ELSEVIER

Journal of Organometallic Chemistry 529 (1997) 171–176

Journal of Organometallic Chemistry

Resorcinol bis(cyclophosphites)

Eduard E. Nifantyev [a,*], Elena N. Rasadkina [a], Larysa K. Vasyanina [a], Vitaliy K. Belsky [b], Adam I. Stash [b]

[a] *Moscow Pedagogical State University, per. Nesvizhskii 3, 119882 Moscow, Russia*
[b] *Karpov Institute of Physical Chemistry, 103064 Moscow, Russia*

Received 4 April 1996; revised 5 July 1996

Abstract

First representatives of resorcinol bis(cyclophosphites) were synthesized, on the basis of which a series of metallocomplexes, including a 16-membered binuclear metallocycle, was obtained. The fact that, in this system, two *m*-phenylene cycles are almost parallel with each other makes possible the creation of complex polynuclear constructions. The phosphocyclane rings in resorcinol bis(cyclophosphites) are readily opened by bromine and chloramine to form bis-(ω-halogenoallylphosphates) of resorcinol.

Keywords: Resorcinol; Phosphorylation; Biscycloalkylenephosphites; Metallocomplexes; X-ray diffraction analysis

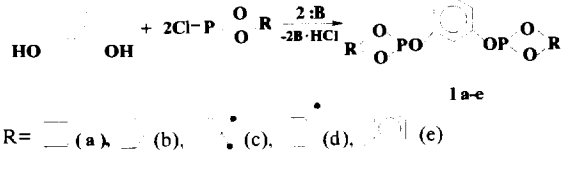
1. Introduction

Molecular systems involving two atoms of trivalent phosphorus are in considerable use for the purpose of fine organic synthesis, coordination chemistry and metallocomplex catalysis. Meanwhile, the circle of compounds of this type is rather narrow. Essentially diphosphines are well known, in which the phosphorus atoms are bonded together by two to four covalent bonds. Diphosphites and aminophosphites, which are available more readily and are convenient in operation, have seldom attracted attention so far. The reason appears to be their capacity for disproportionation: we may mention a very easy transformation of tetraalkylesters of pyrocatecholdiphosphorus acid into pyrocatecholcyclophosphites and trialkyl phosphites [1].

Recently it was clearly demonstrated that diphosphites and aminophosphites can be efficiently stabilized by incorporating phosphocyclic fragments (dioxophospholane and phosphorinane). Thus, cyclic phosphites of pyrocatechol, in contrast to the above mentioned acyclic phosphines, are quite stable; they endure vacuum distillation and prolonged storage [2]. Therefore, the use of cyclic diesters is very promising. It should be remembered that cyclic diphosphites are distinguished by stereochemical and other features, which make them particularly valuable subjects of investigation.

2. Results and discussion

In view of the foregoing, the goal of this work was to study the synthesis of hitherto unknown resorcinol bicyclophosphites [1] and to review the prospects for their application to modern branches of organophosphorus chemistry. We showed that the interaction of resorcinol with acid chlorides and amides of 1,2- and 1,3-alkylenephosphorus acids results in diphosphites (**Ia–e**) readily and with near-quantitative yield:



On evidence derived from thin-layer chromatography

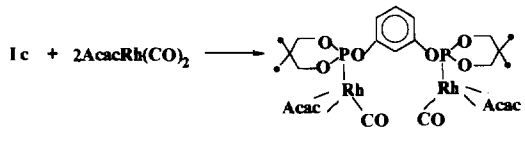
[1] We have reported several data on the chemistry of these compounds in a previous communication [3].

* Corresponding author.

PII S0022-328X(96)06657-0

and ^{1}H and ^{31}P NMR spectroscopy, the products obtained are practically free from impurities and, therefore, can be applied in most subsequent reactions without additional purification. The structure of these compounds as bicyclophosphorylated structures follows from their NMR data and elemental analysis (see Section 3). Attention is drawn to the fact that the ^{31}P NMR spectrum of asymmetric biscyclophosphite **Id** remains unchanged over the temperature range 30–80 °C and exhibits two resonance signals (122 and 125 ppm) corresponding to two geometric forms at equilibrium. At the same time only one form of the corresponding asymmetric diphosphites of hydroquinone [4] and pyrocatechol [2], which are also generated in two forms, is stable at an elevated temperature.

