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XRD DIFFRACTION PATTERN OF THE ADDUCT OF ADIPIC ACID WITH $[NP(OH_2)]_3$

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ABSTRACT

The adduct of adipic acid with $[NP(OH)_2]_3$ assigned as $[P_3N_3(OH)_4][CH_2]_8$ ($(COO)_2$, $(COOH)_2$) *is characterized by EPR and XRD spectra and resultst reveals that the adduct is paramagnetic in character having a triclinic geometrical structure.*

KEYWORDS: Conductor, Geometrical, Paramagnetic, Synthesis, Triclinic.

INTRODUCTION

Synthesis of various adducts of $[NP(OH_2)]_3$ with a number of organic acid such as cinnamic acid, oleic acid, hippuric acid, salicylic acid, nicotinic acid, adipic acid and tannic acid etc. have been reported.[1-7]

EXPERIMENTAL

$[NP(Cl)_2]_3$[8] is used as starting material. When NaOH as hydroxide is treated on $[NP(Cl_2)]_3$ in a non aqueous solvent. $[NP(OH)_2]_3$ is formed and separated as palm coloured mass. To prepare the adduct, $[NP(OH)_2]_3$ and adipic acid in (1.1) ratio are refluxed for 6-8 hrs. at 140^0 -160^0C in presence of 1 ml. conc. H_2SO_4 using alcohol as solvent.

The adduct, obtained, was separated, washed, dried and stored in vacuum desiccator. EPR and XRD spectra were recorded on Varian's X-E-4 band spectrometer at RT and PW-1710 using Cuk_α as source of radiation(λ = 1.5418Å) in the 2θ range 0^0 to 80^0.

RESULTS AND DISCUSSION

Its EPR spectrum[9] consist a medium peak of high intensity (Figure-1) inferring its paramagnetic nature which is supported by the value of magnetic momentum μ_{eff} = 1.6850

BM and magnetic susceptibility χ_A = 1.1833 × 10^{-3} e.s.u. The value of g_z = 2.1710 > 2 indicate the presence of covalency in the adduct as well as ionic valency caused by the reaction of COOH group of adipic acid and P-OH group of hydroxyphosphazene forming P–O^-–C^+=O with the elimination of water molecules which are absorbed by conc. H_2SO_4.

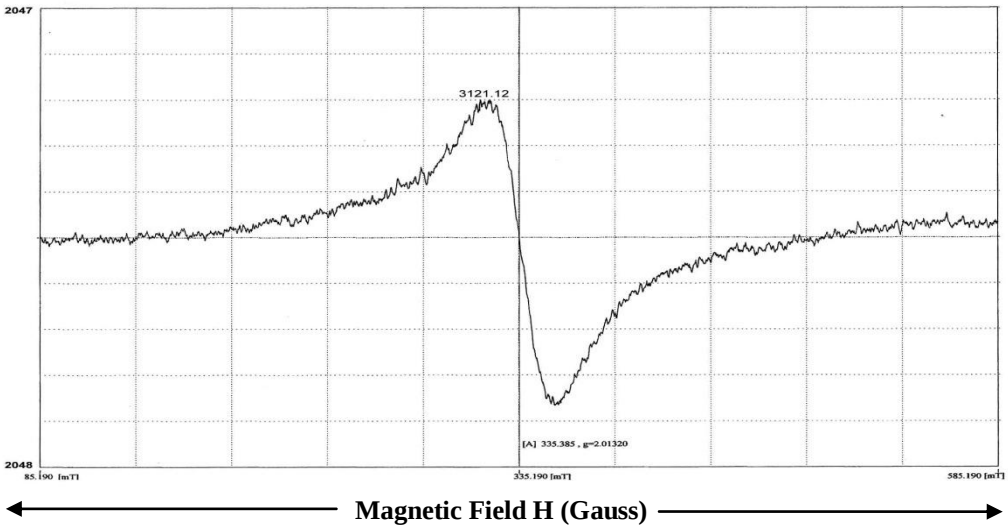

Figure-1: E.P.R Spectrum of Compound

The XRD graph (Figure-2) has a prominent peak of high intensity at 31.83^0 having other peak in its right side at 33.5. This prominent peak is for the P-N bond which repeats again at 36^0, 38^0 and 59^0 & 60^0 with same pattern and low intensity. The peak at 24.33^0 having another peak on its left side at 21.83 is for the C=O bands of adipic acid, which also repeats rights side of the prominent peak, inferring that one molecule of $[NP(OH)_2]_3$ has linked to both side with adipic acid as shown by its proton NMR spectrum (loc.cit.) conferming its reported structure.

