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A QUANTITATIVE SPECTROGRAPHIC STUDY OF MAGNESIUM CONTAMINATION IN CALCIUM OXALATE PRECIPITATES

D. C. McCANN, LOUIS WALDBAUER AND STEPHEN POPOFF

Chemists have attempted quantitative spectrographic analysis repeatedly since 1863 when Miller (1) first conducted his experiments. De Gramont (2) added considerable information in a series of papers of about 1907. Leonard and Pollock, and Hartley (3) also published much material about the same time. These methods depend on comparison with a standard of the same composition, containing the same elements. Gerlack and Schweitzer (4) have recently developed a system which depends on comparison with lines of some other element which occur very nearby. The method used in our determinations is based on the earlier work.

The first slide (57) shows a progressive increase in intensity of the magnesium lines. The wave lengths are 2852.1, 2802.7, 2795.5, and 2783.1, 2781.5, 2779.9, 2778.4, 2776.8. The five lines are very useful in the range of 0.1% to 1%. Below 0.1% we must rely on the three of higher wave lengths. The second point of interest is the uniform intensity of the other lines of the spectra, especially those of calcium. Should there be any variation in these, one could not be certain that variations in the strength of Mg lines were due to change in composition.

Slide two (58) shows solutions of precipitates compared with standards. Again the background, as we may call it, must be uniform. It will be seen that the unknown spectrum is more intense than the standards at the bottom and less intense than those at the top, and falls between 0.3% and 0.5%.

Slide 3 (42) shows a series of standards at the bottom with several unknowns above. A great many of the comparisons were made from plates of this kind.

Slide 4 (54) shows the standards checked against standards prepared from Calcite crystals. Such a check gives one quite a feeling of security.

When we applied spectrographic methods to precipitates we found a number of surprises awaiting us. The precipitates used

were made at the same time as the gravimetric determinations, the only differences being that they were caught in a small Hirsch funnel. They were subsequently ignited to the oxide and then dissolved in distilled 6N HCl.

Slide 5 shows the results of variations in time of digestion with equivalent and twice equivalent weights of $(\text{NH}_4)_2\text{C}_2\text{O}_4$. Using an equivalent weight of ammonium oxalate, the results are found to be too low in comparison with the calculated + the weight of MgO. Twice the amount of Ammonium Oxalate gives one remedy to the case, where the gain is equal to the %MgO.

Slide 6 shows the effect of two digestions by different methods. A longer time of precipitation is seen to be an advantage.

Slide 7 is a continuation of 6 showing twice the amount of Ammonium Oxalate. The $1\frac{1}{2}$, $\frac{1}{4}$ hour digestion is the least contaminated of all precipitates obtained and is the method recommended. (The second digestion has also been varied, and shows that the $\frac{1}{4}$ hour period is best.)

Slide 8 shows the effect of NH_4Cl content. It is apparent that the so-called advantage here lies in the fact that less calcium precipitates. Slide 9 shows several attempts at adding the Ammonium Oxalate to an alkaline solution. As might be expected, they are very similar.

Slide 10 shows the result of a test of the recommended method on a standard CaCl_2 solution prepared by a modification of Richards' atomic weight methods.

Subsequently a determination from the same solution, contaminated with an equivalent amount of Mg ion and precipitated twice gave the same value. The method used here was a $1\frac{1}{2}$ hour digestion with moderate turbidity, followed by a 7 minute precipitation using 3N NH_4OH and filtration after $\frac{1}{4}$ hour. Twice the equivalent weight of Ammonium Oxalate required for the calcium was used. A double precipitation using the $\frac{1}{4}$ hour digestion mentioned previously gave results of similar nature. Each method has its advantages. The longer produces a more granular precipitate, the shorter is far more rapid. Both have been checked by a double precipitation, and found to give results within the experimental error.

LITERATURE CITED

1. MILLER — Phil. Trans., 152, 883 (1863).
2. DEGRAMONT-COMTES REND., 144-161 (1907-1921).
3. HARTLEY, POLLOCK, LENARD, Proc. Roy. Dublin Soc., 1907-9.
4. GERLACH and Z. SCHWEITZER. Anorg. Chem., 142, 383 (1925).