^{19}F NMR study of *cis*- and *trans*-effects of substituted triphenylphosphine ligands in *trans*-(4-fluorophenyl)triarylphosphine-*tri*(4-fluorophenyl)phosphineplatinum 4-fluorothiophenoxides

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A number of compounds of the type of *trans*-4-$FC_6H_4Pt(PAr_3)_2SC_6H_4F$-4, where Ar is a substituted phenyl group, have been prepared starting from the corresponding chlorides. By exchange reactions of *trans*-4-$FC_6H_4Pt[P(C_6H_4F\text{-}4)_3]_2SC_6H_4F$-4 with the above-mentioned compounds or Ar_3P, *trans*-4-$FC_6H_4Pt[P(C_6H_4F\text{-}4)_3][PAr_3]SC_6H_4F$-4 have been generated in solution. For the latter compounds, the effect of Ar_3P on *cis*- and *trans*-ligands has been studied by the ^{19}F NMR technique. It has been shown that the *cis*- and *trans*-effects of Ar_3P run parallel and are well described by pK_a values and ionization potentials of the unshared electron pair in Ar_3P, as well as by σ^0 constants of the aryl groups.

Key words: platinum(II), *trans*-aryl-*bis*-triarylphosphine-platinum thiophenoxides, triarylphosphines, *cis*-effect, *trans*-effect, ^{19}F NMR.

The problem of *cis*- and *trans*-effects of ligands in inorganic and organometallic compounds of bivalent platinum has been intensely studied by various physical methods.[1] However, the influence of neutral ligands has been studied to a considerably lesser degree than that of acido ligands.[1] In addition, the authors of previous studies did not consider a correlation between the physical parameters studied and characteristics of the electron-donating abilities of the corresponding ligands. Finally, despite the fact that the *cis*- and *trans*-effects of neutral ligands have been studied with respect to acido ligands and neutral ligands, these effects have not been compared in terms of the same structural model.

^{13}C and ^{31}P NMR spectroscopies have been used to study the effects of neutral ligands, along with other physical methods. The application of the former method involved a study of ^{13}C chemical shifts for the carbon atoms of the C=C bond and $J(^{195}Pt,^{13}C)$ spin coupling constants in olefinic complexes of platinum,[2] and the application of the latter method involved a study of $J(^{195}Pt,^{31}P)$ spin coupling constants.[3] It should be noted that, though ^{13}C chemical shifts are a sensitive indicator of the variation of the electron density at an aromatic carbon atom under the influence of remote structural changes,[4] the use of ^{13}C NMR for a system containing a large number of aryl groups of similar structures, in the case where a spectrometer operating at 200 MHz is used, may be complicated by superposition of signals and difficulties in their assignment.

However, in our opinion, platinum—element spin coupling constants cannot be regarded as indicators of the effect of one ligand on another, since they also reflect the effect of the ligand on the metal atom. In addition, the direct platinum—element spin coupling constants can exhibit anomalous behavior as the electron density on the electron-donating atom of the ligand varies. In fact, recently it was found[5] that the effects of substituents in the complexes of substituted triphenylphosphines with platinum dichloride on the $J(^{195}Pt,^{31}P)$ spin coupling constants for the *cis*- and *trans*-series are antiparallel: as the electron-withdrawing ability of substituents increase, this constant increases in the case of compounds of the *trans*-series, but it decreases for *cis*-compounds.

In view of the foregoing, ^{19}F NMR spectroscopy combined with the use of neutral and acido ligands containing indicator 4-FC_6H_4 groups seems to be a more promising method for the investigation of the *cis*- and *trans*-effects of neutral ligands. This is caused by the high sensitivity of fluorine chemical shifts (FCS) in 4-substituted fluorobenzenes to the variation of the π-electron density at the neighboring carbon atom, which is equal to 661.5 ppm per electron.[6] Consequently, the 4-FC_6H_4 group is a sensitive indicator of the variation of the electron density at the atom attached to it.[7] These facts are clearly manifested in that the FCS of the $(4\text{-}FC_6H_4)_3P$ (**1a**) change appreciably on going from the free ligand to its complexes.[8] In addition, ^{19}F NMR allows the *cis*- and *trans*-effects of a neutral ligand on neutral and acido ligands to be studied in the framework of one method and one structural model.

