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PHOSPHORUS PENTACHLORIDE REACTIONS WITH CONJUGATED CARBONYL COMPOUNDS AND CONJUGATED OLEFINS

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Phosphorus pentachloride was reacted with conjugated aromatic and carbonyl compounds. The conjugated aromatic compounds were *cis* and *trans* stilbene, and the carbonyl compounds were diphenylketene and 2,3,4,5-tetraphenylcyclopentadiene. Two solvents used for these reactions were 1,1,2,2-tetrachloroethane and methylene chloride. Phosphorus pentachloride can dissociate into phosphorus trichloride and chlorine and our results indicated this was the reaction mechanism for phosphorus pentachloride reacting with 1,1,2,2-tetraphenylcyclopentadiene. With stilbene, *cis* and *trans*, the mechanism with phosphorus pentachloride was the sp^3d^2 octahedral complex mechanism. With diphenylketene the mechanism was the dissociation of phosphorus pentachloride into the phosphorus tetrachloro cation and the phosphorus hexachloro anion.

Key words: Phosphorus pentachloride, stilbene, diphenylketene, and 2,3,4,5-tetraphenylcyclopentadienone.

INTRODUCTION

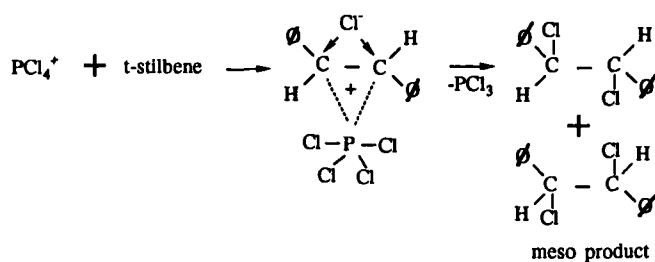
The reactions of phosphorus pentachloride with ketone and olefins structures that are conjugated, i.e. diphenylketene, 2,3,4,5-tetraphenylcyclopentadienone and *cis* and *trans* stilbene are reported here. Two solvents used for these reactions were 1,1,2,2-tetrachloroethane and a polar, lower boiling solvent of methylene chloride.

Phosphorus pentachloride reacts in different ways depending upon the polarity of the solvent and the temperature. Phosphorus pentachloride can dissociate into phosphorus trichloride and a chlorine molecule and form an equilibrium mixture of these components.¹ It also has been reported that phosphorus pentachloride can dissociate into two ions, PCl_4^+ and PCl_6^- , in polar solvents.^{1,2} A third way in which phosphorus pentachloride can react is through the sp^3d^2 octahedral structure.¹

Wyman³ reported a reaction with *trans* stilbene and phosphorus pentachloride to produce meso-1,2-dichlorostilbene in 85% yield and 13% yield of the dl mixture of 1,2-dichlorostilbene. Wyman proposed that an ionic and a free radical reaction were operating simultaneously to produce the yields he obtained. With the ionic mechanism the *trans*-stilbene would produce a meso product as indicated in Figure 1.

In the free radical mechanism with the chlorine free radical, both meso and dl product would result.

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FIGURE 1 Tetrachlorophosphonium cation addition to *trans*-stilbene.

RESULTS AND DISCUSSION

In our laboratory 2,3,4,5-tetraphenylcyclopentadienone reacted with phosphorus pentachloride (1:1 mole ratio) in 1,1,2,2-tetrachloroethane as solvent. In this solvent the optical rotation of the product was zero. The product is evidently a 50-50 mixture of the dextro and levo isomers which produces the zero rotation along with the meso isomers production. This could be explained by the addition of molecular chlorine to the ketone or via the addition of PCl_4^+ to the ketone as shown in Figure 2. Both mechanisms would produce racemic mixtures, but in the case of the PCl_4^+ reaction both the *cis* and *trans* racemic products would be formed. In order to establish which mechanism was occurring with the 2,3,4,5-tetraphenylcyclopentadienone, we reacted this reactant with molecular chlorine under the same experimental conditions and isolated a white product which gave an infrared spectrum which matched peak for peak with the infrared spectrum of the compound from the phosphorus pentachloride reaction with 1,1,2,2-tetraphenylcyclopentadienone, i.e. this proved that no *cis* racemic product occurred (Figure 2).