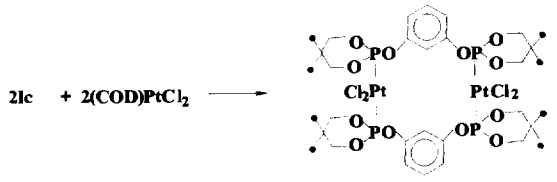
The phosphites obtained readily coordinate to many transition metals. Thus, ester **Ic** interacts with acetylacetonate rhodium(I)dicarbonyl in a 1:2 molar ratio in dichloromethane with the formation of the binuclear complex **II**. The data of thin-layer chromatography and NMR spectroscopy prove the individuality of this complex. Its ^{31}P NMR spectrum exhibits only one doublet with a coupling constant of 289.8 Hz, which is indicative of the direct coordination of phosphorus to rhodium. The presence of a band at 2000 cm^{-1} in the IR spectrum suggests the single substitution of a carbonyl group [5]. The NMR spectrum exhibits the signals of all due protons in corresponding proportions. Summing the foregoing, we can conclude that the complexation occurs as follows:



II

For the purpose of obtaining a phosphometallocycle, we studied the interaction of equimolar amounts of complex **II** and the diphosphite **Ic**. Unfortunately, we failed to substitute the remaining carbonyl groups in the ligand of complex **II**, which appears to be related to a decrease in mobility of these carbonyls because of the *cis*-symbiosis effect [6]. Note that an analogous conclusion has been drawn for a related system [7].

Further efforts to create a phosphometallic system were made on the basis of bivalent platinum. If treated with an equimolar quantity of platinum(II) cyclooctadiene chloride in dichloromethane, ligand **Ic** gives complex **III** of the following composition:



III

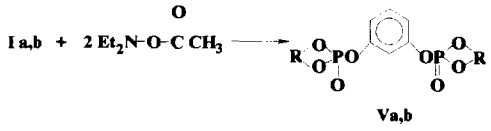
The ^{31}P NMR spectrum of this complex exhibits a signal at δ_P 62.7 ppm (^{1}J(P,Pt) 5815.8 Hz), which corresponds to the *cis*-position of substituents relative to the platinum atom [8]. An X-ray diffraction study proved the dimeric structure of the 16-membered binuclear metallocycle **III** (Fig. 1) and revealed its important structural peculiarities (Table 1). We call attention to the fact that two *m*-phenylene groups are almost parallel with each other (interplanar angle 18.6°, distance between the planes 3.96 Å). This presents an infrequent spatial organization of macrometallocycles. It is important that such a mutual arrangement of two benzene nuclei can lead to the creation of unique supramolecular systems at the expense of π-complexing (see, for example, Ref. [9]).

The resorcinol bis(cyclophosphites) studied are moderately electron-donating species. They easily add sulfur, transforming into the corresponding bisthiophosphoryl derivatives (**IVb–d**):

IVb-d

An exception is provided by the diphosphites **Ia** and **Id**, which are unreactive towards elemental sulfur and disulfides.

At the same time diphosphites are difficult to oxidize by common reagents. Therefore we developed a new, suitable method of oxidation, using *N,N*-diethyl-*O*-acetylhydroxylamine [10]:



