Formation of the cage-like phosphoranes with the P—C and P—N bonds in the reactions of 2-(2-benzylidenamino)phenoxy-4-*tert*-butylbenzo-1,3,2-dioxaphosphol with ethyl mesoxalate and ethyl trifluoropyruvate*

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The reaction of 2-(2-benzylidenamino)phenoxy-4-*tert*-butylbenzo-1,3,2-dioxaphosphol with ethyl mesoxalate and ethyl trifluoropyruvate resulted in the formation of tricyclic phosphoranes with the P—C and P—N bonds. The adduct emerged from the initial reaction of the P^{III} derivative with the activated ketone (1 : 1), further underwent the transformation *via* the intramolecular reaction involving the benzylideniminoaryl substituent, which resulted in the formation of the cage-like phosphoranes.

Key words: organic derivatives of hypercoordinated phosphorus(v), oxidative addition of P^{III} derivatives to heteroatomic multiple bonds, cage-like phosphoranes, trigonal bipyramidal-type structures, stereoselectivity, X-ray crystallography.

Development of new methods for the synthesis of hypercoordinated phosphorus derivatives, key intermediates in phosphorylation and dephosphorylation reactions proceeding in living cells, is an actual direction in the chemistry of organoelement compounds.[1,2] The P^V derivatives are intermediates in many important reactions in organic chemistry of phosphorus, such as nucleophilic substitution at P^{IV} atom, Wittig, Mitsunobu, Evans and other reactions; they are used in organic synthesis for the formation of C—C (oxaphosphorane condensation), C—X (X = O, N, S) and other bonds.[3,4] Synthesis of phosphoranes includes the oxidative addition reactions of P^{III} derivatives to various organic and inorganic agents, to compounds containing heteroatomic multiple bonds, different rearrangements, *etc*.[5,6]

In the last years, we are working on the development of a new approach to the synthesis of derivatives of hypercoordinated phosphorus, which is based on the cascade reactions of P^{III} derivatives containing a carbonyl group in one of the substituents, which are initiated by activated carbonyl compounds, such as chloral, hexafluoroacetone, *etc*.[7] In development of this approach, in the present work we for the first time used in the synthesis of cage-like phosphoranes a P^{III} derivative, *viz*., 2-(2-benzylidenaminophenoxy)-5-*tert*-butylbenzo-1,3,2-dioxaphosphol (**1**) containing a C=N bond in one of the substituents, ethyl esters of mesoxalic and trifluoropyruvic acid (**2a**,**b**) were used as the carbonyl compounds initiating the cascade process.

The reaction of the indicated compounds proceeded under mild conditions and quantitatively led to the formation of stereoisomeric compounds **3**. The process apparently included a nucleophilic attack by the phosphorus atom on the carbonyl carbon atom and the initial formation of the bipolar ion **A**, the further stabilization of which proceeded through the intramolecular attack of the C=N bond by the alkoxide anion and finalized by the formation of the P—N bond (Scheme 1). The *tert*-butyl group in the phenylene fragment plays the role of an anchor, firmly differentiating the axial and the equatorial positions of the oxygen atoms of the benzophosphol fragment in the phosphorus trigonal bipyramid, thus simplifying interpretation of NMR data.

Based on the ^{13}C and ^{31}P NMR spectra, the compounds obtained were identified as spirophosphoranes **3a**,**b** with the phosphorus—carbon and phosphorus—nitrogen bonds. Their structure was also confirmed by electron impact mass spectrometry: the mass spectra contained the molecular ion peaks $[M]^{+\cdot}$ with *m/z* 565 (**3a**) and 561 (**3b**). The general fragmentation process of these molecules upon electron impact is the formation of ions $[M - Me]^+$ (550 and 546, respectively) and ions [M – COOEt] (492 and 488, respectively).

It should be noted that compound **3a** contains two chiral centers (P(1) and C(6)) and is formed as a mixture of two diastereomers (d_1, d_2) of the composition of 3 : 2. In the ^{13}C-{^1H} NMR spectrum, carbon atoms C(8) and

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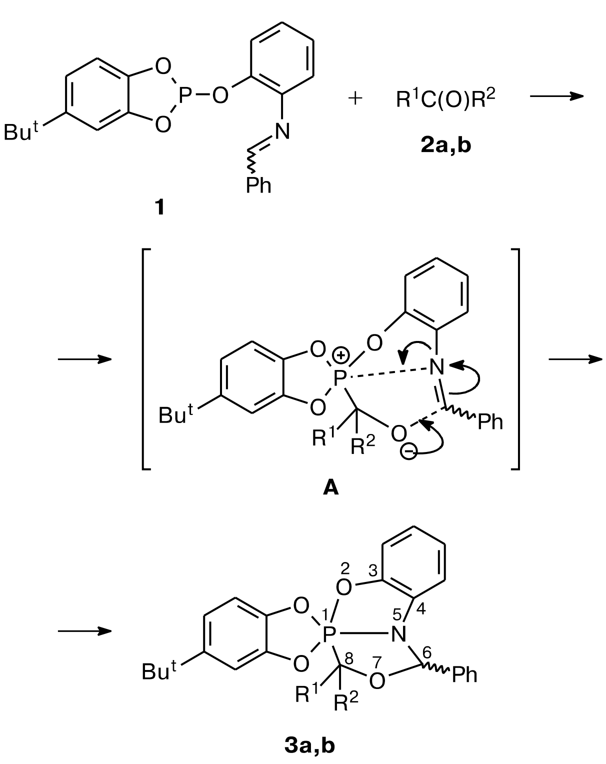
Scheme 1



R^1 = R^2 = COOEt (**a**); R^1 = COOEt, R^2 = CF$_3$ (**b**)

