

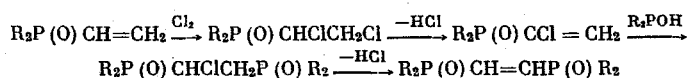
TETRAARYL(ALKYL)VINYLENEDIPHOSPHINES AND  
SOME OF THEIR DERIVATIVES

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Our study of the extraction properties of some tertiary alkylenediphosphine dioxides and, in particular, of the *cis*- and *trans*-isomers of tetraphenylvinylenediphosphine dioxide, revealed that steric factors exert a very great effect on the extraction properties of the ligands: for example, for *cis*-tetraphenylvinylenediphosphine dioxide the distribution coefficient of uranyl nitrate was four orders of magnitude higher than for the *trans*-isomer [1]. Analogous results were obtained when the complexing of these ligands with the alkali metals was studied [2]. In the present paper we report the synthesis and certain properties of some new *cis*- and *trans*-isomers of tetraaryl(alkyl)vinylenediphosphine dioxides, and also of the disulfides and diphosphines that correspond to them.

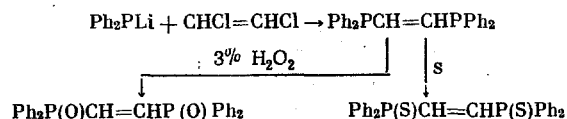
We used our previously proposed synthesis scheme [3] to obtain the trans-vinylenediphosphine dioxides. The chlorination of the vinyl diarylphosphine oxide and subsequent dehydrochlorination gave the chlorovinyl diarylphosphine oxide. The addition of a diarylphosphine oxide to the latter leads to the tetra-arylchloroethylenediphosphine dioxide, the dehydrochlorination of which gives the trans-tetraarylvinylene-diphosphine dioxide.



Trans-ethyltriphenylvinylenediphosphine dioxide (XI), the unsymmetrical trans-diethyldiphenylvinylenediphosphine dioxide (XII), and trans-phenyltriethylvinylenediphosphine dioxide (XIII) were synthesized by the method (Table 1).

The intermediate  $\alpha$ -chlorovinylethylphenylphosphine oxide and ethyltriphenylchloroethylenediphosphine dioxide was isolated in the synthesis of dioxide (XI). Dioxides (XII) and (XIII) were obtained without isolating the intermediate products. As was shown by the IR and NMR spectral data, dioxides (XI), (XII), and (XIII) are the trans-isomers. In particular, the IR spectra are devoid of a band at  $720\text{ cm}^{-1}$ , which is characteristic for the deformation vibrations of the cis-CH = CH group, and have a band at  $980\text{ cm}^{-1}$ , which is characteristic for the deformation vibrations of the trans-CH = CH group [4-6].

The *cis*-isomers of the vinylenediphosphine dioxides cannot be obtained by the described method. The vinylenediphosphines with a *cis*- and a *trans*-structure were obtained by the reaction of lithium diphenylphosphide with either *cis*- or *trans*-dichloroethylene [4, 7].



With the cited scheme we synthesized the trans- and cis-tetra-p-tolylvinylenediphosphines (I) and (II), respectively, the trans- and cis-tetra-p-tolylvinylenediphosphine dioxides (III) and (IV), the trans- and cis-tetra-p-tolylvinylenediphosphine disulfides (V) and (VI), trans-tetra-p-ethylphenylvinylenediphosphine dioxide (VII), the symmetrical trans-diethyldiphenylvinylenediphosphine (VIII), its dioxide (IX), and the disulfide (X).

