

from the above table that the compound was more stable in presence of two moles of β -cyclodextrin than the same amount of γ -cyclodextrin. The half life of 80/53 was almost constant in 80/53: β -CD (1:2) complex. It was found to be about 300 min at all the pH studied as compared to 80/53 itself which varied from 8 minutes at pH 4 to about 143 minutes at pH 7. Thus it can be concluded that β -cyclodextrin increases the stability of 80/53 in solution in 1:2 (80/53: β -cyclodextrin) molar ratio.

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Determination of Some Sulpha Drugs With Potassium Ditelluratocuprate (III) in Alkaline Medium

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A simple and convenient titrimetric method for the determination of sulpha drugs in pure form, at milligram level, is developed using potassium ditelluratocuprate (III) in alkaline medium. The response of the titration is observed to be precise between 1-10 mg drug sample within $\pm 0.5\%$.

Sulpha drugs are widely used in the treatment of infections¹, especially for patients intolerant to antibiotics. Therefore a rapid, accurate and an economic method for their determination is essential. Determination of sulpha drugs is reported by titrating as acids and bases^{2,4}, through bromination^{5,6} and chlorination⁷. Estimation of certain sulpha drugs, in mg quantities with the use of N-bromosuccinimide has been reported⁸. Where as, the halogenation method needs drastic reaction conditions,

the use of N-bromosuccinimide in the other method is cumbersome owing to its instability at room temperature. Though the nitrite titration method^{9,10} has been used for the determination of sulpha drugs, the diazotization may get influenced by several factors including the unstable behaviour of diazo salts and nitrous acid involved. Determination of certain sulpha drugs with ammonium hexanitratocerate (IV)¹¹ in nitric acid medium has been reported. A new method has been proposed for the determination of sulpha drugs through conversion into

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an N-bromoderivative and subsequent iodometric determination¹². Some of the titrimetric determinations of sulpha drugs are reported to use internal indicators^{13,14}.

Potassium ditelluratocuprate (III) has been applied to the oxidimetric determination of many organic^{15,16} and inorganic¹⁷ substance in alkaline medium. The present investigation aims at exploring the application of potassium ditelluratocuprate (III) [Cu(III) reagent] for oxidimetric determination of sulpha drugs, such as sulphanilamide, sulphapyridine, sulphathiazole and sulphadiazine, in an alkaline medium.

Potassium ditelluratocuprate (III) reagent was prepared and standardized following known recipe¹⁵. The requisite amounts of sulphanilamide, sulphapyridine, sulphathiazole and sulphadiazine (Sigma Chemicals, U.S.A., May and Baker Ltd., England) were first dissolved in of 0.05 N NaOH and then diluted with distilled water to obtain the solutions of final concentration of 1 mg/ml. In all titrations, micropipettes (least count 0.01 ml) and micro-burette (least count 0.01 ml) were used.

Aliquots containing 1-5 mg of the sample were taken in 150 ml Erlenmeyer flasks and known excess (5-10 ml) of 0.035 M Cu (III) solution was added. The reaction mixture as heated on a boiling water-bath for 15 minutes and the contents were cooled to room temperature. The un-used Cu (III) was determined by the arsenite method¹⁵. A blank was also run simultaneously using all the reagents excluding sample. The molar ratio i.e. molecularity of the Cu (III) reagent with the sample was ascertained using the volume of the reagent consumed for the sample. The recovery of the sample was calculated in the manner reported earlier^{11,21}.

The procedure applied for the determination of sulphanilamide, sulphapyridine, sulphathiazole and sulphadiazine is observed to be reproducible at milligram level up to 10 mg of the sample in the present instance. The effect of temperature, reaction-time and volume of Cu (III) reagent revealed the fact that best result could be obtained at boiling water bath with reaction to be completed within 15 minutes in all cases. For the best results, 1 ml of sulpha drug requires 5 ml of Cu(III) reagent giving rise to maximum recovery 99.5%. As is evident from the establishment of molecularity of Cu (III) with

sulpha drug, 1 mole of sulphonamide consumes 6 moles of Cu(III) reagent. This indicate that the presence of 6 moles of Cu(III) reagent, the p-aminogroup of the drug usually gets oxidize to the corresponding nitrogroup^{11,19,20}.

The method suggested warrants prior removal of any reducing impurities such as alcohols, phenols, sugars, amines, aldehydes and thioureas from the test analyte to ensure maximum recovery. The proposed method is simple in a sense that the determination can be done in any ordinary laboratory. The work offers an economic and rapid method for the quality control of bulk sulpha drugs at pilot scale.

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