



Quantitative Chemical Analysis of Nitrogen and Sulphur containing Ligand Thiopicolinanilide (TPA), Silver Complex and its Experimental Results.

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ABSTRACT

Quantitative chemical analysis deals with the determination of the amount or percentage of one or more constituents of the compound. The thermogravimetric analysis (TGA) method is used in determination of amount and percentage of constituents of nitrogen and sulphur containing ligand thiopicolinanilide (TPA) with silver complex. The technique of thermogravimetric analysis involves change in weight of a system under examination with increase in temperature at predetermined and preferably at a linear rate study the solid reaction. In the present with the physico-chemical data on kinetic parameters i.e. apparent activation energy, frequency factor, activation entropy and the order of reaction of silver complex with Nitrogen and Sulphur containing ligand Thiopicolinanilide (TPA) have been evaluated from their thermogravimetric curves which have been recorded on an electrobalance with auto recorder.

KEY WORDS

Quantitative Chemical Analysis, Thermogravimetric Analysis (TGA), Thiopicolinanilide (TPA), Silver Complex

INTRODUCTION

The Nitrogen and Sulphur containing ligand Thiopicolinanilide (TPA) was prepared by refluxing a mixture of α -picoline, aniline and solid sulphur on the oil bath. Aniline and α -picoline were primarily distilled 48.2 gram of sulphur, 46.5 of α -picoline and 68.91 gram of aniline were taken in a round bottom flask and refluxed on an oil bath at 162°C for twelve hours. The refluxed mixture was cooled and then distilled in vacuum to remove unreacted aniline and α -picoline. The crude product was transferred in another flask, cooled and redistilled to give a yellow solid. The solid product was dissolved in a mixture of n-hexane and benzene and crystallized. The mass was washed with water and finally with ethanol. It was dried to give a crystalline pale yellow mass with a melting point of 51.53°C.

Complexion of silver (I) Nitrate with ligand TPA

An ethanolic solution of Silver (I) Nitrate (0.18 gram Silver Nitrate dissolved in 20 ml alcohol) was added slowly with constant stirring to the ethanolic solution (0.41 gram dissolved in 20 ml of ethanol) of the ligand. The cream yellow complex was immediately separated. It was digested, filtered and washed with water till free from nitrate ion and dried.

Estimation of Silver

The weighed amount of the complex was decomposed by repeated evaporation with conc.nitric acid and then diluted with water. The solution was heated to approximately 60°C and 0.2N Hydrochloric acid was added slowly in excess amount. The precipitate was allowed to stand in a dark place for several hours and filtered through weighed sintered glass crucible. The residue was washed with 0.1N nitric acid and distilled water until free from chloride ions, it was dried at 130-150°C, cooled and finally weighed. The silver content was known by multiplication of the mass with the factor 0.75264.

INSTRUMENTAL TECHNIQUE

An electrobalance standard Red Craft (TG) Balance Model 750 with a recorder, operating on 1.0mv full scale for obtaining thermograms i.e. temperature vs. loss in weight curves was used. A chrome lalumel thermocouple placed 3-4mm below the sample holder, the platinum boat 2mm deep 8mm diameter was used for recording the sample temperature. A 10°C per minute heating rate was employed for recording the pyrolysis curves.

RESULT AND DISCUSSION

Table – 01

Quantitative Chemical Analysis of Silver Compound

Compound	Molecular Formula	% of Carbon		% of Nitrogen		% of Hydrogen		% of Sulphur		% of Silver	
		Expt	Obsv	Expt	Obsv	Expt	Obsv.	Expt	Obsv	Expt	Obsv
Ligand	C ₁₂ H ₁₀ N ₂ S	67.28	67.23	13.08	13.11	4.67	4.68	14.95	14.92	NIL	NIL
Silver	AgL.2H ₂ O	40.35	40.42	7.84	7.91	3.64	3.62	8.97	8.91	30.21	30.30

Table – 02

Colour and Solubility of the Silver Complex

Complex	Colour	Solubility
AgL.2H ₂ O	Yellow	Meth,Eth,Etr,Acet,N-N Mef

Symbol

Meth = Methyl Alcohol, Eth = Ethyl Alcohol, Eth=Ether, Acet=Acetone-N Mef = N-N Methyl Formamide

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