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Adducts Of Diphenyltin Dichloride With Some Organic Donors

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Adducts Of Diphenyltin Dichloride With Some Organic Donors

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ABSTRACT

Diphenyltin dichloride forms 1:1 adducts with o-phenanthroline, 2,9-dimethylphenanthroline, 2,2'-bipyridyl, 4,4'-dimethyl bipyridyl, 2,2', 2''-tripyridyl pyridine-N-oxide, triphenylphosphine oxide, methyltri-phenylphosphonium chloride, diphenylphosphino ethane, -methane oxides and thianthrene-5-oxide. But it forms 2:1 adduct with diphenylphosphino ethane which on oxidation gives the corresponding oxide. This may be attributed to steric hindrance.

Some physical studies for these adducts are reported. These include infrared spectra, conductivity and molecular weight determinations.

INTRODUCTION

Diphenyltin dichloride does not form any complexes with triphenylphosphine, triphenylphosphine sulphide, diphenyl sulphide, diphenyl sulphone, dimethyl sulphide, dimethyl sulphone or thianthrene. The Sn (IV) in Ph_2SnCl_2 behaves as a typical Lenden Chatt "A" class and is similar to lead (II)[1], thorium (IV)[2] and uranium (IV)[3].

Organotin halides, like tetrachloride of tin, react with certain electron-pair donors to form addition compounds. Although the study of coordination compounds of tin (IV) under other than the anhydrous conditions are difficult because of the extensive hydrolysis and polymerization which results from the great stability of tin (IV) oxygen bonds[4], it is possible to study some complex formation of organo tin (IV) as this class of compounds are less susceptible to such type of reactions[5].

From a survey of the literature, it is found that the majority of adducts are of the type $R_nSn_{4-n}X$, 2L (where R = organic radical, X = halogen and L = an electron pair donor)[6]. The range and stability of the addition compounds formed appear to decrease as the number of organic groups increases and for the tetraorganic tin there is little evidence of Lewis acid behaviour[7]. Scattered reports of complexes of the type R_2SnX_2. L (where R either Me or Ph group) have appeared[5-12], but no systematic study of these derivatives has been made. We felt, it would be of interest to try to prepare a number of Ph_2SnCl_2. nL and to investigate some of their physical properties;

(i) because of the current trend towards the use of such compounds in Mossbauer Spectroscopy[13];

(ii) To compare the complexing ability of Ph_2SnCl_2 with that of tin tetrachloride[14].

Introduction of organic substituents is reported to reduce the alectron-acceptor properties. Thus Germanium halides give complexes with nitrogen donors, but diphenylgermanium dihalides do not react with the same organic bases[15].

Materials and Methods

Diphenyltin (IV) dichloride was prepared[16] from tetraphenyltin (IV) or obtained from BDH. All solvents used were dried carefully by conventional methods[17]. Spectra were recorded on an infrared spectrophotometer Unicam SP 200 G. Melting points were measured using a Unicam melting point apparatus. Analyses for carbon, hydrogen, nitrogen and phosphorus were carried out in the microanalytical Unit El-Nasr Company, Cairo. Chloride was determined by the standard volumetric method.

o-Phenanthroline monohydrate (o-phen.), m.p. 98°C and its 2,9-dimethyl derivative hemihydrate (DM-o-phen.), m.p. 159°C were used without further purification, 2,2'dipyridyl (bipy.), m.p. 70°C and its 4,4'-dimethyl derivative (DM-bipy.), m.p. 169°C, 2,2',2"-tripyridyl (terpy.), m.p. 85°C, bipy; py. and py-N-oxide were obtained from BDH or Sigma.

