

Preparation and identification of di-4-Chloro,3-methylphenyl phosphate Ester (Ba-salt)

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ABSTRACT

Di-4-chloro, 3-methylphenyl phosphate has been synthesized by Auger and Dupis method in a ratio of 1:1 phenol and POCl₃. The compound has been characterized by IR absorption spectra and elemental detection. The spectra study was conducted on KBr disc. IR spectra of di-4-chloro, 3-methylphenyl phosphate ester clearly reveals different stretching frequency of almost all the bonds present in the compound at their respective wave number.

INTRODUCTION

Organophosphates are widely recognized an important fine chemical in different chemical processes. These are derivatives of orthophosphoric acid and infinite revivifications are possibly by making changes in substituent attached to its phosphorus atom through specific linkage likely C-O-P, C-N-P, and C-S-P. They constitutes a family of large number of encumbers which display a great verity of biological activities in later part of the 1930's a number of neutral phosphate derivatives have been developed into practical insecticides. Owing their owing to their high activity and biodegradability, their application to agriculture, public health and related fields have been growing rapidly moreover , the pesticidal activities of organophosphorus compounds, which are not restricted to phosphate derivatives, also include accaricidal, nematocidal, antihelmentic, insect sterilizing, fungicidal, herbicidal and rodenticidal activities.

In view of their practical application there is obvious need to incur continues investigation on both theory and experimental fronts the hydrolytic reaction of orthophosphate. Hydrolysis is basically double decomposition reaction between water ant the

substrate. A vast literature of kinetic investigation of C-O-P linkage is available. Now the attention of scientist is diverted towards other linkage such as C-N-P and C-S-P

Materials and method

It is prepared by Auger and Dupis method in a ratio of 1:1 phenol and POCl₃ 6.52 g of 4-chloro-3-methylphenol (A.R.grade sigma –Aldrich) was dissolved in 20 ml of dry benzene, 366 ml of pocl₃ 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 POCl₃ and the material was stirred for a period of 6 hours at 60-65 degree after few minute 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 obtain was treated with diluted HCl to remove unreacted pyridine as pyridine hydrochloride. The solution was filtered of, first filtrate (very small amount) was rejected .to the clear filtrate barium hydroxide was then added till it become

alkaline and white ppt. began to separate .the ppt. was then washed several times with distilled water (containing few drops of acetic acid) (to remove inorganic phosphate till dark blue colour of phosphorus was obtained in the filtrate by Allen’s test.

Result and Discussion

It is also prepared by Auger and Dupis method ,4-chloro-3-methylphenol (A.R. Grade sigma – Aldrich)and POCl_3 were taken in 2:1 ratio.7 ml of pyridine was added slowly to a stirred solution of 4-chloro-3-methylphenol (6.52 g) and POCl_3 (1.83 ml) in dry benzene (25 ml).pyridine hydrochloride were began to separate immediately with the evolution of heat .The mixture was stirred on a magnetic stirrer at 60 to 65 degree for a periodic of 10 hrs and 30 min. The yellowish oily residue left after stirring was treated H_2O and then with 5% NaOH .the filtrate thus obtain was acidified with diluted HCl to precipitate the chloride

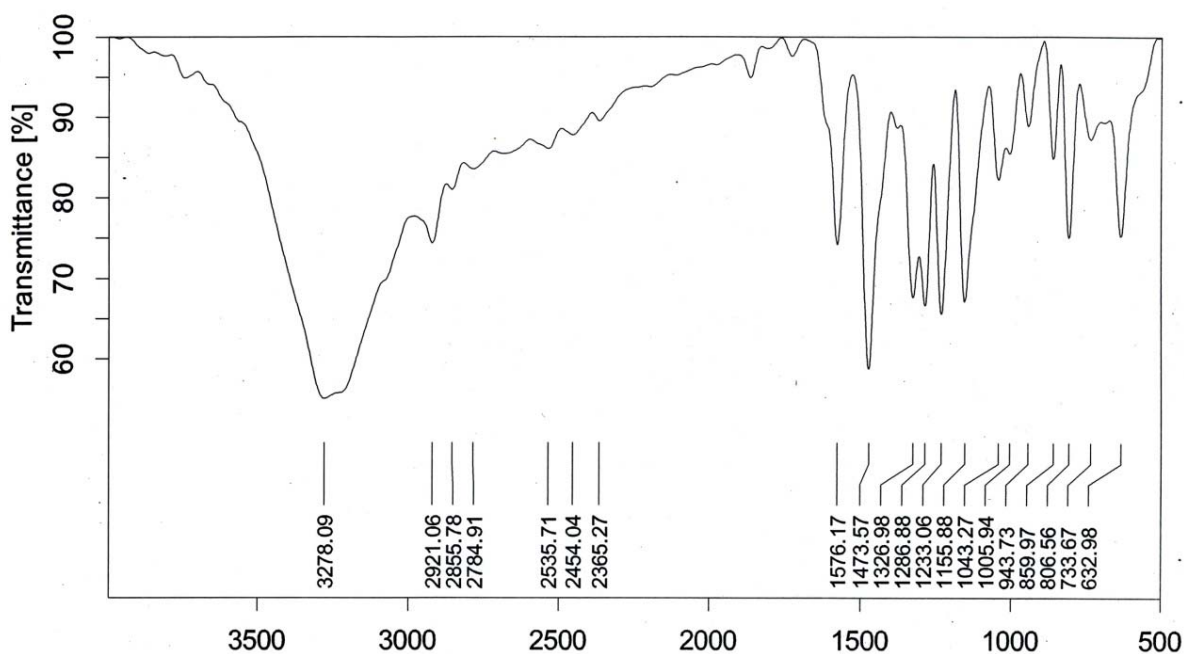
which on washing with distilled water was converted into the free diesters. This free diester was finally dissolved in CCl_4 and filtered off to remove impurities.

Estimation of element

SNO	ELEMENTS	PERCENTAGE	
		THEORETIC AL	OBSERVE B
1.	Carbon	48.31	48.439
2.	Hydrogen	4.23	3.775
3.	Oxygen	18.31	18.436
4.	Chlorine	21.05	20.426
5	Phosphorus	9.31	8.924

I.R. Absorption Spectra: Spectra.4-A.2

I.R spectrum of di-4-chloro 3-methylpheny phosphate



The compound was also identified from its characteristic absorption spectra in KBr pellets IR. Spectral data of di-4-chloro-3-methylphenyl phosphate has been shown in table 4.a-2 (the spectral study was conducted on Nicolet protégé model 460 IR spectrophotometer .SIRT Bhopal)

Table 4.A-2

S.NO.	STRETCHING	I.R v Cm ⁻¹
1.	(C=O) Stretching	1233.061
2.	(P=O) Stretching	1043.271
3.	(C=H) Adjacent Stretching	806.555
4.	(C=H) Isolated Stretching	943.731
5.	(C=Cl) Stretching	632.979

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 application, they are common carriers of organic groups in biosynthesis. They are widely used in a number of ways as fertilizers

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