Va,b

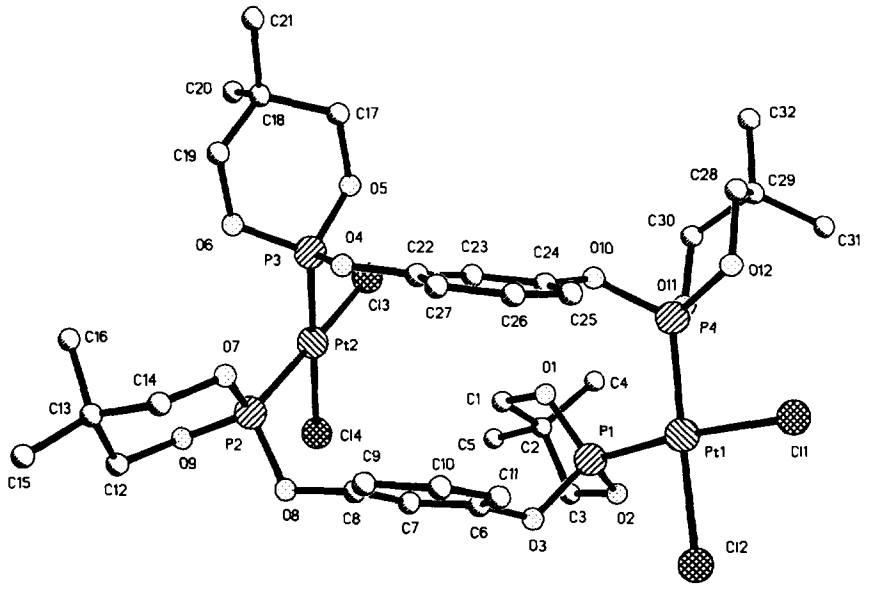
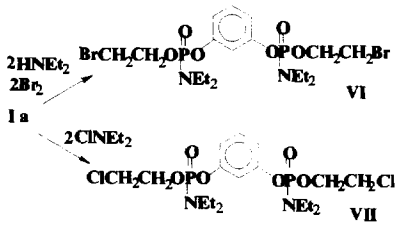


Fig. 1. Molecular structure of complex **III**.

Resorcinol bicyclophosphites very readily react with bromine and chloramine, which appear to react homolytically, for example:



The bis-β-haloethylphosphates of resorcinol then obtained offer promise as bioregulators as well as reagents for the design of phosphorus-containing crown ethers and other complex systems, which easily realize the conformations with the parallel disposition of arylene radicals.

3. Experimental details

All syntheses were carried out under dry argon atmosphere with the use of dry solvents.

Monitoring of reactions and identification of products were by TLC on Silufol plates in benzene–dioxane 3:1 (A) and chloroform–methanol 3:1 (B) systems.

The ^{1}H NMR spectra of compounds **Ia–d**, **II**, **IVb–d**, **Va–b**, **VI** and **VII** in $CDCl_3$ were recorded on a Bruker AM-400 instrument (at 400 MHz). The ^{31}P NMR spectra of **Ia–d** in C_6H_6, **II**, **III**, **IVb–d**, **Va–b**, **VI** and **VII** in CH_2Cl_2 were recorded on a Bruker WP-80SY instrument at 32.4 MHz against 85% H_3PO_4. IR spectra were recorded on a Specord 75 IR instrument in the range 4000–400 cm^{-1} in CH_2Cl_2 solution in NaCl cells.

Table 1
Some bond lengths [Å] and angles [deg] for **III**

Pt(1)–P(4)	2.188(3)	P(4)Pt(1)P(1)	93.20(12)
Pt(1)–P(1)	2.198(3)	P(4)Pt(1)Cl(1)	91.57(14)
Pt(1)–Cl(1)	2.321(4)	P(1)Pt(1)Cl(4)	174.47(14)
Pt(1)–Cl(2)	2.331(4)	P(4)Pt(1)Cl(2)	176.92(13)
Pt(2)–P(3)	2.188(4)	P(1)Pt(1)Cl(2)	86.57(13)
Pt(2)–P(2)	2.197(3)	Cl(1)Pt(1)Cl(2)	88.5(2)
Pt(2)–Cl(3)	2.322(3)	P(3)Pt(2)P(2)	92.87(12)
Pt(2)–Cl(4)	2.346(4)	P(3)Pt(2)Cl(3)	91.26(14)
P(1)–O(2)	1.552(9)	P(2)Pt(2)Cl(3)	174.23(14)
P(1)–O(1)	1.562(8)	P(3)Pt(2)Cl(4)	179.17(12)
P(1)–O(3)	1.598(8)	P(2)Pt(2)Cl(4)	87.14(12)
O(1)–C(1)	1.480(14)	Cl(3)Pt(2)Cl(4)	88.79(14)
O(2)–C(3)	1.460(2)	O(2)P(1)O(1)	106.2(4)
O(3)–C(6)	1.416(14)	O(2)P(1)O(3)	100.7(5)
C(1)–C(2)	1.490(2)	O(1)P(1)O(3)	105.2(4)
C(2)–C(4)	1.490(2)	O(2)P(1)Pt(1)	112.0(3)
C(2)–C(3)	1.500(2)	O(1)P(1)Pt(1)	114.5(3)
C(2)–C(5)	1.560(2)	O(3)P(1)Pt(1)	116.9(3)
		C(1)O(1)P(1)	119.1(8)
		C(3)O(2)P(1)	117.9(8)
		C(6)O(3)P(1)	121.0(7)
		O(1)C(1)C(2)	111.2(10)
		C(1)C(2)C(4)	112.6(11)
		C(1)C(2)C(3)	108.0(10)
		C(4)C(2)C(3)	110.3(11)
		C(1)C(2)C(5)	107.2(11)
		C(4)C(2)C(5)	111.0(11)
		C(3)C(2)C(5)	107.6(11)
		O(2)C(3)C(2)	113.0(10)