Triphenylphoshine (Ph_3P), m.p. 79-80°C was recrystallised from ethyl acetate, triphenylphosphine oxide (Ph_3PO), m.p. 158°C, was prepared either by oxidation of triphenylphosphine by dinitrogen tetroxide[18]or by heating commercial samples of the supposed hemihyrate at 160°C[19]. Triphenylphoshine sulphide (Ph_3PS), m.p. 161°C, was prepared by reaction of triphenylphosphine with sulphur in benzene. Metyltriphenylphosphonium iodide ($MePh_3PI$), m.p. 184°C, was prepared from triphenylphosphine and methyl iodide in in ethanol[20]and $MePh_3PCI$ was obtained by the addition of conc. HCl to the corresponding iodide. Bis (diphenyhosphino)-ethane (DIPhoS), m.p. 141°C, and -methane, m.p. 120°C, were prepared by the method mentioned previously[21], Oxidation with $KMnO_4$in acetone gave the corresponding bis (diphenylphosphinyl)-ethane (DIPhOS oxide), methane. m.p. 158°C. Diphenyl sulphoxide, m.p. 71°C, and diphenyl sulphone, m.p. 128°C were crystallised from ether. Dimethyl sulphone, m.p. 112°C was recrystallised from ethyl acetate. Thianthrene 5-oxide was prepared from thiantrene by oxidation[21].

Preparations of Adducts.- Diphenyltin dichloride was prepared according to the method reported by Folaranmi[16]. By the reaction of iodine monochloride (1.7g., 0.01 mole) in $CCI_4$and Ph_4Sn (0.005 mole) in the same solvent at room temperature. Evaporation of the CCI_4solution, after removal of the formed iodobenzene by extraction, gave Ph_2SnCl_2 (0.7 g., 0.02 mole; 80 % yield). This compound was recrystallised from $CHCI_3$ /pet. ether to give pure diphenyltin dichloride, m.p. 42°C.

Preparation of Complexes:

The 1:1 addition compounds were prepared by adding a solution of diphenyltin dichloride in ethyl acetate or ethyl alcohol to a stoichiometric quantity of the appropriate Lewis base dissolved in the same solvent employed for the diphenyltin compound. In the case of the complexes Ph_2SnCI_2, L (L = Ph_3PO, phen. and dimethylphen.) derivatives, precipitation occurred immediately on mixing the solutions, and the products were suction-filtered, washed with the solvent and dried in air. For the other 1:1 complexes the solvent was removed under reduced pressure and the products dried in vacuo. Preparation of the 1:2 derivatives followed a similar route, except that 100 % excess of base was used. In most cases essentially quantitative yields of the pure complex compopounds resulted, where possible the products were recrystallised from acetonitrile, nitromethane or chloroform. All the adducts precipitated as white stable products, except Ph_2SnCl_2, py-N-oxide which decomposes on standing at room temperature and is best stored under refrigeration.

Attempts to prepare complexes of pyridine and pipyridine with Ph_2SnCl_2produced solids of questionable purity. Analytica data for these two compounds suggest that hydrolysis occurred and they may have the formulae, $Ph_2Sn(OH)$ CI, py. and ph_2 Sn(OH) CI, pipy., as indicated by the ir. spectra.

Recrystallisation of the complexes, $2Ph_2SnCl_2$, L and Ph_2 $SnCl_2$, L, from nitromethane gave the corresponding oxides (L = diphenylphosphinoethane and -methane). Fig. 1 shows the ir. spectra of these adducts.

Analytical data and melting points are given in Table 1.

Some of these adducts have been previously prepared[22], and are reported here for comparative purposes.

Repeated attempts to prepare an addition compound of Ph_2Sn Cl_2 with Ph_3P failed, but oxidation in ethyl acetate takes place and the Ph_3PO adduct was obtained. Failure to isolate a complex with Ph_3P is not surprising in view of the Chatt classification. When a solution of the adduct of diphenyltin dichloride with triphenylphosphine oxide, Ph_2SnCl_2, Ph_3PO, in ethyl acetate is treated with a solution of mercury chloride in the same solvent, phenyl mercury chloride, PhHgCI, is obtained. The same compound is also obtained on adding equimolar solutions of $Ph_2SnCl_2$and $HgCl_2$in ethyl acetate. Similar reactions of organometallic compounds with metal salts are reported[23]

This is in contrast to the behaviour of tetrachlorobis (diphenylphosphine)-tin (IV) which reacts with mercuric chloride to form a halogen bridged dinuclear complex[24].