Additional evidence to support the dissociation of phosphorus pentachloride into phosphorus trichloride and molecular chlorine was obtained by refluxing 2,3,4,5-tetraphenylcyclopentadienone in carbon tetrachloride (b.p. 76°) with phosphorus pentachloride and no reaction occurred. However, when we refluxed 9-fluorenone in carbon tetrachloride with phosphorus pentachloride, we obtained 9,9-dichlorofluorene. If 9-fluorenone reacts with phosphorus pentachloride, the mechanism for converting ketones to gem-dichloro compounds is through the phosphorus tetrachloro anion (PCl_4^+). Since 2,3,4,5-tetraphenylcyclopentadienone is an α - β -unsaturated ketone similar to the 9-fluorenone, one would expect the carbonyl group to react, but we found no evidence for this. Consequently, the carbonium ion mechanism is undoubtedly not operating on the tetraphenyl compound.

When *trans*-stilbene reacted with phosphorus pentachloride in both methylene chloride and 1,1,2,2-tetrachloroethane, we found the *dl* isomer predominates over the meso isomer which is contrary to what Wyman³ reported. As shown in Figure 3, the ionic mechanism with PCl_4^+ as the adduct will produce only the meso isomer. The meso isomer would also be produced from the molecular chlorine addition as also shown in Figure 3. Newman proposed an ionic mechanism for ketones, and if this mechanism were applied to *trans*-stilbene then both meso and *dl* products

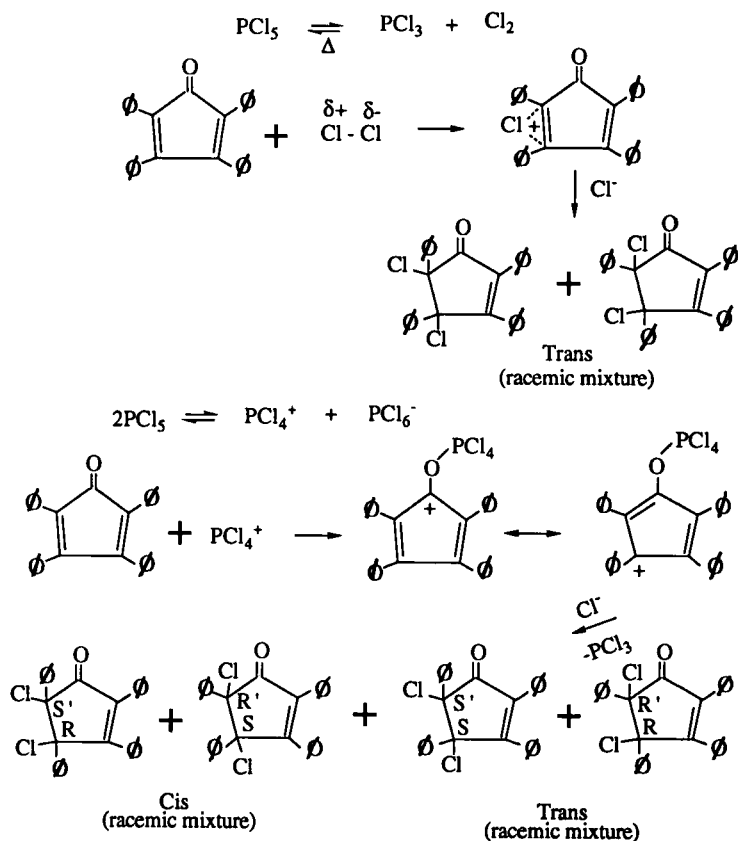


FIGURE 2 Molecular chlorine and ionic mechanism with phosphorus pentachloride.

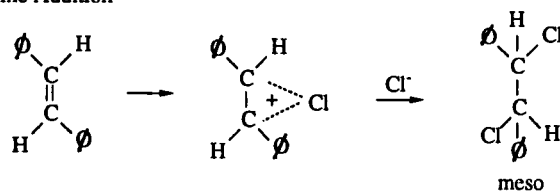
would be formed as illustrated also in Figure 3. Even with the PCl_4^+ mechanism with *cis*-stilbene the meso product should predominate over the dl product (see Figure 4). The PCl_6^- ion will attack from the opposite side of the attached PCl_4^+ ion and both phenyl rings will be on opposite sides because of steric hindrance. Consequently, from a steric hindrance standpoint one would expect the meso product to predominate.

If all these mechanisms (A, B, and C, Figure 3) are occurring simultaneously or independently, the meso/dl isomer ratio should be equal to or greater than one. In no case would the meso/dl ratio be less than one. However, in the results of our experiments with *trans*-stilbene and phosphorus pentachloride our meso/dl isomer ratio was less than one for all reactions.