Table 2
Crystal data and structure refinement for complex **III**

Empirical formula	$C_{32}H_{48}C_{14}O_{12}P_4Pt_2$
Formula weight	648.28
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	$a = 10.228(6)$ Å, $\alpha = 103.78(2)°$
	$b = 13.586(2)$ Å, $\beta = 91.98(3)°$
	$c = 19.361(4)$ Å $\gamma = 92.79(4)°$
Volume	2607(2) Å^3
Z	4
Density (calc.)	1.652 g cm^{-3}
Absorption coefficient	5.737 mm^{-1}
$F(000)$	1256
Crystal size	0.22 × 0.19 × 0.14 mm^3
θ range for date collection	2.00 to 22.47°
Index ranges	$-10 \le h \le 0$, $-14 \le k \le 14$,
	$-20 \le l \le 20$
Reflections collected	3585
Independent reflections	3585 [R(int) = 0.0000]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3585/0/499
Goodness-of-fit on F^2	1.186
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0285$, $wR_2 = 0.0824$
R indices (all data)	$R_1 = 0.0285$, $wR_2 = 0.0824$
Largest diff. peak and hole	1.325 and −0.504 e Å^{-3}

X-ray diffraction data of complex **III** were collected on an automatic Enraf–Nonius CAD-4 diffractometer, using Mo Kα radiation, $\theta/2\theta$ scan technique. Crystal data and structure refinement are as in Table 2.

3.1. Resorcinol biscycloalkylenephosphites

Obtained by the phosphorylation of resorcinol with two moles of alkylenechlorophosphites. A solution of 4.5 mmol of resorcinol and 9.1 mmol of triethylamine in 5 ml of dioxane was added dropwise to a solution of 9.1 mmol of alkylenechlorophosphite in 10 ml of benzene with stirring and cooling to 5 °C. The reaction mixture was stirred for 2 h. The precipitate was filtered off; the solvent was removed in vacuo, and the residue was dried for 2 h (30 °C, 1 mm Hg). Yields were near-quantitative.

3.1.1. Bis(1,3,2-dioxaphospholanyl-2)resorcinol (*Ia*)

Viscous oil. $R_f = 0.9$ (A). ^{1}H NMR spectrum (δ, ppm): 3.32 m (4H, $C^{4,5}H^e$), 3.64 m (4H, $C^{4,5}H^a$), 6.84 m, 7.05 m (4H, CH); δ(P) 128.53. Anal. Found: C, 41.15; H, 4.10; P, 21.28. $C_{10}H_{12}O_6P_2$ Calc.: C, 41.36; H, 4.17; P, 21.35%.

3.1.2. Bis(1,3,2-dioxaphosphorinyl-2) resorcinol (*Ib*)

M.p. 45–46 °C. $R_f = 0.9$ (A). ^{1}H NMR spectrum (δ, ppm): 1.93 m (2H, C^5H^e), 2.45 m (2H, C^5H^a), 4.5 m (2H, $C^{4,6}H^a$), 4.63 m (4H, $C^{4,6}H^a$), 7.11 m, 7.37 m (4H, CH); δ(P) 123.31. Anal. Found: C, 45.01; H, 4.95; P, 19.26. $C_{12}H_{16}O_6P_2$ Calc.: C, 45.30; H, 5.07; P, 19.47%.

3.1.3. Bis(5,5-dimethyl-1,3,2-dioxaphosphorinyl-2) resorcinol (*Ic*)

M.p. 51–52 °C. $R_f = 0.9$ (A). ^{1}H NMR spectrum (δ, ppm): 0.68 s, 1.19 s (12H, CH_3), 3.34 m (4H, $C^{4,6}H^e$), 4.18 m (4H, $C^{4,6}H^a$), 6.72 m, 7.10 m (4H, CH); δ(P) 115.45. Anal. Found: C, 51.34; H, 6.37; P, 16.5. $C_{16}H_{24}O_6P_2$ Calc.: C, 51.33; H, 6.46; P, 16.55%.

