

A repetition of these experiments with commercial ammonia which had not been redistilled gave similar results.

Since it was probable that an excessive amount of amines in the ammonia might influence the solubility of the ferric hydroxide, further experiments were performed in which there was added to the ammonia before precipitation from 1 to 3 grams of the hydrochloride of an amine. The hydrochlorides used were those of methylamine, ethylamine, diethylamine, isoamylamine, ethylenediamine, aniline and phenylhydrazine. As before, in different experiments with the same amine, precipitation was carried out both in hot and in cold solution, and with large quantities of ammonia as well as with a small excess. The filtrates were evaporated to small bulk and tested for ferric iron. In a few cases the filtrate was slightly yellow after evaporation, owing to organic matter, but in these cases the greater part of the color was removed by heating with a considerable quantity of iron-free nitric acid. In the experiments with aniline and phenylhydrazine the oxidizing effect of ferric iron was avoided by nearly neutralizing the ferric solution with ammonia before the addition of the hydrochloride.

The largest quantity of iron found in any one of these filtrates was five one-hundredths of a milligram and in most cases the amount did not exceed one one-hundredth of a milligram. A quantity of precipitate as small as this might well have been mechanically dragged through the filter-paper. At all events the error introduced by the use of an excess of ammonia in precipitating ferric hydroxide is much smaller than the other errors incidental to a quantitative filtration, and the expulsion of the excess of ammonia, at the risk of solution of a portion of the precipitate through hydrolysis of the ammonium salts, should be omitted.

Evidently the solubility of ferric hydroxide in ammonia which was previously observed was caused by the presence of some organic impurity of unusual occurrence in ammonia.

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A New Qualitative Test for Calcium.—The difficulty of testing for calcium in the presence of barium and strontium, led the

writer to turn his attention to a test for this metal which would not be answered by barium and strontium.

In this connection the action of potassium ferrocyanide has been tried with surprisingly good results so good in fact, that it seems almost impossible that the reaction has not been used before, in this connection.

As only one reference could be found to the action of potassium ferrocyanide on calcium compounds,¹ the fear of repetition is braved and the test described, in the hope that it may prove useful.

In separating barium, strontium and calcium, the commonly accepted method seems to be precipitation with ammonium carbonate in ammoniacal solution, solution of the washed precipitate in acetic acid, removal of barium by potassium chromate or bichromate, reprecipitation of strontium and calcium by ammonium carbonate in ammoniacal solution, re-solution of the precipitate in acetic or hydrochloric acid, removal of strontium by ammonium sulphate and finally testing for calcium by addition of ammonium oxalate in ammoniacal solution.

The weak point in this scheme is the danger of traces of barium and strontium remaining in solution, with the well-known effect on the calcium test.

The suggested procedure is the same as the foregoing, up to the point of the re-solution of the precipitate after barium has been removed, (except that acetic acid only should be used).

At this point the solution is divided and calcium sulphate added to one portion to test for strontium.

To the other portion, an equal volume of ammonium chloride is added and a few cubic centimeters of potassium ferrocyanide. The presence of calcium is indicated by the formation of a light yellowish green precipitate.

The sensitiveness of the test has been tried on standard solutions of calcium salts and when applied directly to the calcium solutions was found to indicate 1 part of calcium in 7,000 parts. The ammonium oxalate test was also tried and was found to indicate 1 part in 140,000 parts.

The oxalate test was also applied to a series of standard calcium solutions to which strontium had been added. The strontium was removed by ammonium sulphate as in the first method out-

¹ Prescott and Johnson, 1903, p. 212.

lined. Under these conditions the addition of ammonium oxalate caused no precipitate in solutions more dilute than 1 part in 14,000.

The difference is accounted for by the fact that the ammonium sulphate precipitates out a large amount of calcium with the strontium, and by the fact that calcium oxalate is probably more soluble in ammonium sulphate than in water.

The ferrocyanide test was also tried in the regular scheme of analysis and clearly indicated 1 part in 7,000. This 1 part in 7,000 seems to be the solubility limit of the calcium-potassium ferrocyanide.

Under similar conditions and within the limits of the ferrocyanide test, the volume of the precipitate is about four times as great with ferrocyanide as with oxalate.

It might be added that comparatively fresh solutions of potassium ferrocyanide seem to give the best results.

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Note on the Coloration of Didymium Glass by Radium Chloride.—About six-tenths of a gram of radium chloride, 7,000 activity, were sealed in a small tube of didymium glass and allowed to remain six months. The colorless glass acquired an exquisite pink color. Unfortunately on analysis, the glass was found to contain manganese. No variation in the absorption spectrum of the glass was observed after the exposure. No electrical or sparking effects were noted when the tube was opened.

Note on the Preparation of Rubber Samples for Analysis.—In the course of construction of the buildings of the College of the City of New York, the writer has had to pass upon a number of items involving large contracts. Among these were rubber and cable insulations. The analysis of rubber and insulating material at best is not attended with satisfaction. To eliminate at least one source of error we desired the sample in a pulverulent condition. This was accomplished by thorough chilling with liquid air and grinding under that refrigerant in an ample agate mortar. The analyses were made by Dr. L. H. Friedburg, of this department.

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