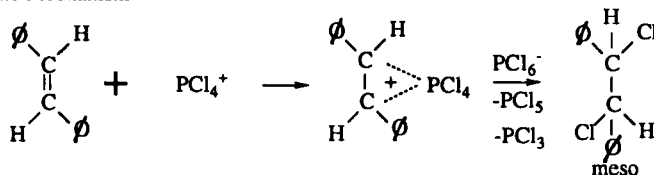
To explain our results one can postulate an olefin complex of the phosphorus pentachloride which is an sp^3d^2 octahedral structure as illustrated in Figure 5.

We ran the reaction with phosphorus pentachloride and stilbene at the reflux temperature of both 1,1,2,2-tetrachloroethane (b.p. 147°) and methylene chloride (b.p. 40°). The mole ratio and the length of the reflux time were varied. Mole ratios were run at 1:1, 2:1, 3:1 and 4:1 for PCl_5 /*trans* stilbene and each mole fraction ratio was run for 1 hour reflux, 2 hour reflux and 3 hour reflux. The only major

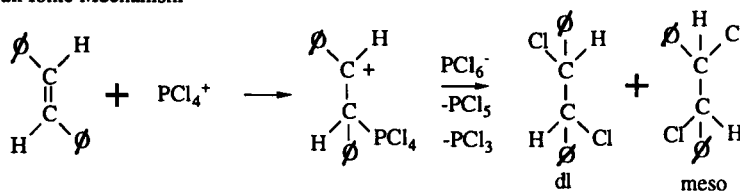
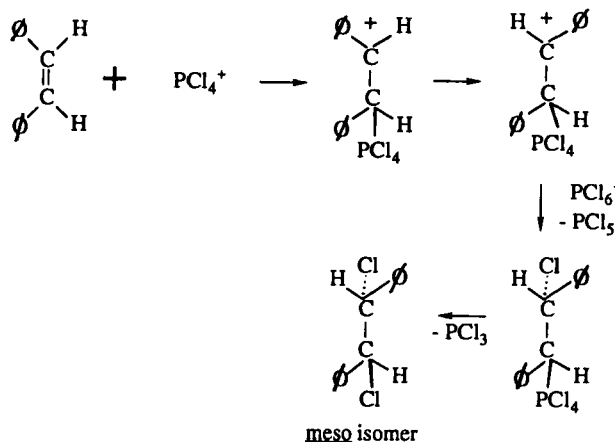
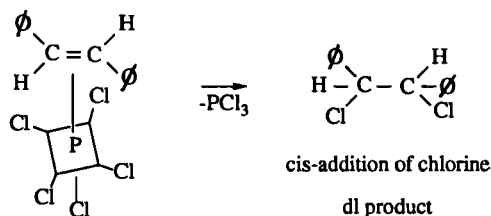
A. Molecular Chlorine Addition



B. Wyman Ionic Mechanism



C. Newman Ionic Mechanism

FIGURE 3 Mechanisms for chlorine addition to *trans*-stilbene with phosphorus pentachloride.FIGURE 4 Meso isomer formation with phosphorus pentachloride ionic mechanisms with *cis*-stilbene.FIGURE 5 sp^3d^2 Octahedral complex of PCl_5 and *trans*-stilbene.

difference we noticed in these reactions was that in the 1:1 mole ratio not all of the *trans*-stilbene reacted regardless of whether it was refluxed for 1 hr. or 3 hrs. These results are illustrated in Table I. As can be seen from Table I, the average ratio of dl to meso dichlorostilbene was 1.42 to 1.

Some of the *trans*-stilbene isomerized in these reactions and the amount of *cis*-stilbene formed is given in Table I. Not all of the *trans*-stilbene and products added up to 100% because on the gas chromatograph these unknown peaks occurred which would account for the difference between 100% and the products produced and reactant recovered.

We analyzed the reaction of phosphorus pentachloride with *trans*-stilbene in methylene chloride after one hour of reflux. Again the *trans*-stilbene did not all react after one hour of reflux in a mole ratio of 1:1, PCl_5 :*trans*-stilbene. However, when the mole ratio was increased to 2:1 and 3:1 all of the phosphorus pentachloride reacted as illustrated in Table II.