C(6) resonate as two doublets at δ_C 83.65 and 83.56 ($^1J_{PC}$ = 145.6 and 144.5 Hz), 88.12 and 88.06 ($^2J_{PNC}$ = = 17.2 Hz), which belong to the two diastereomers. The signals of the *ipso*-carbon atoms C(3), C(4), C(9), C(14), C(23) are also doubled in pairs. The ethoxy groups in the ethoxycarbonyl substituents are equivalent in one diastereomer (d_2) and considerably differ in the other one (d_1), whereas the carbonyl groups themselves are nonequivalent in both diastereomers. The interpretation of the signals for the carbon atoms of the dioxa- and oxazaphenylene fragments was made with allowance for the splitting of the signals in the ^{13}C NMR spectrum, as well as based on the differing screening effects of *ortho*-, *meta*-, and *para*-carbon atoms by the *tert*-butyl substituent and the O and N atoms. Phosphorane **3b** with three chiral centers (P(1), C(6), C(8)) is a mixture of four diastereomers (d_1—d_4), which are formed in the ratio of (43 : 37 : 10 : 10). Diastereomers d_1 and d_2 gradually crystallize, and the structure of one of them (solvate with CCl_4 of the composition 1 : 1) was confirmed by X-ray diffraction analysis (Fig. 1, Table 1). The configurations of atoms C(6), C(8), and P(1)[8] in the crystal of the isolated diastereomer (racemate) are *S*(*R*), *R*(*S*), and *R*(*S*), respectively. The phosphorus atom has a nearly right trigonal-bipyramidal configuration. The structure has a specific feature of the antiapicophilic arrangement of the P(1)—N(5) bond, which can be probably explained by two reasons. First, this is a compromise reached for the arrangement of two highly apicophilic five-membered heterocycles annulated with respect to this bond: in such an arrangement each of them has the axial-equatorial orientation. The second reason seems more important, this is the competition for the second axial place of the P—O bond of the *tert*-butylbenzodioxaphosphol ring P(1)O(1)C(9)C(14)O(3), which has the flattened *envelop* conformation (the fragment O(1)C(9)C(14)O(3) is planar within 0.002(3) Å, whereas the P(1) atom deviates from this plane by 0.1224(6) Å). The base of the trigonal bipyramid O(2)P(1)O(3)C(8) is almost ideally planar (within 0.0023(6)); the axial atoms O(1) and N(5) deviate from it by −1.700(2) and 1.732(2) Å, respectively. The cycle P(1)O(2)C(3)C(4)N(5) also has the flattened *envelop* conformation (the fragment O(2)N(5)C(3)C(4) is planar within 0.013(2) Å, atom P(1) deviates from the plane by 0.0633(6) Å). The rigidity of the structure, apparently, forces the cycle P(1)C(8)O(7)C(6)N(5) to adopt a similar conformation (the fragment C(8)O(7)C(6)N(5) is planar within 0.036(2) Å, atom P(1) deviates by 0.4222(6) Å), in which the C(29)F_3 group and atom O(3) occupy the axial positions (they deviate by 1.450(3) and −1.944(2) Å), whereas the C(30)(O)OEt group and atom O(2) are equatorial (the deviations of C(30) and O(2) are of −0.993(3)

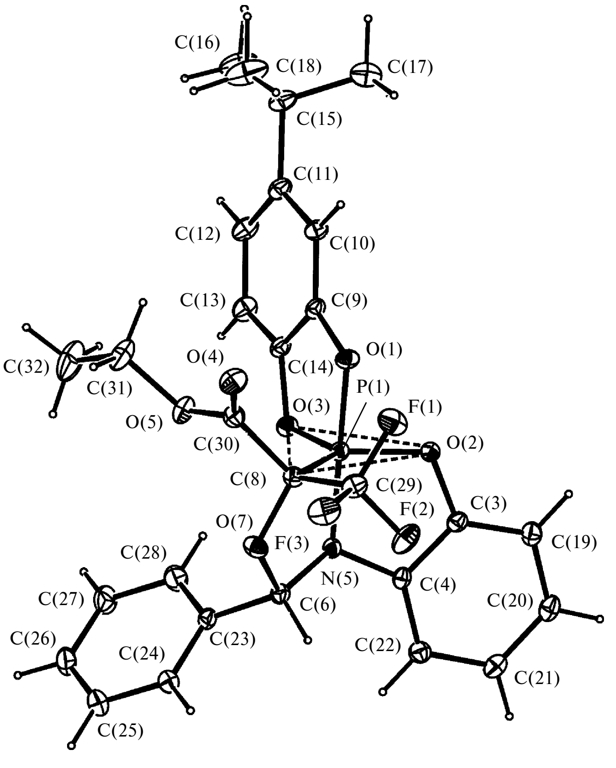


Fig. 1. Spatial structure of one of the diastereomers of compound **3b** in crystal (the solvent molecule of CCl_4 is not shown).

Table 1. Selected bond distances (*d*), bond (ω) and torsional (φ) angles (deg)

Parameter	Value
Bond	*d*/Å
P(1)—O(1)	1.698(2)
P(1)—O(2)	1.618(2)
P(1)—O(3)	1.623(2)
P(1)—N(5)	1.738(2)
P(1)—C(8)	1.894(2)
O(1)—C(9)	1.378(3)
O(2)—C(3)	1.398(3)
O(3)—C(14)	1.394(3)
O(7)—C(8)	1.401(3)
O(7)—C(6)	1.463(3)
N(5)—C(4)	1.399(3)
N(5)—C(6)	1.431(3)
C(3)—C(4)	1.379(3)
C(9)—C(14)	1.374(4)
Bond angle	ω/deg
O(1)—P(1)—O(2)	89.50(9)
O(1)—P(1)—O(3)	92.01(9)
Bond angle	ω/deg
O(1)—P(1)—N(5)	174.6(1)
O(1)—P(1)—C(8)	89.0(1)
O(2)—P(1)—O(3)	113.23(9)
O(2)—P(1)—N(5)	91.3(1)
O(2)—P(1)—C(8)	126.2(1)
O(3)—P(1)—N(5)	92.6(1)
O(3)—P(1)—C(8)	120.5(1)
N(5)—P(1)—C(8)	86.2(1)
P(1)—O(1)—C(9)	111.6(2)
P(1)—O(2)—C(3)	114.1(2)
P(1)—O(3)—C(14)	113.0(2)
P(1)—C(8)—C(29)	110.9(2)
P(1)—C(8)—C(30)	110.4(2)
C(6)—O(7)—C(8)	115.3(2)
P(1)—N(5)—C(4)	111.5(2)
P(1)—N(5)—C(6)	118.1(2)
C(4)—N(5)—C(6)	120.7(2)
Bond angle	ω/deg
O(2)—C(3)—C(4)	112.7(2)
N(5)—C(4)—C(3)	110.2(2)
O(7)—C(6)—N(5)	106.9(2)
P(1)—C(8)—O(7)	108.7(2)
Torsion angle	φ/deg
O(2)—P(1)—N(5)—C(4)	−2.4(2)
O(2)—P(1)—C(8)—C(29)	9.3(2)
O(2)—P(1)—C(8)—C(30)	133.0(2)
N(5)—P(1)—C(8)—C(29)	98.2(2)
N(5)—P(1)—C(8)—C(30)	−138.2(2)
P(1)—O(2)—C(3)—C(4)	−4.6(3)
C(31)—O(5)—C(30—C(8)	179.8(2)
P(1)—N(5)—C(4)—C(3)	0.2(2)
P(1)—N(5)—C(6)—C(23)	107.2(2)
C(29)—C(8)—C(30—O(5)	179.2(2)

and 0.596(2) Å). In the absence of strong intermolecular interactions, the crystal packing of compound **3b** is stabilized by intra- and intermolecular short contacts of the C—H···O type: C(28)—H(28)···O(3) (*d*(C—H) 0.95 Å, *d*(H···O) 2.46 Å, *d*(C···O) 3.290(4) Å, ∠CHO 146.0°) and C(22)—H(22)...O(4) (*d*(C—H) 0.95 Å, *d*(H···O) 2.55 Å, *d*(C···O) 3.484(3) Å, ∠CHO 170.0°, symmetry operation $1 + x, y, z$, respectively).

