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Reaction of 2-Ethyl-1,3,2-bezoxaphospholine with Calix[4]resorcinarenes

S. A. Terent'eva, I. L. Nikolaeva, A. R. Burilov, D. I. Kharitonov, E. V. Popova, M. A. Pudovik, I. A. Litvinov, A. T. Gubaidullin, A. I. Konovalov

Arbuzov Institute of Organic and Physical Chemistry, Kazan Scientific Center, Russian Academy of Sciences, Kazan, Tatarstan, Russia

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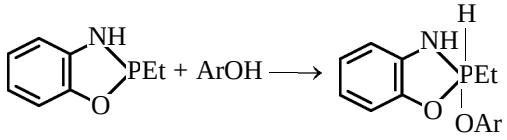
Abstract — By reaction of calix[4]resorcinarene with 2-ethyl-1,3,2-benzoxazaphospholine in 1 : 1 ratio we prepared a cavitand possessing one dioxaphosphocinic fragment on the upper circle of the molecule. Reaction of the same compounds in ether solution in 1 : 4 and 1 : 8 ratio according to the data of spectral methods leads initially to formation of addition product, the P—H phosphorane with composition 1 : 4. This compound is not stable and on keeping forms 2-oxo-2-ethyl-1,3,2-benzoxazaphospholine, spirophosphorane, *o*-aminophenol and phosphorylated calixarene. In the reaction of the aminoalkylated calixarene with 2-ethyl-1,3,2-benzoxazaphospholine we isolated a compound possessing four hydrophoshonyl fragments on the upper circle of the calixarene matrix.

Calixarenes and their derivatives with calyx-like form that capable of bonding both organic molecules and ions [1–4] attract enhanced attention of explorers. The calix[4]resorcinarenes can be easily modified by phosphorylation of their hydroxy groups located on the upper circle of the molecule. As the phosphorylating agents were mainly used tetracordinated phosphorus acid chlorides; the complete phosphates (phosphonates and phosphinates) such obtained as a rule do not capable of further transformations [5]. Further development of the chemistry of *O*-phosphorylated calixarenes, in our opinion, is connected with insertion to the molecules of reactive phosphorus-containing groups including those with a P–H bond. Involving of such objects to chemical transformations can result in formation of complexing reagents which contain simultaneously a macro ring (formation of *guest–host* type complexes) and chelating fragments, as well as new container-type compounds with unusual properties and so on.

Recently we first prepared a calixarene possessing four hydrophosphoryl fragments on the upper circle of the molecule [6]. The current investigation is aimed at the attempted synthesis of earlier unknown P–H-phosphoranes on a calixarene matrix. Earlier [7] we showed that at 20°C compound **I** easily adds to phenols to form the stable phosphoranes with a P–H bond, capable of distillation in a vacuum without decomposition.



I **II**

Taking this in account we hoped to prepare stable cavitands by reaction of phospholine **I** with calix[4]-resorcinarenes. We expected that the number of phosphorane fragments with a P–H bond located on the upper circle of the calixarene matrix would defined by the ratio of the reagents.

Reaction of phospholine (**I**) and calixarene (**III**) (R = C_6H_{13}) in equimolar amounts proceeds smoothly in ethereal solution at cooling and leads to formation of cavitand **VI**, which possess one dioxaphosphocynic fragment.

^{31}P NMR spectrum of the reaction mixture shows a signal of P–H-phosphorane **IV** (δ_P –28.31 ppm, $^1J_{PH}$ 617.9 Hz), which, however, soon disappears and strong singlet peak of the final reaction product appears at δ_P 189.5 ppm; the product was isolated and characterized. Its elemental analysis corresponds to the presence of one phosphorus atom in a molecule. In its IR spectrum occurs strong absorption of phenol hydoxy groups in the region of 3100–3300 cm^{-1}. ^{31}P NMR spectrum of reaction mixture contains also signals of 5-ethyl-1,6-dioxa-4,9-diaza-2,3,7,8-dibenzo-5-phosphaspiro[4.4]nonane **VII** (δ_P –23.97 ppm) and