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TABLE 1

Compound	Yield, %	Mp, °C	Found Calculated %			Empirical formula	$\delta_{31P}$ , ppm
			C	H	P		
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ trans (I)	55	117–119			13,5 13,7	$\text{C}_{30}\text{H}_{30}\text{P}_2$	–8,6
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ cis (II)	30,5	81–82	79,6 79,6	6,6 6,6	13,9 13,7	$\text{C}_{30}\text{H}_{30}\text{P}_2$	–24,6
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ trans (III)	84	292–293	74,1 74,4	6,3 6,2	12,8 12,8	$\text{C}_{30}\text{H}_{30}\text{P}_2\text{O}_2$	–21,7
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ cis (IV)	80	227–229			12,5 12,8	$\text{C}_{30}\text{H}_{30}\text{P}_2\text{O}_2$	–20,6
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ trans (V)	86	224,5–225,5			12,4 * 12,4	$\text{C}_{30}\text{H}_{30}\text{P}_2\text{S}_2$	–35,9
$(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2$ cis (VI)	67	225–226			12,6 * 12,4	$\text{C}_{30}\text{H}_{30}\text{P}_2\text{S}_2$	–32,1
$(p\text{-C}_6\text{H}_5\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{C}_6\text{H}_5)_2$ trans (VII)	29	264–266	75,0 75,5	7,1 7,0	11,9 11,5	$\text{C}_3\text{H}_{18}\text{P}_2\text{O}_2$	–22,8
$(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_6\text{H}_5)(\text{C}_2\text{H}_5)_2$ trans (VIII)	30,5	Bp 166–169(2)			20,2 20,7	$\text{C}_{18}\text{H}_{22}\text{P}_2$	–8,6
$(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_6\text{H}_5)(\text{C}_2\text{H}_5)_2$ trans (IX)	79,2	204–205	65,0 65,1	6,9 6,6	18,7 18,7	$\text{C}_{18}\text{H}_{22}\text{P}_2\text{O}_2$	–30,0
$(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_6\text{H}_5)(\text{C}_2\text{H}_5)_2$ trans (X)	64,2	193–194			17,6 * 17,6	$\text{C}_{18}\text{H}_{22}\text{P}_2\text{S}_2$	–42,4
$(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_6\text{H}_5)(\text{C}_2\text{H}_5)_2$ trans (XI)	63	214–215	63,1 63,1	5,8 5,8	16,2 16,3	$\text{C}_{22}\text{H}_{22}\text{P}_2\text{O}_2$	–21,5 –30,0
$(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_2\text{H}_5)_2$ trans (XII)	82	154–155	65,5 65,4	6,7 6,6	18,8 18,7	$\text{C}_{18}\text{H}_{22}\text{P}_2\text{O}_2$	–22,1 –41,7
$(\text{C}_6\text{H}_5)(\text{C}_2\text{H}_5)_2\text{PCH=CHP}(\text{C}_2\text{H}_5)_2$ trans (XIII)	63	135–136	58,4 59,1	7,7 7,7	21,7 21,8	$\text{C}_{11}\text{H}_{22}\text{P}_2\text{O}_2$	–30,0 –40,7
$[(\text{C}_6\text{H}_5)_2\text{PCH=CHP}(\text{C}_6\text{H}_5)_2] \cdot 2\text{I}^-$ trans (XIV)	70	178–231 (decompn)	49,3 49,4	4,2 4,1	9,2 9,1	$\text{C}_{22}\text{H}_{22}\text{P}_2\text{I}_2$	–20,1
$[(p\text{-CH}_3\text{C}_6\text{H}_4)_2\text{PCH=CHP}(\text{C}_6\text{H}_4\text{CH}_3)_2] \cdot 2\text{I}^-$ trans (XV)	66	267–269 (decompn)	34,4 † 34,5		8,2 8,4	$\text{C}_{32}\text{H}_{36}\text{P}_2\text{I}_2$	–19,2

\* S, %.

† I, %.

We also studied the reaction of lithium ethylphenylphosphide with cis-dichloroethylene. However, in this case, after oxidation and the addition of sulfur, the trans-dioxide (IX) and disulfide (X) were isolated, which is confirmed by the IR and NMR spectra. It is known [5] that the reaction of lithium di-n-butylphosphide with cis-dichloroethylene (at  $-78^\circ\text{C}$ ) gives a mixture of the cis- and trans-isomers; however, the cis-isomer of tetra-n-butylvinylenediphosphine was not isolated in the pure form. As a result, trans-isomerization proceeds very easily in vinylenediphosphines where alkyl radicals are attached to the phosphorus atoms. The configuration of the isomers is retained when sulfur adds to vinylenediphosphines.