Experimental

NMR spectra were recorded on a Bruker Avance-400 spectrometer relative to the signal of the solvent ($CDCl_3$) residual protons (^1H) and the carbon atoms (^{13}C), or relative to the external standard H_3PO_4 (^{31}P). GLC-MS spectrometric studies were carried out on a DFS Thermo Electron Corporation instrument (Germany) with an Agilent DB-5MS capillary column (length 30 m, diameter 0.254 mm). Ionization by electron impact, the energy of ionizing electrons was 70 eV, the temperature of the source of ions was 280 °C, helium was the carrier gas. The mass spectral data were processed using the Xcalibur program. A sample under study before injection into the instrument was diluted in the chromatographically pure carbon tetrachloride in the concentration of ~0.1 wt.% Elemental analysis was performed on a EuroVector-3000 analyzer (C, H, N), as well as manually by pyrolysis of the sample in the flow of oxygen (P).

2-(2-*N*-Benzylidenaminophenoxy)-5-*tert*-butylbenzo-1,3,2-dioxaphosphol (1). 5-*tert*-Butyl-2-chlorobenzo-1,3.2-dioxaphosphol (2.05 g, 8.89 mmol) in benzene (10 mL) was added dropwise over 10 min to a mixture of 2-*N*-benzylidenaminophenol (1.75 g, 8.88 mmol) and triethylamine (1 g, 9.90 mmol) in anhydrous benzene (30 mL) with stirring under argon at room temperature. After all the chlorophosphite was added, the reaction a mixture was stirred for another 1 h and allowed to stand for 16 h. The following day, a precipitate formed was filtered off and washed with benzene (3 mL). The salt obtained after drying weighed 1.20 g. The solvent was evaporated from the filtrate, the residue was kept for 1 h *in vacuo* (0.1 Torr) to obtain compound **1** 3.33 g (96%) as light yellow oil. Found (%): C, 70.43; H, 5.77; N, 3.61; P, 8.06. $C_{23}H_{22}NO_3P$. Calculated (%): C, 70.59; H, 5.63; N, 3.58; P, 7.93. ^{31}P-{^1H} NMR (CH_2Cl_2): δ_P 128.4.

3,4-Benzo-1,1-(4-*tert*-butylphenylenedioxy)-3,3-bis(ethoxycarbonyl)-6-phenyl-5-aza-2,7-dioxa-1-phosphabicyclo[3.3.0^{1,5}]octane (3a). A solution of compound **2a** (1.16 g, 6.67 mmol) in CCl_4 (5 mL) was added dropwise to a solution of 2-(2-*N*-benzylidenaminophenoxy)-5-*tert*-butylbenzo-1,3,2-dioxaphosphol **1** (2.6 g, 6.65 mmol) in anhydrous CCl_4 (40 mL) with stirring under argon over 30 min. After all the chlorophosphite was added, the reaction a mixture was stirred for another 1 h and allowed to stand for 16 h. The following day, the solvent was evaporated *in vacuo* (10 Torr) and than was kept for 1 h at 0.1 Torr. The residue was a light yellow oily liquid, which upon prolong (more than 1 month) storage under the layer of dry pentane crystallized. The mass of the crystals after drying *in vacuo* was 3.27 g (87%), m.p. 130—132 °C (from pentane). Found (%): C, 63.89; H, 5.45; N, 2.31; P, 5.64. $C_{30}H_{32}NO_8P$. Calculated (%): C, 63.72; H, 5.66; N, 2.48; P, 5.49. MS, *m/z* (relative intensity (%), max.), ion: 565 (80.0) $[M]^{+\cdot}$, 550 (7.5) $[M - CH_3]^+$, 519 (5.9) $[M - C_2H_6O]^+$, 492 (17.6) $[M - C(O)OC_2H_5]^+$, 391 (57.7) $[M - C_7H_{10}O_5]^+$, 195 (72.2) $[C_{13}H_9NO]^+$, 91 (100.0) $[C_6H_5CH_2]^+$, 77 (13.9) $[C_6H_5]^+$. ^1H NMR ($CDCl_3$), δ: 0.74, 0.77 (both t, H(31), H(34), $^3J_{HH} = 7.1$ Hz); 1.17, 1.18 (both t, H(31), H(34), $^3J_{HH} = 7.1$ Hz (d_1, d_2)); 1.29, 1.30 (both s, C(16)—C(18) (d_1, d_2)); 3.73—3.74, 3.86—3.88, 4.23—4.25, 4.30—4.33 (all m, H(30), H(32), *AB*-parts of four ABX_3 spectra, $^3J_{H(A)H(B)} = 10.0$—12.0 Hz, $^3J_{H(A)H(X)} = 7.1$ Hz, $^3J_{H(B)H(X)} = 7.1$ Hz (d_1, d_2)); 7.75 (br.m, H(24), $^3J_{HH} = 7.1$ Hz (d_1, d_2)); 7.47 (m, H(25), $^3J_{HH} = 7.5$ Hz, $^3J_{HH} = 7.1$ Hz (d_1, d_2)); 7.43 (m, H(26), $^3J_{HH} = 7.5$ Hz