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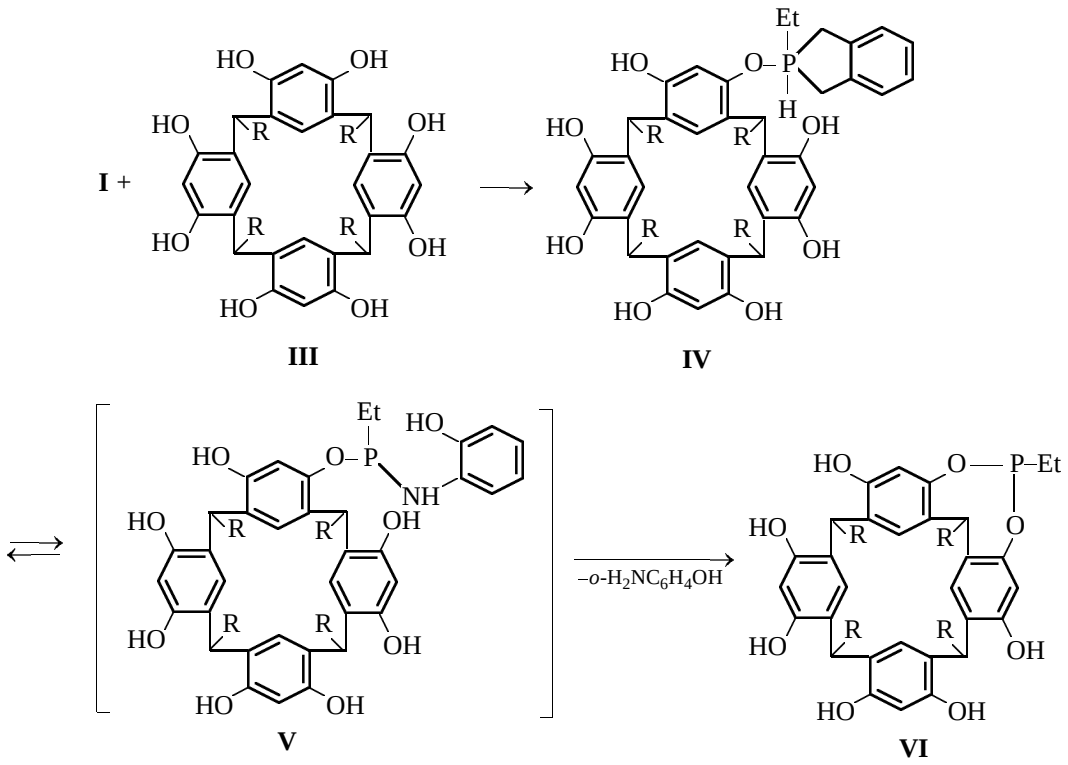
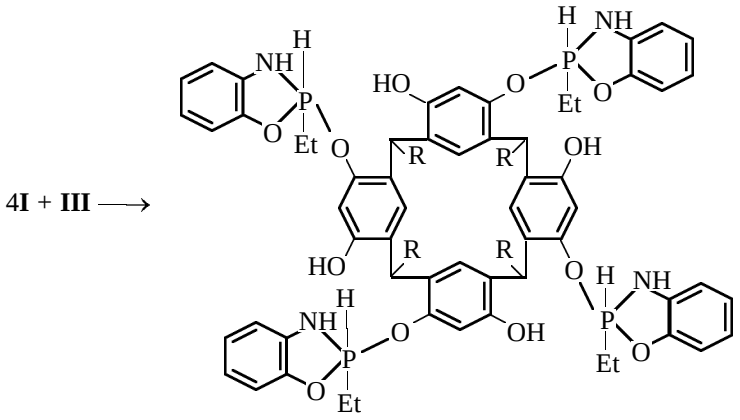


