

crotonic acid and isocrotonic acid, which are oxidized, respectively, to *dl-threo- α,β* -dihydroxybutyric acid and to *dl-erythro- α,β* -dihydroxybutyric acid.^{1,6}

Hitherto, silver chlorate has been prepared by passing chlorine gas into a suspension of silver oxide,² silver carbonate,⁷ or an aqueous solution of silver fluoride⁴ or by the action of chloric acid on silver oxide,⁸ finely divided silver,⁵ or silver carbonate.³ These methods are neither dependable nor satisfactory.

The procedure outlined below is based upon a consideration of the solubilities of silver nitrate, silver chlorate, and the corresponding sodium salts. Under appropriate conditions simple metathesis involving interaction of concentrated solutions of silver nitrate and sodium chlorate leads to the desired product.

Salt	Soly., g./100 ml. H ₂ O			
	0°	15°	80°	100°
AgClO ₃	...	10	50	
AgNO ₃	122	952
NaClO ₃	79	230
NaNO ₃	73	180

Procedure

A product of sufficient purity for use in the oxidation reactions mentioned above may be prepared by dissolving 170 g. (1 mol) of silver nitrate and 106 g. (1 mol) of sodium chlorate each in 100 ml. of water. After the two solutions are heated to 85°, they are mixed and allowed to cool slowly to 0°, and at this temperature the mother liquor is carefully decanted. Fifty milliliters of distilled water, previously cooled to 0°, is added to the solid product, and the crystals are separated by suction filtration. Yield 150 g. (78.5 per cent). The product contains 95.2 per cent AgClO₃ as indicated by a silver determination.

For further purification the product obtained after the

initial filtration is dissolved in 125 ml. of distilled water at 90°, the solution cooled to 0°, and the product filtered by suction. This procedure is repeated with 120 ml. of water for the second recrystallization. The product is dried in a desiccator. Yield 118.7 g. (62 per cent); purity 99.7 per cent AgClO₃.

Since silver chlorate decomposes slightly (darkens in color) when exposed to light, it should be stored in a dark bottle. While the opinion of the authors, based on their own experience, is that silver chlorate is perfectly safe to handle, attention is directed to the fact that chlorates in general are powerful oxidizing agents. Caution would dictate the advisability of keeping silver chlorate away from organic vapors and other easily oxidizable materials.

Properties

Silver chlorate forms heavy white crystals, which melt at 230°. The compound decomposes when heated above its melting point.

References

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3. RECOVERY OF SILVER AND IODINE FROM SILVER IODIDE RESIDUES¹

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Considerable quantities of silver iodide-silver oxide residues sometimes accumulate in the laboratory, for

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example, in methylation studies involving the use of methyl iodide and silver oxide.² While silver alone is usually recovered from silver residues, iodine is valuable enough to warrant its recovery when the residue contains a large proportion of silver iodide. In the method described, the presence of silver iodide and of silver salts soluble in concentrated aqueous ammonia is assumed. If significant quantities of contaminating salts, such as silver sulfide, are present, suitable modification is required.

Procedure

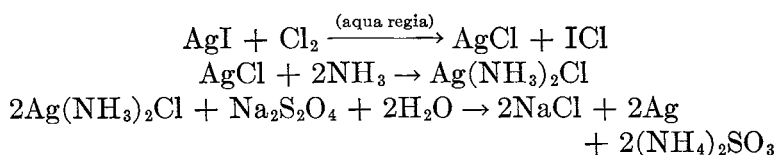
A. PRELIMINARY TREATMENT OF RESIDUE



The residue containing silver iodide is freed from organic matter by extraction with a suitable solvent, dried, and ground to pass a 40-mesh sieve. The powdered residue is shaken with sufficient concentrated ammonia (sp. gr. 0.90) to dissolve all soluble silver salts. The suspension is filtered on a Büchner funnel and the filtrate (I) is reserved for subsequent recovery of silver by reduction with sodium dithionite solution.

The insoluble silver iodide is washed with water on the filter, dried, and weighed.*

B. RECOVERY OF SILVER



The powdered silver iodide (40-mesh) is treated with excess aqua regia (under hood). A solution consisting of 81 ml. of concentrated nitric acid (sp. gr. 1.42) and 216 ml. of concentrated hydrochloric acid (sp. gr. 1.19) is suitable

* It is desirable to know the weight of silver iodide because a subsequent reaction involving the reduction of iodic acid with sodium dithionite requires exact quantities.