(d_1, d_2)); 7.04 (m, H(10), $^{4}J_{HH}$ = 2.2 Hz, $^{4}J_{POCCH}$ = 1.2 Hz, $^{5}J_{HH}$ = 1.1 Hz); 6.98 (m, H(10), $^{4}J_{HH}$ = 2.2 Hz, $^{4}J_{POCCH}$ = 1.2 Hz, $^{5}J_{HH}$ = 1.2 Hz (d_1, d_2)); 5.70 (br.s, H(6) (d_1, d_2)); 6.37, 6.39 (both br.d, H(22) (d_1, d_2)); 7.01 (br.d, H(13), $^{3}J_{HH}$ = 8.4 Hz); 6.91 (dm, H(13), $^{3}J_{HH}$ = 8.4 Hz, $^{4}J_{POCCH}$ = 1.2 Hz, $^{5}J_{HH}$ = 1.1 Hz (d_1, d_2)); 6.83, 6.93 (both br.d, H(19), $^{3}J_{HH}$ = 7.0—8.0 Hz (d_1, d_2)); 6.79 (br.m, H(20) (d_1, d_2)); 6.83 (br.m, H(21) (d_1, d_2)); 6.81 (br.m, H(12), $^{3}J_{HH}$ = 8.3 Hz (d_1, d_2)). ^{13}C NMR (CDCl$_3$, 150.9 MHz), δ_C: 108.05 (ddd (d), C(22), $^{1}J_{HC}$ = 160.2 Hz, $^{3}J_{PNCC}$ = 11.0 Hz, $^{3}J_{HC(20)CC(22)}$ = 7.8 Hz (d_1)); 109.08 (ddd (d), C(22), $^{1}J_{HC}$ = 160.2 Hz, $^{3}J_{PNCC}$ = 14.7 Hz, $^{3}J_{HC(20)CC(22)}$ = = 7.7 Hz (d_2)); 109.92 (br.ddm (br.d), C(10), $^{1}J_{HC}$ = 164.0 Hz, $^{3}J_{POCC}$ = 8.9 Hz, $^{3}J_{H(12)CC(10)}$ = 6.5—7.0 Hz (d_1, d_2)); 110.70 (dd (d), C(13), $^{1}J_{HC}$ = 164.1 Hz, $^{3}J_{POCC}$ = 15.0 Hz (d_1)); 109.61 (dd (d), C(13), $^{1}J_{HC}$ = 163.9 Hz, $^{3}J_{POCC}$ = 10.6 Hz (d_2)); 111.58 (dddm (d), C(19), $^{1}J_{HC}$ = 165.0 Hz, $^{3}J_{POCC}$ = 13.2 Hz, $^{3}J_{HC(21)CC(19)}$ = 9.4 Hz (d_1 + d_2)); 111.58 (dddm (d), C(19), $^{1}J_{HC}$ = 165.0 Hz, $^{3}J_{POCC}$ = 13.2 Hz, $^{3}J_{HC(21)CC(19)}$ = 9.4 Hz (d_1 + d_2)); 120.44 (dd (d), C(12), $^{1}J_{HC}$ = 162.0 Hz, $^{3}J_{HC(10)CC(12)}$ = 7.8 Hz (d_1 + d_2)); 123.70 (dd (s), C(21), $^{1}J_{HC}$ = 161.1 Hz, $^{3}J_{HC(19)CC(21)}$ = 7.3 Hz (d_1)); 123.64 (dd (s), C(21), $^{1}J_{HC}$ = 160.9 Hz, $^{3}J_{HC(19)CC(21)}$ = 7.4 Hz (d_2)); 117.90 (dd (s), C(20), $^{1}J_{HC}$ = = 159.1 Hz, $^{3}J_{HC(22)CC(20)}$ = 6.5 Hz (d_1)); 119.79 (dd (s), C(20), $^{1}J_{HC}$ = 157.5 Hz, $^{3}J_{HC(22)CC(20)}$ = 5.3 Hz (d_2)); 147.22 (br.m (br.s), C(11) (d_1)); 144.67 (br.m (br.s), C(11) (d_2)); 143.13 (m (d), C(3), $^{3}J_{POCC}$ = 1.5 Hz (d_2)); 143.12 (m (d), C(3), $^{3}J_{POCC}$ = 1.5 Hz (d_1)); 140.32 (br.m (br.s), C(14) (d_1)); 142.41 (br.m (br.s), C(14) (d_2)); 147.61 (br.m (br.s), C(9) (d_1)); 145.57 (br.m (br.s), C(9) (d_2)); 138.33 (br.m (br.s), C(4) (d_1, d_2)); 133.62 (m (d), C(23), $^{3}J_{PNCC}$ = 18.7 Hz, $^{3}J_{HCCC}$ = 7.2 Hz, $^{2}J_{HCC}$ = 6.1 Hz (d_1)); 133.55 (m (d), C(23), $^{3}J_{PNCC}$ = 18.7 Hz, $^{3}J_{HCCC}$ = 7.2 Hz, $^{2}J_{HCC}$ = 6.1 Hz (d_2)); 164.67 (m (d), C(29), $^{2}J_{PCC}$ = 7.7 Hz, $^{3}J_{HCOC}$ = 3.7 Hz (d_1)); 164.75 (m (d), C(29), $^{2}J_{PCC}$ = 7.3 Hz, $^{3}J_{HCOC}$ = 3.7 Hz (d_2)); 164.55 (m (br.d), C(32), $^{2}J_{PCC}$ = 2.6 Hz, $^{3}J_{HCOC}$ = 3.6 Hz (d_1)); 164.48 (m (br.d), C(32), $^{2}J_{PCC}$ = 2.5 Hz, $^{3}J_{HCOC}$ = 3.6 Hz (d_2)); 88.12 (ddt (d), C(6), $^{1}J_{HC}$ = 158.6 Hz, $^{3}J_{PNCC}$ = 17.2 Hz, $^{3}J_{HCCC}$ = 4.8 Hz (d_1)); 88.06 (ddt (d), C(6), $^{1}J_{HC}$ = 158.6 Hz, $^{3}J_{PNCC}$ = 17.2 Hz, $^{3}J_{HCCC}$ = 4.8 Hz (d_2)); 83.65 (d (d), C(8), $^{1}J_{PC}$ = 145.6 Hz (d_2)); 83.56 (d (d), C(8), $^{1}J_{PC}$ = 144.5 Hz (d_1)); 62.74 (tq (s), C(30), $^{1}J_{HC}$ = 148.7 Hz, $^{2}J_{HCC}$ = = 4.5 Hz (d_1)); 62.68 (tq (s), C(30), $^{1}J_{HC}$ = 148.7 Hz, $^{2}J_{HCC}$ = 4.5 Hz (d_1)); 62.87 (tq (s), C(30), $^{1}J_{HC}$ = 149.1 Hz, $^{2}J_{HCC}$ = 4.5 Hz (d_2)); 164.67 (m (d), C(29), $^{2}J_{PCC}$ = 7.7 Hz, $^{3}J_{HCOC}$ = 3.7 Hz (d_1)); 164.55 (m (br.d), C(29), $^{2}J_{PCC}$ = 2.6 Hz, $^{3}J_{HCOC}$ = 3.6 Hz (d_1)); 164.75 (m (d), C(29), $^{2}J_{PCC}$ = 7.3 Hz, $^{3}J_{PCOC}$ = 3.7 Hz (d_2)); 164.48 (m (br.d), C(29), $^{2}J_{PCC}$ = 2.5 Hz, $^{3}J_{PCOC}$ = 3.6 Hz (d_2)); 34.78 (m (s), C(15), $^{3}J_{HC(10)CC(15)}$ = 3.7 Hz, $^{3}J_{HC(12)CC(15)}$ = = 3.7 Hz, $^{3}J_{HCCC}$ = 3.7 Hz (d_1)); 34.64 (m (s), C(15), $^{3}J_{HC(10)CC(15)}$ = 3.7 Hz, $^{3}J_{HC(12)CC(15)}$ = 3.7 Hz, $^{3}J_{HCCC}$ = 3.7 Hz (d_2)); 31.58 (q.sept (s), C(16)—C(18), $^{1}J_{HC}$ = 125.7 Hz, $^{3}J_{HCCC}$ = = 4.9 Hz); 31.52 (q.sept (s), C(16)—C(18), $^{1}J_{HC}$ = 125.7 Hz, $^{3}J_{HCCC}$ = 4.9 Hz (d_1 + d_2)); 13.17 (qt (s), C(31), C(34), $^{1}J_{HC}$ = = 127.4 Hz, $^{3}J_{HCCC}$ = 2.5 Hz (d_2)); 13.96 (qt (s), C(31), $^{1}J_{HC}$ = = 127.7 Hz, $^{3}J_{HCCC}$ = 2.5 Hz (d_1)); 13.98 (qt (s), C(34), $^{1}J_{HC}$ = = 127.7 Hz, $^{3}J_{HCCC}$ = 2.5 Hz (d_1)); 127.79 (br.dm (s), C(24), $^{1}J_{HC}$ = 159.7 Hz (d_1, d_2)); 128.86 (C(25), $^{1}J_{HC}$ = 160.3 Hz, $^{3}J_{HCCC}$ = 7.5 Hz (d_1, d_2)); 129.78 (dt (s), C(26), $^{1}J_{HC}$ = 160.4 Hz, $^{3}J_{HCCC}$ = 7.5 Hz (d_1, d_2)). ^{31}P-{^{1}H} NMR (161.9 MHz, CDCl$_3$), δ_P: –8.2 (s, d_1, 60%); –7.9 (s, d_2, 40%).