2-oxo-2-ethyl-1,3,2-benzoxazaphospholine **VIII** (δ_P 50.67 ppm). From the reaction mixture we isolated *o*-aminophenol. Formation of compound **VI** probably proceeds according to the scheme above. Reaction of phospholine **I** with calixarene **III** involves only one hydroxy group and leads to P–H-phosphorane **IV**. The latter compound exists in equilibrium with linear phosphonite **V** due to cleavage of endocyclic P–O bond [8]. Cyclization of the latter involving neighboring phenolic hydroxy group leads to release of *o*-aminophenol molecule and formation of dioxaphosphocine **VI**. Note that earlier attempts to introduce one phosphite (phosphonite, phosphinite) fragment to calixarene molecule by traditional methods failed. Phosphorylation of calixarenes by the derivatives of P(III) acids, e.g., hexaalkyltriaminophosphines, led to symmetrically substituted cavitands regardless of the reagent ratio [9].

^{31}P NMR spectrum of the reaction mixture of phospholine **I** with calixarene **III** in 8 : 1 ratio in ethereal solution, contained signals of equal intensity of the parent phospholine **I** (δ_P 160 ppm) and addition product, P–H phosphorane **IX** (δ_P –28.38 ppm, $^1J_{PH}$ 617.7 Hz). The spectral data obtained show, first, that only four hydroxy groups enter to the reaction and introduction of eight phosphorane fragments to the calixarene molecule probably impossible by steric reasons. Second, occurrence of only one signal characterizing hydrophosphorane fragments evidences in favor of formation of symmetrical molecule **IX**.

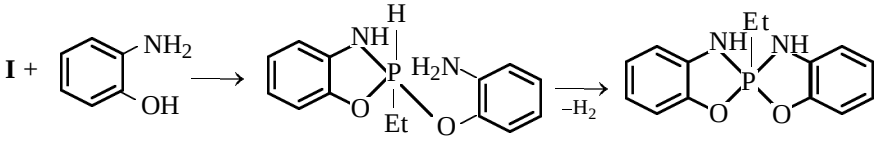
We studied in detail reaction of phospholine **I** with calixarene **III** in a 4 : 1 ratio. The reaction is exotermic in ethereal solution, and the calixarene dissolves quickly while the solutin becomes brick-red. From ^{31}P data follows that like the case of the reagent ratio 8 : 1 adduct, P–H-phosphorane **IX**, is formed instantly after mixing (δ_P –28.38 ppm, $^1J_{PH}$ 617.7 Hz). However, in this case the signal of parent phospholine **I** (δ_P 160 ppm) is not observed in the spectrum of the solution. IR spectrum of crude product contains absorption bands of P–H (2420 cm^{-1}) and N–H (3275 cm^{-1}) bonds, typical of the phosphoranes of similar structure [7], as well as broad band of unused O–H bonds (3100–3400 cm^{-1}). On keeping, the reaction mixture becomes colorless and a precipitate drops down, consisting of spirophosphorane **VII** (δ_P –23.77 ppm) and *o*-aminophenol. According to ^{31}P data, the filtrate after removing of the precipitate contains minor amounts of the oxidation product of the parent phospholine **I**, 2-ethyl-2-oxo-1,3,2-benzoxazaphospholine **VIII** (δ_P 50.58 ppm) [10], 5-ethyl-1,6-dioxa-4,9-diaza-2,3,7,8-dibenzo-5-phosphaspiro-



IX

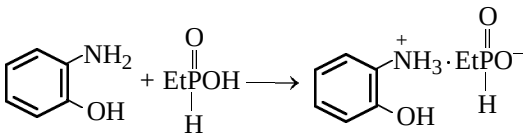
[4.4]nonane **VII** (δ_P –23.77 ppm), and dioxaphosphocine (δ_P 189.87 ppm).

Formation of spirophosphorane **VII** can be explained by reaction of *o*-aminophenol appearing in the reaction mixture with parent phospholine **I**. Earlier we showed [11] that this process goes through intermediate formation of P–H-phosphorane **X** which easily transforms to spirophosphorane **VII** eliminating a molecule of hydrogen.