Results and Discussion

In this paper, we report the preparations of a number of addition compounds of diphenyltin (IV) chloride, Ph_2SnCl_2 with Lewis bases in which coordination of tin is via a nitrogen or oxygen

TABLE 1

Compound	Yield	M.P.	Required %				Formula	Found %			
	%	oC	C	H	N/P	CI		C	H	N/P	CI
1) Ph_2SnCI_2,o-phen.,H_20*	83.5	280	53.2	3.7	5.2		$C_{24}H_{20}OCI_2N_2Sn$	53.6	3.7	4.9	
2) Pb_2SnCI_2,DM-o-phen.,H_20	97.5	157	54.8	4.2			$C_{26}H_{24}OCI_2N_2Sn$	54.7	4.5		
3) Ph_2SnCI_2, biby.*	92.7	>230	52.8	3.6			$C_{22}H_{18}CI_2N_2Sn$	52.2	3.7		
4) Ph_2SnCI_2, DM-bipy.	98.7	228	54.6	4.2	5.3		$C_{24}H_{22}CI_2N_2Sn$	53.5	4.0	5.8	
5) Ph_2SnCI_2,2,2,2-terpy.2H_20	92.0	174	52.9	4.1			$C_{27}H_{25}OCI_2N_3Sn$	52.1	4.2		
6) 2Ph_2SnCI_2,2,2,2-terpy.	95.0	105	51.2	3.2	4.9		$C_{39}H_{31}CI_4N_3Sn_2$	50.8	3.4	4.6	
7) Ph_2SnCI_2,py-N-oxide	84.0	167	46.7	3.4	3.2		$C_{17}H_{15}0\ CI_2NSn$	47.0	3.6	3.6	
8) Ph_2Sn (OH) CI, py	66.0	148	50.6	3.8	3.4	8.8	$C_{17}H_{16}0\ CI_2NSn$	50.6	4.1	3.2	8.7
9) Ph_2Sn (OH) CI, pipy	62.0	242	49.7	5.4	3.4	8.6	$C_{17}H_{22}0\ CI\ NSn$	49.5	5.9	3.9	8.8
10) Ph_2SnCI_2, Ph_3PO	89.7	175	57.9	4.1			$C_{30}H_{25}OCIPSN$	57.3	4.0		
11) Ph_2SnCI_2, $MePh_3PCI$	88.0	137	56.8	4.2			$C_{31}H_{27}CI_3PSn$	56.0	4.3		
12) 2Ph_2SnCI_2, DIPhOS	84.1	168	55.3	4.1			$C_{50}H_{44}CI_4P_2Sn_2$	55.9	4.3		
13) 2Ph_2SnCI_2,DIPhOS oxide	82.0	>230	53.7	4.0			$C_{50}H_{44}0_2C_4lP_2Sn$	54.1	4.1		
14) Ph_2SnCI_2,DIPhOS met.	80.0	>230	61.0	4.4	8.5		$C_{37}H_{32}CI_2P_2Sn$	61.3	4.1	8.1	
15) Pb_2SnCI_2, DIPhOS methane oxide	86.0	248	58.4	4.2	8.1		$C_{37}H_{32}0_2CI_2P_2Sn$	57.9	4.2	8.2	
16) Ph_2SnCI_2, Ph_2SO	95.7	93	52.8	3.7			$C_{24}H_{20}OCI_2SSn$	52.9	4.0		
17) Ph_2SnCI_2, 2Me_2SO	94.0	130	38.4	4.4	S		$C_{16}H_{22}0_2CI_2S_2Sn$	38.9	4.3	S	
18) Ph_2SnCI_2, Thianox	82.0	>230	50.0	3.1	11.1		$C_{24}H_{18}OCI_2S_2Sn$	49.8	2.8	11.0	
19) Ph HgCI	65.0	245	23.0	1.6			C_6H_5CIHg	23.4	1.6		

Ref. 22

atom. There generally seems to be little difficulty in obtaining these adducts with the ligands employed here.