TABLE I
Gas chromatographic analysis of *trans*-stilbene with phosphorus pentachloride in 1,1,2,2-tetrachloroethane

One Hour Reflux				
Mole Ratio	1:1	2:1	3:1	4:1
PCl_5 : <i>trans</i> -Stilbene				
<i>cis</i> -Stilbene	2.0 \pm 0.2	6.6 \pm 0.8	1.5 \pm 0.2	0.8 \pm 0.1
<i>trans</i> -Stilbene	26.3 \pm 1.5 ^a	00.0 \pm 0.0	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	37.4 \pm 0.3	50.3 \pm 2.3	51.8 \pm 1.7	47.0 \pm 1.4
meso-Dichlorostilbene	25.1 \pm 0.9	31.0 \pm 1.0	37.6 \pm 1.3	35.5 \pm 1.2
Two Hour Reflux				
Mole Ratio	1:1	2:1	3:1	4:1
PCl_5 : <i>trans</i> -Stilbene				
<i>cis</i> -Stilbene	2.0 \pm 0.5	0.1 \pm 0.01	00.0 \pm 0.0	00.0 \pm 0.0
<i>trans</i> -Stilbene	20.7 \pm 1.8	00.0 \pm 0.0	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	42.0 \pm 2.7	47.5 \pm 1.8	47.6 \pm 1.0	44.8 \pm 1.5
meso-Dichlorostilbene	26.1 \pm 1.8	37.9 \pm 1.2	33.4 \pm 0.6	36.3 \pm 1.6
Three Hour Reflux				
Mole Ratio	1:1	2:1	3:1	4:1
PCl_5 : <i>trans</i> -Stilbene				
<i>cis</i> -Stilbene	0.8 \pm 0.7	00.0 \pm 0.0	00.0 \pm 0.0	00.0 \pm 0.0
<i>trans</i> -Stilbene	12.0 \pm 2.2	00.0 \pm 0.0	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	45.6 \pm 1.9	48.1 \pm 1.1	48.5 \pm 2.1	48.0 \pm 1.6
meso-Dichlorostilbene	33.1 \pm 1.3	36.8 \pm 1.5	35.1 \pm 1.8	30.0 \pm 1.4

^a average and average deviations for two reactions

TABLE II
Gas chromatographic analysis of *trans*-stilbene reaction with phosphorus pentachloride in methylene chloride (1 hr reflux)

Mole Ratio	1:1	2:1	3:1
PCl_5 : <i>trans</i> -stilbene			
<i>trans</i> -Stilbene	57.5 \pm 0.9 ^a	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	14.1 \pm 1.1	44.5 \pm 0.5	44.5 \pm 1.1
meso-Dichlorostilbene	13.9 \pm 1.6	37.3 \pm 1.2	39.4 \pm 1.7

^a average and average deviations for two determinations

For the methylene chloride solvent the mole ratio of dl isomer versus the meso isomer was 1.11:1 compared to the 1,1,2,2-dichloroethane solvent which was 1.42:1.

We also carried out the same reactions in 1,1,2,2-tetrachloroethane with *cis*-stilbene. These results are shown in Table III. We checked the reaction before refluxing and noticed that there was an immediate reaction with phosphorus pentachloride with isomerization to *trans*-stilbene occurring. These results are documented in Table III.

Table III indicates that formation of *trans*-stilbene occurs more readily at a 2:1 ratio than at a 3:1 ratio. This can be explained by the Newman mechanism where the PCl_4^+ reacts with the olefin to make a carbon cation, but since the PCl_6^- is at a low concentration at the 2:1 ratio than at the 3:1 ratio. Consequently, the PCl_4^+ cation complex can complex with *cis*-stilbene and can revert to the *trans*-stilbene before the low concentration of PCl_6^- anion can react with the cation.

TABLE III
Gas chromatographic analysis of *cis*-stilbene reaction with phosphorus pentachloride in 1,1,2,2-tetrachloroethane

