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Unusual reaction of 8-chloro-2-cyclohexyl-4-phenylbenzo[*e*]-1,2-oxaphosphinine-2-oxide with tetramethylenebis(magnesium bromide)

Dmitry A. Tatarinov,[a,b] Vladimir F. Mironov,*[a,b] Elena N. Varaksina,[a] Dmitry B. Krivolapov,[a] Igor A. Litvinov,[a] Rashid Z. Musin,[a] Boris I. Buzykin[a] and Alexander I. Konovalov[a,b]

[a] *A. E. Arbuzov Institute of Organic and Physical Chemistry, Kazan Scientific Centre of the Russian Academy of Sciences, 420088 Kazan, Russian Federation. Fax: +7 843 275 5322; e-mail: mironov@iopc.knc.ru*
[b] *A. M. Butlerov Chemical Institute, Kazan State University, 420008 Kazan, Russian Federation*

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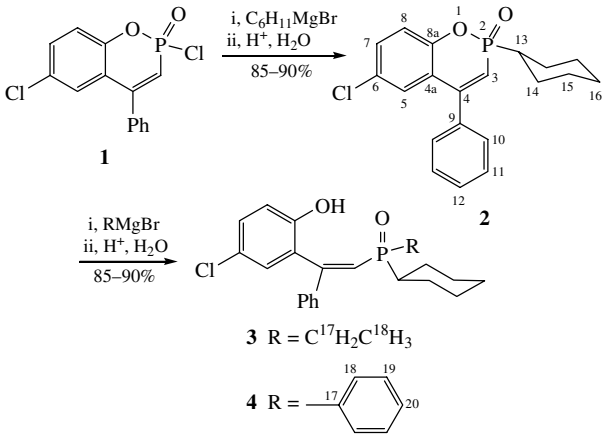
The reaction of 8-chloro-2-cyclohexyl-4-phenylbenzo[*e*]-1,2-oxaphosphinine-2-oxide with ethyl- and phenylmagnesium bromides and tetramethylenebis(magnesium bromide) is a versatile approach to the synthesis of unsymmetrical phosphine oxides. With the latter Grignard reagent, an unusual phosphorus–carbon cleavage also occurs to yield a pentacoordinate phosphorus derivative, 2,3,8,9-bis(4-chlorobenzo)-6-cyclohexyl-6,9-diphenyl-1,7-dioxa-6-phosphaspiro[5.5]deca-1,9-diene.

Achiral ($R_3P=O$), prochiral ($R_2R^1P=O$) and chiral ($R^1R^2R^3P=O$) phosphine oxides are the main precursors in the syntheses of corresponding phosphines,[1–11] which are widely used as ligands in metal complex catalysts[2,4,6,10,12] and neutral extracting agents for rare-earth metals.[13–17] Phosphine oxides can also be used as ligands in reactions catalysed by palladium and molybdenum complexes.[18–20] The syntheses of phosphine oxides include reactions of organolithium and organomagnesium compounds with acyclic derivatives of tetracoordinate phosphorus containing alkoxy groups or chlorine atoms[21–23] or addition of hydrophosphoryl compounds to C=C bonds.[24] Reactions of organolithium and organomagnesium compounds with phosphorus derivatives containing both alkoxy groups and chlorine atoms are characterised by medium or low chemoselectivity. The use of Grignard reagents results in phosphine oxide mixtures, which are difficult to separate.

In this work, we attempted to obtain unsymmetrical phosphine oxides containing dissimilar substituents at the phosphorus atom, *viz*., six-membered phosphorus-containing heterocycles with one phosphorus–oxygen bond, such as 2,8-dichloro-4-phenylbenzo[*e*]-1,2-oxaphosphinine-2-oxide **1**. Compound **1** also contains an active phosphorus–chlorine bond and can be easily obtained by the reaction of phenylacetylene with 2,2,2-trichlorobenzo-1,3,2-dioxaphosphole.[25] Phosphinine **1** selectively reacts with cyclohexylmagnesium bromide to give only 8-chloro-2-cyclohexyl-4-phenylbenzo[*e*]-1,2-oxaphosphinine-2-oxide **2**, in a high yield. Note that introduction of the second cyclohexyl radical does not occur due to the sterical effect of this substituent in the Grignard reagent. The structure of compound **2** was confirmed by NMR spectroscopy and mass spectrometry.† Derivative **2** containing a chiral phosphorus atom is capable of reacting with Grignard reagents obtained from non-branched alkyl halides or aryl halides; this reaction affords phosphine oxides **3**, **4** with three different substituents through the cleavage of the oxaphosphinine heterocycle (Scheme 1).†

