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## The Solubility of Ignited Ferric and Chromic Oxides

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used to show the apparatus used (including the wedge sector), typical spectrograms, and also to show some of the data obtained in graphical form.

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### FREEZING POINT CURVES OF IODINE MONOCHLORIDE MIXED WITH IODINE, ACETIC ACID, OR CARBON TETRACHLORIDE

JACOB CORNOG AND LEONARD OLSON

The curve obtained with iodine differs from the curve obtained by previous workers; the acetic acid curve approximates expectation based on Raoult's Law; the carbon tetrachloride curve indicates the formation of solid substances.

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### THE SOLUBILITY OF IGNITED FERRIC AND CHROMIC OXIDES

JACOB CORNOG AND DOROTHY BUCK

In the gravimetric determination of iron the metal is frequently precipitated as the hydroxide by ammonia. It is then collected by filtration, dried, ignited and weighed as the oxide. Such precipitates when wet, as hydroxides, dissolve instantaneously in acids. After the precipitate has been ignited it often becomes practically insoluble. Such ignition gives a range of temperature of 600°-1000°C, depending on the type of heating device used. Kolthoff and Sandell suggest the fusion of this ignited ammonia precipitate with alkali pyrosulfate "in order to convert the oxides into sulfate which then can be dissolved." This procedure is troublesome because the alkali must then be removed before the iron is determined either volumetrically or gravimetrically.

Chromium is not usually determined in this way because the

strong heating forms some  $\text{Cr}_2(\text{CrO}_4)_3$  which gives high results unless the oxide is ignited in hydrogen.

Different forms of ferric oxide can be prepared. The ordinary quantitative procedure of preparation and determination of the oxide is the precipitation of the hydroxide from a ferric salt solution by ammonia. This precipitate is then filtered and washed with ammonium nitrate or chloride. The precipitate is then dried overnight in an oven at  $150^\circ\text{C}$ . It has an indefinite amount of water combined with it. Upon ignition the oxide becomes less soluble in acids. When this oxide is heated to temperatures up to  $1000^\circ\text{C}$  the oxide will completely dissolve in concentrated hydrochloric acid in a little over an hour and give 100 per cent ferric by analysis.

As the temperature of ignition is increased the solubility of the oxide decreases. Heating of the oxide for as short a time as four hours does not greatly effect the solubility. Longer than four hours heating is required. This shows that as the temperature (for a constant period of time) is increased, the solubility decreases.

	900°	1000°	1100° each for four hours
Given 2 hours to dissolve in HCl at 70 C.	100%	100%	98.5%
	800°	900°	— 1200° each for four hours
Given 1 hour to dissolve in HCl at 31 C.	14.1%	10.9%	7.67%

Using a constant temperature and varying the time of ignition, the following at  $1180^\circ\text{C}$ , it is seen that the solubility of the oxide is decreased as the time of ignition at a constant temperature is increased.

Time of ignition	Percentage dissolved in 30 minutes at 80 C.
9 hours	98.28
13	77.88
25	64.15
32	61.95
35½	59.64
41	53.55
53	50.41
70	37.57

Hydrated chromic oxide, prepared in the same manner as the ferric oxide, by precipitation of the hydroxide from a chromic salt solution by ammonia and dried in the oven at  $100^\circ\text{C}$ , when heated becomes insoluble in mineral acids at a much lower tem-

perature than does the ferric oxide. The hydrated chromic oxide when heated to 400°C for even as short a time as four hours is soluble to no appreciable extent in contact with concentrated hydrochloric acid for 48 hours at 80°C. The same oxide when heated for two hours at 300°C is almost completely dissolved in concentrated hydrochloric acid in one hour.

At a temperature of 350°C the oxide shows increase in the time necessary for solution.

Time of heating at 350°C.	Time for 100% solution of 0.1g sample in H <sub>2</sub> SO <sub>4</sub> at 80° C.
1 hour	3 hours
2 hours	5
3	8
6	15
13	25

In each case a much longer time was taken for solution in hydrochloric acid, and as the time of ignition increases, the oxide approaches insolubility in hydrochloric acid.

This shows that for both ferric oxide and for chromic oxide, the solubility in acids is dependent on the temperature of ignition, the time of ignition, and the time allowed for solution.

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## THE EFFECT OF EGG ALBUMIN ON THE SOLUBILITY OF THALLOUS SALTS AT 25°C.

W. G. EVERSELE AND CAROLD F. BJORK

The solubilities of TlCl, TlCNS, and Tl<sub>2</sub>SO<sub>4</sub> were determined in solutions of egg albumin at 25°C.

The Tl<sup>+</sup> was titrated to Tl<sup>+++</sup> with standard KIO<sub>3</sub> using the IC1 endpoint. The albumin concentration was determined by a modified Kjeldahl method. The pH of each solution was obtained with a glass electrode using M/20 potassium acid phthalate as a standard (pH = 3.97).

In solutions of egg albumin dialyzed free of chlorides and sulphates and adjusted to a pH of 3.3 it was found that the greater the albumin concentration the greater was the amount of dissolved salt per 1000 g. of water.