Therefore, in the present work we have used the ^{19}F NMR method to study the *cis*- and *trans*-effects of substituted triphenylphosphines on neutral and acid lig-

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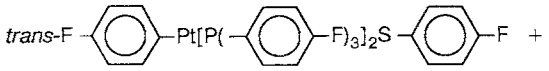
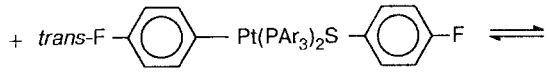
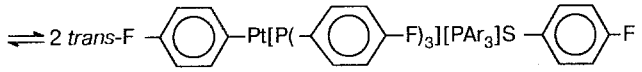
ands. We chose *trans*-4-fluorophenyl-*tri*(4-fluorophenyl)phosphinetriarylphosphineplatinum 4-fluorothiophenoxides (**2**), which were generated in a solution by two methods. The first method was based on exchange reactions of **3a** with **3b—e**:



3a



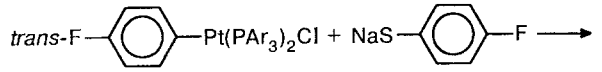
3b—e



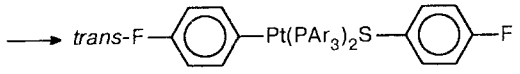
2b—e

Ar = Ph (**b**), 4-MeC_6H_4 (**c**), 4-$Me_2NC_6H_4$ (**d**), 3,4-$Cl_2C_6H_3$ (**e**).

Compounds **3** were prepared by the reactions of the corresponding chlorides (**4**) with $NaSC_6H_4F$-4:



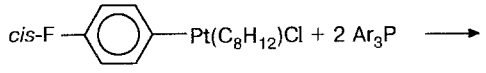
4c—e



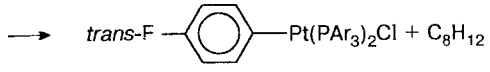
3c—e

Ar = 4-$CH_3C_6H_4$ (**c**); 4-$Me_2NC_6H_4$ (**d**); 3,4-$Cl_2C_6H_3$ (**e**).

In turn, chlorides **4** were synthesized by the reaction of the 1,5-cyclooctadiene complex (**5**) with substituted triphenylphosphines:



5 **1c—e**



4c—e

Ar = 4-$CH_3C_6H_4$ (**c**); 4-$Me_2NC_6H_4$ (**d**); 3,4-$Cl_2C_6H_3$ (**e**).

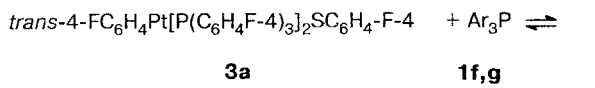
The *trans*-configurations of compounds **3** and **4** were confirmed by the ^{31}P NMR spectra. The data of the ^{31}P and ^{19}F NMR spectra of this type of compounds are presented in Table 1.

Table 1. ^{19}F and ^{31}P chemical shifts and $J(^{195}Pt,^{31}P)$ spin coupling constants for compounds **3** and **4** in $CHCl_3$

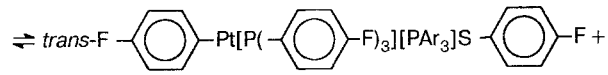
Compound	FCS, ppm		$\delta^{31}P$	$J(^{195}Pt,^{31}P)$
	4-FC_6H_4	4-FC_6H_4S		Hz
3b*	12.68	11.56	—	—
3c	13.17	12.27	19.80	3032
3d	14.93	13.67	16.66	2935
3e	8.59	8.36	20.27	3183
3f	13.12	12.09	—	—
3g	10.88	10.14	—	—
4c	13.47	—	22.86	3069
4d	15.21	—	19.53	2983
4e	8.60	—	23.97	3177

* The compound was described previously.[14]

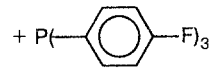
The second method of generation of compounds **2** was based on exchange reactions of **3a** with **1f—g**:



3a **1f,g**



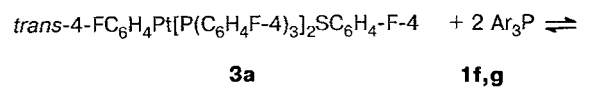
2f,g



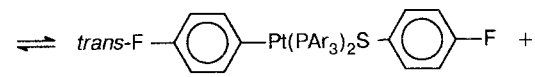
1a

Ar = 4-$Me_3OC_6H_4$ (**f**), 4-ClC_6H_4 (**g**).