The obtained aryl(alkyl)vinylenediphosphine dioxides and disulfides are colorless crystalline compounds that are insoluble in water, and difficultly soluble in nonpolar solvents. The compounds with alkyl radicals on the phosphorus atoms have a better solubility. The chemical shifts were measured for all of the synthesized compounds, and also for the previously described diphosphorus compounds [4, 7]: trans-tetraphenylvinylenediphosphine (XVI)  $\delta_{31P}$  –7.0 ppm; cis-tetraphenylvinylenediphosphine (XVII)  $\delta_{31P}$  –22.5 ppm; cis-tetraphenylvinylenediphosphine dioxide (XVIII)  $\delta_{31P}$  –20.5 ppm; trans-tetraphenylvinylenediphosphine dioxide (XIX)  $\delta_{31P}$  –21.6 ppm. The constants, analysis data, yields, and chemical shifts of the obtained compounds are given in Table 1.

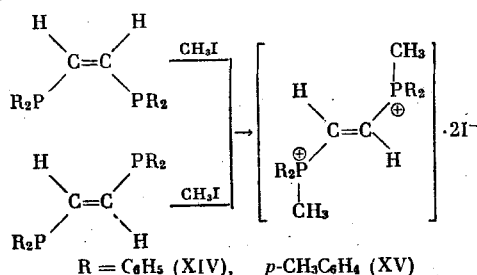
Attempts to effect the thermal, catalytic (in the presence of benzoyl peroxide), and photochemical isomerization of trans-tetraphenylvinylenediphosphine dioxide to the cis-isomer proved unsuccessful. The reverse transition of the cis-dioxide to the trans-isomer is accomplished with relative ease. Previously [8] the analogous isomerization of the cis-dioxide (XVIII) to the trans-isomer (XIX) was observed by refluxing in THF in the presence of  $\text{PCl}_3$ .

TABLE 2

Dienophile	Yield, %	Mp, °C	Found, % Calculated			Empirical formula	$\delta_{31P}$ , ppm
			C	H	P		
(IV)-cis	47,3	238—240 (decompn.)	76,2 76,4	6,6 6,5	11,3 11,3	C <sub>35</sub> H <sub>36</sub> P <sub>2</sub> O <sub>2</sub>	—40,5
(XII)-trans	46	210—211 (decompn.)	69,4 69,3	7,0 7,0	15,0 15,6	C <sub>23</sub> H <sub>28</sub> P <sub>2</sub> O <sub>2</sub>	
(XVIII)-cis	50,8	257—260 (decompn.)	74,9 75,3	5,7 5,7	12,6 12,5	C <sub>31</sub> H <sub>28</sub> P <sub>2</sub> O <sub>2</sub>	—56,5
(XIX)-trans	72	>200 (decompn.)	75,5 75,3	5,7 5,7	12,5 12,5	C <sub>31</sub> H <sub>28</sub> P <sub>2</sub> O <sub>2</sub>	—28,6* —31,8

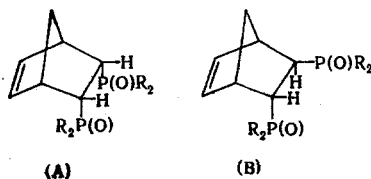
\* Nonequivalent phosphorus atoms of AB systems,  $J_{AB} = 9.8 \pm 1$  Hz.

The dimethiodides, corresponding to only one isomer, were isolated in both cases when the dimethiodides of the trans- and cis-vinylenediphosphines (I), (II), (XVI), and (XVII) were prepared, and specifically: tetraphenylvinylenediphosphine dimethiodide (XIV),  $\delta_{31P}$  –20.1 ppm (CH<sub>3</sub>OH), and tetra-*p*-tolylvinylenediphosphine dimethiodide (XV),  $\delta_{31P}$  –19.2 ppm.



Based on the IR spectra (presence of a band at  $980\text{ cm}^{-1}$  in the vinylenediphosphine dimethiodides) and the mixed melting points of the obtained vinylenediphosphine methiodides it may be assumed that in both cases the structure of the dimethiodides corresponds to the trans-structure.