X **VII**

One cannot exclude also direct transformation of phospholine **I** to spirophosphorane **VII** [12]. *o*-Aminophenol can be formed as a result of chemical transformation on the upper circle of the calixarene matrix with involvement of phosphorane P–H fragment and neigboring hydroxy group (sf. **V** → **VI** transformation). Besides, the observed reactions are probably involve water which is contained in the calixarene molecular cavity. According to the published data, such type calixarenes can contain from one to several tens of bounded water molecules forming a complex system of H-bonds with phenol hydroxyl [13]. Participating of water in the observed transformations is also confirmed by formation in small amount of *o*-hydroxyphenylammonium hydroxyethylphosphinite **XII**, the product of reaction of *o*-aminophenol with ethylphosphonite **XI**. Appearance of phosphonite **XI** in the reaction mixture is probably a result of hydrolysis of the formed in the reaction phosphorylated calixarenes possessing one (compound **VI**) or several dioxaphosphocine fragments.



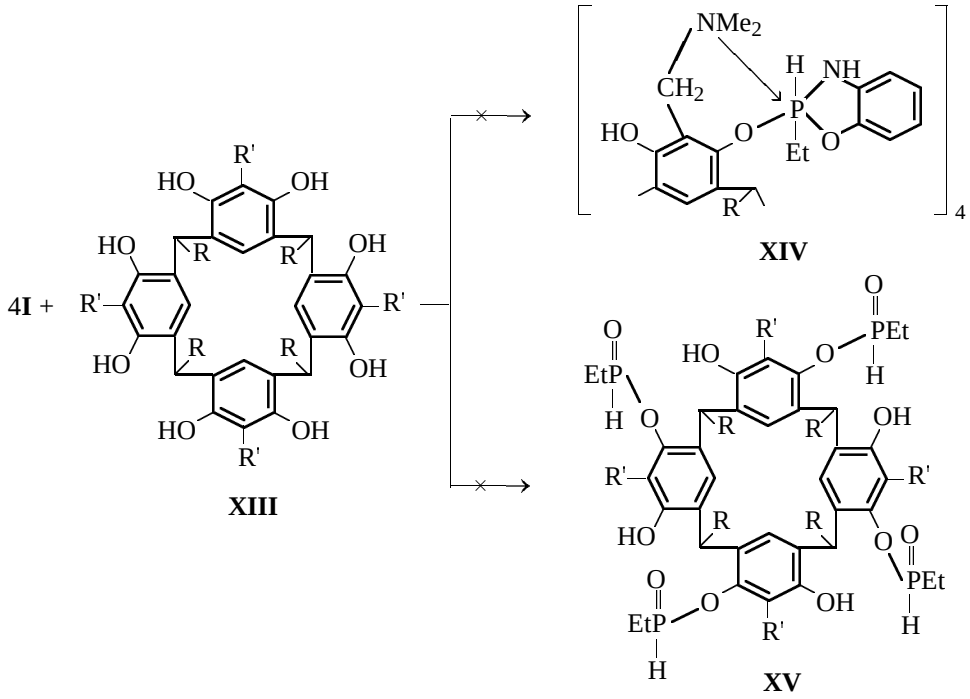
XI **XII**

As follows from x-ray crystallography, of a crystal compound **XII** has form of a salt, the anion–cation pair (Fig. 1). The *o*-aminophenol cation and ethylphosphonate anion have ordinary structures. The P–O bond lengths are equal within the experimental error [average 1.505(3) Å] and fall to the range between bond lengths of double and single bonds. The P–H bond length is 1.21 Å. A feature of the crystal structure of **XII** is its system of hydrogen bonds. All three hydrogen atoms of the protonated amino group and the hydroxyl hydrogen are involved in hydrogen bonding with the oxygen atoms of the phosphoryl group. Therewith, the contactcs of P–H group obeying the criterion of hydrogen bonding have not been