3,4-Benzo-1,1-(4-*tert*-butylphenylenedioxy)-3-ethoxycarbonyl-6-phenyl-3-trifluoromethyl-5-aza-2,7-dioxa-1-phosphabicyclo[3.3.0^{1,5}]octane 3b was obtained by similar procedure from 2-(2-*N*-benzylidenaminophenoxy)-5-*tert*-butylbenzo-1,3,2-dioxaphosphol **1** (2.13 g, 5.45 mmol) and compound **2b** (0.93 g, 5.45 mmol) as light yellow oily liquid, which crystallized upon standing under the layer of anhydrous pentane. The weight of the crystals (a mixture of diastereomers d_1, d_2 of the composition 1 : 1, a solvate with CCl$_4$ of the composition 1 : 1) after drying *in vacuo* was 2.74 g (70%), m.p. 108—110 °C (from CCl$_4$). Found (%): C, 48.85; H, 3.33; N, 2.35; P, 4.71. $C_{28}H_{27}NO_6F_3P \cdot CCl_4$. Calculated (%): C, 48.67; H, 3.78; N, 1.96; P, 4.34. MS, *m/z* (relative intensity (%) max.), ion (the peaks of ions containing the most often encountered isotopes are reported): 561 (100.0) $[M]^{+\cdot}$, 546(69.3) $[M - CH_3]^+$, 488 (14.3) $[M - C(O)OC_2H_5]^+$, 391(57.7) $[M - C_5H_5O_3F_3]^+$, 195(93.5) $[C_{13}H_9NO]^+$, 77(23.3) $[C_6H_5]^+$. ^{1}H NMR of the mixture of diastereomers d_1—d_4, δ: 0.83 (t, H(32), $^{3}J_{HH}$ = 7.1 Hz); 0.84 (t, H(32), $^{3}J_{HH}$ = 7.1 Hz (d_1, d_2)); 1.10 (t, H(32), $^{3}J_{HH}$ = 7.1 Hz); 1.11 (t, H(32), $^{3}J_{HH}$ = = 7.1 Hz (d_3, d_4)); 1.31 (s, H(16)—H(18)); 1.32 (s, H(16)—H(18) (d_1, d_2)); 1.33 (s, H(16)—H(18)); 1.35 (s, H(16)—H(18) (d_3, d_4)); 3.62 (m, H(31), *AB*-part of the ABX_3 spectrum, $^{3}J_{H(A)H(B)}$ = = 10.7 Hz, $^{3}J_{H(A)H(X)}$ = 7.1 Hz, $^{3}J_{H(B)H(X)}$ = 7.1 Hz); 3.95 (m, H(31), *AB*-part of the ABX_3 spectrum, $^{3}J_{H(A)H(B)}$ = 10.7 Hz, $^{3}J_{H(A)H(X)}$ = 7.1 Hz, $^{3}J_{H(B)H(X)}$ = 7.1 Hz (d_1, d_2)); 4.17 (m, H(31), two *AB*-parts of two ABX_3 spectra (d_3, d_4)); 7.81 (m, H(24) (d_1, d_2)); 7.73 (m, H(24) (d_3, d_4)); 7.52 (m, H(25) (d_1—d_4)); 7.46 (m, H(26) (d_1—d_4)); 6.03 (d, H(6), $^{3}J_{PNCH}$ = 4.4—4.5 Hz); 6.02 (d, H(6), $^{3}J_{PNCH}$ = 4.4—4.5 Hz (d_1, d_2)); 6.13 (br.d, H(6), $^{3}J_{PNCH}$ = 4.4—4.5 Hz (d_3, d_4)); 6.22 (v.br.d, H(22), $^{3}J_{HH}$ = = 7.0—8.0 Hz); 6.18 (v.br.d, H(22), $^{3}J_{HH}$ = 7.0—8.0 Hz (d_3, d_4)); 6.68 (br.d, H(22), $^{3}J_{HH}$ = 7.0—8.0 Hz); 6.71 (br.d, H(22), $^{3}J_{HH}$ = 7.0—8.0 Hz (d_1, d_2)); 7.14 (dd, H(10), $^{4}J_{HH}$ = 1.5 Hz, $^{4}J_{POCCH}$ = 1.5 Hz); 7.12 (dd, H(10), $^{4}J_{HH}$ = 1.5 Hz, $^{4}J_{POCCH}$ = = 1.5 Hz (d_1, d_2)); 7.04 (d, H(13), $^{4}J_{HH}$ = 8.3 Hz); 7.03 (d, H(13), $^{4}J_{HH}$ = 8.3 Hz (d_1, d_2)); 7.02 (d, H(13), $^{4}J_{HH}$ = 8.3 Hz); 7.01 (d, H(13), $^{4}J_{HH}$ = 8.3 Hz (d_3, d_4)); 7.10 (dd, H(19), $^{3}J_{HH}$ = = 7.8 Hz, $^{4}J_{POCCH}$ = 1.8 Hz); 7.09 (dd, H(19), $^{3}J_{HH}$ = 7.8 Hz, $^{4}J_{POCCH}$ = 1.6 Hz (d_1, d_2)); 6.92—6.95 (m, H(20), H(12), H(21) (d_1, d_2)). ^{13}C NMR of the mixture of diastereomers d_1—d_4, δ_C (here and further, the splitting of the signal in the ^{13}C-{^{1}H}) NMR spectrum is given in parentheses: 108.36 (ddd (d), C(22), $^{1}J_{HC}$ = 160.8 Hz, $^{3}J_{PNCC}$ = 11.5 Hz, $^{3}J_{HC(20)CC(22)}$ = 7.5 Hz); 109.21 (dddd (d), C(22), $^{1}J_{HC}$ = 160.7 Hz, $^{3}J_{PNCC}$ = 15.7 Hz, $^{3}J_{HC(20)CC(22)}$ = 8.1 Hz, $^{2}J_{HC(21)C(22)}$ = 1.2 Hz (d_1, d_2)); 108.72 (ddm (d), C(22), $^{1}J_{HC}$ = 160.8 Hz, $^{3}J_{PNCC}$ = 12.3 Hz); 109.16 (ddm (d), C(22), $^{1}J_{HC}$ = 160.7 Hz, $^{3}J_{PNCC}$ = 14.1 Hz (d_3, d_4)); 110.90 (ddd (d), C(13), $^{1}J_{HC}$ = 164.5 Hz, $^{3}J_{POCC}$ = 15.5 Hz, $^{3}J_{H(12)CC(13)}$ = 1.0 Hz); 110.01 (ddd (d), C(13), $^{1}J_{HC}$ = 164.8 Hz, $^{3}J_{POCC}$ = 11.3 Hz, $^{3}J_{H(12)CC(13)}$ = 1.1 Hz (d_1, d_2)); 110.82 (br.dd (d), C(13), $^{1}J_{HC}$ = 165.0 Hz, $^{3}J_{POCC}$ = 14.0 Hz); 110.51 (br.dd (d), C(13), $^{1}J_{HC}$ = 164.7 Hz, $^{3}J_{POCC}$ = 16.3 Hz (d_3, d_4)); 111.15 (br.ddd (d), C(10), $^{1}J_{HC}$ = 161.7 Hz, $^{3}J_{POCC}$ = 9.2 Hz, $^{3}J_{HC(12)CC(10)}$ = 8.3 Hz); 111.20 (br.ddd (d), C(10), $^{1}J_{HC}$ = = 161.6 Hz, $^{3}J_{POCC}$ = 9.4 Hz, $^{3}J_{HC(12)CC(10)}$ = 8.3 Hz (d_1, d_2)); 111.37 (d, C(10), $^{3}J_{POCC}$ = 9.4 Hz); 111.31 (d, C(10), $^{3}J_{POCC}$ = = 9.4 Hz (d_3, d_4)); 112.13 (dddd (d), C(19), $^{1}J_{HC}$ = 166.0 Hz, $^{3}J_{POCC}$ = 13.0 Hz, $^{3}J_{HC(21)CC(19)}$ = 8.4 Hz, $^{2}J_{HC(20)C(19)}$ = 1.3 Hz (d_1 + d_2)); 111.85 (ddm (d), C(19), $^{1}J_{HC}$ = 162.0 Hz, $^{3}J_{POCC}$ = = 13.5 Hz, $^{3}J_{HC(21)CC(19)}$ = 8.3 Hz); 111.86 (ddm (d), C(19), $^{1}J_{HC}$ = 162.0 Hz, $^{3}J_{POCC}$ = 13.5 Hz, $^{3}J_{HC(21)CC(19)}$ = 8.3 Hz (d_3, d_4)); 121.41 (br.dd (d), C(12), $^{1}J_{HC}$ = 165.5—166.0 Hz, $^{3}J_{HC(10)CC(12)}$ = 8.1—8.3 Hz, $^{4}J_{POCCC}$ = 1.3 Hz (d_1 + d_2)); 120.86