The reaction of 8-chloro-2-cyclohexyl-4-phenylbenzo[*e*]-1,2-oxaphosphinine-2-oxide with tetramethylenebis(magnesium bromide) is unusual. The formation of diphosphine oxide derivative **5** is not the major pathway of the reaction (Scheme 2).‡ The main pathway is the unexpected formation of spirophosphorane derivative **9** (its content is 47% in the reaction mixture after hydrolysis). We failed to find data on a similar behaviour of Grignard reagents toward phosphorus derivatives, though examples of sulfur–carbon bonds cleavage were reported.[26]



Scheme 1

Scheme 2 shows a possible pathway for the formation of this compound, which involves a reaction of phosphinine oxide **2** with an organomagnesium compound to yield organodimagnesium derivative **6** or its cyclic form **6a** (examples of similar organodimagnesium derivatives are known[27]). A similar reaction may be considered as the ligand replacement of the basic radical by more acidic one at a magnesium atom. This process should also give a molecule of 2-cyclohexyltetrahydrophosphole-2-oxide **7**, which has not been isolated. However, its presence follows from the chemical shift of the corresponding signal in the ^{31}P NMR spectrum (δ_P 78.2 ppm) of the reaction mixture, which agrees with the corresponding values for similar compounds, containing

† Melting points (uncorrected) were measured with a Boetius melting point apparatus. NMR spectra were recorded with Bruker Avance-600 (^1H, 600 MHz; ^{13}C, 150.9 MHz), Bruker Avance-400 (^1H, 400 MHz; ^{13}C, 100.6 MHz) and Bruker CXP-100 (^{31}P, 36.48 MHz) spectrometers. The δ_H and δ_P values were determined relative to internal (HMDS) or external (H_3PO_4) standards. The IR spectrum was recorded with a Bruker Vector-22 instrument in Nujol. EI mass spectra were obtained with a TRACE MS Finnigan MAT instrument; the ionization energy was 70 eV and the ion source temperature was 200 °C. The samples were introduced into the ion source *via* a direct inlet system. The evaporating ampoule was heated from 35 to 150 °C at a rate of 35 K min^{-1}. The mass spectrometric data were processed using the Xcalibur program.

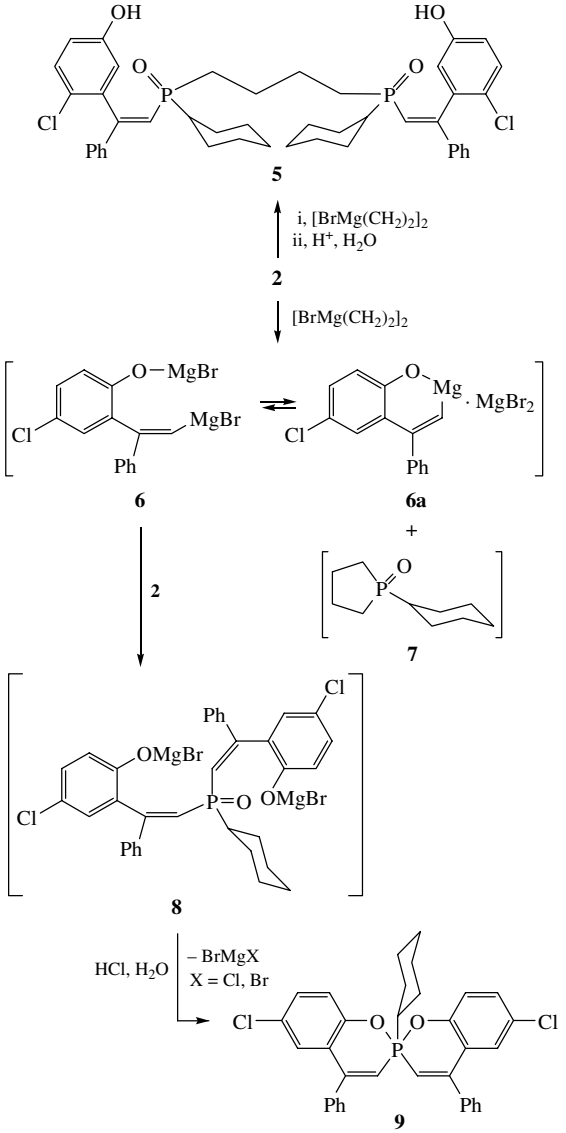
For syntheses and characteristics of compounds **2**–**4** see Online Supplementary Materials.

three P–C bonds.[28] Presumably, organomagnesium compound **6** subsequently reacts with phosphinate **2** to give magnesium-containing phosphine oxide **8**; the signal with δ_P 49.9 ppm (diethyl ether/benzene) in the ^{31}P NMR spectrum, which disappears after treatment of the reaction mixture with water, can be assigned to compound **8**. After water treatment, final compound **9** appears; it has a characteristic peak with δ_P –65.0 ppm (benzene) in the ^{31}P NMR spectrum.

