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Determination of Calcite-Dolomite Ratios in Carbonate Rocks

By RALPH W. LAWSON

When studying a large number of carbonate rock samples, the usual methods for determining the calcite or dolomite content are time-consuming and tedious. To identify and count grains in thin-section is difficult for the most experienced. If sections or powders of the rock are subjected to various stains or dyes grain counts, still, must be made.

In working with the silver nitrate-potassium chromate method (Lemberg, 1892), the author noticed a definite difference in the colors produced in several samples of powdered limestone by the stain. In this method the calcite is stained a very dusky red, whereas the dolomite is unaffected. Therefore, it is obvious that the mass color of a powder will be related to the relative proportions of dolomite and calcite.

On this basis, a set of eleven standard samples was prepared from mineralogically pure calcite and dolomite. These samples range from 100 percent calcite to 100 percent dolomite. Intervening samples are mixtures of calcite and dolomite varying in 10 percent increments. Each sample then underwent precisely the same staining procedure. The result is a series of powders varying in color from very dusky red, through several lightness values of grayish red and pale red, to light gray. The color names are those which appear in the Rock-Color Chart distributed by the National Research Council, 1948.

PROCEDURE

After a number of experiments the following procedure was adopted as the most reliable. The pure dolomite was reduced to a fineness that would just pass the No. 200 screen of the Tyler Standard screen scale. This material was caught on the pan. Calcite was reduced in the same way and the two powders were used to prepare eleven 1 gram samples. The first 1 gram sample contains only calcite and is labeled No. 0. to indicate zero percent dolomite. The second sample containing 0.9 gram calcite and 0.1 gram dolomite is labeled No. 1. In this manner standard samples No. 0. to No. 10. were prepared.

Solutions found to be most effective for short-time immersion are as follows: (1) a 10 percent solution of silver nitrate in distilled water, (2) a 35 percent neutral solution of potassium chromate.

Lemberg (1892) used a silver nitrate solution warmed to between 60 and 70 degrees C., and a more dilute chromate solution may be as effective in such a procedure.

The staining method is somewhat critical and the most uniform results were achieved as follows: (1) immerse sample in the silver nitrate solution, and agitate the mixture with a rubber-tipped rod for 1.5 minutes; (2) allow powder to settle for 30 seconds; (3) immediately transfer the solution to a ceramic, or glass, filter above an evacuating flask; (4) wash powder thoroughly with distilled water to remove the nitrate and excess silver, and transfer powder to filter; (5) cover powder with potassium chromate solution and agitate mixture for 45 seconds; (6) develope vacuum and draw off chromate solution immediately; (7) wash thoroughly with distilled water and transfer powder to a beaker; (8) dry powder at room temperature. This part of the procedure requires about 7 or 8 minutes per sample, thus fifty powdered rock samples can be run in one day. They will dry overnight.

PRECAUTIONS

The silver nitrate reacts with calcium carbonate to precipitate silver carbonate on the calcite surfaces. The silver carbonate in turn reacts with potassium chromate to precipitate silver chromate, which is a dark reddish brown compound. Dolomite also tends to react with the silver nitrate but much less readily than calcite. Longer treatment will cause some staining of the dolomite and produce a darker color on the calcite.

Samples larger than 1 gram make filtering a slower process, allowing a longer reaction time. Results are less uniform. Each filter should be placed in an acid bath after a sample is treated in order to dissolve out any carbonate in the pores. If excess silver is not completely removed, silver chromate precipitates in the pores of the filter. This slows up filtering and may cause non-uniform results.

Powdered samples of the rocks to be tested should be treated in the same manner as prescribed for the standards. The unknowns can be compared visually with the standards, and the amount of dolomite can be determined with an accuracy of about 10 percent. There are other sources of error which should be considered. Other factors being equal, the final color of the powdered rock will depend upon the original rock color, the fineness of the powder, and certain critical values of the iron and clay content. The fineness of the powder may produce either negative or positive anomalies. The necessary mechanical reduction should be predetermined by petro-

graphic study of the calcite-dolomite relations. For best results the grain size of the powder should be small enough to eliminate mixed grains. If all of the dolomite and calcite does not contribute to the mass color the results are misleading. The fineness must be such that a homogeneous coloration is produced rather than a mottled effect.

Clay minerals in the rock may absorb silver and cause precipitation of silver chromate. This effect would produce negative anomalies for dolomite and positive anomalies for calcite, if the clay content is relatively high. G. H. Otto (1949) believes that iron may substitute for silver and that this may be an important source of error if the iron content is greater than five or ten percent.

The stability of the silver chromate has not been investigated by this author, and it may be advisable to keep the standards in a dry, dark place. The colors of the standards and their Munsell numerical designations, as determined from the Rock-Color Chart, are listed in Table 1.

VERIFICATION

Twenty-one of the stained rock powders were checked under the petrographic microscope. Of these, fourteen had dolomite percentages that fell within the range determined by visual comparison with the standards. Five had percentages that fell within the next interval above or below that determined by visual comparison. In none was the variation in percentage greater than twenty.

Since counting grains, even in a stained sample, is not infallible, such a check is not indicative of the accuracy of the visual method.

Table 1
Color of Standards

Standard Number	Percent Dolomite	Munsell Designation	Color Name
0	0	10 R 2.5/2	Very Dusky Red
1	10	10 R 3/2	
2	20	10 R 3.5/2	Grayish Red
3	30	5 R 3/2	
4	40	5 R 4/2	Grayish Red
5	50	5 R 4.5/2	
6	60	5 R 5/2	
7	70	5 R 5.5/2	
8	80	5 R 6/2	Pale Red
9	90	5 R 7/2	
10	100	N 7	Light Gray

However, it is evident that comparable results are obtained by either method. Chemical analyses were made on three rocks, after their dolomite content had been determined by visual comparison. The results are recorded in Table 2.

Table 2
Dolomite Content

Sample	Visual method (% wt.)	Chemical analyses (% wt.)
1	20 — 30	26.9
2	40 — 50	45.6
3a*	90 — 100	
3b	60 — 70	83.7

* Rock not reduced sufficiently to expose calcite included within dolomite grains. Sample 3b was reduced to a finer state.

Literature Cited

Lemberg, J. (1892) Zur mikrochemischen Untersuchung einiger Minerale: Zeitschr. d. Deutsch. Geol. Gesellsch., vol. 44, p. 231.
 Otto, G. H., Illinois Institute of Technology, Chicago, Illinois. (1949) Information on the silver chromate stain. [Private communication]

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