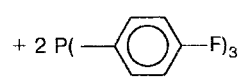
For similar triphenylphosphines, both methods gave conforming results. Compounds **3f** and **3g** were also formed by the following reactions:



3a **1f,g**



3f,g



1a

Ar = 4-$MeOC_6H_4$ (**f**), 4-ClC_6H_4 (**g**).

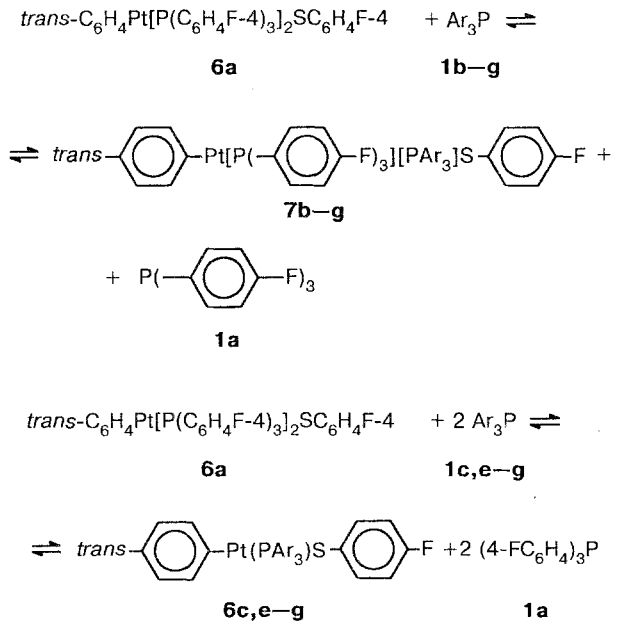
We determined the FCS for compounds **2** in $CHCl_3$ with respect to external C_6H_5F (Table 2). The positive sign corresponds to an upfield shift with respect to the standard. It should be noted that the fluorine signals of

Table 2. ^{19}F chemical shifts for compounds **2, 3a, 6,** and **7** in $CHCl_3$

Compound	FCS, ppm		
	$(4\text{-}FC_6H_4)_3P$	$4\text{-}FC_6H_4$	$4\text{-}FC_6H_4S$
2b	−3.69	11.71	10.79
2c	−3.46	11.99	11.16
2d	−2.85	12.98	11.91
2e	−4.69	9.68	9.19
2f	−3.40	12.25	11.11
2g	−4.23	10.60	9.95
3a*	−4.13	10.85	10.12
6a*	−3.69	—	10.46
6b**	—	—	11.86
6c	—	—	12.65
6e	—	—	8.73
6f	—	—	12.30
6g	—	—	10.07
7b	−3.27	—	11.14
7c	−3.00	—	11.55
7d	−2.46	—	12.24
7e	−4.26	—	9.57
7f	−3.07	—	11.38
7g	−3.85	—	10.25

* The compound was described previously.[15]
** The compound was described previously.[14]

the $4\text{-}FC_6H_4$ and $4\text{-}FC_6H_4S$ ligands in compounds **2** are located in the similar regions that also contain the signals of compounds **3**, which complicates the assignment of the signals. Therefore, compounds **6** and **7** were generated in a solution by exchange reactions involving compounds **6a** and **1b**. The FCS for compounds **6** and **7** are given in Table 2.



Ar = Ph (**b**); $4\text{-}MeC_6H_4$ (**c**); $4\text{-}Me_2NC_6H_4$ (**d**); $3,4\text{-}Cl_2C_6H_3$ (**e**); $4\text{-}MeOC_6H_4$ (**f**); $4\text{-}ClC_6H_4$ (**g**).

Consideration of the data of Table 2 indicates that the natures of the Ar_3P ligands in compounds **2** have parallel effects on the shielding of fluorine in the $(4\text{-}FC_6H_4)_3P$-, $4\text{-}FC_6H_4$-, and $4\text{-}FC_6H_4S$-ligands. The electron-donating substituents increase the shielding of fluorine and the electron density on the ligands of all three types, and the electron-withdrawing substituents exert the opposite effect. This indicates that the *cis*- and *trans*-effects of the Ar_3P ligands run parallel and that they increase with an increase in the electron-donating ability of this type of ligands.

