 2, following). In each case, the sample was re-constituted. Reproducibility of readings is of a much higher order. Figures given are averages of four positional readings. This, plus the ability to read half-points accurately, accounts for the decimals.

Table 2

Light Mica Standerd, Revised 300N Test Series:
(Run by Refined Technique)

	Green	Amber	Blue
Run 1	56.5	59.3	48.5
Run 2	57.0	59.1	48.4
Run 3	57.0	58.9	48.3
Run 4	56.6	59.1	48.9
Run 5	56.9	58.7	48.5

Whereas a variation of two points was formerly usual (Table No. 1) in Table No. 2 a variation of 0.6 point is maximum. Under the latter conditions, a variation in a given average reading, for a color, of one point between one test and another can be assigned some validity, although a variation of 1.5 points is probably a better magnitude to heed.

While no attempt was made to run a screen analysis on all the micas tested, it can be surmised that, since they were of the same origin their general screen analysis was quite similar. A typical screen analysis ran as follows:

Table 3

Screen Analysis of Composite Mica Sample
Tests 302N, 305N, 333N

Mesh Size	+28	28-35	35-48	48-70	70-100	100-150	150-200	200-270	270-325	-325
Percent	1.0	6.0	24.0	16.0	24.0	15.0	7.0	3.0	2.0	2.0

It is recognized that each screen fraction would very likely give a different set of reflectance readings from the one adjacent, the tendency being for higher readings to occur in the finer fractions.

As an interesting final note on this project, there is cited (Table No. 4) a comparison between the reflectance readings from a series of tests, placing these in descending order of reflectance, and the subjective visual rating of these same samples by a well known expert in the mica-producing industry with a lifetime of experience in evaluating mica on the basis of color.

Table 4

Reflectance Readings Vs. Trained Visual Evaluation

Test No.	Green	Amber	Blue	Visual Rating
1	57.1	59.0	48.0	1
2	54.0	56.2	45.7	3
3	54.3	56.2	45.3	2
4	53.0	55.1	44.6	5
5	52.2	54.3	44.5	4
6	51.9	53.8	44.2	6
7	51.3	52.6	42.1	7

It would appear that proper and careful use of this reflection meter can result in the fairly accurate rating of the color characteristics which are criteria of quality in the mica industry.