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Retention of a six-membered ring in the reaction of 2-dialkylaminobenzo[*e*]-1,3,2-dioxaphosphinin-4-ones with pentafluorobenzaldehyde: O,N-exchange at phosphorus

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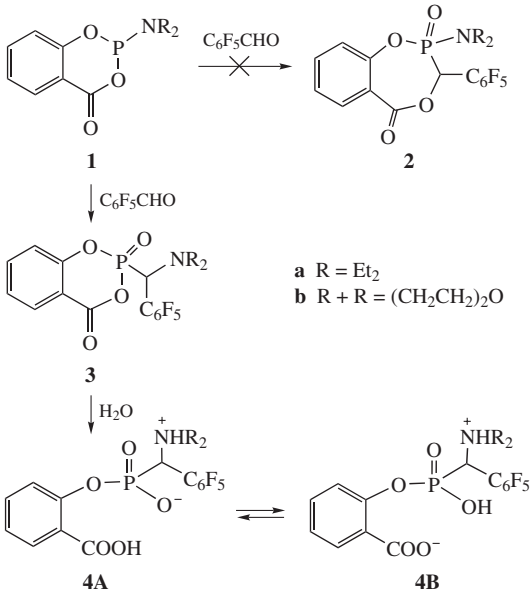
The title reaction leads to the formation of diastereoisomeric 2-[(dialkylamino)(pentafluorophenyl)methyl]benzo[*e*]-1,3,2-dioxaphosphinine-2,4-diones in a ratio of 70 : 30. The configuration of chiral centres for the preferable diastereoisomer was determined by single crystal X-ray diffraction analysis.

2-Alkylbenzo[*d*]-1,3,2-dioxaphosphinin-4-ones containing the reactive fragment P–O–C(O) readily interact with carbonyl compounds and imines containing acceptor substituents with the formation of tetracoordinated phosphorus derivatives – benzo[*e*]-1,3,2(1,4,2)-dioxa(oxaza)phosphepines, seven-membered phosphorus heterocycles.[1–7] Among them are the isosteres of 1,4-benzodiazepine-2,5-diones, which exhibit high biological activity.[8] The method is simple; the process is highly stereoselective, and the starting compounds are available. In some cases, for example, with the use of ethyl trifluoropyruvate and diethyl mesoxalate, spirophosphoranes are formed, and the fragment P–O–C(O) remains unaffected.[9,10]

We found that, unlike chloral,[1,2] ethyl trifluoropyruvate and diethyl mesoxalate, pentafluorobenzaldehyde forms 1,3,2-dioxaphosphinane derivatives **3**† instead of expected benzo[*e*]-1,4,2-dioxaphosphepines **2** in reactions with 2-NR_2-benzo[*d*]-1,3,2-dioxaphosphinin-4-ones **1** (Scheme 1).† In this case, the starting heterocycle remains unchanged, and an unusual O,N-exchange process[11–16] occurs in the course of reaction under mild conditions to afford a P–C bond and a phosphoryl group. The reaction stereoselectivity is ~70%.



Scheme 1

The ^{31}P NMR spectra (CH_2Cl_2) of compounds **3** exhibit characteristic signals with δ_P 11–13 ppm (d, $^2J_{PCH}$ 28.0–29.0 Hz), which corresponds to a P^{IV} derivative containing a P–C bond. Their IR spectra exhibit a characteristic band at 1773–1775 cm^{-1},

† *Synthesis of 2-dialkylaminobenzo[e]-1,2-dioxaphosphinin-4-ones* **1**. Trimethylsilylamine (0.1 mol) was added dropwise to a solution of 0.1 mol of 2-chlorobenzo[*e*]-1,3,2-dioxaphosphinin-4-one in 50 ml of CH_2Cl_2 with bubbling of argon (10 °C). Upon completion of the addition, the solvent and the resulting trimethylchlorosilane were removed *in vacuo*. A mixture of 10 ml of CH_2Cl_2 and 20 ml of pentane was added to the resulting mass, and the mixture was kept at –10 °C for one to three days for the freezing of an amine hydrochloride impurity, which was filtered off in an argon atmosphere. The residue as a viscous yellowish oily substance of 2-R-amino-4-oxobenzo[*e*]-1,2-dioxaphosphinin-4-ones **1** was used without additional purification. ^{31}P NMR spectrum (161.0 MHz, CH_2Cl_2) δ: 137.7 (s, quint., $^3J_{HCNP}$ 10.8 Hz) for **1a**, 133.5 (s, quint., $^3J_{HCNP}$ 7.3 Hz) for **1b**.