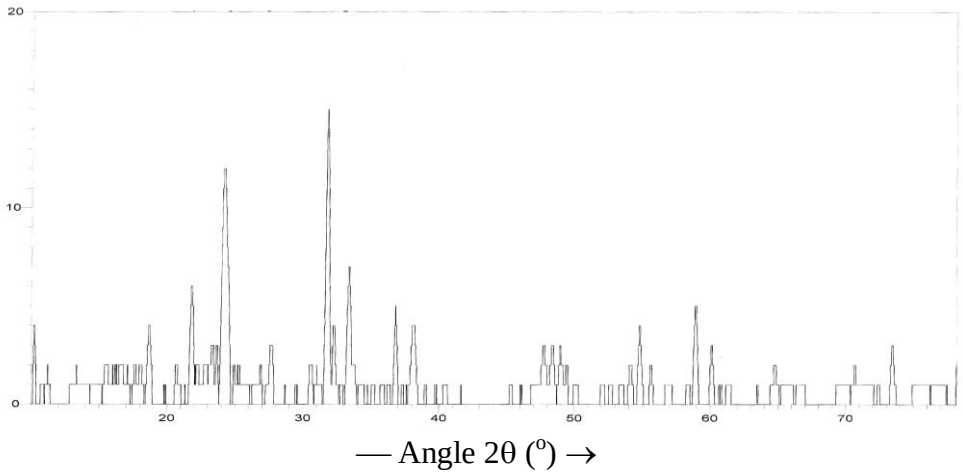

Figure -2: X.R.D. Spectrum of Compound

The values[10] of $\sin^2\theta$, milar indexes hkl and interplaner distance 'd'.

Table-1 XRD Pattern of Compound

S. No.	2θ (degree)	$Sin^2\theta$	$q(h^2 + k^2 + l^2)$	hkl	d(Å)		I / I_o %
					Obs.	Theo.	
1.	17.50	0.02314	0.02314 x (1)	100	5.0683	5.0633	20
2.	21.83	0.03585	0.01792 x (2)	110	4.0712	4.0678	30
3.	24.33	0.04440	0.0222 x (2)	110	3.6587	3.6552	60
4.	31.83	0.07519	0.02506 x (3)	111	2.8114	2.8089	75
5.	33.50	0.08305	0.02076 x (4)	200	2.6753	2.6727	35
6.	36.83	0.09979	0.01996 x (5)	210	2.4407	2.4382	25
7.	38.00	0.10599	0.01766 x (6)	211	2.3679	2.3659	20
8.	48.33	0.16758	0.02095 x (8)	220	1.8832	1.8815	15
9.	54.83	0.21199	0.02355 x (9)	300	1.6744	1.6729	20
10.	59.00	0.24248	0.02424 x (10)	310	1.5655	1.5642	25
11.	60.17	0.25128	0.02284 x (11)	311	1.5379	1.5365	15
12.	64.83	0.28734	0.02394 x (12)	222	1.4382	1.4369	10
13.	73.50	0.35799	0.02557 x (14)	321	1.2884	1.2873	15

$q_{avg} = 0.28779$

Calculated, from its XRD spectrum are in close agreement to that of theortical ones. The values of axial distances a_o 1.4370Å, b_o 0.7869Å, c_o 1.8175Å and axial angles α 65.91°, β 150.01°, γ 135° are corresponding to triclinic geometrical packing of the molecule..

CONCLUSIONS

From the results it is evident that the adduct has ionic as well as covalent bond with formation of P-O-C=O linkage showing good conductivity due to presence of unpaired electrons in it,supported by its paramagnetism.The published(loc.cite)structure as (Figure-3).

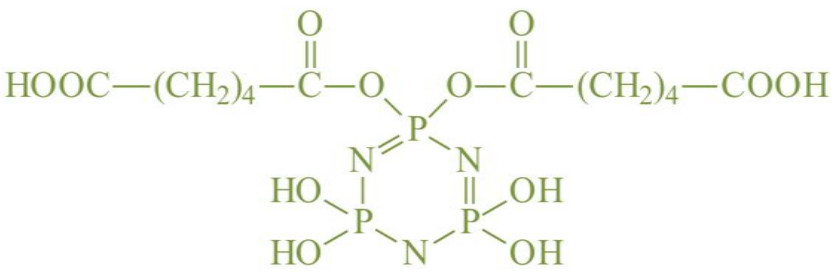

Figure-3: Structure of Compound

Acknowledgement

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