Before Reflux			
Mole Ratio PCl_5 : <i>cis</i> -Stilbene	1:1	2:1	3:1
<i>cis</i> -Stilbene	96.6 \pm 3.0	7.9 \pm 0.4	3.4 \pm 0.1
dl-Dichlorostilbene	0.0 \pm 0.0	15.7 \pm 0.8	29.5 \pm 1.5
meso-Dichlorostilbene	1.8 \pm 0.9	40.8 \pm 2.60	43.5 \pm 1.47
<i>trans</i> -Stilbene	1.6 \pm 0.8	30.0 \pm 0.8	19.3 \pm 1.0
Two Hour Reflux			
Mole Ratio PCl_5 : <i>cis</i> -Stilbene	1:1	2:1	3:1
<i>cis</i> -Stilbene	21.6 \pm 1.2	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	25.8 \pm 0.2	35.2 \pm 1.55	35.2 \pm 1.56
meso-Dichlorostilbene	37.9 \pm 1.3	54.6 \pm 2.7	55.0 \pm 2.7
<i>trans</i> -Stilbene	7.6 \pm 0.0	00.0 \pm 0.0	00.0 \pm 0.0
Three Hour Reflux			
Mole Ratio PCl_5 : <i>cis</i> -Stilbene	1:1	2:1	3:1
<i>cis</i> -Stilbene	22.9 \pm 1.2	00.0 \pm 0.0	00.0 \pm 0.0
dl-Dichlorostilbene	25.5 \pm 1.3	33.9 \pm 1.61	34.4 \pm 1.51
meso-Dichlorostilbene	36.6 \pm 1.43	54.6 \pm 2.7	52.0 \pm 2.6
<i>trans</i> -Stilbene	7.6 \pm 0.4	00.0 \pm 0.0	00.0 \pm 0.0

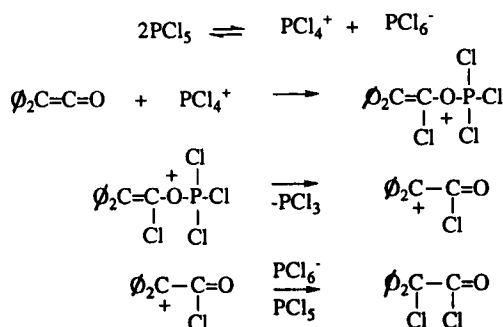


FIGURE 6 Diphenylketene reaction with phosphorus pentachloride.

In these experiments with *cis*-stilbene, the meso:dl ratio came out to be 1.65, almost the reverse with the *trans*-stilbene results. These results were not unexpected because if the chlorine atoms are added *cis* to the *trans*-stilbene as suggested for a sp^3d^2 complex, the product would be a dl pair. However, with *cis*-stilbene the *cis* addition of chlorine would produce the meso adduct.

A third reaction studied was phosphorus pentachloride with diphenylketene in a 1:1 mole ratio of diphenylketene and phosphorus pentachloride in refluxing methylene chloride. The reaction produced a 65% yield of 1-chlorodiphenylacetyl chloride. These results can be explained by Newman's ionic mechanism as illustrated below in Figure 6.

Ketenes are known to react with molecular chlorine to produce acyl halides in low yields.⁵ However, since our yields were high and methylene chloride dissociates PCl_5 into ions, the ionic mechanism is justified for diphenylketene.

We have examined the literature for phosphorus pentachloride reactions with conjugated olefins and found where stilbene reacts in weakly polar solvents to produce meso-dichlorostilbene, but the dl product increased with solvent polarity.⁶ In the proposed mechanism the pi orbital of stilbene was attaching to the equatorial chlorine of phosphorus pentachloride.

Phosphorus pentachloride reacts with divinyl ether⁷ to form a cyclic ring containing phosphorus with two chlorines attached, two double bonds conjugated with an ether functional group all in a six membered ring. This compound was produced in 75% yield, but no discussion of the mechanism was discussed.

In the stereochemical reaction mechanism to form phosphate esters, styrene reacted with phosphorus pentachloride to form an adduct with the phosphorus tetrachloro cation (PCl_4^+), which reacted further with phosphorus pentachloride and hydrochloric acid to form a styrene phosphorus oxychloride derivative, but no chlorination of the double bond was reported.⁸

EXPERIMENTAL

Methylene Chloride. J. T. Baker Chem. Co., practical grade methylene chloride was purified by first washing with concentrated sulfuric acid followed by washing with water. The wet solvent was dried over calcium chloride for 3 hours and distilled from phosphorus pentoxide through a 36-inch glass helices fractionating column.

1,1,2,2-Tetrachloroethane. This solvent was treated in the exact manner as methylene chloride and distilled through the 36-inch glass helices fractionating column.

Preparation of Diphenylketene. A chilled solution of triethylamine [Eastman White Label distilled from calcium oxide; 31 ml (0.20 mole)] in 100 ml of anhydrous ether (Mallinckrodt Chem. Co.) was dropped into a chilled solution of diphenylacetyl chloride (46 g; 0.26 mole) in 100 ml of anhydrous ether. The solution was stirred for 1 hr. and filtered, and the precipitate was washed with anhydrous ether. The solution, after adding a small crystal of hydroquinone, was rotary evaporated to 75 ml at room temperature and vacuum distilled under nitrogen. The ketene (40 ml) was collected at 0.88 mm between 94–104° (lit.⁹ bp 119–121°C at 3.5 mm).

