Analytical conditions for the quantitative estimation of these elements have been determined and their limitations discussed.

Apparatus and procedure for the rapid and accurate determination of solubilities have been developed for future work.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, RESEARCH INSTITUTE OF CUTANEOUS MEDICINE]

SODIUM AUROTHIOSULFATE. A SIMPLE METHOD FOR ITS PREPARATION

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The preparation first described by Fordos and Gélis[1] as sodium aurothiosulfate has recently attained considerable prominence through the claims by Møllgaard and his associates[2] for its curative properties in tuberculosis. The value of the compound in the treatment of *lupus erythematosus*, which is believed by many to be of tuberculous origin, has been indicated by Schamberg and Wright[3] and during the past year hundreds of ampoules of sodium aurothiosulfate have been distributed from this Institute to various dermatologists.

In view of the increasing demand, an economical method for preparing this compound assumed considerable importance. Møllgaard's method not being available, the procedure of McCluskey and Eichelberger,[4] employing cesium auric chloride as the intermediate, was used at first. However, the price of cesium nitrate soon increased enormously and it became difficult to obtain, making desirable the development of a method of preparation which would not require cesium. The procedure described below presents several advantages over that of McCluskey and Eichelberger, in that the use of cesium is eliminated, the process is carried out in one continuous operation and is completed within a few hours. The yields are over 95%.

To 41.2 g. (0.1 mole) of hydrochloro-auric acid dissolved in 75 cc. of water is added 40% of sodium hydroxide solution, dropwise, until a faint alkaline reaction is obtained to litmus, and the gold hydroxide is precipitated. The entire contents of the beaker is then added to 102 g. (0.4 mole plus 10% excess) of hydrated sodium thiosulfate dissolved in 200 cc. of water, and the mixture is stirred mechanically. At this point no apparent change takes place in the gold hydroxide suspension. After about five minutes, and while stirring, approximately 4 *M* nitric acid is

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[1] Fordos and Gélis, *Ann. chim. phys.*, **13**, 394 (1845).

[2] Møllgaard, "Chemotherapy of Tuberculosis," *Nyt Nordisk Forlag,* **1924.**

[3] Schamberg and Wright, *Arch. Derm. Syph.*, **15**, 119 (1927).

[4] McCluskey and Eichelberger, THIS JOURNAL, **48**, 136 (1926).

slowly added from a dropping funnel, the rate of addition of the acid being so regulated that the red color formed in the mixture from any one drop is nearly or completely discharged before the next drop enters. The red color is probably due to the formation of the sodium salt of auric acid, since the same color may be noted upon the addition, in the cold, of a fixed alkali to a solution of hydrochloro-auric acid before the gold hydroxide is precipitated. About 45 cc. of acid is required. After about half of the acid is added, all of the gold hydroxide has dissolved. The end of the reaction is indicated when the red color is no longer produced; care should be taken to avoid an excess of acid. At the end-point the solution is nearly colorless, and neutral to litmus paper. Stirring is now continued for about five minutes longer or until the first appearance of a faint turbidity, due to sulfur and some gold sulfide.

The reactions involved may be represented by the equations $Au_2O_3 + 4Na_2S_2O_3 + 2H_2O \longrightarrow 4NaOH + 2Na_2S_4O_6 + Au_2O$; $Au_2O + 4Na_2S_2O_3 + H_2O \longrightarrow 2NaOH + 2Na_3Au(S_2O_3)_2$.

The solution is then filtered and the filtrate treated with four volumes of alcohol. The precipitate is white but is contaminated with sulfur and, as a rule, is not crystalline. It is filtered, dissolved in a minimum amount of water, filtered from the sulfur and precipitated with four volumes of alcohol. This precipitate is crystalline and, ordinarily, is sufficiently pure for use. A very pure product is obtained if the compound is again treated as above, with water and alcohol. The final product is sucked as dry as possible on a Büchner funnel and dried in the dark, in a vacuum desiccator over sulfuric acid. Overnight drying is sufficient to give a dry, finely crystalline, white powder soluble to a water-clear solution in two parts of water, and giving nearly theoretical values for gold and sulfur.

Anal. Subs., 0.5: Au, 0.1861; $BaSO_4$, 0.8932. Calcd. for $Na_3Au(S_2O_3)_2.2H_2O$: Au, 37.4; S, 24.36. Found: Au, 37.22; S, 24.54.

It is unnecessary to preserve the compound under anhydrous ether, as recommended by McCluskey and Eichelberger. We have found that decomposition is greatly retarded by merely keeping in well-stoppered amber bottles in the refrigerator. Such specimens have been maintained unchanged for over five months. In amber ampoules, sealed under slightly reduced pressure, the compound will keep for at least three months, at room temperature.

Summary

1. A new, rapid method, avoiding the use of cesium, has been developed for the preparation of sodium aurothiosulfate.

2. Simple procedures for preserving and distributing the compound are indicated.

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