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Another Way for the Laboratory Preparation of Anhydrous Chromium(III) Chloride

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Two methods for the laboratory preparation of anhydrous chromium(III) chloride are described in the standard handbooks of inorganic syntheses, *i. e.* (i) the chromium metal is heated in a porcelain tube in a stream of chlorine at high temperature¹, (ii) the green aquo-chloride $[\text{CrCl}_2(\text{H}_2\text{O})_4]\text{Cl} \cdot 2\text{H}_2\text{O}$ is heated and the resulting mass of oxide and basic chloride is then chlorinated with carbon tetrachloride².

The method (i) is convenient owing to its simplicity but the method (ii) may be preferred in many laboratories because the starting material is easier available, although it is not as simple as the previous one. Hence we tried to modify the method (ii) and we succeeded in applying the thionyl chloride as dehydrating agent in a manner described previously³ for the preparation of the anhydrous chlorides of nickel(II) and cobalt(II).

In a 1-1. round bottomed flask with a ground joint, fitted with an efficient reflux condenser, the upper end of which was protected with a calcium chloride drying tube, 100 g. of finely pulverized hydrated chromium(III) chloride $[\text{CrCl}_2(\text{H}_2\text{O})_4]\text{Cl} \cdot 2\text{H}_2\text{O}$ and 325 ml. of thionyl chloride were placed. The mixture was gently refluxed on a water bath for six hours. After this time no traces of hydrogen chloride could be detected to escape from the condenser through the drying tube. The colour of the powder in the flask turned completely from green to violet. The thionyl chloride was distilled over and then completely removed by heating on a water bath in a stream of dry air and eventually under reduced pressure.

The crude anhydrous chloride can be easily shaken out of the flask and stored in a tightly stoppered bottle. It deliquesces on free air since it contains chromous chloride and must be purified by sublimation in a stream of dry chlorine. The sublimation was carried out in a vitreous silica tube (2—3 cm. in diameter, 80 cm. in length) in portions of 8—10 g. of the crude material placed in a porcelain boat, at a temperature of 950°C of an electric furnace. The sublimate is easily separated from the impurities deposited at the end of the tube and can be eventually purified by boiling with concentrated hydrochloric acid and washed with water.

REFERENCES

1. *Handbuch der präparativen anorganischen Chemie*, herausgegeben von G. Brauer, Stuttgart 1954, p. 1004.
2. *Inorganic Syntheses*, edit. by W. C. Fernelius, Vol. II., New York and London 1946, p. 193.
3. See ref. (1) pp. 1131 and 1154, resp.

IZVOD**Još jedan postupak za laboratorijsku preparaciju bezvodnog krom-(III)-klorida***D. Grdenić i M. Đorđević*

Opisuje se postupak za dobivanje bezvodnog krom-(III)-klorida iz zelenog hidrata $[\text{CrCl}_2(\text{H}_2\text{O})_4]\text{Cl} \cdot 2\text{H}_2\text{O}$. Potonji se oslobada vode grijanjem s tionil-kloridom uz povratno hladilo, a sirovi bezvodni klorid sublimira se u struji suhog klora, u cijevi od kvarcnog stakla, na temperaturi od 950°C .

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