At the same time, according to theoretical estimates,[1,9] *cis*-effects of ligands may be either parallel or antiparallel to their *trans*-effects. In an experimental study of the *cis*- and *trans*-isomers of $[Bu_3P]LPtCl_2$ type complexes carried out by ^{31}P NMR using the $J(^{195}Pt,^{31}P)$ spin coupling constants for the bond of platinum with the Bu_3P ligand, it was found[3] that in the case of *trans*-isomers, *trans*-effects and the electron-donating abilities of the phosphorus-containing ligands increase in the sequence $(PhO)_3P < Ph_3P < Et_3P$, while in the case of *cis*-isomers, the *cis*-effects of these ligands and their phenomenological electron-donating abilities increase in the order $Et_3P < Ph_3P < (PhO)_3P$. However, in view of the data obtained in the present work and in view of the above-mentioned antiparallel effects of similar variations of the donating abilities of substituted triphenylphosphines on the $J(^{195}Pt,^{31}P)$ spin coupling constants in *cis*- and *trans*-isomers of $(Ar_3P)_2PtCl_2$, it is likely that the above-mentioned case of antiparallel *cis*- and *trans*-effects of the phosphorus-containing ligands is associated with the specific character of $J(^{195}Pt,^{31}P)$ spin coupling constants as an indicator, which may be due to the difference in their signs for *cis*- and *trans*-isomers or *cis*- and *trans*-systems of bonds, rather than to the antiparallel effects of the phosphorus-containing ligands on the electron densities at *cis*- and *trans*-ligands.

Let us consider now some quantitative characteristics of the *cis*- and *trans*-effects of the triarylphosphine ligands. Comparison of the FCS of free compound **1a** in $CHCl_3$, which is equal to −1.12 ppm, with the FCS of this ligand in compounds **2** implies that the coordination of phosphine **1a** to the platinum atom results in that the shielding of fluorine decreases and the FCS changes by 1.6—3.4 ppm. These data indicate that the coordination is accompanied by the dominating transfer of the electron density of the unshared electron pair of phosphine **1a** on the vacant orbital of platinum, and the degree of this transfer increases as the electron requirements of triarylphosphines in compounds **2** increase. In this connection, it might be assumed that the *cis*- and *trans*-effects of triarylphosphine would be mostly determined by the donor ability of its unshared electron pair, and that the value of pK_a or the ionization potential (IP) of the unshared electron pair can be used as a measure of this ability.

In fact, examination of Table 3 shows that the FCS in *cis*- and *trans*-ligands correlate well with the pK_a[10]

Table 3. Parameters of the correlation equations $y = \rho x + c$ for compounds **2**, **3**, **6**, and **7**

y		*x*	ρ±Δρ	*r*	*c*
Compound	L				
2	$P(C_6H_4F\text{-}4)_3$	$pK_a(Ar_3P)$	0.182±0.002	0.961	−4.31
2	$4\text{-}FC_6H_4$	$pK_a(Ar_3P)$	0.308±0.008	0.941	10.54
2	$4\text{-}FC_6H_4S$	$pK_a(Ar_3P)$	0.255±0.005	0.946	9.87
2	$P(C_6H_4F\text{-}4)_3$	$IP(Ar_3P)$	−1.08±0.03	0.982	4.71
2	$4\text{-}FC_6H_4$	$IP(Ar_3P)$	−1.84±0.16	0.968	25.93
2	$4\text{-}FC_6H_4S$	$IP(Ar_3P)$	−1.52±0.08	0.971	22.55
2	$P(C_6H_4F\text{-}4)_3$	$\sigma^0(Ar)$	−1.98±0.02	0.997	−3.73
2	$P(C_6H_4F\text{-}4)_3$	σ(Ar)	−1.33±0.12	0.955	−3.84
2	$4\text{-}FC_6H_4$	$\sigma^0(Ar)$	−3.42±0.13	0.992	11.54
2	$4\text{-}FC_6H_4$	σ(Ar)	−2.25±0.50	0.935	11.36
2	$4\text{-}FC_6H_4S$	$\sigma^0(Ar)$	−2.82±0.05	0.995	10.69
2	$4\text{-}FC_6H_4S$	σ(Ar)	−1.86±0.31	0.940	10.54
2	$4\text{-}FC_6H_4$	FCS **2**, $P(C_6H_4F\text{-}4)_3$	1.81±0.01	0.996	18.26
2	$4\text{-}FC_6H_4S$	FCS **2**, $P(C_6H_4F\text{-}4)_3$	1.47±0.01	0.998	16.18
3	$4\text{-}FC_6H_4$	FCS **2**, $(4\text{-}FC_6H_4)$	1.84±0.03	0.992	−8.96
3	$4\text{-}FC_6H_4S$	FCS **2**, $(4\text{-}FC_6H_4S)$	1.93±0.01	0.998	−9.26
6	$4\text{-}FC_6H_4S$	FCS **7**, $(4\text{-}FC_6H_4S)$	1.98±0.01	0.999	−10.26

