

Proceedings of the Iowa Academy of Science

Volume 11 | Annual Issue

Article 17

1903

Some Features in the Analysis of Dolomite Rock

Nicholas Knight

Copyright ©1903 Iowa Academy of Science, Inc.

Follow this and additional works at: <https://scholarworks.uni.edu/pias>

Recommended Citation

Knight, Nicholas (1903) "Some Features in the Analysis of Dolomite Rock," *Proceedings of the Iowa Academy of Science*, 11(1), 127-131.

Available at: <https://scholarworks.uni.edu/pias/vol11/iss1/17>

This Research is brought to you for free and open access by the Iowa Academy of Science at UNI ScholarWorks. It has been accepted for inclusion in Proceedings of the Iowa Academy of Science by an authorized editor of UNI ScholarWorks. For more information, please contact scholarworks@uni.edu.

SOME FEATURES IN THE ANALYSIS OF DOLOMITE ROCK.

BY NICHOLAS KNIGHT.

The purpose of this work was to compare different methods of determining silica in dolomite, and also to ascertain the amount of magnesium oxalate that will precipitate with calcium oxalate in connection with the analysis of dolomite rock by different students. These rocks abound in Iowa and in many other portions of the United States. The specimens under examination were variable mixtures of calcium and magnesium carbonates, with silica, ferric oxide and aluminum oxide. All were obtained from the Mount Vernon quarry and belonged to the Niagara period of geologic history.

At first a complete analysis of each specimen was made. Miss Grace Bradshaw determined the silica by two different methods to see how the results might agree.

1. A gram of the fine powder was placed in a small beaker, and, covered with a watch glass, it was dissolved in pure, dilute hydrochloric acid, by gently heating to the boiling point. The insoluble residue consisting of silica was filtered and the amount determined.

2. A gram of the substance was placed in a porcelain evaporating dish, and while covered with a watch glass, dilute hydrochloric acid was added. It was warmed on the water bath until there was no more evolution of gas.

The contents of the watch glass were rinsed into the dish and evaporation continued until crystals began to appear. Then as the drying continued it was stirred with a glass rod until a fine dry powder resulted. This was moistened with a few drops of concentrated hydrochloric acid and then about 20 c.c. dilute hydrochloric acid and the same quantity of water were added. The precipitate was filtered and determined as silica.

Analyses by the first method of the same specimen gave 0.75 per cent and 0.76 per cent silica, and by the second gave 0.78 per cent. As the results were similar, and as the determinations could be made much more easily and quickly by the first method, it was the one usually practiced.

About two grams of ammonium chloride were added to the filtrate from the silica, and at the boiling temperature it was treated with a small excess of ammonia to precipitate the iron and alumina. The filtrate from these, moderately diluted, was heated to boiling and precipitated with a $N/2$ solution of ammonium oxalate, care being taken to avoid an excess of the reagent. The oxalate was slowly added from a pipette, and with rapid stirring, to prevent as far as possible the occlusion of magnesium with the calcium oxalate. The precipitate was allowed to stand eight to twelve hours before filtering. The well washed precipitate of calcium oxalate, with a small quantity of magnesium oxalate was dissolved in warm, dilute hydrochloric acid and the solution was rendered alkaline with ammonia; this precipitates the calcium oxalate and leaves the magnesium in solution. This amount of magnesium and the main portion from the first calcium-magnesium precipitate were determined separately by precipitating each with disodium phosphate and weighed as magnesium pyrophosphate. The carbon dioxide was determined by the Bunsen method. After the complete analysis was made only the small amount of magnesium, that which precipitates with the calcium, was determined.

I. Determinations by C. C. Carhart.

The specimen was nearly a typical dolomite, containing:

	PER CENT.
Ca CO ₃	53.78
Mg CO ₃	44.96
Si O ₂	0.88
Fe ₂ O ₃ and Al ₂ O ₃	0.38
	<hr/>
	100.00

The small amount of Mg O—i. e. the quantity precipitated with the calcium was 0.08 per cent.

