

Synthesis of Mono-(4-Chlorothio) Phenyl Phosphate Ester and Its Characterization from IR Absorption Spectra

Asha Verma

Professor, Department of Chemistry
Govt. Science and Commerce College, Benazir, Bhopal.
dr.ashaverma@ymail.com

Firdous Andleep

Research Scholar, Department of Chemistry
Govt. Science and Commerce College, Benazir, Bhopal.
andleebshah000@gmail.com

Abstract: Mono-4-chlorothiophenyl phosphate ester has been synthesized by Auger and Dupis method in a ratio of 1:1 thiol and POCl_3 . The compound has been characterized by IR absorption spectra and elemental detection. The spectral study was conducted on KBr disc. IR spectra of mono-4-chlorothiophenyl phosphate ester clearly reveals different stretching frequencies of almost all the bonds present in the compound at their respective wave numbers.

Keywords: Mono-4-chlorothiophenyl phosphate, Auger and Dupis, absorption spectra, elemental detection, stretching.

1. INTRODUCTION

An organophosphate refers to the ester of phosphoric acid. Phosphates are most probably the most prevalent organ phosphorus compounds. The chemistry of phosphate esters continues to draw the attention of chemists due to their versatile applications in the analytical^{1,2}, biological^{3,4} and industrial fields^{5,6}. They are essentially the constituent components of DNA and protoplasm and play a vital role for the maintenance of life. Phosphates having C-S-P linkage are of great importance. They are used for antiviral activity⁷, radioactive tracer techniques⁸ for biological investigations, insecticidal activity⁹ and textile commodities¹⁰. Due these multiple uses it is important to get knowledge about their stability and bond cleavage.

2. METHODOLOGY

The method of synthesis of phosphate ester (mono-4-chlorothiophenyl phosphate) has been illustrated as ;

It is prepared by Auger and Dupis method in a ratio of 1:1 phenol and POCl_3 . 6.52 g of 4-chlorothiophenol (A.R. grade) was dissolved in 20 ml of dry benzene; 3.66 ml of POCl_3 was taken in a conical flask and kept on a magnetic stirrer. Then a very small amount of the phenol (parent compound) was added slowly to POCl_3 and the material was stirred for a period of 6 hrs at 60 to 65°. After few minutes of each addition, 3 ml of pyridine was added to the stirred material in installments, pyridine hydrochloride began to separate at once with the evolution of heat. After the stirring is completed the stirred material was kept open so as to evaporate the solvent. Then the oily residue left in the flask was treated with water. The milky solution thus obtained was treated with diluted HCl to remove unreacted pyridine as pyridine hydrochloride. The solution was filtered off, first filtrate (very small amount) was rejected. To the clear filtrate Barium hydroxide was then added till it became alkaline and white precipitate began to separate. The precipitate was then washed several times with distilled water (containing few drops of acetic acid) to remove inorganic phosphate till dark color of phosphorus was obtained in the filtrate by Allen's test.

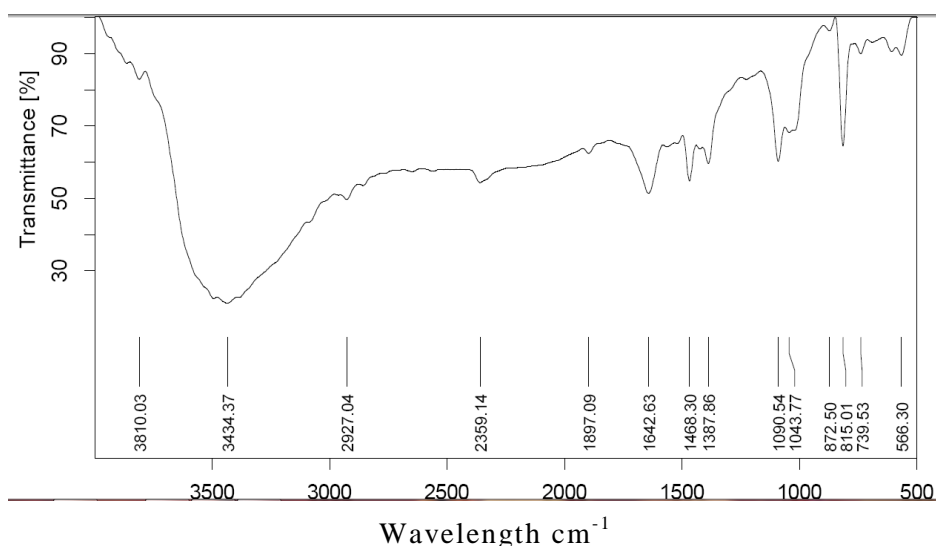
3. CHARACTERIZATION OF THE COMPOUND

3.1. Estimation of Elements

Table A-1

S.No	Element	Percentage	
		Theoretical	Observed
1.	Carbon	32.08	31.15
2.	Chlorine	15.78	15.27
3.	Phosphorus	13.72	13.07
4.	Hydrogen	2.69	2.02
5.	Oxygen	21.37	20.92
6.	Sulphur	14.27	13.98