On the example of dioxides (IV), (XII), (XVIII), and (XIX) it was shown that they are capable of entering into the diene synthesis reaction with cyclopentadiene. Based on the known stereochemical rules of the diene synthesis [9–11], it is possible to assign the cis-endo-(A)-configuration to the dioxide adducts obtained from the cis-compounds, and the trans-(B)-configuration to the adducts obtained from the trans-dioxides. In the  $^{31}P\{^1H\}$  NMR spectrum of compound (A) the singlet signal is an additional argument in support of the cisoid conformation, where the phosphorus nuclei are magnetically equivalent. In the spectrum of compound (B) the quadruplet testifies to the transoid conformation. As a result, these reactions proceed with a retention of the conformation. The constants, analysis data, yields, and chemical shifts of the obtained compounds are given in Table 2.



## EXPERIMENTAL METHOD

The purity of the dichloroethylene cis- and trans-isomers was checked by GLC and the IR spectra. The THF was freshly distilled over  $LiAlH_4$  prior to use. The  $^{31}P$  NMR spectra were taken on a Bruker HX-90 spectrometer.

**Trans-tetra-*p*-tolylvinylenediphosphine (I).** To a suspension of 2.2 g (0.32 g-atom) of Li in 75 ml of THF was added in drops at  $\sim 20^\circ$ , in an argon stream, 19 g (0.08 mole) of di-*p*-tolylchlorophosphine in 30 ml of THF, after which 30 ml of THF was added and the mixture was stirred at  $\sim 20^\circ$  for 1.5 h, and at THF reflux for 0.5 h (the color of the solution was dark cherry-red). Then the mixture was cooled, the excess Li was filtered in an argon stream, and to the filtrate was slowly added in drops 3.9 ml (0.04 mole) of trans-dichloroethylene in 15 ml of THF at  $0-5^\circ$ . Then the reaction mixture was stirred at  $\sim 20^\circ$  for 2 h.

After distilling off the solvent the residue was poured into water. The obtained crystalline precipitate was filtered, and washed in succession with water and alcohol. The yield of the crude product was 11.2 g, and 10.1 g after a double recrystallization from alcohol.

Cis-tetra-p-tolylvinylenediphosphine (II). From 9 g (0.037 mole) of di-p-tolylchlorophosphine, 1 g (0.145 g-atom) of Li, and 1.8 ml (0.018 mole) of cis-dichloroethylene at  $-10^{\circ}$  we obtained 3.6 g of crude product. Extraction with hot hexane gave 2.5 g of (II).

Trans-tetra-p-tolylvinylenediphosphine Dioxide (III). To 0.85 g (1.8 mmoles) of trans-tetra-p-tolylvinylenediphosphine in 30 ml of acetone at  $\sim 20^{\circ}$  was added 30 ml of 3%  $\text{H}_2\text{O}_2$  solution. The obtained precipitate was recrystallized from alcohol to give 0.72 g of (III).

Cis-tetra-p-tolylvinylenediphosphine Dioxide (IV). From 0.4 g (0.8 mmole) of cis-tetra-p-tolylvinylenediphosphine in 20 ml of acetone and 10 ml of 3%  $\text{H}_2\text{O}_2$  solution we obtained 0.33 g of (IV) recrystallized from a benzene-petroleum ether mixture).

Trans-tetra-p-tolylvinylenediphosphine Disulfide (V). A mixture of 1 g (2.0 mmoles) of trans-tetra-p-tolylvinylenediphosphine and 0.16 g (5.0 mmoles) of sulfur in 2 ml of benzene was heated in an ampul at  $120^{\circ}$  for 2 h. The obtained crystals were recrystallized twice from benzene to give 0.98 g of (V).

Cis-tetra-p-tolylvinylenediphosphine Disulfide (VI). A mixture of 0.3 g (0.6 mmole) of cis-tetra-p-tolylvinylenediphosphine and 0.08 g (2.5 mmoles) of sulfur in 2 ml of benzene was heated at  $120^{\circ}$  for 0.5 h. After recrystallization from benzene we obtained 0.22 g of (VI).