Note: *y* is the chemical shift of fluorine (FCS) in the ligand L, Δρ is the error ρ for a 95% confidence level, and *r* is the correlation coefficient.

and IP[11] values of triarylphosphines. The correlation with IP proves to be somewhat better than that with pK_a. This may indicate the fact that the IP is a better characteristics of the electron-donating ability of a substituted triphenylphosphine than the value of pK_a.

Another approach to the evaluation of the *cis*- and *trans*-effects of triarylphosphines was based on the use of polar constants of substituents. For this purpose, the σ Hammett constants and the σ^0 Taft constants were chosen.[12] As follows from Table 3, the FCS in the *cis*- and *trans*-ligands really exhibit good correlations with the σ and σ^0 values and this correlation is appreciably better in the latter case. These data indicate that the variation of the *cis*- and *trans*-effects of substituted triphenylphosphines is best described by the inductive effects of the aryl groups.

Furthermore, the correlation between the FCS of the $4\text{-}FC_6H_4$ and $4\text{-}FC_6H_4S$ ligands and the FCS of the $(4\text{-}FC_6H_4)_3P$ ligand shows that the effects of triarylphosphine ligands on the shielding of fluorine in the former two cases are substantially greater than that in the latter case. This is governed by the distinctions in the direction in which the electron effects of the triarylphosphine ligands are transferred, in the nature of the transferring chain, and in the number of indicator 4-fluorophenyl groups. Finally, the correlation of the FCS in the $4\text{-}FC_6H_4$ and $4\text{-}FC_6H_4S$ ligands for the series of compounds **2** and **3** and also **5** and **6** (Table 3) indicates that the *cis*-effect of triarylphosphine ligands is additive and exhibits no saturation.

Thus, the data obtained in the present work imply that the use of the ^{19}F NMR method with an indicator 4-fluorophenyl group is a highly efficient technique for investigating the *cis*- and *trans*- effects of neutral ligands in organometallic compounds of bivalent platinum.

Experimental

^{31}P and ^{19}F NMR spectra were recorded on a Bruker WP-200 SY spectrometer operating at 81.03 MHz (^{31}P) or 188.3 MHz (^{19}F) at 25 °C for 0.1 *M* solutions in $CHCl_3$. The resonance conditions for the ^{31}P NMR spectra were stabilized using the signal of D_2O, placed between the wall of the ampule (10 mm) and the wall of the inserted tube (8 mm) containing the solution of a sample. The values of $\delta^{31}P$ were measured by the method of substitution with respect to external 85% H_3PO_4 and are presented without corrections for the bulk magnetic susceptibility. The sign "plus" corresponds to a downfield shift of the signal. The errors in determining $\delta^{31}P$ did not exceed 0.1 ppm, and those in determining $J(^{195}Pt,^{31}P)$ were no more than ±2 Hz.

The FCS values were measured by the method of substitution relative to a solution of external fluorobenzene in the same solvent and at the same concentration, as the compound studied. The sign "plus" corresponds to an upfield shift of the signal. The errors in determining FCS did not exceed ±0.01 ppm.