Reaction of compound **1a** *with pentafluorobenzaldehyde*. A solution of pentafluorobenzaldehyde (8.48 g, 0.043 mol) in 5 ml of CH_2Cl_2 was added dropwise to a solution of compound **1a** (10.34 g, 0.043 mol) in 10 ml of CH_2Cl_2. After three days, a portion of the solvent was removed, and the residue was kept at 0 °C for a day. The resulting crystals of 2-[(diethylamino)(pentafluorophenyl)methyl]benzo[*e*]-1,3,2-dioxaphosphinine-2,4-dione **3a** were washed with dry diethyl ether and dried; yield, 95%; mp 146–147 °C (main diastereoisomer). IR (Vaseline oil, ν/cm^{-1}): 1775, 1700, 1650, 1605, 1580, 1525, 1500, 1460, 1380, 1305, 1290, 1240, 1200, 1155, 1110, 1060, 1010, 990, 975, 940, 920. ^{31}P NMR (121.4 MHz, CH_2Cl_2) δ: 13.0 (br. d, $^2J_{PCH}$ 28.0 Hz). MS, *m/z*: 435 $[M]^{\bullet+}$. Found (%): C, 51.28; H, 4.04; P, 7.49. Calc. for $C_{18}H_{15}F_5NO_4P$ (%): C, 51.23; H, 3.57; P, 7.39.

Compound **3a** was ground in wet acetone; in this case, the immediate formation of a snow-white precipitate occurred. This precipitate was filtered off, washed with diethyl ether and dried *in vacuo*. The yield of 2-carboxyphenyl[(diethylammonio)(pentafluorophenyl)methyl]phosphonate **4a** was 95%; mp 192–193 °C (DMF). IR (Vaseline oil, ν/cm^{-1}): 3400, 2660–2680, 2450–2550, 1680, 1600, 1510, 1455, 1330, 1300, 1280, 1262, 1220–1230, 1162, 1100, 1090, 1070, 1010, 953, 918, 784, 766, 643, 610, 520. ^1H NMR (400 MHz, DMSO-d_6) δ: 1.22 (t, Me, 6H, $^3J_{HH}$ 7.2 Hz), 3.46 (m, 4H, NCH_2, $^3J_{HH}$ 6.4 Hz), 3.13 (br. m, 1H, NH), 4.97 (d, 1H, PCH, $^2J_{PCH}$ 17.3 Hz), 7.14 [ddd, 1H, H(6), $^3J_{HH}$ 8.1 and 6.2 Hz, $^4J_{HH}$ 2.3 Hz], 7.46 [m, H(7), H(8)], 7.70 [br. d, 1H, H(5), $^3J_{HH}$ 7.4 Hz], 11.41 (ws, 1H, COOH). ^{31}P NMR (161.9 MHz, DMSO-d_6) δ: 3.0 (d, $^2J_{HCP}$ 20.0 Hz). Found (%): C, 47.54; H, 3.87; P, 6.72. Calc. for $C_{18}H_{17}F_5NO_5P$ (%): C, 47.68; H, 3.75; P, 6.84. For ^{13}C NMR spectrum of compound **4a**, see Online Supplementary Materials.

attributed to the anhydride carbonyl group C(O)OP. In the ^{1}H NMR spectra (DMSO-d_6) of freshly prepared crystalline substances **3** a doublet ($^2J_{PCH}$ 28.0–29.0 Hz) in the region of 5.43–5.48 ppm, which corresponds to the P–CH fragment of the main diastereomer, is observed.

The predominant diastereomers of compounds **3** were isolated in a crystalline form, and their structures were determined by X-ray diffraction analysis.‡ Figures 1 and 2 show the molecular geometries in crystals and specify selected bond lengths and bond and torsion angles.