Trans-tetra-p-ethylphenylvinylenediphosphine Dioxide (VII). From 4.5 g (16 mmoles) of di-p-ethylphenylchlorophosphine, 0.7 g (0.1 g-atom) of Li, and 9 g (0.09 mole) of trans-dichloroethylene, without isolating the corresponding diphosphine, by oxidation with 3%  $\text{H}_2\text{O}_2$  solution we obtained 1.28 g of (VII).

Symmetrical Trans-diethyldiphenylvinylenediphosphine (VIII). From 8.8 g (0.05 mole) of ethylphenylchlorophosphine, 1 g (0.145 g-atom) of Li, and 2.4 g (0.025 mole) of trans-dichloroethylene, after distilling off the solvent, we isolated 2.2 g of (VIII) by distillation.

Sym-trans-diethyldiphenylvinylenediphosphine Dioxide (IX). From 0.8 g (2.6 mmoles) of diphosphine (VIII) and 10 ml of 3%  $\text{H}_2\text{O}_2$  solution we obtained 0.65 g of (IX) (recrystallized from a benzene-petroleum ether mixture).

Sym-trans-diethyldiphenylvinylenediphosphine Disulfide (X). A mixture of 1.2 g (4 mmoles) of diphosphine (VIII) and 0.6 g (18 mmoles) of sulfur in 20 ml of benzene was heated in an ampul at  $120^{\circ}$  for 2 h. After recrystallization from acetone we obtained 0.9 g of (X).

$\alpha$ -Chlorovinylethylphenylphosphine Oxide. A stream of chlorine was passed into a solution of 9.4 g (0.05 mole) of vinyl ethylphenylphosphine oxide [12] in 75 ml of  $\text{CCl}_4$  for 2 h at  $40-50^{\circ}$ . Then the solvent was distilled off and 50 ml of benzene and 6.6 g of triethylamine were added to the residue. The reaction mixture was refluxed for 3 h. After separating the triethylamine hydrochloride (5.6 g) the residue was distilled. We obtained 6.2 (49%) of a hygroscopic substance, mp  $48-49^{\circ}$  (in a sealed capillary). Found: Cl 16.3%.  $\text{C}_{10}\text{H}_{12}\text{POCl}$ . Calculated: Cl 16.5%.

Ethyltriphenylchloroethylenediphosphine Dioxide. A mixture of 1.9 g (8 mmoles) of  $\alpha$ -chlorovinylethylphenyldiphosphine oxide and 1.9 g (9 mmoles) of diphenylphosphine oxide in 4 ml of benzene was heated in an ampul for 5 h at  $100^{\circ}$ . After distilling off the solvent the residual glassy mass was rubbed in absolute toluene and then recrystallized from toluene. We obtained 1.8 g (36%) of substance, mp  $175-176^{\circ}$ . Found: Cl 8.6%.  $\text{C}_{22}\text{H}_{23}\text{P}_2\text{O}_2\text{Cl}$ . Calculated: 8.5%.

Trans-ethyltriphenylvinylenediphosphine Dioxide (XI). A mixture of 0.54 g (1 mmole) of ethyltriphenylchloroethylenediphosphine dioxide and 0.5 g (5 mmoles) of triethylamine in 4 ml of toluene was heated in a sealed ampul for 2 h at  $110^{\circ}$ . The crystalline substance was filtered, washed on the filter with water, and recrystallized from an alcohol-benzene mixture. We obtain 0.31 g of (XI).

Unsym-trans-diethyldiphenylvinylenediphosphine Dioxide (XII). A mixture of 4 g (0.015 mole) of  $\alpha$ -chlorovinyl diphenylphosphine oxide [3] and 2.2 g (0.02 mole) of diethylphosphine oxide in 6 ml of benzene was heated in an ampul for 8 h at  $100^{\circ}$ . Then the benzene was distilled off and 2 g (0.019 mole) of triethylamine in 5 ml of toluene was added to the residue. The obtained hydrochloride was filtered, the solvent was distilled off, and the residue was recrystallized twice from a benzene-ether mixture. We obtained 1.9 g of (XII).