3.1.4. Bis(4-methyl-1,3,2-dioxaphosphorinyl-1) resorcinol (*Id*)

Obtained as a mixture of two isomeric forms. δ(P): 121.58, 124.36 ppm. Anal. Found: C, 48.44; H, 5.71; P, 17.51. $C_{14}H_{20}O_6P_2$ Calc.: C, 48.56; H, 5.82; P, 17.89%.

3.1.5. Bis(1,3,2-dioxa-4,5-benzophospholanyl-2) resorcinol (*Ie*)

Viscous oil. $R_f = 0.85$ (A). δ(P): 127.49 ppm. Anal. Found: C, 55.90; H, 3.08; P, 16.07. $C_{18}H_{12}O_6P_2$ Calc.: C, 55.97; H, 3.13; P, 16.03%.

3.1.6. μ-[Bis(5,5-dimethyl-1,3,2-dioxaphosphorinyl-2)resorcinol]– bis[acetylacetonatocarbonylrhodium(I)] (*II*)

A solution of 0.26 mmol of $AcacRh(CO)_2$ in 2 ml of dichloromethane was added at once to 0.13 mmol of diphosphite **Ic** in 5 ml of dichloromethane and stirred for 1 h. The solvent was stripped off in vacuo; the residue was washed with hexane, dissolved in dichloromethane and evaporated again. Complex **II** was obtained as yellow transparent plates. Yield 91%, m.p. 98–99 °C. $R_f = 0.8$ (A). ^{1}H NMR spectrum (δ, ppm): 0.82 s, 1.35 s (12R, CH_3), 1.78 s, 2.0 s (12H, CH_3 in Acac), 3.72 m (4H, $C^{4,6}H^e$ in dioxaphosphorinane), 4.34 m (4H, $C^{4,6}H^a$), 5.44 s (2H, CH in Acac), 7.11 m, 7.27 m, 7.35 m (4H, CH). ^{31}P NMR spectrum (δ, ppm): 118.98 d, ^1J(PRh) 284.83 Hz. IR spectrum: ν(CO–Rh) 2000 cm^{-1}. Anal. Found: C, 40.52; H, 4.57; P, 7.86; Rh, 23.68. $C_{28}H_{38}O_{12}P_2Rh_2$ Calc.: C, 40.30; H, 4.59; P, 7.42; Rh, 24.69%.

3.1.7. Di-μ-[bis(5,5-dimethyl-1,3,2-dioxaphosphorinyl-2)resorcinol]– bis[dichloroplatinum(II)] (*III*)

A solution of 0.13 mmol of $Pt(COD)Cl_2$ in 1.5 ml of dichloromethane was added at one time to a solution of 0.13 mmol of diphosphite **Ic** in 4 ml of dichloromethane with stirring. The reaction mixture was stirred for 0.5 h. The solvent was evaporated in vacuo (25 mm Hg); the residue was washed with benzene and hexane and dried for 1 h (50 °C, 1 mm Hg). Transparent crystals were formed from acetone. Yield 95.6%, m.p. 309–310 °C with dec. $R_f = 0.7$ (B). δ(P): 62.72 ppm, ^1J(PtP)

5815.78 Hz. Anal. Found: P, 11.21; Pt, 35.86. $C_{32}H_{48}Cl_4O_{12}P_4Pt_2$ Calc.: P, 11.37; Pt, 35.85%.

3.2. Biscycloalkylenethiophosphates of resorcinol

Obtained according to the following general procedure: 7.7 mmol of sulfur was added to a solution of 3.9 mmol of diphosphite **Ib–d** in 7 ml of benzene, and the mixture was refluxed for 2 h. The solvent was removed in vacuo (20 mm Hg), and the residue was chromatographed on a column packed with silica gel; the thiophosphates **IVb–d** were eluted with benzene. Yields 70–80%.