(br.dd (d), C(12), $^{1}J_{\mathrm{HC}}$ = 161.0 Hz, $^{3}J_{\mathrm{HC(10)CC(12)}}$ = 7.5 Hz, $^{4}J_{\mathrm{POCCC}}$ = 1.3 Hz); 120.81 (br.dd (d), C(12), $^{1}J_{\mathrm{HC}}$ = 161.0 Hz, $^{3}J_{\mathrm{HC(10)CC(12)}}$ = 7.5 Hz, $^{4}J_{\mathrm{POCCC}}$ = 1.4 Hz (d_3, d_4)); 124.20 (dd (s), C(21), $^{1}J_{\mathrm{HC}}$ = 161.2 Hz, $^{3}J_{\mathrm{HC(19)CC(21)}}$ = 7.7 Hz); 124.17 (dd (s), C(21), $^{1}J_{\mathrm{HC}}$ = 161.2 Hz, $^{3}J_{\mathrm{HC(19)CC(21)}}$ = 7.8 Hz (d_1, d_2)); 123.67 (dd (s), C(21), $^{1}J_{\mathrm{HC}}$ = 161.0 Hz, $^{3}J_{\mathrm{HC(19)CC(21)}}$ = 7.7 Hz); 123.64 (dd (s), C(21), $^{1}J_{\mathrm{HC}}$ = 161.0 Hz, $^{3}J_{\mathrm{HC(19)CC(21)}}$ = 7.6 Hz (d_3, d_4)); 120.41 (dd (s), C(20), $^{1}J_{\mathrm{HC}}$ = 158.8 Hz, $^{3}J_{\mathrm{HC(22)CC(20)}}$ = 7.2 Hz); 117.73 (dd (s), C(20), $^{1}J_{\mathrm{HC}}$ = 159.6 Hz, $^{3}J_{\mathrm{HC(22)CC(20)}}$ = 7.1 Hz (d_1, d_2)); 120.19 (dd (s), C(20), $^{1}J_{\mathrm{HC}}$ = 160.8 Hz, $^{3}J_{\mathrm{HC(22)CC(20)}}$ = 8.2 Hz); 118.57 (dd (s), C(20), $^{1}J_{\mathrm{HC}}$ = 164.0 Hz, $^{3}J_{\mathrm{HC(22)CC(20)}}$ = 7.4 Hz (d_3, d_4)); 148.0 (d.decet (s), C(11), $^{3}J_{\mathrm{HC(13)CC(11)}}$ = 7.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 3.6—3.8 Hz); 145.14 (d.decet (s), C(11), $^{3}J_{\mathrm{HC(13)CC(11)}}$ = 7.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 3.6—3.8 Hz (d_1, d_2)); 148.66 (m (s), C(11)); 148.67 (m (s), C(11) (d_3, d_4)); 147.37 (dd (s), C(9), $^{3}J_{\mathrm{HC(13)CC(9)}}$ = 6.5 Hz, $^{2}J_{\mathrm{HC(10)C(9)}}$ = 4.1 Hz); 145.24 (dd (s), C(9), $^{3}J_{\mathrm{HC(13)CC(9)}}$ = 6.9 Hz, $^{2}J_{\mathrm{HC(10)C(9)}}$ = 3.4 Hz (d_1, d_2)); 142.88 (m (s), C(3), $^{3}J_{\mathrm{HCCC}}$ = 8.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.6 Hz, $^{2}J_{\mathrm{HC(19)C(3)}}$ = 4.0 Hz, $^{4}J_{\mathrm{HC(21)CCC(3)}}$ = 1.1 Hz (d_1, d_2)); 142.0 (m (d), C(14), $^{3}J_{\mathrm{HC(12)CC(14)}}$ = 10.7 Hz, $^{3}J_{\mathrm{HC(10)CC(14)}}$ = 7.0 Hz, $^{2}J_{\mathrm{POC}}$ = 3.8 Hz, $^{2}J_{\mathrm{HC(13)C(14)}}$ = 2.4 Hz); 139.88 (m (d), C(14), $^{3}J_{\mathrm{HC(12)CC(14)}}$ = 8.0 Hz, $^{3}J_{\mathrm{HC(10)CC(14)}}$ = 4.6 Hz, $^{2}J_{\mathrm{POC}}$ = 4.0 Hz, $^{2}J_{\mathrm{HC(13)C(14)}}$ = 1.4 Hz (d_1, d_2)); 139.27 (m (d), C(4), $^{3}J_{\mathrm{HCCC}}$ = 7.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.5 Hz, $^{2}J_{\mathrm{PNC}}$ = 2.7 Hz, $^{2}J_{\mathrm{HC(22)C(4)}}$ = 2.2 Hz); 139.25 (m (d), C(4), $^{3}J_{\mathrm{HCCC}}$ = 7.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.5 Hz, $^{2}J_{\mathrm{PNC}}$ = 2.7 Hz, $^{2}J_{\mathrm{HC(22)C(4)}}$ = 2.2 Hz (d_1, d_2)); 134.68 (m (d), C(23), $^{3}J_{\mathrm{PNCC}}$ = 18.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz); 134.61 (m (d), C(23), $^{3}J_{\mathrm{PNCC}}$ = 18.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz (d_1, d_2)); 133.68 (m (d), C(23), $^{3}J_{\mathrm{PNCC}}$ = 18.4 Hz); 133.61 (m (d), C(23), $^{3}J_{\mathrm{PNCC}}$ = 18.2 Hz (d_3, d_4)); 123.17 (qd (qd), C(29), $^{1}J_{\mathrm{FC}}$ = 287.6 Hz, $^{2}J_{\mathrm{PCC}}$ = 8.4 Hz ($d_1 + d_2$)); 90.32 (ddt (d), C(6), $^{1}J_{\mathrm{HC}}$ = 161.2 Hz, $^{3}J_{\mathrm{PNCC}}$ = 19.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 5.0 Hz); 90.27 (ddt (d), C(6), $^{1}J_{\mathrm{HC}}$ = 161.2 Hz, $^{3}J_{\mathrm{PNCC}}$ = 19.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 5.0 Hz (d_1, d_2)); 88.16 (ddt (d), C(6), $^{1}J_{\mathrm{HC}}$ = 164.0 Hz, $^{3}J_{\mathrm{PNCC}}$ = 17.