In agreement with the related work of Alleston and Davies[22], we find that Ph_2SnCI_2readily forms 1:1 adducts with chelate ligands such as o-phen. and bipy. However, all our attempts to prepare the 1:2 adduct with Ph_3PO-Ph_2SnCl_2, $2Ph_3PO$- (using five fold excess of the ligand) afforded only the mono addition compound; despite the reported[25]preparation of the 1:2 complex. Mullins[11] and Liengma[26]have also been unable to obtain the bis adduct.

Irving and Cox[27]have observed that certain organometallic cations like the dialkyltin (IV) and the alkylmercury (II) ions form very stable complexes with sulphur donors such as the conjugated base of 3- 3–mercapto-1,5-diphenyl formazon. The stability of this complex was attributed to $d\pi$–$P\pi$ back donation from the tin atom. During this work no adducts were obtained with ligands containing sulphur atoms.

Infrared Spectra

The infrared spectrum of Ph_2SnCI_2 is briefly mentioned. There are two bands of medium intensity at 1070 and 1020 cm^{-1} which we attribute to a phenyl in-plane C-H deformation mode, and a ring vibration at 997 cm^{-1}, of medium intensity Strong bands assigned as out-of-plane C-H deformation[28]occurs at 730 and 965 cm^{-1}, the former band being split in several of the addition compounds. The bands assigned[29,30] as Vas (Sn-Ph) at 285 cm^{-1} and Vs (Sn-Ph) at $\backsim$ 232 cm^{-1} for the parent dipheyltin dichloride appear to be relatively insensitive to complex formation.

A comparion of the spectra of the present studied complexes with those of analogous complexes with metal halides[31,32] confirms coordination of these ligands to Sn (IV). An important feature in the spectra of the complexes studied is the absence of any vibration corresponding to pure ligand, indicating that these ligands act as bidentate donors.

The ir. spectra of the complexes, Ph_2SnCI_2, L (L = bidentate ligand) mentioned in this work, possess two strong Sn-CI stretching bands at $\backsim$ 325 $cm^{-1}$and 265 cm^{-1}, which is associated with the non linear Cl-Sn-CI moiety. These complexes most likely have octahedral configuration with the Ph groups trans to each other. As stated[26], such structure is more stable.

The most important features of these complexes are discussed in terms of the donor atom concerned.

Nitrogen Donors.- With bipy. and o-phen., the reaction of Ph_2 $SnCI_2$ with the ligands in a polar non-aqueous solvent (e.g. ethyl acetate) gave products of the type 1:1 adduct.

Changes were observed in the infrared spectra of the ligands when it was complexed with Ph_2SnCI_2. Thus, with 2,2′-bipy., the 760 cm^{-1}band (out-of-plane) bending of C-H bonds, is higher by 10-20 $cm^{-1}$and splitted to two bands, and medium bands at 1600 and 1610 cm^{-1}replace those at 1558 and 1620 cm^{-1}in the pure ligand. Similar changes have been reported for other bipyridyl complexes[30]. In the o-phen. complex, little change ($\sim$10 cm^{-1}) is seen in the C-H bending frequencies (737, 850^{-1}) but the ring vibration at 1500 cm^{-1}moves to 1560 cm^{-1}, this is again similar to previous results[32].

The spectrum of the adducts with DM-o-phen. show slight change from the ligand, shift of the band at 850 cm^{-1}in the ligand to 865 cm^{-1}in the complex and multiple splitting in the region 1500-1650 cm^{-1}.

In case of the complex with DM-dipy. there are great change in the spectrum and multiple splitting is obvious in the region 1400-1600 cm^{-1}, shift of these bands are clear also the shift of the bands in the region 740-850 cm^{-1}. In case of the terpy-adduct, strong bands in the pure ligand at 760, 1457 and 1580 cm^{-1} were replaced by bands at 790, 1475 and 1610 cm^{-1}in the spectrum of the complex.