3.2.1. Bis(2-thio-1,3,2-dioxaphosphorinyl-2) resorcinol (IVb)

M.p. 124–125 °C. $R_f = 0.63$ (A). ^{1}H NMR spectrum (δ, ppm): 1.84 m (2H, C^5U^e), 2.41 m (2H, C^5H^a), 4.46 m (4H, $C^{4,6}H^e$), 4.60 m (4H, $C^{4,6}H^a$), 7.11 m, 7.37 m (4H, CH); δ(P) 55.81. Anal. Found: C, 37.51; H, 4.12; P, 14.12. $C_{12}H_{16}O_6P_2S_2$ Calc.: C, 37.69; R, 4.21; P, 16.20%.

3.2.2. Bis(2-thio-5,5-dimethyl-1,3,2-dioxaphosphorinyl-2) resorcinol (IVc)

M.p. 140–141 °C. $R_f = 0.66$ (A). ^{1}H NMR spectrum (δ, ppm): 0.91 s, 1.32 s (12H, CH_3), 4.0 m (4H, $C^{4,6}H^e$), 4.3 m (4H, $C^{4,6}H^a$), 7.07 m, 7.09 m, 7.33 m (4R, CH); δ(P) 54.33. Anal. Found: C, 43.68; H, 5.41; P, 14.12. $C_{16}H_{24}O_6P_2S_2$ Calc.: C, 43.83; H, 5.52; P, 14.12%.

3.2.3. Bis(2-thio-4-methyl-1,3,2-dioxaphosphorinyl-2) resorcinol (IVd)

M.p. 136–137 °C. $R_f = 0.65$. ^{1}H NMR spectrum (δ, ppm): 0.91 m (2H, C^5H^e), 0.97 d (6H, CH_3), 1.34 m (2H, C^5H^e, ^2J(HH) 10.3 Hz, ^3J(PH) 20.6 Hz), 3.77 m (4H, C^6H^a), 6.88 m, 7.01 m, 7.45 m (4H, CH); δ(P) 58.42. Anal. Found: C, 40.89; H, 4.85; P, 15.01. $C_{14}H_{20}O_6P_2S_2$ Calc.: C, 41.00; H, 4.91; P, 15.10%.

3.3. Biscycloalkylenephosphates of resorcinol (Va–b)

Obtained according to the following general procedure: 1 mmol of *N*-chloro-diethylamine [11] was added to 2 mmol of sodium acetate in 10 ml of dry dioxane at room temperature and stirred for 0.5 h. The reaction mixture was cooled to 0 °C, and a solution of 1 mmol of diphosphite (**Ia–b**) in 3 ml of dioxane was added. After stirring for 1.5 h, the mixture was heated to room temperature; acetamide precipitated from the solution and an excess of sodium acetate were filtered; the solution was evaporated to a minimum, and hexane was added. The precipitate of oxodicyclophosphates formed was filtered off and dried in vacuo. Yields up to 70%.

3.3.1. Bis(2-oxo-1,3,2-dioxaphospholanyl-2) resorcinol (Va)

M.p. 130–131 °C. $R_f = 0.72$ (B). ^{1}H NMR spectrum (δ, ppm): 4.25 m (4H, $C^{4,6}$ H^e), 4.45 m (4H, $C^{4,6}H^a$), 6.85 m, 7.02 m (4H, CH); δ(P) 11.73. Anal. Found: C, 37.20; H, 3.69; P, 19.24. $C_{10}H_{12}O_8P_2$ Calc.: C, 37.20; H, 3.75; P, 19.23%.

3.3.2. Bis(2-oxo-1,3,2-dioxaphosphorinyl-2) resorcinol (Vb)

M.p. 148–149 °C. $R_f = 0.76$ (B). ^{1}H NMR spectrum (δ, ppm): 2.05 m (2H, C^5H^e), 2.43 m (2H, C^5H^a), 4.6 m (4H, $C^{4,6}H^e$), 4.81 m (4H, $C^{4,6}H^a$), 6.81 m, 7.10 m (4H, CH); δ(P) −13.17. Anal. Found: C, 41.10; H, 4.58; P, 17.70. $C_{12}H_{16}O_8P_2$ Calc.: C, 41.13; H, 4.65; P, 17.68%.

