Colorimetric Determination of Aluminum with Aurintricarboxylic Acid[1]

By Paul S. Roller[2]

The aurintricarboxylic test of Hammett and Sottery[3] has been shown by Yoe and Hill[4] to be adaptable to the quantitative estimation of aluminum. In the recommended quantitative procedure, as directed for the qualitative test, the reaction mixture is made alkaline with NH_4OH–$(NH_4)_2CO_3$ before colorimetric comparison. The resultant gradual fading and the evolution of bubbles have been found troublesome.[5] By comparison with a slightly acid solution, it is found, as shown below, that the alkaline treatment also greatly reduces the sensitivity. The alkaline solution masks the effect of Cr^{+++}, but since this effect is relatively slight, and since the manner of its complete elimination is indicated, it is proposed that in general the colorimetric determination of aluminum with aurintricarboxylic acid be carried out at a fixed acid P_H.

The yellow color of the dye in alkaline solution and its red color in acid solution practically neutralize each other at a P_H of 6.3 in buffered solution. Hence a P_H of 6.3 was adopted as the fixed acid P_H at which comparison is made.

In the present procedure, adapted to colorimeter comparison, X cc. of the aluminum solution at about P_H 6.3 is diluted with $(12-X)$ cc. of water, and 5 cc. of a buffer (P_H 6.3) is added. The buffer is 4 M ammonium acetate containing some hydrochloric acid. After shaking, 1 cc. (per 0.01 mg. of aluminum) of a 0.1% solution of the ammonium salt of the dye is added and the mixture is again thoroughly shaken. The maximum color intensity is reached in about fifteen minutes. The color is stable over a period of many hours. As in alkaline solution,[5,6] the measured intensity is not linearly proportional to the quantity of aluminum. For amounts of aluminum less than 0.002 mg., comparison must be made in Nessler tubes.

The non-interference of other elements originally observed is found to obtain also under the present conditions. A blank result was given by the following: 10 mg. of Ba^{++}, Ca^{++}, Mg^{++}, Zn^{++}, Pb^{++}; 0.1 mg. of Co^{++}, Cu^{++}; 5 mg. of $PO_4^{\equiv}$. One mg. of SiO_2 from a solution of a crystallized silicate gave a color equivalent to 0.001 mg. of aluminum, no doubt due to an impurity of aluminum.

As is well known, Fe^{+++} is a major source of interference and must be eliminated. Measured in a colorimeter, 0.010 mg. of Fe gave a color equivalent to 0.005 mg. of aluminum.

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(3) Hammett and Sottery, This Journal, **47**, 142 (1925).

(4) Yoe and Hill, *ibid.*, **49**, 2395 (1927).

(5) Myers, Mull and Morrison, *J. Biol. Chem.*, **78**, 595 (1928); Schwartze and Hann, *Science*, **69**, 169 (1929).

(6) Winter, Thrun and Bird, This Journal, **51**, 2721 (1929).

Cr^{+++} in chrome alum solution was found to react slowly with the dye. The green Cr^{+++} reacts more rapidly than the blue; increase of temperature hastens the reaction. At room temperature, 0.10 mg. of green Cr^{+++} gave after fifteen minutes a color equivalent to 0.0005 mg. of aluminum, after thirty minutes 0.001 mg. of aluminum, and after eighteen hours 0.008 mg. of aluminum. It is seen that Cr^{+++} is but a slight source of interference under the conditions. Complete elimination of this interference is indicated by an increase in *P*H dependent on the quantity of Cr^{+++} with, of course, a resultant decrease in over-all sensitivity of the test.

Under the present conditions, the aurin reaction with aluminum is extremely delicate. A faint pink is obtained with 0.0001 mg. of aluminum, so that the sensitivity is about twenty times that reported by Yoe and Hill under alkaline conditions. Besides the elimination of fading and of carbon dioxide evolution, turbidity in the presence of foreign ions such as that observed by Yoe and Hill is also obviated.

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Structure of the Chloraloses, Alpha and Beta-Glucochloraloses

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The literature regarding the structure of the chloral derivatives of glucose and of xylose was reviewed in the previous publications[1] of this series. The experimental data of these previous publications as well as the data presented below are summarized for glucose in Table I and for xylose in Table II.

Referring to Table I, the condensation of glucose with chloral under the specified conditions yields a crystalline mixture from which can be isolated dichloralglucose (A), dichloralglucose (C), and dichloralglucose (D). Boiling the mother liquor from this separation causes β-glucochloralose to separate. Neutralization or ether extraction of the mother liquor remaining after the separation of the β-glucochloralose yields α-glucochloralose. The condensation of β-glucochloralose with chloral yields dichloralglucose (A) and dichloralglucose (C). The condensation of α-glucochloralose with chloral yields dichloralglucose (E). A fifth dichloralglucose (B) is reported by Pictet and Reichel.[2]

Repeated methylations[1a] of β-glucochloralose introduced only three methyl groups to give trimethyl-β-glucochloralose. This indicated that

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(1) (a) Coles, Goodhue and Hixon, THIS JOURNAL, **51**, 519 (1929); (b) Goodhue, White and Hixon, *ibid.*, **52**, 3191 (1930).

(2) Pictet and Reichel, *Helv. Chim. Acta*, **6**, 621 (1923). The dichloralglucoses A and B were so designated by Pictet and Reichel. Further descriptions of dichloralglucoses A, C and D are given by Ross and Payne, THIS JOURNAL, **45**, 2363 (1923).