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 5.4 Hz); 88.09 (ddt (d), C(6), $^{1}J_{\mathrm{HC}}$ = 164.0 Hz, $^{3}J_{\mathrm{PNCC}}$ = 17.5 Hz, $^{3}J_{\mathrm{HCCC}}$ = 5.4 Hz (d_3, d_4)); 78.88 (dq (dq), C(8), $^{1}J_{\mathrm{PC}}$ = 135.3 Hz, $^{2}J_{\mathrm{FCC}}$ = 29.3 Hz ($d_1 + d_2$)); 80.16 (dq (dq), C(8), $^{1}J_{\mathrm{PC}}$ = 142.7 Hz, $^{2}J_{\mathrm{FCC}}$ = 30.5 Hz (d_3, d_4)); 62.94 (tq (s), C(31), $^{1}J_{\mathrm{HC}}$ = 149.1 Hz, $^{2}J_{\mathrm{HCC}}$ = 4.5 Hz); 62.84 (tq (s), C(31), $^{1}J_{\mathrm{HC}}$ = 149.1 Hz, $^{2}J_{\mathrm{HCC}}$ = 4.5 Hz (d_1, d_2)); 63.40 (tq (s), C(31), $^{1}J_{\mathrm{HC}}$ = 149.5 Hz, $^{2}J_{\mathrm{HCC}}$ = 4.5 Hz); 63.37 (tq (s), C(31), $^{1}J_{\mathrm{HC}}$ = 149.5 Hz, $^{2}J_{\mathrm{HCC}}$ = 4.5 Hz (d_3, d_4)); 163.33 (br.m (dq), C(30), $^{2}J_{\mathrm{PCC}}$ = 1.9—2.0 Hz, $^{3}J_{\mathrm{FCCC}}$ = 1.9—2.0 Hz); 163.31 (br.m (dq), C(30), $^{2}J_{\mathrm{PCC}}$ = 1.9—2.0 Hz, $^{3}J_{\mathrm{FCCC}}$ = 1.9—2.0 Hz (d_1, d_2)); 164.02—164.05 (br.m (m), C(30), $^{2}J_{\mathrm{PCC}}$ = 5.2 Hz, $^{3}J_{\mathrm{FCCC}}$ = 1.6 Hz (d_3, d_4)); 35.01 (m (s), C(15), $^{3}J_{\mathrm{HC(10)CC(15)}}$ = 3.7 Hz, $^{3}J_{\mathrm{HC(12)CC(15)}}$ = 3.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 3.7 Hz); 34.83 (m (s), C(15), $^{3}J_{\mathrm{HC(10)CC(15)}}$ = 3.7 Hz, $^{3}J_{\mathrm{HC(12)CC(15)}}$ = 3.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 3.7 Hz (d_1, d_2)); 35.15 (m (s), C(15)); 34.85 (m (s), C(15) (d_3, d_4)); 31.66 (q.sept (s), C(16)—C(18), $^{1}J_{\mathrm{HC}}$ = 125.8 Hz, $^{3}J_{\mathrm{HCCC}}$ = 4.8 Hz); 31.62 (q.sept (s), C(16)—C(18), $^{1}J_{\mathrm{HC}}$ = 125.8 Hz, $^{3}J_{\mathrm{HCCC}}$ = 4.8 Hz (d_1, d_2)); 31.67 (m (s), C(16)—C(18)); 31.52 (m (s), C(16)—C(18) (d_3, d_4)); 13.30 (qt (s), C(32), $^{1}J_{\mathrm{HC}}$ = 127.6 Hz, $^{3}J_{\mathrm{HCCC}}$ = 2.6 Hz); 13.27 (qt (s), C(32), $^{1}J_{\mathrm{HC}}$ = 127.6 Hz, $^{3}J_{\mathrm{HCCC}}$ = 2.6 Hz (d_1, d_2)); 13.75 (qt (s), C(32), $^{1}J_{\mathrm{HC}}$ = 127.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 2.7 Hz); 13.73 (qt (s), C(32), $^{1}J_{\mathrm{HC}}$ = 127.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 2.7 Hz (d_3, d_4)); 127.65 (dm (s), C(24), C(28), $^{1}J_{\mathrm{HC}}$ = 160.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz); 127.63 (dm (s), C(24), C(28), $^{1}J_{\mathrm{HC}}$ = 160.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz, $^{3}J_{\mathrm{HCCC}}$ = 6.0—7.0 Hz (d_1, d_2)); 128.03 (C(24), C(28), $^{1}J_{\mathrm{HC}}$ = 160.4 Hz); 127.99 (dm (s), C(24), C(28), $^{1}J_{\mathrm{HC}}$ = 160.4 Hz (d_3, d_4)); 129.02 (C(25), C(27)), $^{1}J_{\mathrm{HC}}$ = 160.4 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.3 Hz); 129.03 (C(25), C(27), $^{1}J_{\mathrm{HC}}$ = 160.4 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.3 Hz (d_3, d_4)); 128.94 (dd (s), C(25), C(27), $^{1}J_{\mathrm{HC}}$ = 160.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.4 Hz); 128.92 (dd (s), C(25), C(27), $^{1}J_{\mathrm{HC}}$ = 160.3 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.4 Hz (d_1, d_2)); 127.79 (dt (s), C(26), $^{1}J_{\mathrm{HC}}$ = 160.6 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.6 Hz); 127.77 (dt (s), C(26), $^{1}J_{\mathrm{HC}}$ = 160.6 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.6 Hz (d_1, d_2)); 130.17 (dt (s), C(26), $^{1}J_{\mathrm{HC}}$ = 160.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.6 Hz); 130.15 (dt (s), C(26), $^{1}J_{\mathrm{HC}}$ = 160.7 Hz, $^{3}J_{\mathrm{HCCC}}$ = 7.6 Hz (d_3, d_4)). ^{31}P-{^{1}H} NMR of the mixture of diastereomers d_1—d_4, δ_{P}: −5.8 (q, d_1, $^{3}J_{\mathrm{FCCP}}$ = 5.1 Hz); −6.3 (q, d_2, $^{3}J_{\mathrm{FCCP}}$ = 5.1 Hz, 80%); −7.4 (br.m, d_3, d_4, 20%).