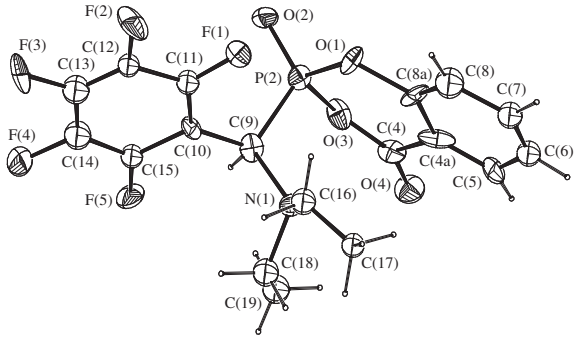


Figure 1 Molecular structure of compound **3a** (thermal ellipsoids are shown with an oscillation probability of 30%). Selected bond lengths (Å) and bond and torsion angles (°): P(2)–O(1) 1.561(9), P(2)–O(2) 1.44(1), P(2)–O(3) 1.637(9), P(2)–C(9) 1.78(1), O(3)–C(4) 1.32(2), O(4)–C(4) 1.18(2), C(4a)–C(8a) 1.39(2), O(1)–P(2)–O(2) 113.2(6), O(1)–P(2)–O(3) 103.3(5), O(1)–P(2)–C(9) 110.3(6), O(2)–P(2)–O(3) 111.8(6), O(2)–P(2)–C(9) 115.7(6), O(3)–P(2)–C(9) 101.2(6), P(2)–O(1)–C(8a) 123.7(8), P(2)–O(3)–C(4) 127.1(8), O(2)–P(2)–C(9)–N(1) –178.7(1), O(2)–P(2)–C(9)–C(10) –33(1).

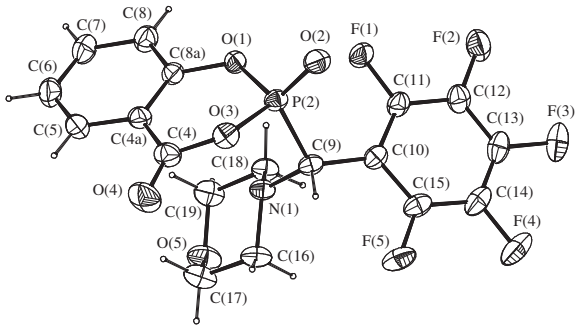


Figure 2 Molecular structure of compound **3b** (thermal ellipsoids are shown with an oscillation probability of 30%). Selected bond lengths (Å) and bond and torsion angles (°): P(2)–O(1) 1.588(2), P(2)–O(2) 1.445(2), P(2)–O(3) 1.593(2), P(2)–C(9) 1.818(2), O(1)–C(8a) 1.389(3), O(3)–C(4) 1.389(4), O(4)–C(4) 1.185(3), C(4)–C(4a) 1.461(3), C(4a)–C(8a) 1.383(3), N(1)–C(9) 1.467(3), C(9)–C(10) 1.511(3), O(1)–P(2)–O(2) 112.24(9), O(1)–P(2)–O(3) 103.70(8), O(1)–P(2)–C(9) 109.0(1), O(2)–P(2)–O(3) 113.5(1), O(2)–P(2)–C(9) 117.2(1), O(3)–P(2)–C(9) 99.83(9), P(2)–O(1)–C(8a) 124.5(1), P(2)–O(3)–C(4) 125.9(2), O(2)–P(2)–C(9)–N(1) –170.4(1), O(2)–P(2)–C(9)–C(10) –34.1(2).