found. The H-bond parameters are as follows: (1) O^1–H···$O^{22'}$ (*x*, *y* – 1, *z*), O^1–H 1.06, H···$O^{22'}$ 1.58, O^1···$O^{22'}$ 2.643(3) Å, angle O^1–H···$O^{22'}$ 178°; (2) N^2–H^{21}···$O^{22''}$ (*x*, 3/2 – *y*, –1/2 + *z*), N^2–H^{21} 1.01, H^{21}···$O^{22''}$ 1.81, N^2···$O^{22''}$ 2.797(3) Å, angle N^2–H^{21}···$O^{22''}$ 164°; (3) N^2–H^{22}···$O^{21'''}$ (1 – *x*, –1/2 + *y*, 1/2 – *z*), N^2–H^{22} 0.89, H^{22}···$O^{21'''}$ 1.87, N^2···$O^{21'''}$ 2.747(3) Å, angle N^2–H^{22}···$O^{21'''}$ 168°; (4) N^2–H^{23}···O^{21}, N^2–H^{23} 1.02, H^{23}···O^{21} 1.72, N^2···O^{21} 2.736(3) Å, angle N^2–H^{23}···O^{21} 175°.

These H-bonds form in the crystal a layer (hydrogen bond net), parallel to the *yz* plane (Fig. 2.)

For extension of these studies, we involved calixarene **XIII** which possess dimethylaminomethyl fragments in *ortho* position to the hydroxy groups of the benzene rings, in the reaction with phospholine **I** We expected that the addition product, P–H phosphorane **XII** will be much more stable due to intramolecular N→P donor–acceptor interaction. Examles of stabilization of the such type phosphoranes were published earlier [14, 15].

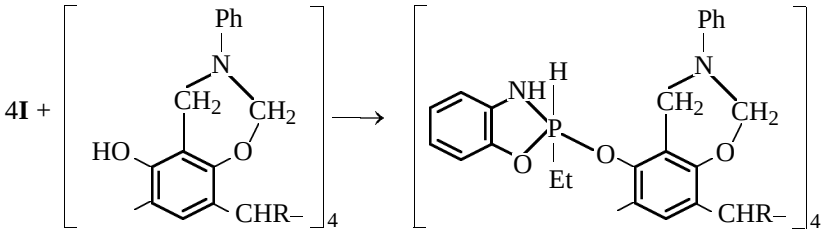


R = C_6H_{13}, R' = CH_2NMe_2.

^{31}P NMR spectrum of ethereal solution of phospholine **I** and calixarene **XIII** mixture (4:1 ratio) directly after mixing of the reagents contains a group of signals, δ_P, ppm: 25.64, $^1J_{PH}$ 496 Hz (hydrophosphonite **XV**), –28.34, $^1J_{PH}$ 618 Hz (P–H phosphorane, similar to compound **IX**), 50.18 (phospholine **VIII**), 188.22 (the calixarene including dioxaphosphocine fragments). After 6-h boiling of the reaction mixture, the final product **XV** was isolated (δ_P 25.30 ppm, $^1J_{PH}$ 496 Hz), which contained four hydrophoshonyl fragments. Probably the formation of phosphonite **XV** is a result of hydrolysis of intermediately formed phosphorus-containing compounds with a water from the cavity. In the course of the study of properties of the phosphorylated calixarenes we repeatedly noted "activation" of the water inside a cavity when tertiary amine or dialkylaminomethyl group constructed a part of calixarene matrix, like this case.

From the data obtained follows that introducing of dimethylaminomethyl groups to the *ortho* position to the hydroxy groups at benzene rings results in decrease in reactivity of calix[4]resorcinarenes with phospholine **I** and makes possible hydrolysis of the formed compounds with the intercavital water.

Reaction of phospholine **I** with calixarene **XVI** (4:1 ratio) which has four hydroxy groups and four oxazine fragments on the upper circle of its calyx proceeds much slower than with calixarene **XIII**.



