

EXPERIMENT NINE

Part I - The Standardization of Thiosulfate Solutions

In general, thiosulfate solutions are standardized by indirect methods. Primary-standard oxidizing agents such as KIO_3 , As_2O_3 , or $\text{K}_2\text{Cr}_2\text{O}_7$ are used to liberate an equivalent amount of I_2 from fairly concentrated I^- solutions. The resulting I_3^- 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



For this reaction to be quantitative the H^+ and I^- concentrations, and the time must all be controlled. The equilibrium in reaction (1) lies well to the right at $\text{pH} < 4$, 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,



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 Cr^{3+} . For this reason tartaric acid is added to the system to complex the Cr^{3+} 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,

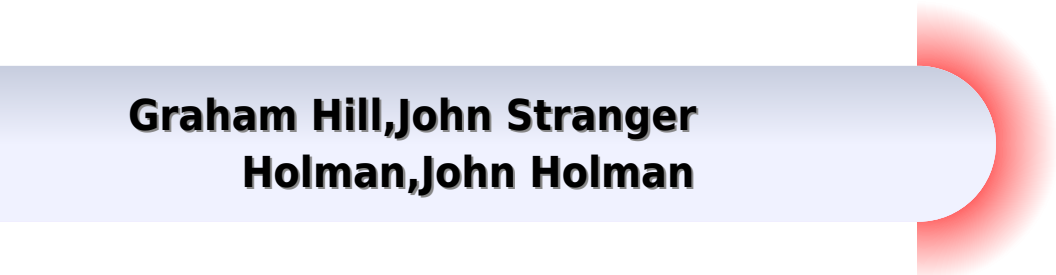


Triiodide solutions are reasonably stable. Moreover, the starch-triiodide complex is much more intensely colored (blue) than its I_2 counterpart (pale yellow-brown) so the end point of the titration is sharpened.

The soluble fraction of vegetable starches, B-amylase, is believed to hold I_3^- inside the helical chains of its polymeric (mol. wt. 10,000-50,000) structure. The formation of the blue complex is reversible provided the I_3^- 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

**Graham Hill, John Stranger
Holman, John Holman**



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Chemistry in the Laboratory James M. Postma, Julian L. Robert, J. Leland Hollenberg, 2004-03-12 This clearly written class tested manual has long given students hands on experience covering all the essential topics in general chemistry Stand alone experiments provide all the background introduction necessary to work with any general chemistry text This revised edition offers new experiments and expanded information on applications to real world situations **Laboratory Manual in**

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chemical principles as well as techniques The manual helps students understand the timing and situations for the various techniques The Beran lab manual has long been a market leading lab manual for general chemistry Each experiment is presented with concise objectives a comprehensive list of techniques and detailed lab intros and step by step procedures

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