$CHCl_3$ was purified by the standard procedure and distilled in an argon flow. To generate **2** by the exchange reactions of **3a** with **3b—e** or **1f—g**, $CHCl_3$ solutions of equimolar amounts of reactants were mixed and kept at ~20 °C until equilibrium was achieved. Compounds **6c,e—g** and **7b—g** were generated in a similar way. Known compounds **1** were prepared by reported procedures[10] and identified by melting points. The purity of these compounds was checked by ^{31}P NMR spectra. The synthesis of the previously unknown compound **1e** is described below. Compound **5** was synthesized by arylation of $cis\text{-}(C_8H_{12})PtCl_2$ by the action of $Me_3SnC_6H_4F\text{-}4$ according to the known procedure.[13] Compounds **3a—b** and **6a—b** were

Table 4. Characteristics of compounds **3** and **4**

Compound	Yield (%)	M.p. °C	Molecular formula	Found / Calculated (%)	
				C	H
3c	50	242—246	$C_{54}H_{50}F_2P_2PtS$	63.21 63.13	4.91 4.93
3d	40	265—266	$C_{60}H_{68}F_2N_6P_2PtS$	60.04 59.95	5.71 5.59
3e	40	253—254	$C_{48}H_{26}Cl_{12}F_2P_2PtS$	42.52 42.12	1.93 2.08
4c	70	242	$C_{48}H_{46}ClFP_2Pt$	61.63 61.83	4.96 5.03
4d	60	266—268	$C_{54}H_{64}ClN_6P_2Pt$	58.50 58.01	5.81 5.73
4e	60	220—224	$C_{42}H_{22}Cl_{13}FP_2Pt$	39.90 39.71	1.75 1.72

prepared previously.[14,15] Below we present typical examples of the synthesis of compounds **3** and **4**. All of the reactions were carried out under dry argon.

Synthesis of *trans*-4-fluorophenyl-*bis*[tri(4-tolyl)phosphine]-platinum chloride (4c). A mixture of compound **5** (0.9 g, 2 mmol) and compound **1c** (1.2 g, 4 mmol) in 50 mL of anhydrous benzene was stirred at 80 °C for 2 h. The solvent was removed *in vacuo*, and the residue was washed with ether, dissolved in warm benzene, and filtered. Hexane was added to the filtrate until it became turbid, and the mixture was allowed to stand in a refrigerator for 12 h. The precipitate was filtered off and dried *in vacuo* at 50 °C for 5 h to give 1.1 g (70 %) of a colorless crystalline material.

Synthesis of *trans*-4-fluorophenyl-*bis*[tri(4-tolyl)phosphine]-platinum 4-fluorothiophenoxide (3c). A solution of $NaSC_6H_4F$-4 in a mixture of 30 mL of anhydrous benzene and 5 mL of anhydrous ethanol prepared from HSC_6H_4F-4 (0.4 g, 2.2 mmol) and Na (0.07 g) in 5 mL of anhydrous ethanol was added to a warm solution of **4c** (0.93 g, 1.1 mmol) in 100 mL of anhydrous benzene. The reaction mixture became transparent and turned yellow. Then it was heated to 50 °C for 2 h. The precipitate of NaCl was filtered off, the solvent was removed *in vacuo* and the residue was dissolved in anhydrous benzene and filtered. Hexane was added to the filtrate, until it became turbid, and the mixture was allowed to stand for 12 h. The resulting precipitate was filtered off and again reprecipitated from benzene with hexane to give 0.65 g (50 %) of a light yellow crystalline material. The yields, melting points, and analysis data of compounds **3** and **4** are given in Table 4.

Synthesis of tri(3,4-dichlorophenyl)phosphine (1e). At 0 °C, a solution of PCl_3 (4.73 g, 34 mmol) in 50 mL of anhydrous ether was added to a stirred solution of the Grignard reagent prepared from of 3,4-$Cl_2C_6H_3Br$ (23.5 g, 104 mmol) and Mg (2.5 g) in 250 mL of anhydrous ether. Then the reaction mixture was boiled for 1.5 h, cooled, and decomposed by an aqueous solution of NH_4Cl. The organic layer was separated and dried with Na_2SO_4, and the solvent was evaporated to give 28.1 g (60 %) of light-yellow crystals, which were purified by crystallization from ethanol. Crystallization gave 3.5 g of colorless crystals, m.p. 125 °C. Found (%): C, 46.38; H, 2.44; P, 6.20. $C_{18}H_9Cl_6P$. Calculated (%): C, 46.07; H, 1.93; P, 6.60. ^{31}P NMR ($CHCl_3$), δ: −6.60.

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