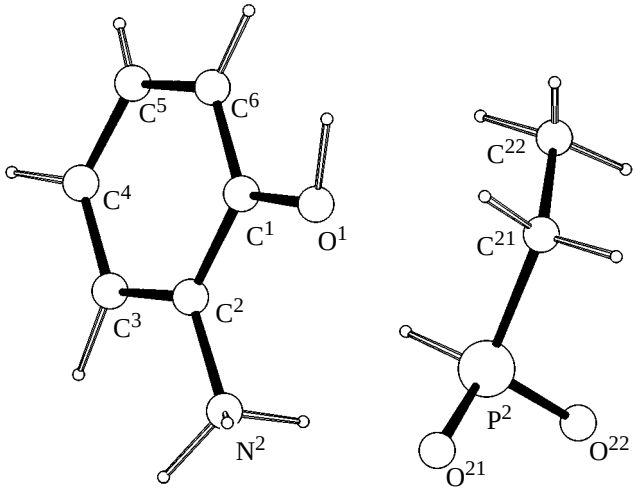


Fig. 1. Geometry of the anion-cation oair in the crustal of salt **XII**.

In first two hours the reaction, as follows from the data of ^{31}P NMR, was not found to occur at all. After 4 h, in the spectrum was registered, besides great amount of parent phospholine (I), appearance of P–H phosphorane **XVII** (δ_P –27 ppm, $^1J_{PH}$ 617 Hz) (10% of the total signal intensity), phospholine **VIII** (20%), the product of oxidation of the parent compound, and ~5% of spirophosphorane **VII** (δ_P –23 ppm). Further keeping results in significant complication of the picture. Note that change of methyl groups on the lower circle of the molecule by hexyl influences significantly the calixarene reactivity with phospholine **I**: instantly after mixing **I** the spectrum contains equal in intensity signals of parent compound **I** and the produt of its oxidation **VIII**.

The data concerning calixarene properties published to the moment do not allow argue explaining of the obtained experimental results. Generally, the features of reactivity of calix[4]resorcinarenes in a great extent are defined by their association in solution and macroring conformation, the later depends substantially on the length of the alkyl group on the lower circle of the calyx.

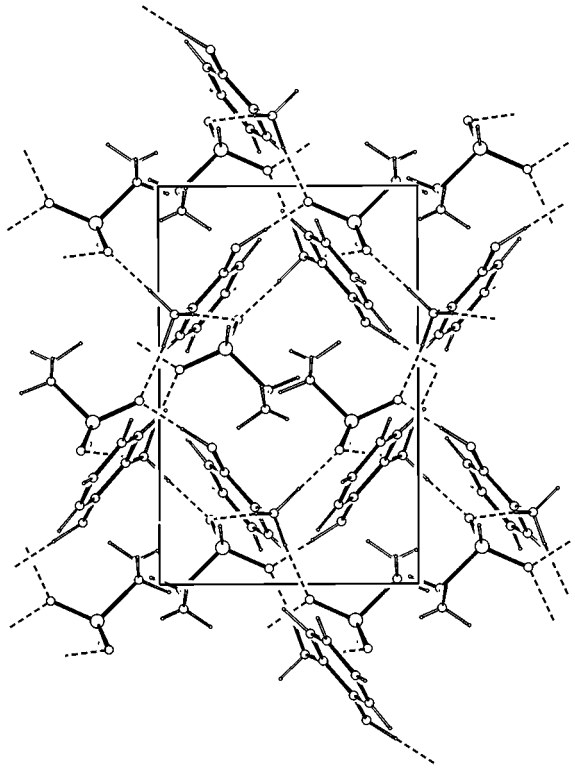


Fig. 2. Hydrogen bonding system in a crystal of salt **XII**. Projection along 0*x* axis.

EXPERIMENTAL

IR spectra were registered on a UR-20 spectrometer in the 400–3600 cm^{-1} range, compounds studied were taken as suspensions in mineral oil. ^{31}P NMR spectra were registered on a Fourier NMR spectrometer Bruker MSL-400, operating frequency 161.97 MHz, external reference 85% H_3PO_4.

