

Some Observations on Reactions between Certain Metallic Ions and the Ammonium Salt of Aurintricarboxylic Acid.—Several years ago Hammett and Sottery¹ discovered that the ammonium salt of aurintricarboxylic acid could be used as a very sensitive reagent for the detection of aluminum. The new reagent is called "Aluminon." In acetic acid-acetate solution aluminum forms a bright red lake with this dye, which is fairly stable in ammonium hydroxide—ammonium carbonate solution. Observations on most of the common metallic ions, as well as silicic acid and phosphate, were made with this reagent. Ferric iron was the only one found to give a colored precipitate, the others either gave white precipitates or none at all. Iron, therefore, must be completely removed from the aluminum. This may be accomplished by co-precipitation of the iron with relatively large amounts of elements such as copper. Precipitation with sodium hydroxide, or sodium sulfide, followed by digestion for thirty to sixty minutes on a steam-bath, is satisfactory.

Middleton² reports that "Aluminon" forms lakes with the hydroxides or basic acetates of beryllium, cerium, erbium, lanthanum, neodymium, thorium, yttrium and zirconium. These lakes are deeper red than the aluminum lake but all, except that of beryllium, are either dissolved or decolorized by ammonium carbonate.

Corey and Rogers³ have studied the reactions of "Aluminon" with gallium, germanium, indium, scandium and thallium. With the exception of germanium, these elements form red lakes, but they are destroyed by ammonia or ammonium carbonate solutions.

Lundell and Knowles⁴ made use of the "Aluminon" reagent in the quantitative determination of small amounts of aluminum in non-ferrous alloys.

Yoe and Hill⁵ made an extensive study of the various factors involved in the use of this reagent and developed a method for the colorimetric determination of aluminum in potable water. The method is also applicable to the determination of small quantities of aluminum in salts, minerals, rocks, etc.

In this note we report further studies with "Aluminon." Observations with nineteen elements, most of them rare elements, have been made. In most cases the chloride or nitrate was used; in a few instances the sulfate or oxalate. The concentration of the metallic ion was approximately twenty parts per million in the final dilution, *i. e.*, 0.24 mg. per 12 ml. The "Aluminon" reagent was 0.1% in water solution.

The following procedure was observed. To a measured amount of a given salt the reagents were added in the order listed: (1) 2 ml. of 1 *N* HCl; (2) 2 ml. 3 *N* NH₄Ac; (3) 2 ml. "Aluminon," allowed to stand five to

¹ Hammett and Sottery, *THIS JOURNAL*, **47**, 142 (1925).

² Middleton, *ibid.*, **48**, 2125 (1926).

³ Corey and Rogers, *ibid.*, **49**, 216 (1927).

⁴ Lundell and Knowles, *Ind. Eng. Chem.*, **18**, 60 (1926).

⁵ Yoe and Hill, *THIS JOURNAL*, **49**, 2395 (1927).

ten minutes; (4) 2 ml. 6 *N* NH₄OH; (5) 4 ml. 6 *N* (NH₄)₂CO₃. The results are recorded in the following table.

TABLE I
ANALYTICAL RESULTS

Ion	"Aluminon"	NH ₄ OH	(NH ₄) ₂ CO ₃
Arsenic, As ⁺⁺⁺	Red soln.	Almost decolorizes soln.	Decolorizes soln.
Columbium, Cb ⁺⁺	Brownish-red ppt.	Ppt. dissolves giving pale yellow soln.	Decolorizes soln.
Dysprosium, Dy ⁺⁺⁺	Red soln.	Purple soln.	Decolorizes soln.
Gadolinium, Gd ⁺⁺⁺	Red ppt.	Ppt. dissolves giving purple soln.	Decolorizes soln.
Hafnium, Hf ⁺⁺⁺⁺	Deep pink soln.	Light pink soln.	Decolorizes soln.
Iridium, Ir ⁺⁺⁺⁺	Red soln.	Pale yellow soln.	Soln. almost colorless
Molybdenum, ^a Mo ⁺⁺⁺⁺⁺	Red ppt.	Ppt. dissolves giving pale yellow soln.	Soln. almost colorless
Praseodymium, Pr ⁺⁺⁺	Red ppt.	Ppt. dissolves giving pink soln.	Decolorizes soln.
Rhenium ^b	Red soln.	Pale yellow soln.	Soln. almost colorless
Rhodium, Rh ⁺⁺⁺	Brownish-red ppt.	Ppt. dissolves giving pale yellow soln.	Soln. almost colorless
Ruthenium, Ru ⁺⁺⁺	Pink soln.	Pale yellow soln.	Dingy yellow soln.
Samarium, Sm ⁺⁺⁺	Red soln.	Pale purple soln.	Decolorizes soln.
Tantalum, Ta ⁺⁺⁺⁺⁺	Brownish-red ppt.	Ppt. dissolves giving pale yellow soln.	Soln. almost colorless
Tellurium, ^a Te ⁺⁺⁺⁺⁺	Red soln.	Pale yellow soln.	Soln. almost colorless
Thulium, Tm ⁺⁺⁺	Red soln.	Purple soln.	Decolorizes soln.
Tungsten, W ⁺⁺⁺⁺⁺	Brownish-red ppt.	Ppt. dissolves giving pale yellow soln.	Soln. almost colorless
Uranyl, (UO ₂) ⁺⁺	Red soln.	Pale yellow soln.	Soln. almost colorless
Vanadium, V ⁺⁺	Scarlet ppt. and soln.	Ppt. partly dissolved, soln. pale pink	Dissolves ppt.
Ytterbium, Y ⁺⁺⁺	Deep pink soln.	Pink soln.	Decolorizes soln.

^a The metal was dissolved in *aqua regia*. The ions are assumed to be in the highest state of oxidation.

^b The metal was treated with *aqua regia* and the mixture evaporated to dryness. Concentrated nitric acid was added to the residue and evaporated to dryness. This treatment was repeated and the residue taken up in distilled water.

An inspection of the table shows that gadolinium, molybdenum, praseodymium and vanadium give red precipitates. All are dissolved and decolorized by ammonia or ammonium carbonate. Columbium, rhodium, tantalum and tungsten give brownish-red precipitates soluble in ammonia and decolorized to a pale yellow. The remaining elements give red or deep pink solutions with "Aluminon," but all were decolorized by ammonia or ammonium carbonate. It is evident that none of the above elements will interfere with the detection of aluminum by the "Aluminon" reagent. Beryllium forms a lake similar to the aluminum lake and hence the reagent does not distinguish between these two elements.

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