X-ray diffraction analysis of a single crystal of compound **3b** was carried out on a Bruker Smart APEX II CCD automatic diffractometer: graphite monochromator; λ(Mo-Kα) = 0.71073 Å; ω-scan technique; temperature of 150 K. Absorption was allowed for semiempirically using the SADABS program.[9] The structure was solved by direct method using the SIR program[10] and refined first in isotropic and then in anisotropic approximation using the SHELXL-97 program.[11] Hydrogen atoms in the structure of **3b** were placed in the geometrically calculated positions and included into refinement using a riding model. All the calculations were performed using the WinGX[12] and APEX2[13] programs. All the figures and analysis of the intermolecular in-

Table 2. Parameters of the crystal of compound **3b** and conditions of X-ray diffraction experiment

Parameter	Value
Color	Colorless
Gabitus	Prism-like form
Molecular formula	$C_{28}H_{27}F_3NO_6P$, CCl_4
Molecular weight	715.29
Crystal system	Monoclinic
Space group	*P*21/*c*
Parameters of unit cell	
a/Å	8.8033(9)
b/Å	19.658(2)
c/Å	18.716(2)
β/deg	95.414(1)
V/Å^3	3224.5(6)
Z	4
d_{calc}/g cm^{-3}	1.473
Absorption coefficient/μMo cm^{-1}	4.77
F(000)	1464
Number of measured reflections	7745
R_{int}	0.0420
Number of observed independent reflections with $I > 2\sigma(I)$	5636
Divergence factor values, $I > 2\sigma(I)$	$R = 0.0557$, $R_w = 0.1434$
GOOF	0.896
Number of refined parameters	401
Region of indices measurements	$-11 \le h \le 11$, $-25 \le k \le 25$, $-24 \le l \le 24$
Maximum and minimum peaks/e Å^{-3}	1.007 and −1.134

teractions were performed using the PLATON[14] and ORTEP[15] programs. The single crystal of compound **3b** was studied in the Community Federal Spectro-analytical Center of the A. E. Arbuzov Institute of Organic and Physical Chemistry Kazan Scientific Center of the Russian Academy of Sciences, the Laboratory of Diffractional Methods of Study. Crystallographic characteristics of compound, parameters of experiment and refinement of the structure are given in Table 2.

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