Preparation of Diphenylacetyl Chloride. Diphenylacetic acid [Aldrich Chem. Co., 55 g. (0.26 mole)] was placed in a 250 ml flask. Thionyl chloride [Eastman White Label, 28 ml (0.29 mole)] was dropped through a condenser onto the acid. The solution was refluxed for 2 hrs. Crystals were obtained upon cooling which were recrystallized from ligroin yielding 53 g (86%) mp 55–56° (lit.¹⁰ mp 56–57°C).

Phosphorus Pentachloride. J. T. Baker Chem. Co., reagent grade. All phosphorus pentachloride was sublimed before use.

Gas Chromatograph. Varian Aerograph Model 90-P with a 1 mv Honeywell recorder, 6 ft., 1/4 m o.d. Porapak Q Column, 180° column temperature at 60 ml/min.

Preparation of 2,3,4,5-Tetraphenylcyclopentadienone. Freshly distilled dibenzyl ketone [Eastman Yellow Label 15.5 g (0.074 mole)] was added to recrystallized benzil [Eastman Yellow Label 15.5 g (0.074 mole)] in 115 ml of absolute ethanol and potassium hydroxide (2.3 g). The reaction was refluxed for 15 min. Crystals were recrystallized from ethanol, mp 220–222°C (lit.¹¹ mp 219–220°).

Reaction of Trans-Stilbene with Phosphorus Pentachloride in 1,1,2,2-Tetrachloroethane. Sublimed phosphorus pentachloride was placed with 49 ml of 1,1,2,2-tetrachloroethane in a 100 ml, round-bottom flask with a condenser and calcium chloride tube attached. The amounts of phosphorus pentachloride used was 0.577 g (0.00277 mole), 1.154 g (0.00554 mole), 1.731 g (0.00831 mole) and 2.308 g (0.0111 mole) and to each of these solutions was added 0.500 g (0.00277 mole) of *trans*-stilbene. Each series of reactions was refluxed for 1, 2, 3, and 4 hrs. as indicated in Table I. Similar reactions were run with *trans*-stilbene in methylene chloride as documented in Table II.

Cis-stilbene was run with phosphorus pentachloride in 1,1,2,2-tetrachloroethane under the same conditions as described above for *trans*-stilbene and the results have been shown in Table III.

Reaction of Phosphorus Pentachloride with Diphenylketene. Diphenylketene (14.2 g; 0.073 mole) was dissolved in 100 ml of methylene chloride. To this solution was added phosphorus pentachloride (15.8 g; 0.0759 mole) dissolved in 100 ml of methylene chloride, and the solution was refluxed for 10 hrs during which time the orange solution changed to a clear yellow solution. The solution was rotary evaporated, and the residue was recrystallized from petroleum ether yielding 12.5 g (65% yield) of product, mp 47–49°C (lit.¹², mp 50–51°).

Reaction of Phosphorus Pentachloride with 2,3,4,5-Tetraphenylcyclopentadienone. 2,3,4,5-Tetraphenylcyclopentadienone (18 g; 0.047 mole) was placed in a 100 ml of 1,1,2,2-tetrachloroethane containing 20 g (0.095 mole) of sublimed phosphorus pentachloride. The solution was heated to reflux, cooled and rotary evaporated. The residue was recrystallized from absolute ethanol which yielded 7 g. (33%) of crystals mp, 182–84°C. Anal. Calcd for C₂₉H₂₀O₁Cl₂: C, 76.50; H, 4.40; O, 3.50; Cl, 15.60. Found. C, 76.7; H, 4.43; O, 3.32; Cl, 15.55.

CONCLUSIONS

Phosphorus pentachloride reacted with conjugated olefins (*cis* and *trans*-stilbene) to produce a dl/meso isomer ratio of 0.606 and 1:42, respectively, which can be explained through an sp³d² octahedral complex of phosphorus pentachloride with the conjugated olefins. The ionic mechanism (PCl₄⁺, PCl₆⁻) would generate the chloro derivatives in the reverse order, i.e., the meso/dl ratio would be greater than one. However, the ionic mechanism does occur with a reactive conjugated carbonyl such as diphenylketene, but does not occur with conjugated carbonyls such as 1,2,3,4-tetraphenylcyclopentadienone where the molecular chlorine is reacting from the dissociation of phosphorus pentachloride into phosphorus trichloride and chlorine.

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