The phosphorus atoms of both molecules have a distorted tetrahedral configuration. The configuration of chiral atoms is $P(2)_RC(9)_R/P(2)_SC(9)_S$ (**3a**,**b**). The phosphorus heterocycles are noticeably flattened, and they have a sofa conformation [the atoms P(2)O(1)C(8a)C(4a)C(4) are arranged approximately in a plane to within 0.07(1) Å (**3a**) and 0.034(2) Å (**3b**), and the O(3) atom is deviated from the plane by 0.28(1) and –0.311(2) Å, respectively]; the torsion angle P(2)O(3)C(4)C(4a) is –21(1)° (**3a**) or –16.9(2)° (**3b**). The carbonyl group and the *o*-phenylene fragment occur approximately in a plane [the torsion angle O(4)C(4)C(4a)C(8a) is –179(1)° (**3a**) or –172.1(3)° (**3b**)], which is favourable for conjugation between them. The phosphoryl group in compounds **3a**,**b** occupies a pseudo-equatorial position [it is deviated from the plane P(2)O(1)C(8a)C(4a)C(4) by 0.842(8) or –0.881(2) Å, respectively], whereas the *N*,*N*-diethylamino(morpholino)pentafluorophenylmethyl substituent occurs in a pseudo-axial position [the C(9) atom is deviated from the plane P(2)O(1)C(8a)C(4a)C(4) by –1.77(1) (**3a**) or 1.765(2) Å (**3b**)]. In both of the molecules, the diethylamide substituent hangs over the oxaphosphinine heterocycle.

Reaction of compound **1b** *with pentafluorobenzaldehyde*. A solution of pentafluorobenzaldehyde (5.42 g, 0.027 mol) in 5 ml of CH_2Cl_2 was added to a solution of compound **1b** (6.99 g, 0.027 mol) in 10 ml of CH_2Cl_2. The reaction mixture was heated for 2 h (40 °C). The solution was evaporated to a half volume, diluted with 15 ml of diethyl ether and allowed to stand at 0 °C for two months. The resulting crystals of 2-[(morpholino)(pentafluorophenyl)methyl]benzo[*e*]-1,3,2-dioxaphosphinine-2,4-dione **3b** were washed with dry diethyl ether and dried *in vacuo*. Yield, 80%; mp 163–166 °C (main diastereomer). ^{1}H NMR (300 MHz, DMSO-d_6) δ: 5.48 (d, PCH, $^2J_{PCH}$ 28.0 Hz) and 5.43 (d, PCH, $^2J_{PCH}$ 28.0 Hz) in a ratio of 2 : 3. ^{31}P NMR (121.42 MHz, CH_2Cl_2) δ: 15.4 (d, $^2J_{PCH}$ 28.0 Hz) and 15.1 (d, $^2J_{PCH}$ 28.0 Hz) in a ratio of 2 : 3. MS, *m*/*z*: 449 $[M]^{\bullet+}$. IR (ν/cm^{-1}): 1720, 1680, 1605, 1580, 1520, 1460, 1385, 1280, 1240, 1140, 1120, 1080, 1005, 985, 960, 910, 890, 690, 650. Found (%): C, 48.19; H, 3.11; P, 6.27. Calc. for $C_{18}H_{15}F_5NO_4P$ (%): C, 48.11; H, 2.90; P, 6.60.

Compound **3b** was ground in wet acetone; in this case, the immediate formation of a snow-white precipitate occurred. This precipitate was filtered off, washed with diethyl ether and dried *in vacuo*. The yield of 2-carboxyphenyl[(morpholino)(pentafluorophenyl)methyl]phosphonate **4b** was 85%; mp 198 °C. IR (KBr pellet, ν/cm^{-1}): 3432, 2993, 2953, 2903, 2859, 2675–2750, 2575–2590, 2502, 2468, 2261, 2128, 1708, 1658, 1632, 1600, 1580, 1513, 1485, 1455, 1446, 1390, 1559, 1303, 1261, 1229, 1161, 1125, 1083, 1049, 1027, 998, 982, 954, 915, 883, 822, 799, 794, 724, 667, 641, 553, 517, 495, 470, 441, 415. ^{1}H NMR (300 MHz, DMSO-d_6) δ: 3.20 (br. m, 4H, NCH_2), 3.61 (br. m, 1H, NH), 3.74 (br. m, 4H, OCH_2), 5.00 (d, 1H, PCH, $^2J_{PCH}$ 20.0), 7.14 [m, 1H, H(6)], 7.46 [m, 2H, H(8), H(7)], 7.68 [d, 1H, H(5), $^3J_{HH}$ 7.6 Hz], 11.40 (ws, 1H, COOH). ^{19}F NMR (282.4 MHz, DMSO-d_6) δ: –145.77 [br. m, 1F, F(3)], –153.86 [br. m, 2F, F(1)], –162.55 [br. m, 2F, F(2)]. ^{31}P NMR (121.42 MHz, DMSO-d_6) δ: 4.5 (d, $^2J_{HCP}$ 19.0 Hz). Found (%): C, 46.07; H, 3.41; P, 6.72. Calc. for $C_{18}H_{15}F_5NO_4P$ (%): C, 46.25; H, 3.21; P, 6.64.

