



# Elemental Analysis of Thiopicolinanilide (TPA) and its Lead (II) Complex.

Dr.Rudra Narayan Sharma

## ABSTRACT

The term elemental analysis is typically defined as the determination of the amount of an element in given sample. Elemental analysis determining what element are present and determining how much each are present. The elemental analysis of Thiopicolinanilide (TPA) and its lead (II) complex carried out by an electrobalance Stanton Red Craft (TG) Balance Model with a reader, operating 1.0mv full scale for obtaining thermograms i.e. temperature vs. loss in weight curves.

## KEY WORDS

Elemental Analysis, Thermograms, Lead (II) Complex, Thiopicolinanilide (TPA)

## INTRODUCTION

The elemental analyses of Thiopicolinanilide (TPA) and Lead (II) complex were carried out by conventional method to assign the formula. The different methods employed as follows Freeman-Carroll Method, Coats-Redfern Method, Karkhanawala-Dharwadkar Method and Zsako Method. The breaking of bonds, distribution of lattice of the solid product consists of formation of crystallization centres and the growth of their centres and adsorption, desorption of the gaseous product, diffusion and heat transfer. In view of the complexity of the thermal decomposition, it is often difficult to determine the various kinetic parameters i.e. order of reaction, activation energy, apparent frequency factor and apparent activation energy. 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.

## PREPARATION OF LIGAND

The Nitrogen and Sulphur containing ligand Thiopicolinanilide (TPA) was prepared by refluxing a mixture of  $\alpha$ -picoline, aniline and solid sulphur on the oil bath.

Aniline and  $\alpha$ -picoline were primarily distilled 48.2 gram of sulphur ,46.5 of  $\alpha$ -picoline and 68.91gram of aniline were taken in a round bottom flask and refluxed on as oil -bath at 162<sup>0</sup>C for twelve hour. The refluxed mixture was cooled and then distilled in vacuum to remove to remove off an unreacted aniline and  $\alpha$ -picoline. The crude product was transferred in another flask, cooled redistilled to give 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 crystalline pale yellow mass of melting point 51.53<sup>0</sup>C.

**PREPARATION OF LEAD (II) COMPLEX WITH LIGAND THIOPICOLINANILIDE (TPA)**

About 0.01M ligand was dissolved in ethanol (50ml) and was treated with Lead (II) acetate solution in ethanol, in warm condition with constant stirring. The mixture was refluxed on water bath for half an hour and then filtered, washed thoroughly with hot alcohol and finally with distilled water. The precipitate was dried and collected in glass Stoppard bottle.

**ESTIMATION OF CARBON, HYDROGEN, NITROGEN AND SULPHUR OF LEAD (II) COMPLEX**

The carbon, Hydrogen, Nitrogen and Sulphur content of Thiopicolinanilide (TPA) ligand and the lead (II) complex was carried out in CDRI, Lucknow. The lead complex was decomposed and metal content was determined by the standard procedure. The Lead complex was decomposed by boiling the concentrate nitric acid and was evaporated till syrupy mass water and lead precipitate as lead molybdate was filtered crucible, dried at 500-600°C muffle furnaces and weighed as  $PbMoO_4$ . The gravimetric factor for lead is 0.56436

**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 alumel 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****Elemental Analysis of Thiopicolinanilide (TPA) and Lead (II) Complex**

Compound	Molecular Formula	% of Carbon		% of Nitrogen		% of Hydrogen		% of Sulphur		% of Metal	
		Expt	Obsv	Expt	Obsv	Expt	Obsv.	Expt	Obsv	Expt	Obsv
Ligand	$C_{12}H_{10}N_2S$	67.28	67.23	13.08	13.11	4.67	4.68	14.95	14.92	NIL	NIL
Lead	$PbL_2.2H_2O$	31.46	31.48	6.12	6.13	2.02	2.04	7.13	7.19	45.3	45.45

**Table – 02****Colour and Solubility of the Lead(II) Complex**

Complex	Colour	Solubility
$PbL.2H_2O$	Blackish	Meth,Eth,Etr,Acet,N-N Mef

**Symbol**

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

**REFERENCES**

1. Vogel, A.I, "A Text Book of Quantitative Inorganic Analysis" Longmans Green and Co.Ltd London (1952)
2. Eugene Lieber, Edwin Oftedhal, C.N Pillai and Ralphd Hites pp-441 April (1957)
3. Furman, N.H and Scotts "Standard Method of Chemical Analysis" 6<sup>th</sup> Edition Vol I D, Von, Norstrand Co.INC.901 (1962)
4. N, Raman, J.Dhavecethu Raja and A.Sakthivd, J.Chemical Society.Vol-119 No – 4,303-310(2007)
5. Debra, C.Q, Kathy, R.K.Earl.Antivir, Res.71 (1)24, (2006)
6. Chandra, S,Sangeetika,X,Spectrochim,Acta A.60.147, (2004)
- 7.Bhattachargee,C.R,Deo,M,Karmakar,Su,C.D,Lu,C.Z,DI Fallah,M.S,Mitra,S,New J.Chem 27.1360(2003)
8. Prasad, T.P Thermo Chem Acta.65, 147, 1983
9. Dahiya, J.B and Sushila Rana, India. Journal of Chemistry Vol.A.October.2024-2029(2005)
10. Agarwal, R.K, Journal of Indian Chemical Society.72.263 (1996)
11. Siovasankar, B.N; Indian Journal of Chemistry. Vol.44 (A) Sept.1806-1811(2005)

