

## THE FILTRATION OF BARIUM SULPHATE

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Krak<sup>1</sup> has recently stated that to filter barium with suction seems a rather daring experiment even when specially prepared filters are used, but that it may be done as follows:

"Pour the supernatant liquid through the filter using suction. Before the precipitate begins to follow, add about 10 cc of a saturated, slightly acid, solution of ammonium acetate to the precipitate and stir it up well. The fine BaSO<sub>4</sub> changes instantly into a coarse precipitate and can be washed instantly while suction is going full speed without ever passing through the filter. The results thus obtained are not influenced by the use of the filter solution."

This sounded as though the barium sulphate were coagulated by the ammonium acetate solution. It seemed desirable to verify this and to see what would be the effect of adding solid ammonium acetate to a solution from which barium sulphate had been precipitated cold. A number of experiments were therefore made to determine what happens and what are the limitations of the method, if any.

Equivalent solutions of barium chloride and of sulphuric acid were prepared and 100 cc of the barium chloride solution were added a little at a time to 100 cc sulphuric acid, stirring continuously and keeping the solutions at 75°–80°. The precipitate is coarse enough for ordinary filtration by gravity but is sucked through a filter paper when suction is applied. In the next experiment the supernatant liquor was decanted, as recommended by Krak, and 10 cc saturated ammonium acetate solution were stirred up with the residue. There seems to be a solution of the barium sulphate which reprecipitates as coarser crystals which are not carried through a filter paper by suction. There is no difficulty at all in duplicating Krak's result.

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<sup>1</sup> Chemist Analyst, 1912, 26.

In another experiment the barium sulphate was precipitated hot as before but the supernatant liquid was not decanted. About 75 grams of solid ammonium acetate were added to the solution, making the concentration with respect to this salt practically the same as in the preceding experiment. Filtration by suction was not possible. The whole mixture was boiled for 9–10 hours, but even then the barium sulphate ran through the filter under suction. This showed that a mixture of ammonium acetate and hydrochloric acid behaves quite differently from ammonium acetate alone. This might be due to the free acid or to the chloride preventing the growth of the crystals, or it might be that the chloride had a peptonizing action and disintegrated the barium sulphate.

A hot solution of ammonium sulphate was precipitated by adding just sufficient hot barium chloride solution. Since there were about 110 cc of solution, approximately 100 grams of solid ammonium acetate was added and the mixture was boiled for 8 hours, 4 hours at a time with several hours of standing between the two periods. The barium sulphate could then be filtered with suction. This experiment shows that ammonium chloride retards but does not prevent the growth of the barium sulphate crystals.

Some barium sulphate was made filterable by Krak's method. This was boiled for 9 hours with an ammonium chloride solution. At the end of this time the crystals filtered as well as before, thus showing that the ammonium chloride solution had no peptonizing or disintegrating action.

To determine whether Krak's method worked well with neutral and acid solutions, three sulphate solutions were made up of the same strength, sodium sulphate, sodium sulphate acidified with hydrochloric acid, ammonium sulphate. These solutions were heated to 80° and sufficient hot barium chloride solution added to ensure complete precipitation. The mixtures were brought to boiling for 5 minutes and were then allowed to stand over night. In each case the supernatant liquid was decanted, 5 cc of saturated ammonium acetate

added, and the solution boiled for 5 minutes. The precipitates did not filter well, so 10 cc more of ammonium acetate solution were added to each beaker. Each solution was boiled for 1 minute and then allowed to stand. There was no difficulty in filtering with suction, the three lots behaving practically alike.

The last experiments dealt with cold precipitation of sulphuric acid by barium chloride. After decantation, ammonium acetate solution was added and boiling was kept up for 8-10 hours. It was quite impossible to filter the barium sulphate. The experiment was repeated with 20 cc ammonium acetate solution, boiling steadily for 96 hours, using a reflux cooler. At the end of this time the barium sulphate filtered nicely with full suction.

These experiments clear up the theory of the process. Barium sulphate is soluble in ammonium acetate solution and the crystals therefore grow in a hot solution. If the barium sulphate is precipitated under conditions such that the crystals are nearly large enough to permit of filtration with suction, a short heating is enough to make them grow to the required size. If the barium sulphate is precipitated cold, the crystals are small and a very much longer heating is necessary to give the required size. Since ammonium chloride and hydrochloric acid do not peptonize or disintegrate the barium sulphate crystals and yet do retard the growth, it is clear that these substances decrease the solubility of barium sulphate in ammonium acetate solutions.

This work was suggested by Professor Bancroft and has been carried on under his supervision.

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