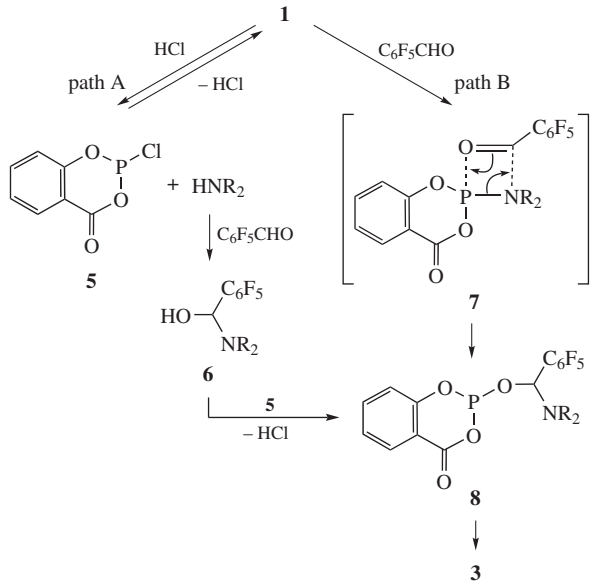
For ^{13}C NMR spectrum of compound **4b**, see Online Supplementary Materials.

‡ *Crystal data*. The X-ray data for the crystals of compound **3a** ($C_{18}H_{15}F_5NO_4P$) were collected on a CAD-4 Enraf-Nonius automatic diffractometer [graphite monochromator, λ(MoKα) = 0.71073 Å; $\omega/2\theta$-scanning]. At 293 K: crystals are monoclinic, space group $P2_1/c$, a = 8.182(4), b = 13.879(4) and c = 16.422(5) Å, β = 90.26(1)°, V = 1864.9(12) Å^3, Z = 4, d_{calc} = 1.55 g cm^{-3}, μ(MoKα) = 2.22 cm^{-1}, F(000) = 888, 0° ⩽ θ ⩽ 26.3°, R_{int} = 0.071. 3872 reflections were measured, 1017 of which were independent, 1002 reflections with $I > 2\sigma(I)$. The final R indexes are R = = 0.114, R_w = 0.110, GOF = 2.88, the number of refined parameters is 202. Bad data was observed because of a small and weak crystal. Empirical absorption correction was not applied.

X-ray diffraction analysis of the crystals of compound **3b** ($C_{18}H_{13}F_5NO_5P$) was performed on a Bruker Smart APEX II CCD automatic diffractometer [graphite monochromator, λ(MoKα) = 0.71073 Å, ω-scanning]. At 293 K: crystals are monoclinic, space group C_2/c, a = 20.922(1), b = 13.667(1) and c = 16.162(1) Å, β = 128.161(1)°, V = 2058.5(6) Å^3, Z = 8, d_{calc} = = 1.642 g cm^{-3}, μ(MoKα) = 2.34 cm^{-1}, F(000) = 1824, 1.9° ⩽ θ ⩽ 27.00°, R_{int} = 0.042; 24 065 reflections were measured, 3970 of which were independent, 2839 reflections with $I > 2\sigma(I)$. The final R indexes are R = = 0.0377, R_w = 0.0880, R_{all} = 0.0603, $R_{w\,all}$ = 0.1006, GOF = 1.05, the number of refined parameters is 271. An absorption correction was performed using the SADABS program.[17]

The structures were solved by a direct method using the SIR program[18] and refined by the full matrix least-squares using the SHELXL-97 program.[19] Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed into the geometrically calculated positions and refined as riding atoms. Calculations were performed using the WinGX[20] program. Figures and analysis of intermolecular interactions were performed using the PLATON[22] program.