Eleven other determinations of this amount resulted as follows:

	PER CENT.
1.....	0.26
2.....	0.16
3.....	0.20
4.....	0.29
5.....	0.31
6.....	0.11
7.....	0.13
8.....	0.11
9.....	0.17
10.....	0.20
11.....	0.14

This is an average of 0.18 per cent Mg O for the twelve determinations equivalent to 0.378 per cent Mg CO₃. The amounts obtained are small and indicate that a sufficient quantity of ammonium chloride was added after removing the silica, and also that the ammonium oxalate was slowly added and not in large excess in the precipitation of the calcium. The ammonium chloride forms a double salt with magnesium which renders the latter less likely to precipitate as oxalate.

II. Determinations by F. E. Welstead.

A complete analysis of the specimen gave:

	PER CENT.
Ca CO ₃	50.91
Larger quantity Mg CO ₃	42.01
Smaller quantity Mg O = 0.70% = Mg CO ₃	1.47
Si O ₂	2.08
Fe ₂ O ₃ and Al ₂ O ₃	3.51
	<hr/>
	99.98

2 Mg O = 0.59% }
 3 Mg O = 0.47% } An average of 0.63 per cent.
 4 Mg O = 0.75% }

III. Determinations by C. A. Utt.

1. The analysis of the specimen gave:

	PER CENT.
Ca CO ₃	52.92
Mg CO ₃	37.56
Mg O = 0.47 = Mg CO ₃	1.84
Fe ₂ O ₃ and Al ₂ O ₃	6.26
Si O ₂	1.44
	100.02

2 Mg O = 0.96 per cent.
 3 Mg O = 0.15 per cent.
 4 Mg O = 1.28 per cent.
 5 Mg O = 0.47 per cent.
 6 Mg O = 0.228 per cent.

In 1, 2 and 4 of this series after dissolving the calcium and magnesium oxalates with hydrochloric acid and precipitating with ammonia the calcium precipitate was at once filtered. It is therefore evident that the calcium was not all precipitated, and afterwards came down and was determined as magnesium. This easily accounts for the large amounts obtained in these determinations.

Next such a mixture of pure Iceland spar and dolomite was taken that the calcium carbonate largely predominated. The analysis of the mixture is given below.

	PER CENT.
Ca CO ₃	87.035
Mg CO ₃	10.828
Mg O = 0.47 = Mg CO ₃	0.990
SiO ₂	0.06
Fe ₂ O ₃ and Al ₂ O ₃	1.08
	99.993

2. MgO = 0.15 per cent.

Instead of a gram but 0.2033 gram, not of the mixture but of the original substance was used. The small quantity Mg O was 0.66 per cent. From the foregoing it appears that it makes no difference if the percentage of magnesium is relatively small, or if a small amount of substance is used. About the same quantity of magnesium falls down with the calcium.

IV. Determinations by Miss L. B. Safely.

1. The analysis of the substance showed its composition as follows:

	PER CENT.
Ca CO ₃	41.92
Mg CO ₃	42.10
Mg O = 0.81 = Mg CO ₃	1.70
Fe ₂ O ₃ and Al ₂ O ₃	13.62
Si O ₂	0.88
	100.22

- 2 Mg O = 0.81 per cent.
- 3 Mg O = 0.78 per cent.
- 4 Mg O = 0.80 per cent.
- 5 Mg O = 0.72 per cent.
- 6 Mg O = 1.20 per cent.

In the last case some calcium precipitated with the magnesium because the calcium precipitate was not allowed to stand. Next used a mixture of Iceland spar and dolomite in which the calcium carbonate largely predominated.

- 1 Mg O = 0.47 per cent.
- 2 Mg O = 0.47 per cent.
- 3 Mg O = 0.47 per cent.
- 4 Mg O = 0.54 per cent.

Instead of a gram, used 0.3518 gram of the original dolomite, Mg O = 0.25 per cent.

Again 0.2133 gram gave 0.03 per cent.

Next a specimen of magnesite was used in which the amount of Ca CO₃ was only 1.30 per cent. The small amount of Mg O was 0.26 per cent. The conclusion seems to be that the magnesium precipitated with the calcium varies from an almost inappreciable amount to a considerable quantity. It is therefore always better to dissolve the unwashed precipitates of calcium and magnesium in warm hydrochloric acid, then to add ammonia to precipitate the calcium. After standing a sufficient time the calcium may be filtered, and the filtrate can be added to the solution containing the main portion of the magnesium, or the two portions can be separately treated.