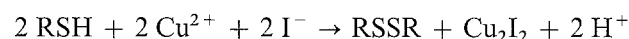


Titration of Thiol Groups with Cupric Ion

Krishna K. Verma, Jameel Ahmed, and Sameer Bose
Department of Chemistry, University of Jabalpur, India
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Titration von Thiolgruppen mit Kupfer(II)-Ionen
Best. von Thiolen; Volumetrie; Kupfer(II).

A method of determining thiols with cupric ion is suggested. Samples are titrated directly in the presence of potassium iodide with a standard solution of cupric sulphate:



The procedure is convenient and rapid. The results are favourably comparable with those of iodimetric determinations.

Mercaptans tested were: mercaptoacetic acid, 2-mercaptoethanol, 1-butanethiol, benzenethiol, thio-

nalide, 2-naphthalenethiol, p-chlorobenzenethiol and 2-diethylaminoethanethiol hydrochloride.

Procedure. Take an aliquot of sample dissolved in water or dimethylformamide containing 0.1–0.3 meq of mercaptan in a 150-ml Erlenmeyer flask. Add 25 ml of water, 1 g of potassium iodide and 1 ml of 1% starch solution. Titrate with 0.02 M solution of cupric sulphate taken in a 10-ml burette (graduated at 0.02 ml). Shake the solution vigorously during titration. At the end point a blue colour is formed.

Under the described conditions of titration no cuprous mercaptide is formed. Cuprous iodide precipitates during the course of titration.

The present method is accurate and precise to 0.1%. It has the advantage that only one solution is required. Like the micro-iodimetric procedure which involves use of potassium iodate, standard solution of cupric sulphate can be prepared from a primary reagent grade sample and can be used for a long period.

Dr. Krishna K. Verma, 363 Chhoti Omti, Jabalpur 482002, India

Spectrophotometric Determination of Aromatic m-Polynitro Compounds using Sodium Borohydride

G. Srivastava, M. Gopal, U. C. Pande, and R. D. Tiwari
Dept. of Chemistry, University of Allahabad, India
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Spektralphotometrische Bestimmung von aromatischen m-Polynitroverbindungen unter Verwendung von Natriumborhydrid

Best. von Polynitroverbindungen mit Borhydrid; Spektralphotometrie; aromatische m-Polynitroverbindungen.

The colour formation between m-di or polynitro compounds and sodium borohydride in absolute ethanol has been made the basis for a spectrophotometric method of determination for these compounds. In comparison with other methods this procedure has the advantage of being applicable to a specific type of aromatic nitro compounds. Other

types of nitro compounds do not interfere. Beer's law is obeyed in the range of 1–700 µg, accuracy being ± 2%. The following compounds (wavelength of maximum absorption in parentheses) have been investigated: 2,4-dinitrobenzene (520 nm), 2,4-dinitrobenzaldehyde (520 nm), 1-chloro-2,4-dinitrobenzene (525 nm), 3,5-dinitrobenzoic acid (520 nm), 2,4-dinitrotoluene (530 nm), 1,8-dinitronaphthalene (530 nm), 2,4,7-trinitrofluorenone (525 nm), 1,3,5-trinitrobenzene (520 nm).

Procedure. Deliver 4 ml of the ethanolic sample solution into a 25 ml volumetric flask, add 8 ml of ethanolic sodium borohydride solution (2 mg/ml), keep for 25 min to develop the colour (in the case of sym-trinitrobenzene and trinitrofluorenone the reaction is complete within 10 min only), shake and make up to the mark with absolute ethanol. Measure the absorption at the wavelengths given above. Prepare a calibration curve accordingly.

Dr. (Mrs.) G. Srivastava, Chemistry Department, University of Allahabad, Allahabad-211002, India