Oxygen and Phosphorus Donors.- The P-O stretching frequency in the product obtained with Ph_3PO occurs at 1140 cm^{-1}, a lowering of about 50 $cm^{-1}$from the 1190 $cm^{-1}$of the pure ligand, as found in other cases of coordination of this ligand via oxygen.[33],[34] The same shift was found in the spectrum of the adduct obtained from the reaction of Ph_3P and Ph_2SnCI_2, which proved by analysis to be the Ph_3PO adduct.

The reaction of Ph_2SnCI_2 with $MePh_3PCI$ gave a water soluble salt which seems to have the ionic structure, $MePh_3P^+$ $Ph_2SnCI^-_3$ as indicated from the conductivity of its solution in dimethyl formamide. Sîmilar salts containing the $SnCI^-_3$ anion are reported[35]. A complex was produced by the reaction with DIPhOS shown, by analysis to be $2Ph_2SnCI_2$,DIPhOS; the infrared spectrum was very similar to that of the ligand, apart from a shift of the band at 770 cm^{-1} to lower frequency, 755 cm^{-1}. Recrystallisation of this compound from nitromethane gave the DIPhOS oxide compound. An authentic sample of this compound was obtained from the reaction of DIPhOS oxide and Ph_2SnCI_2in ethyl acetate. The spectrum of this oxide adduct shows very strong bands at 1130 and 1180 cm^{-1}corresponding to the P-O stretching frequency of the coordinated DIPhOS oxide. The DIPhOS and DIPhOS oxide in such adducts are not considered to be chelated ligands. This may be due to the large size of the ligands and is attributed to steric hindrances. A complex of $Me_2SnCI_2$and terpy. is reported to have the same stoichiometry ratio[35].

The $S=0$ stretchîng frequency occurs at 1040 cm^{-1}in Ph_2SO and at 1055 cm^{-1}in Me_2SO[35], these bands were found at 975 and 1030 cm^{-1}, respectively in the adducts with Ph_2SnCI_2. The shift to lower frequencies can be taken as an indication that bonding occurs through oxygen rather than sulphur[37]. Some of these adducts contain water of crystallisation and their ir. spectra indicate a broad band in the region 3500-3400 cm^{-1}.

The spectra of the adducts of Ph_2SnCI_2 with py. and pipy. indicate a broad band in the region 3000-2500 cm^{-1}, which corresponds to bonded OH.

The trigonal bipyramidal structure is expected for the complexes of $Ph_2SnCI_2$with monodentate ligands, Ph_2SnCI_2, L ($L=$ Ph_3PO Ph_2SO and py.-N-oxide). There is thus the possibility of competition between the halogens and the donor molecule for one of the apical positions presumably depending upon the nature of the Lewis base.

Mullins[38], on the bases of dipole moment measurements, suggested that the ligand occupies one of the apical positions.

Ph_3P does not react with $Ph_2SnCI_2$and no product was obtained by any of the solution techniques used previously. Equally, none of the methods that are successful with other ligands gave any reaction with Ph_3PS, Ph_2S, Ph_2SO_2, Me_2SO_2 Me_2S or thianthrene $(C_5H_4S)_2$, It is therefore, concluded that the acceptor properties of Sn (IV) in $Ph_2SnCI_2$is similar to that in $SnCI_4$.

Conductivity Studies.- The diphenyltin dichloride adducts reported in this paper show low molar conductivities in organic solvents. The expectional compound, Ph_2SnCI_2, $MePh_3PCI$, having the conductivity characteristic of a 1:1 electrolyte in DMF, 64 $ohm^{-1}cm^2$, is presumably $Ph_2SnCI_3^-$ $MePh_3^+$.

The Molecular Weights.- Table 2 indicates the molecular weights of some adducts of Ph_2SnCI_2 in organic solvents.

