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Rapid Spectrophotometric Determination of Manganese(II) with 4-(2-Thiazolylazo)-resorcinol

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With 1 Figure

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Introduction

Heterocyclic azodyes find considerable application\textsuperscript{1-5} in analytical chemistry, due to the sensitivity and/or selectivity of their reactions with various metal ions. The resulting metal chelates are either water soluble or can be extracted into nonaqueous phase. 4-(2-pyridylazo)-resorcinol, PAR, and (4-(2-thiazolylazo)-resorcinol, TAR, are most useful among pyridylazo and thiazolylazo phenol dyes, TAR being more reactive to metal ions in acid medium than PAR. 4-(2-Thiazolylazo)-resorcinol has been reported for spectrophotometric determination\textsuperscript{4} of some metal ions. It has been found to react slowly\textsuperscript{6-8} with certain transition metal ions, heating being sometimes required.

Manganese(II) was determined\textsuperscript{9,10} spectrophotometrically using PAR. Since PAR and TAR form metal chelates of comparable sensitivity and stability, it was deemed worthwhile to study the reaction of 4-(2-thiazolylazo)-resorcinol with manganese(II). This has resulted in a rapid method for the spectrophotometric determination of manganese(II), which has been applied for the determination of manganese content in alloy steels.
Experimental

Apparatus

A spektromom 204 spectrophotometer was used for absorption measurements. The absorption spectrum was recorded on a Varian Superscan 3 U. V.-Visible Spectrophotometer. A Cambridge pH meter was used for pH measurements.

Reagents

Manganese sulphate. Stock solution of manganese(II) containing 5.33 mg/ml of manganese was prepared from manganese(II) sulphate heptahydrate (BDH AnalaR), and standardised titrimetrically. Experimental solutions were obtained by appropriate dilution of stock solution.

4-(2-Thiazolylazo)-resorcinol (TAR). 0.1% solution of 4-(2-thiazolylazo)-resorcinol (Fluka AG, Purum) was prepared by dissolving 100 mg of TAR in 100 ml of methyl alcohol.

Buffer solutions of various pH values were prepared by dropwise addition of 1.0 N HCl (AR) to 0.05 M Na₂B₄O₇ (BDH AnalaR). All other chemicals were of BDH (AnalaR).

Procedure

Take an aliquot (2 to 5 ml) of the faintly acidic solution containing ≤40 μg of Mn(II), add 10 ml of borate buffer of pH 8.8, 5 ml of tert-butyl alcohol and 1.5 ml of 0.1% TAR solution. Make up the contents to 25 ml with distilled water and measure the absorbance of the red coloured complex at 540 nm after 30 s against a reagent blank prepared under identical conditions.

Results and Discussion

Manganese(II) forms a red coloured complex with TAR in alkaline media.

Effect of pH and buffer concentration. The pH of the reaction mixture was varied from 3.5 to 10, and absorbance was found to be maximum in the range 8.3—9.0. Hence, all studies were carried out at pH 8.8, and pH was maintained with borate buffer. It was also noted that maximum absorbance was obtained on addition of ≥9.0 ml of buffer. Thus, 10 ml of buffer solution was used in all studies.

Effect of reagent concentration. To an aliquot of manganese solution containing 25 μg of manganese per 25 ml, varying amounts of 0.1% TAR solution were added. Absorption was found to be maximum on addition of 0.9 to 1.5 ml of the reagent, and was