CCDC 936015 and 936016 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2013.



Scheme 2

Thus, unlike reactions with chloral, bromal and ethyl trifluoropyruvate, the interaction of salicylamidophosphites with pentafluorobenzaldehyde results in unusual compounds, 2-(dialkylaminopentafluorophenyl)methyl-5,6-benzo-1,3,2-dioxaphosphinine-2,4-diones **3**, which can be formed in accordance with two hypothetic reaction pathways (Scheme 2). According to reaction path A, catalysis with the participation of acidic impurities (amine hydrochlorides), which can be in an amidophosphite, can occur. In this case, it is likely that the P–N bond is cleaved with the formation of chlorophosphite **5** and an amine. This compound can be detected based on a characteristic value of δ_P 146.3 ppm in the $^{31}P-\{^1H\}$ NMR spectrum of the reaction mixture. The amine is readily added to pentafluorobenzaldehyde with the intermediate formation of semiaminal **6**, which, in turn, readily forms phosphite **8**. It is likely that two low-intensity signals with δ_P 123.9 and 124.2 ppm, which disappeared upon the completion of interaction, in the $^{31}P-\{^1H\}$ NMR spectrum correspond to this phosphite. This compound contains two acceptor substituents and readily undergoes the Arbuzov rearrangement to furnish the end product **3**.

According to pathway B, the insertion reaction can be represented as a concerted process, which occurs through four-membered transition state **7** with the simultaneous formation of C–N and P–O bonds and the cleavage of P–N and C=O bonds. Intermediate phosphite **8** is rearranged into reaction product **3**, as in reaction pathway A. It is difficult to prefer either of these reaction paths.

Compounds **3** are insoluble in a number of organic solvents including CCl_4, $CHCl_3$ and C_6H_6 and very prone to hydrolysis because they contain the macroergic anhydride bond P–O–C(O). Thus, very rapid ring opening and the formation of salts **4**, which can occur as tautomeric species **A** and **B**, takes place on the dissolution of compounds **3** in DMSO without moisture protection.

In the $^{13}C-\{^1H\}$ NMR spectrum of compound **4a**, the C(9) atom (PCH) has a characteristic chemical shift δ_C 55.11 ppm (d, $^1J_{PC}$ ~135 Hz). The *ipso*-carbon atoms C(4a) and C(8a) manifest themselves as doublets with δ_C 124.30 and 150.37 ppm and coupling constants $^3J_{POCC(4a)}$ 5.5 Hz and $^2J_{POC(8a)}$ 7.5 Hz, respectively.

The $^{13}C-\{^1H\}$ NMR spectrum of phosphonate **4b** exhibits a doublet with δ_C 42.08 ppm ($^3J_{PCNC(16)}$ 4.9 Hz), which corresponds to C(16) in the morpholine ring. The carbon of the fragment PC(9)H reveals itself at δ_C 48.56 ppm ($^1J_{PC(9)}$ 142.0 Hz). Signals due to the *ipso*-carbons C(4a), C(8a), and C(10) also manifest themselves as doublets. IR ($\nu_{C=O}$ 1680–1685, ν_{COO-H} 3400 cm^{-1}), ^{13}C NMR (δ_C 167.2–167.6 ppm) and ^1H NMR (δ_{COOH} 11.4 ppm) spectroscopic data suggest the occurrence of betaine form (**4A**) with an uncharged carboxyl group in a crystal.

Thus, the reaction of salicylamidophosphites with pentafluorobenzaldehyde unexpectedly leads to the formation of compounds with the retention of a dioxaphosphinine ring – corresponding 2-[(dialkylamino)(pentafluorophenyl)methyl]benzo[*e*]-1,3,2-dioxaphosphinine-2,4-diones – with high regio- and stereoselectivity; this process includes an unusual O,N-exchange reaction.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.mencom.2013.05.018.

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