Table 2

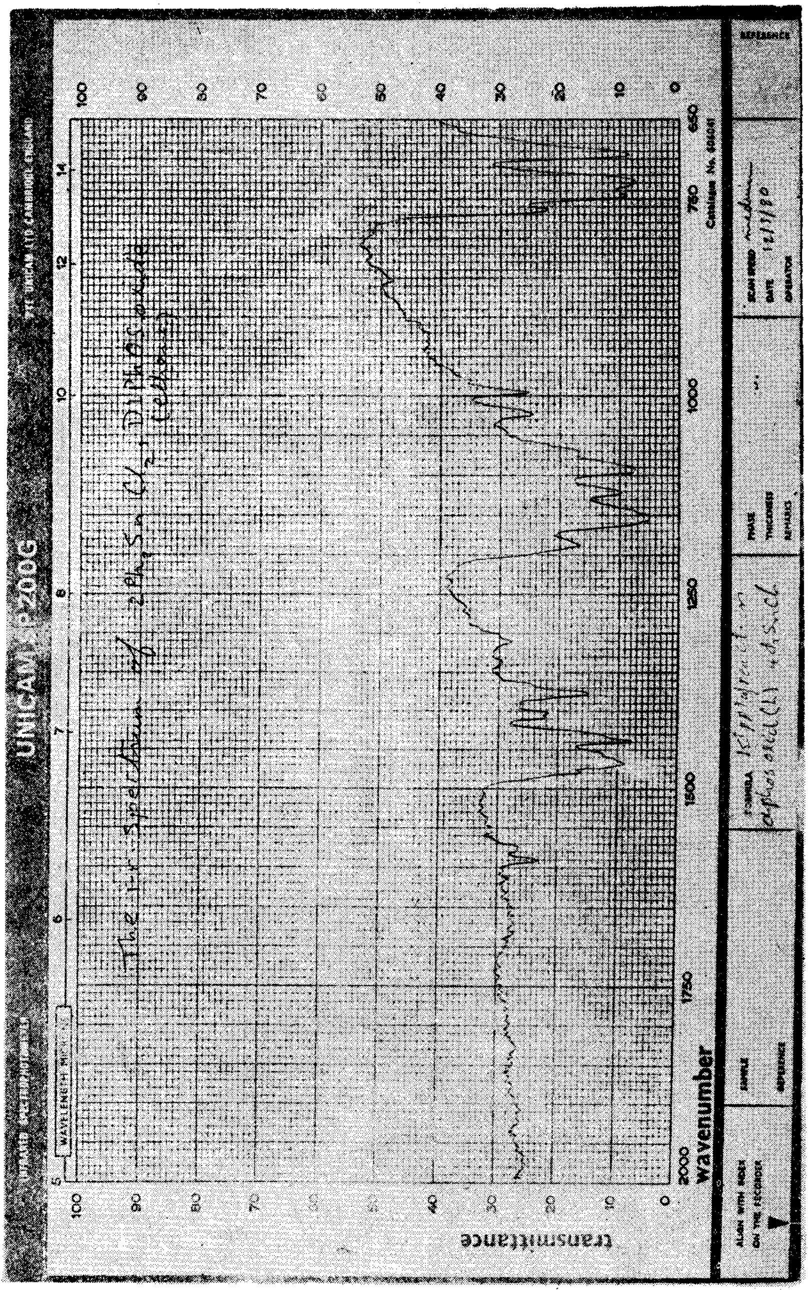
Molecular Weights Determination of Some Sn (IV) Complexes

Compound	M obs.	M cal.		Solvent
1. Ph_2SnCI_2,o-phen., H_20	163	542.0	3.3	nitromethane
2. Pb_2SnCI_2, DM-o-phen., H_20	244	470.1	2.3	nitromethane
3. Ph_2SnCI_2, bipy.	414	500	1.2	acetonitrile
4. Ph_2SnCI_2, DM-bipy.	279	528.0	1.9	acetonitrile
5. Ph_2SnCI_2, Py-N-oxide	182	438.9	2.4	nitromethane
6. Ph_2SnCI_2, Ph_3PO	260	622.1	2.4	acetonitrile
7. Ph_2SnCI_3 $MePh_3P$	200	656.5	3.3	DMF
8. Ph_2SnCl_2, DIPhOS	223	1085.0	4.9	DMF
9. Ph_2SnCI_2, Ph_2SO	156	546.1	3.5	acetonitrile
10. Ph_2SnCI_2, $2Me_2SO$	153	500	3.3	acetonitrile

Where M: is the molecular weight (obs: observed and cal. calculated). F: is the dissociation factor.