Trans-ethyltriphenylvinylenediphosphine Dioxide (XIII). From 1 g (4.6 mmoles) of  $\alpha$ -chlorovinyl-ethylphenylphosphine oxide, 0.6 g (5 mmoles) of diethylphosphine oxide, and 1 g (9.8 mmoles) of triethylamine we obtained 0.8 g of (XIII) (recrystallized from a benzene-ether mixture).

Reaction of Cis-tetraphenylvinylenediphosphine (XVII) with Methyl Iodide. To 0.6 g (1.7 mmoles) of cis-tetraphenylvinylenediphosphine in 25 ml of alcohol was added 1 g (7 mmoles) of  $\text{CH}_3\text{I}$ . The crystals obtained on standing were filtered and washed in succession with alcohol and absolute ether. The yield of (XIV) was 0.7 g, mp 280-282° (decompn.).

Reaction of Trans-tetraphenylvinylenediphosphine with  $\text{CH}_3\text{I}$ . From 0.6 g (1.7 mmoles) of trans-tetraphenylvinylenediphosphine and 1.1 g (8 mmoles) of  $\text{CH}_3\text{I}$  we obtained 0.67 g of (XIV) with mp 278-281° (decompn.). The mixed melting point of (XIV) with the above described compound was not depressed.

Reaction of Cis-tetra-p-tolylvinylenediphosphine (II) with  $\text{CH}_3\text{I}$ . To 0.8 g (1.7 mmoles) of diphosphine (II) in 10 ml of absolute ether was added 1 g (7 mmoles) of  $\text{CH}_3\text{I}$ . We obtained 0.75 g of (XV) with mp 264-267° (decompn.).

Reaction of Trans-tetra-p-tolylvinylenediphosphine with  $\text{CH}_3\text{I}$ . From 0.4 g (0.85 mmole) of diphosphine (I) and 0.5 g (3.5 mmoles) of  $\text{CH}_3\text{I}$  we obtained 0.43 g of substance, mp 267-269° (decompn.). The mixed melting point with the substance obtained in the preceding experiment was 265-267° (decompn.).

Trans-tetraphenyl-bicyclo[1, 2, 2]-5-heptene-2, 3-diphosphine Dioxide. A mixture of 1 g (2 mmoles) of trans-tetraphenylvinylenediphosphine dioxide (XVI) and 5 g (7 mmoles) of cyclopentadiene was heated in an ampul for 2 h at 160°. We obtained 0.8 g of substance, which was purified on a column containing  $\text{Al}_2\text{O}_3$  (II activity) by elution with a 4:1 benzene-alcohol mixture.

Cis-endo-tetraphenyl-bicyclo[1, 2, 2]-5-heptene-2, 3-diphosphine Dioxide. From 0.5 g (1 mmole) of cis-tetraphenylvinylenediphosphine dioxide (XVII) and 3.3 g (50 mmoles) of cyclopentadiene we obtained 0.28 g of substance, which was purified on  $\text{Al}_2\text{O}_3$  by elution with a 4:1 benzene-alcohol mixture.

Cis-endo-tetra-p-tolyl-bicyclo[1, 2, 2]-5-heptene-2, 3-diphosphine Dioxide. From 0.34 g (7 mmoles) of dioxide (IV) and 3.3 g (50 mmoles) of cyclopentadiene we obtained 0.18 g of substance.

Unsym-trans-diethyldiphenyl-bicyclo[1, 2, 2]-5-heptene-2, 3-diphosphine Dioxide. From 0.4 g (1 mmole) of (XII) and 5 g (75 mmoles) of cyclopentadiene we obtained 0.22 g of substance, which was purified on  $\text{Al}_2\text{O}_3$  by elution with a 7:1 benzene-alcohol mixture.

## CONCLUSIONS

1. We synthesized a number of new tetraaryl(alkyl)vinylenediphosphines, and also their dioxides and disulfides with a cis- and a trans-structure.
2. The adducts of the dioxides with cyclopentadiene were obtained.

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