3.2. I.R. Absorption Spectra



The compound was also identified from its characteristic absorption spectra in KBr disc. IR spectral data of Mono-4-chlorothiophenyl phosphate has been shown in Table A-1. The spectral study was conducted on BRUKER ALPHA FT-IR SPECTROPHOTOMETER (SIRT Bhopal).

IR Spectral Data of Mono-4-chlorothiophenyl Phosphate

Table A-2

S.No	IRv cm^{-1} P-O Stretching	IRv cm^{-1} (O-H) stretching	IRv cm^{-1} (P- S) stretching	IRv cm^{-1} Ar(C-C) stretching	IRv cm^{-1} (C- S) stretching	IRv cm^{-1} Ar(C-Cl) stretching
1.	888.67	3433.174	566.30	1433.15	2448.057	1003.46

4. APPLICATIONS

Organophosphates refer to a group of insecticides acting on the enzyme Acetylcholine esterase. Some of their pesticides irreversibly inactivate ACHE which is essential to nerve functioning in insects and many other animals. Due to their versatile applications, they are common carriers of organic groups in biosynthesis. They are widely used in a number of ways as fertilizers¹¹, fire retardants¹², pesticides¹³, smoke generation¹⁴, chemosterilants^{15,16,17}, synthetic lubricants¹⁸, plasticizers¹⁹ and oil additives²⁰. Due to their multipurpose applications, most of the work has been concentrated on studying their pharmacological and toxicological effects and biochemical action and hence are the most important class of organic compounds.

REFERENCES

- [1] L.D. Quin., A Guide to Organophosphorus Chemistry, John Wiley & Sons. Inc, New York, P.P. 2,375(2000).
- [2] L.F. Audrieth & A.D.F. Toy., J. Am. Chem. Soc., 64,1553(1942).
- [3] B. Holmstedt, Pharmacol. Rev., 11,567-688(1959).

Synthesis of Mono-(4-Chlorothio) Phenyl Phosphate Ester and Its Characterization from IR Absorption Spectra

- [4] H. Arnold, F. Bourseaux & N. Brock, *Arzneim Forsch*, 11, 143 (1961).
- [5] R. A. Moss & H. Morales – Rojas, *J. Am. Chem. Soc.*, 123, 7457-7458 (2001).
- [6] Y. Tonaka, S. Kano & K. Odawara, *Nipponsoda Co. Ltd.*, D.O.S. 2.416.178 (1974).
- [7] Synthesis and antiviral activity, *Chem. Astr.*, 54, 1303 (1961).
- [8] G. Gardner & B. A. Kilby, *J. Chem. Soc.*, 3, 1769 (1950).
- [9] A. H. Schlesinger, *Chem. Abstr.*, 49, 5517 (1955).
- [10] H. N. Dayer, *Chem. Abstr.*, 53, 1772 (1959).
- [11] Mattingly, G. E. G. and Failibudeen, O. "Topics in Phosphorus Chemistry", 4, 157 (1967).
- [12] C. A. Vernon, V. A. Welch, *J. Chem. Soc., Sec. B: Physical Organic Chemistry* (1966).
- [13] Grapov A. F. and Melnikov N. N., *Russ. Chem. Revs.*, 42, 772 (1973).
- [14] GOSS, M. F. Jr., *J. Chem. Ed.*, 18, 3 (1941).
- [15] Knipling E. F., *J. Econ. Entomol.*, 53, 415 (1960).
- [16] Knipling E. F., *ibid*, 55, 782 (1962).
- [17] Knipling E. F., *Agr. Res.*, 14(9); 3 (1966).
- [18] E. F. Norman, Hitchcock, A. C. Robert, Rohar Corsswell and E. Derbysire, 1, 042, 391 (1965).
- [19] Fisher, E. B. and Van Wazer, J. R., *Uses of Org. Phos. Compounds in phosphorus and its comp.*, Vol. II, Van Wazer, J. R. (ed.), Interscience, N. Y. (1961).
- [20] Fisher, J. and Van Wazer (1961).

AUTHORS' BIOGRAPHY

Miss. Asha Verna is working as a professor in the department of Chemistry Govt. Science and Commerce College Benazir Bhopal.



Miss. Firdous Andleep is working as a research scholar in the department of Chemistry Govt. Science and Commerce College Benazir Bhopal.