X-ray structural investigation of salt XII. Crystals of the salt **XII**, $C_6H_8NO^+ \cdot C_2H_6O_2P^-$, mp 155°C, monoclinic. At 20°C a = 11.3413(2), b = 7.673(3), c = 12.142(5) Å; β = 101.038(5)°, V = 1037(1) Å^3, d_{calc} = 1.30 g cm^{-3}, Z = 4, steric group $P2_1/c$.

Atomic coordinates and equivalent isotropic thermal parameters of nonhydrogen atoms $B = 4/3\sum_{i=1}^{3}\sum_{j=1}^{3}(a_i a_j)B(i,j)$ (Å^2) and isotropic thermal factors of hydrogen atoms B_{iso} (Å^2) in **XII**

Atom	*x*	*y*	*z*	*B*
P^2	0.34078(7)	–0.2569(1)	0.40793(6)	2.39(1)
O^1	0.3853(2)	0.2025(3)	0.3436(2)	3.21(5)
O^{21}	0.4311(2)	–0.3032(3)	0.3368(2)	3.32(5)
O^{22}	0.3533(2)	–0.0783(3)	0.4605(2)	2.84(4)
N^2	0.3946(2)	0.4398(4)	0.1777(2)	2.20(5)
C^1	0.2796(3)	0.2506(5)	0.2817(2)	2.38(5)
C^2	0.2810(3)	0.3689(4)	0.1939(2)	2.14(5)
C^3	0.1746(3)	0.4189(5)	0.1254(3)	2.84(7)
C^4	0.0653(4)	0.3550(5)	0.1436(3)	3.91(8)
C^5	0.0640(3)	0.2439(6)	0.2306(3)	4.09(8)
C^6	0.1695(3)	0.1911(5)	0.2998(3)	3.43(7)
C^{21}	0.3348(4)	–0.4175(5)	0.5123(3)	4.03(9)
C^{22}	0.2230(6)	–0.4089(8)	0.5605(4)	7.2(1)
H^1	0.370(5)	0.090(8)	0.390(4)	6(2)
H^2	0.240(3)	–0.268(4)	0.353(2)	2.9(7)
H^3	0.178(3)	0.522(5)	0.063(3)	4.9(9)
H^4	–0.028(4)	0.390(6)	0.087(4)	6(1)
H^5	0.007(4)	0.208(6)	0.243(3)	6.1(9)
H^6	0.161(4)	0.111(5)	0.364(3)	4.9(9)
H^{21}	0.395(3)	0.482(5)	0.099(3)	3.4(8)
H^{22}	0.458(3)	0.372(4)	0.178(3)	2.9(7)
H^{23}	0.404(4)	0.534(5)	0.238(3)	5.7(9)
H^{211}	0.418(4)	–0.413(6)	0.569(4)	6(1)
H^{212}	0.32(2)	–0.53(3)	0.48(1)	5(7)
H^{221}	0.13(1)	–0.39(1)	0.505(7)	19(3)
H^{222}	0.246(7)	–0.306(9)	0.606(5)	10(2)
H^{223}	0.21(1)	–0.49(1)	0.585(8)	19(3)

The cell parameters and intensities of 2395 reflections, of which 1607 characterized by $I \geq 3\sigma(I)$, were measured on an automatic four-circle Enraf–Nonius CAD-4 difractometer (λCuK_α radiation, graphite monochromator, $\theta/2\theta$ scanning ($\theta \leq 76.3°$). No decrease in intensity occurred during the experiment in three testing measurements. Absorption was not accounted for (μCu 22.0 cm^{-1}). The structure was decoded by a direct method using SIR package [16] and refined in isotropic and then in anisotropic approximations. All hydrogen atoms were revealed from the electron density difference series and their contributions to the structural amplitudes were accounted for in the final step of the refinement with fixed position and isotropic termal parameters. Final divergence factors are R 0.044, R_W 0.057 on 1110 independent reflections with $F^2 \geq 3\sigma$.

Atomic coordinates in the structure of **XII** are listed in the table, geometry of cation-anion pair and the system of hydrogen bonding in the crystal of **XII** are shown in Figs. 1 and 2. All calculations were conducted on a DEC Alpha Station-200 computer with the MolEN [17] program package. Figures of hydrogen bond system in the crystal and calculation of intermolecular contacts were performed with PLATON 98 [18] package.