The dissociation factors, F, show that the addition compounds, Ph_2SnCI_2, bipy., is a monomer with in the limits of the experimental error. The other complexes undergo dissociation in these solvents.

The overall conclusion from these studeisis that the molecular weight determination imply a more complete dissociation than do the conductometric results for the tin (IV) complexes. Similar results are reported[39].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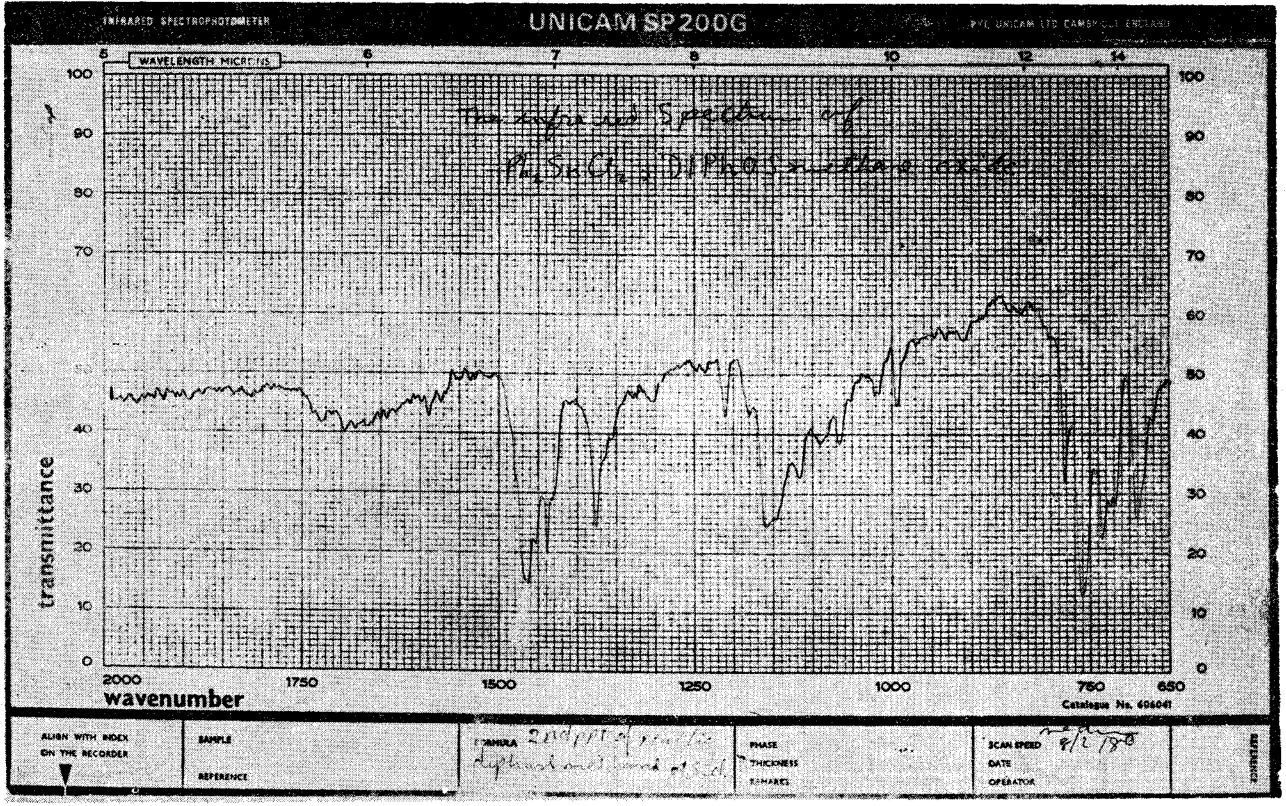
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ALIGN WITH INDEX ON THE RECORDER
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FORMULA
2nd ppt of reaction
diphosphine oxide and at 5.C.
PHASE
THICKNESS
REMARKS
SCAN SPEED
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OPERATOR
9/2/'80
REFERENCE

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