

Fourier's series to determine the electron density at points in the unit cell of a crystal, the coefficients of the series being evaluated from the measured intensities of X-ray reflection. This method, when applied to mercurous chloride, mercurous bromide and mercurous iodide, sets the values of the parameters at $u = 0.116$, $v = 0.347$ in the unit cell containing two molecules of mercurous halide with mercury atoms at $00u$; $00\bar{u}$; $1/2, 1/2, 1/2 + u$; $1/2, 1/2, 1/2 - u$; and halogen atoms at $00v$; $00\bar{v}$; $1/2, 1/2, 2/2 + v$; $1/2, 1/2, 1/2 - v$.

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NOTES

Reaction of "Aluminon" with Hydroxides of Beryllium, Rare Earths, Zirconium and Thorium.—The new reagent for aluminum, proposed by Hammett and Sottery¹ and now on the market under the trade name "Aluminon," has proved very satisfactory in our Laboratory. Its value for the detection of very small amounts of aluminum has been shown recently by Lundell and Knowles.²

It appears worth recording that lakes are formed by this reagent with the hydroxides or basic acetates of beryllium, yttrium, lanthanum, cerium, neodymium, erbium, zirconium and thorium. All these are deeper red than the aluminum lake, the color being a rich bright crimson, that with cerous hydroxide much darker than the others. All, except that of beryllium, are either dissolved or decolorized by moderate additions of ammonium carbonate. Accordingly, the reagent does not distinguish aluminum from beryllium in mixtures of the two hydroxides. The lakes are not affected by moderate concentrations of ammonia except that of zirconium which is partially decolorized and flocculates as a rose-pink precipitate. All are distinctly more insoluble than the corresponding hydroxides or basic acetates.

From the commercial pure nitrates, solutions were made up containing approximately 10, 1 and 0.1 mg. of the element per cc., except the beryllium solutions which contained 2, 0.2 and 0.02 mg. of the element per cc. To 1 cc. of these solutions was added 5 cc. of 1 *N* hydrochloric acid, 5 cc. of 3 *N* ammonium acetate, 5 cc. of 0.1% Aluminon solution and, after mixing, 3 cc. of 6 *N* aqueous ammonia. Further addition of 2 cc. of 9 *N* aqueous ammonium carbonate sufficed to dissolve or decolorize the precipitates except in the case of beryllium. Distinct precipitates were formed by 1 mg. of each of the elements tested (0.2 mg. of beryllium). Pink solutions, but no precipitates formed when 0.1 mg. (0.02 mg. of beryllium) was taken. The pink color of these very dilute solutions was not

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

² Lundell and Knowles, *Ind. Eng. Chem.*, **18**, 60 (1926).

altered by aqueous ammonia even after several days, except in the case of zirconium where aqueous ammonia rapidly discharged the color.

The following experiments show the smaller solubilities of the lakes compared with the solubilities of the corresponding hydroxides or basic acetates. Mixtures of 1 cc. of solution (10 mg. of the element), 5 cc. of 1 *N* hydrochloric acid and 5 cc. of 3 *N* ammonium acetate became distinctly turbid as soon as 5 cc. of Aluminon solution was added, except in the cases of aluminum and erbium which remained clear for several hours, but on standing overnight deposited precipitates. Precipitation in all these cases was incomplete and the supernatant liquids remained red or pink. The fraction precipitated seemed to vary between roughly one-fifth and one-third. The zirconium lake precipitated under these conditions was brilliant scarlet-red in contrast to the rose-pink color of that formed in the presence of ammonia. Likewise the aluminum lake similarly precipitated is deep crimson, much darker than that formed in the presence of ammonia. It appears that ammonia exerts an appreciable bleaching action on both of these lakes. Mixtures in which the Aluminon solution was replaced by an equal volume of water all remained perfectly clear after 24 hours.

The beryllium lake is much less soluble in ammonium carbonate than is beryllium hydroxide. A precipitate of the latter containing 2 mg. of the element dissolved completely when the liquid was made 3 *N* in ammonium carbonate while the lake containing the same amount of element was only partially dissolved when the liquid was made 5.5 *N* in carbonate.

A peculiar action was observed in the case of the lanthanum solution and in this alone. In the presence of ammonia the crimson lake settled rapidly, leaving the supernatant liquid colorless. Thereafter a considerable amount of pure white precipitate gradually flocculated which settled slowly and tended to cling to the walls of the vessel. After 24 hours a sharp line of demarcation between the heavier red and lighter white precipitates was visible. The cause of this phenomenon is not immediately apparent since impurities likely to be found in a lanthanum preparation should adsorb the dye equally well.

Thallic hydroxide is not precipitated in the presence of ammonium acetate but when the usual addition of Aluminon was made before adding ammonia the solution remained red for some days and a small amount of reddish precipitate was gradually deposited.

A scandium salt was not available. It would be of interest to know the behavior of Aluminon toward scandium hydroxide.