

1938

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Recommended Citation

Kadesch, Richard G. and Getchell, R. W. (1938) "The Single Precipitation of Calcium Oxalate in the Presence of Magnesium," *Proceedings of the Iowa Academy of Science*: Vol. 45: No. 1 , Article 19.
Available at: <https://scholarworks.uni.edu/pias/vol45/iss1/19>

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THE SINGLE PRECIPITATION OF CALCIUM OXALATE IN THE PRESENCE OF MAGNESIUM

RICHARD G. KADESCH AND R. W. GETCHELL

It has been known for a long time that when calcium is precipitated as the oxalate in the presence of magnesium some magnesium is likely to be found in the precipitate. Therefore, it is customary to perform a double precipitation whenever the content of magnesium amounts to more than a few milligrams. With a view toward eliminating the time-consuming second precipitation whenever possible, it is the purpose of the present work to determine to what extent reliable results may be obtained for calcium using merely a single precipitation.

Many special methods have been developed for the separation of calcium from large amounts of magnesium. However, it has not been our intention to evolve a wholly new method. The method as ordinarily given in quantitative analysis texts for precipitation with ammonium oxalate in slightly alkaline solution has been followed.

Several investigations have been made in connection with magnesium contamination of calcium oxalate precipitates. These have covered variations in technique such as the quantity of precipitant used, time of precipitation, and time of digestion. It was thought best for our purposes, however, since the standard procedure was to be followed, to determine the effects of varying the MgO-CaO ratio for varying quantities of CaO. A few tests were also made to determine the effect of allowing the precipitate to stand at room temperature in contact with the solution before filtration.

It is desired that the amounts of calcium and magnesium taken for analysis be such that they are representative of the proportions actually encountered in limestones. Accordingly, the solutions taken for analysis were made up to 100 ml. on the basis of a .5g sample of limestone as given in Mahin (1), the text used for quantitative analysis at Iowa State Teachers College. On this basis Table I shows the effects of increasing the amount of MgO present from .02g (4 per cent) to .1g (20 per cent) while holding the CaO content constant at .2g (40 per cent). Forty per cent CaO is most nearly characteristic for limestones while the MgO percentage may run as high as 22 per cent in dolomites. The precipitations were

Table I

Amt. CaO Present	Amt. MgO Present	Amt. CaO Obtained
.2000	.0202	.2002
.2000	.0303	.2010
.2000	.0405	.2021
.2000	.0507	.2015
.2000	.0606	.2031
.2000	.0810	.2027
.2000	.1014	.2031

made using .6g of ammonium oxalate in 15 ml. of solution added over a period of 5-7 minutes.

Table I clearly shows that the results as obtained for calcium in the presence of magnesium cannot be relied upon in any case where the MgO content is above 4 per cent (2.5 per cent Mg) and when, as is often the case, the CaO amounts to about 40 per cent. If the MgO present is about 4 per cent or less, a reliable determination may be made by dispensing with the customary procedure of re-dissolving and reprecipitating. It is seen that for the most part the positive error increases as the amount of MgO is increased up to 12 per cent. The positive error of three milligrams noted at this point is increased no further for greater magnesium concentrations.

Table II shows the effects of varying amounts of MgO with

Table II

Amt. CaO Present	Amt. MgO Present	Amt. CaO Obtained
.0500	.0103	.0497
.1000	.0204	.1003
.1000	.0307	.1000
.1000	.0501	.0995
.1500	.0204	.1509
.1500	.0307	.1519
.1500	.0374	.1508
.2500	.0517	.2503
.2500	.0504	.2501

each of several quantities of CaO. Amounts of CaO taken were .05g (10 per cent), .1g (20 per cent), .15g (30 per cent), and .25g (50 per cent). The amount of ammonium oxalate used and the time of precipitation for each quantity of CaO are as follows:

Amt. CaO	Amt. amm. ox.	Time of precip.
.05	.20	3-4 min.
.10	.40	4-5
.15	.60	5-7
.25	.80	7-9

In every case the amount of ammonium oxalate used is approx-

imately equivalent to both the calcium and magnesium present. Popoff, Waldbauer, and McCann (2) state that the complete precipitation of calcium is dependent upon the presence of sufficient ammonium oxalate for both calcium and magnesium. Blasdale (3) found that when enough oxalic acid was added to combine with all the calcium and one-half the magnesium, the precipitation was incomplete. This was remedied, however, by adding more than enough to combine with all the calcium and magnesium.

Here it is seen that when smaller amounts of CaO are used (.05 and .1g) results are obtained which are in fair agreement with the theoretical values, although the magnesium content runs as high as one-half that of CaO. In table I the maximum MgO-CaO ratio was found to be one-tenth. However, when .15g CaO were taken the opposite effect was noted. No longer is an amount of MgO corresponding to 4 per cent allowable. The positive error noted when solutions containing amounts of CaO not differing too widely (.15 and .20g) seems to depend on the ratio of MgO to CaO.

Table III shows the effects of allowing the precipitate to stand

Table III

Amt. CaO Present	Amt. MgO Present	Amt. CaO Obtained
24 hrs. .2000	.0202	.1998
.2000	.0410	.2002
.2000	.0614	.2004
48 hrs. .2000	.0614	.2007

before filtration at room temperature in contact with the solution following the usual one-half hour digestion at about 90° C.

It is to be observed that the results obtained are in fair agreement with the theoretical values for a twenty-four hour standing period when the MgO content is as high as 12 per cent in the presence of 40 per cent CaO. This is in agreement with Kolthoff's (4) statement that the amount of co-precipitated ions always decrease with the time of standing before filtration. However, no further advantage was obtained when the standing time was lengthened to forty-eight hours.

SUMMARY

1. A satisfactory determination of calcium may be made in the presence of magnesium using merely a single precipitation when the CaO content is .2g in 100 ml. of solution and the amount of MgO present is not more than one-tenth this amount.

2. If the amount of CaO present is one-half as great or less,

results of fair reliability are obtained when the MgO content is as much as .05g.

3. The errors found when .15g and .20g amounts of CaO are taken and the results compared, seem to depend on the relative amounts of MgO and CaO present.

4. Reliable results are obtained using .2g CaO when the MgO content is as high as .06g by allowing the precipitate to stand for 24 hours before filtration. No further advantage is obtained by standing 48 hours.

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