3.3.3. Bis(2-bromoethyl-N-diethylamidophosphate) resorcinol (VI)

A solution of bromine (2.8 mmol) in 5 ml of dichloromethane was added to a solution of diphosphite **Ia** (1.4 mmol) in 3 ml of dry dichloromethane with stirring and cooling to 0 °C. The mixture was stirred at room temperature for 1 h. The ^{31}P NMR spectrum of the reaction mixture exhibited a singlet at −13.17 ppm typical for bromophosphates. Diethylamine (5.6 mmol) in 3 ml of dichloromethane was added to the reaction mixture, and the mixture was stirred for 15 min. The solvent was removed in vacuo, and the residue was purified by column chromatography on silica gel, eluting the amidophosphate with chloroform. The compound was isolated as an oil. Yield 60%. $R_f = 0.74$ (B). ^{1}H NMR spectrum (δ, ppm): 1.05 t (12H, CH_3), 3.13 m (8H, CH_2N, ^3J(PH) 12.1 Hz), 3.51 t (4H, CH_2Br), 4.29 m (4H, CH_2OP, ^3J(PH) 8.1 Hz), 7.15 m (4H, CH); δ(P) 5.64. Anal. Found: C, 36.30; H, 5.31; P, 10.40. $C_{18}H_{32}Br_2N_2O_6P_2$ Calc.: C, 36.41; H, 5.43; P, 10.43%.

3.3.4. Bis(2-chloroethyl-N-diethylamidophosphate) resorcinol (VII)

N-Chlorodiethylamine (4.0 mmol) was added to a solution of 2.0 mmol of diphosphite **Ia** in 4 ml of dry dichloromethane at 0 °C with stirring for 15 min. The compound was isolated analogously to **VI** and obtained as an oil. Yield 70%. $n_D^{20} = 1.517$, $R_f = 0.8$ (B). ^{1}H NMR spectrum (δ, ppm): 1.09 t (12H, CH_3), 3.15 m (8H, CH_2N, ^3J(PH) 11.96 Hz), 3.73 t (4H, CH_2Cl), 4.3 m (4H, CH_2OP, ^3J(PH) 7.82 Hz), 7.11 m (4H, CH); δ(P) 5.82. Anal. Found: C, 42.70; H, 6.31; P, 12.25. $C_{18}H_{32}C_{12}N_2O_6P_2$ Calc.: C, 42.81; H, 6.38; P, 12.27%.

4. Supplementary material available

Atomic coordinates and equivalent isotropic displacement parameters, anisotropic displacements, bond

lengths and angles, and observed and calculated structure factors (Tables S1–S4 respectively) (13 pages). Ordering information is given on any current masthead page.

Acknowledgements

The support of this research by the foundation "Russian Universities" and Russian Foundation for Basic Research (Grant 95-03-09030) is gratefully acknowledged.

References

[1] A.N. Pudovik and I.M. Adzhaeva, *Zh. Obshch. Khim.*, *32* (1962) 2005.
[2] E.N. Rasadkina, N.S. Magomedova, V.K. Belsky and E.E. Nifantyev, *Zh. Obshch. Khim.*, *65* (1995) 214.
[3] E.E. Nifantyev, E.N. Rasadkina, V.K. Belsky and A.I. Stash, *Dokl. RAN*, *341* (1995) 491.
[4] E.E. Nifantyev, E.N. Rasadkina, E.V. Ronkova, A.R. Bekker, N.S. Magomedova and V.K. Belsky, *Zh. Obshch. Khim.*, *65* (1995) 1448.
[5] A. Maisonnat, P. Kalk and R. Poilblanc, *Inorg. Chem.*, *13* (1974) 661.
[6] A.A. Grinberg, M.M. Singkh and Yu.S. Varshavskii, *Zh. Neorg. Khim.*, *13* (1968) 2716.
[7] E.E. Nifantyev, A.T. Teleshev and T.A. Shikovets, *Zh. Obshch. Khim.*, *56* (1986) 298.
[8] S.D. Pastor and A. Togni, *Inorg. Chim. Acta*, *159* (1989) 3.
[9] F. Inokuchi and S. Shinkai, *3rd Int. Calixarene Conf., 21–25 May 1995, Fort Worth, TX, USA*, Abstr. P-17, p. 73.
[10] E.N. Rasadkina, E.V. Ronkova, L.K. Vasyanina and E.E. Nifantyev, *Zh. Obshch. Khim.*, *65* (1995) 1951.
[11] H. Bock and K.L. Kampa, *Chem. Ber.*, *99* (1966) 1347.