EXPERIMENT NINE

Part I - The Standardization of Thiosulfate Solutions

In general, thiosulfate solutions are standardized by indirect methods. Primary-standard oxidizing agents such as KIO₃, As₂O₃, or K₂Cr₂O₇ are used to liberate an equivalent amount of I₂ from fairly concentrated Γ solutions. The resulting I₃ is then titrated with thiosulfate to a starch endpoint. In this course we shall use potassium dichromate as the oxidizing agent.

Iodine is liberated according to the equation

(1)
$$Cr_2O_7^{2-} + 6\Gamma + 14H^4 \rightarrow 2Cr^{3+} + 3I_2 + 7H_2O$$

For this reaction to be quantitative the H* and Γ concentrations, and the time must all be controlled. The equilibrium in reaction (1) lies well to the right at pH < 4, but the rate of the oxidation is very low unless the hydrogen ion concentration is greater than 0.2 M and the iodide concentration is greater than 0.05 M. Under these conditions the reaction is quantitatively complete in 5 minutes.

If the H* concentration exceeds 0.4 M, air oxidation of iodide can also occur by the reaction,

(2)
$$O_2 + 4 \Gamma \rightarrow 2 I_2 + 2 H_2O$$

Up to 0.4 M acid the rate of reaction (2) is low enough that solutions can stand in air up to 10 minutes without appreciable amounts of iodine appearing. Reaction (2) is, however, catalyzed by light and various metal ions, including Cr³⁺. For this reason tartaric acid as added to the system to complex the Cr³⁺ formed by reaction (1).

Saturated solutions of I_2 in pure water are only 1.3×10^{-3} M, and the vapor pressure above these solutions is so great that large amounts of iodine escape. In the presence of excess iodide ion, iodine becomes soluble by the reaction.

(3)
$$I_2 + \Gamma \rightarrow I_3$$

Triiodide solutions are reasonably stable. Moreover, the starch-triiodide complex is much more intensely colored (blue) than its I₂ counterpart (pale yellow-brown) so the end point of the titration is sharpened.

The soluble fraction of vegetable starches, B-amylose, is believed to hold I₃' inside the helical chains of its polymeric (mol. wt. 10,000-50,000) structure. The formation of the blue complex is reversible provided the I₃' concentration is rather low, otherwise a red-violet complex is formed irreversibly and the end point may be obscured. Starch solutions customarily are prepared with traces of mercuric iodide or boric acid to impede bacterial decomposition of the solutions.

Thiosulfate Standardization Chemistry Lab

Gary D. Christian, Purnendu K. Dasgupta, Kevin A. Schug

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