5,11,17,26-tetrahexyl-3,7,9,13,15,19-hexahydroxy-22,24-dioxa-23-phospha[19.3.3.1^{4,2,5}.1^{6,10}.1^{12,16}.1^{18,26}]hexacyclotriginta[1,3,6,8,12,14,18,20,10(29),16(28),26(27),25(30)]dodecaene (VI). 0.82 g of calixarene **II** was mixed with a solution of 0.17 g of phospholine **I** in 3 ml of anhydrous ether at 5°C. The calixarene completely dissolved to form transparent solution. After 8-h keeping 0.7 g (43%) of *o*-aminophenol was filtered off, mp 171–174°C. From the filtrate, after removing of solvent in a vacuum dropped crystals which were separated, washed with ether and dried. Yield 0.3 g (34%) of compound **VI**, mp 182–190°C. Found, %: C 68.32; H 9.11; P 3.94. $C_{54}H_{75}O_8P$. Calculated, %: C 68.02: H 8.84; P 3.51.

Reaction of 2-ethyl-1,3,2-benzoxazaphospholine I with calix[4]resorcinarene II (4 : 1). To 1.67 g of phospholine **I** at cooling to 10°C was slowly added a suspension of 2.06 g of calixarene **II** in 10 ml of ether. The calixarene was completely dissolved, the solvent became of brick-red color. During 5 h we observed the solution decolorization, evolution of a gas in small bubbles and dropping of a precipitate. The solid part was fractionally recrystallized and the following compounds were isolated: 0.12 g (11%) of *o*-aminophenol, mp 174°C; 0.11 g (8%) of 5-ethyl-1,6-dioxa-4,9-diaza-2,3,7,8-dibenzo-5-phosphaspiro-[4.4]nonane **VII**, mp 121–123°C [11]; 0.10 g (5%) of *o*-hydroxyphenylammonium hydroethylphosphonite **XII**, mp 155°C. Found P, %: 15.03. $C_8H_{14}NO_3P$. Calculated P, %: 15.27.

2,8,14,20-tetraethylpentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacoza-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-4,10,16,22-tetrahydroxy-5,11,17,19-tetra(dimethylaminomethyl)-6,12,18,24-tetra(ethylhydrophosphinat) (XV). *a*. A mixture of 0.8 g of phospholine **I** and 0.63 g of calixarene **XIII** (8 : 1) in 5 ml of anhydrous ether was refluxed for 6 h. We obtained 0.64 g (79%) of compound **XV**, mp 92°C. IR spectrum, ν, cm^{-1}: 1250 (P=O), 1600 (aromatic ring), 3200–3400 (OH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm(J, Hz): 0.89 t [12H, CH_3–(CH2)4, $^3J_{HH}$ 6.9), 1.11 t (12H, CH_3–CH$_2$P), 1.20–1.38 m [32H, CH$_3$–(CH_2)$_4$], 2.18 m (CH_2–P), 2.38 s (24H, CH_3–N), 2.58–2.64 s (8H, CH_2–N),

2.83 m (8H, CH_2–CH), 4.18–4.24 m (4H, CH–CH_2), 7.13 with (4H, H_m-calix). ^{31}P NMR spectrum, δ_P, ppm (J, Hz): 25.64 ($^1J_{PH}$ 496). Found, %: C 63.34; H 8.13; P 8.67. $C_{72}H_{120}N_4O_{12}P_4$. Calculated, %: C 63.71; H 8.84; P 9.14.

b. A mixture of 0.8 g of phospholine **I** and 1.26 g of calixarene **XIII** (4 : 1) in 5 ml of anhydrous ether was refluxed for 6 h. 1.3 g (81%) of compound **XV** was isolated, mp 92°C. ^{31}P NMR spectrum, δ_P, ppm (J, Hz): 25.36 ($^2J_{PH}$ 494).

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