Compound **5** contains two chiral centres and might be expected to form two diastereomers (*d*,*l*- and *mezo*-). However, the signals of its atoms in NMR spectra are broadened, which does not allow us to conclude on the process stereoselectivity. Only one diastereoisomer was isolated after crystallization from $CHCl_3$.

The structures of compounds **5** and **9** were confirmed by NMR spectroscopy, mass spectrometry‡ and single-crystal X-ray diffraction.§ Figures 1 and 2 show the geometry of the molecules.

Diphosphine oxide **5** (*mezo*-form, P_SP_R) was isolated as a solvate with thichloromethane and water (Figure 1). The phosphorus atoms have a slightly distorted tetrahedral configuration. The butylene fragment with two phosphorus atoms is almost planar. The cyclohexyl substituents have a chair conformation. In the crystal of compound **5**, intramolecular OH···O hydrogen bonds between the phosphorus-containing molecules and solvate water were revealed. As a result, infinite hydrogen-bonded



Scheme 2

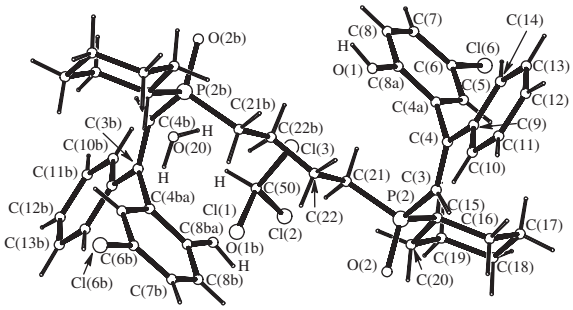


Figure 1 Molecular geometry of compound **5** in a crystal (a solvate with $CHCl_3$ and water). Selected bond lengths (Å): C(3)–C(4) 1.338(5), C(3)–P(2) 1.792(4), C(4)–C(9) 1.483(6), C(4)–C(4a) 1.503(5), C(15)–P(2) 1.801(4), C(21)–C(22) 1.528(6), C(21)–P(2) 1.791(4), C(22)–C(22b) 1.523(8), O(2)–P(2) 1.501(3); bond angles (°): C(4)–C(3)–P(2) 131.6(3), C(9)–C(4)–C(4a) 116.5(3), C(22)–C(21)–P(2) 113.4(3), C(22b)–C(22)–C(21) 112.0(4), O(2)–P(2)–C(21) 111.4(2), O(2)–P(2)–C(3) 109.9(2), C(21)–P(2)–C(3) 107.6(2), O(2)–P(2)–C(15) 111.9(2), C(21)–P(2)–C(15) 106.8(2), C(3)–P(2)–C(15) 109.2(2).

chains are formed along the 0*c* crystal axis. The hydrogen bond parameters are as follows: O(1)–H(1A)···O(20)′ (–*x*, –1/2 + *y*, 3/2 – *z*), *d*(O–H) 0.83(6) Å, *d*(H···O′) 1.88(6) Å, *d*(O···O′) 2.692(5) Å, ∠(O–H–O′) 166(6)°; O(20)–H(201)···O(2)′ (–*x*, 1/2 + *y*, 1/2 – *z*), *d*(O–H) 0.86(5) A, *d*(H···O′) 1.93(5) Å, *d*(O···O′) 2.777(4) Å, ∠(O–H–O′) 174(8)°; O(20)–H(202)···O(2)′ (*x*, 1/2 – *y*, 1/2 + *z*), *d*(O–H) 0.9(1) Å, *d*(H···O′) 2.02(11) Å, *d*(O···O') 2.853(5) Å, ∠(O–H–O′) 163(8)°.

The phosphorus atom in **9** (a solvate with two benzene molecules, Figure 2) has an almost regular trigonal-bipyramidal

‡ *1,10-Bis(5-chloro-2-hydroxyphenyl)-3,8-bis(cyclohexyl)-1,10-diphenyl-3,8-diphosphadeca-1,9-diene-3,8-dione* **5**. The Grignard reagent obtained from magnesium (0.48 g, 0.02 mol) and 1,4-dibromobutane (1.2 ml, 0.01 mol) in diethyl ether (15 ml) was added dropwise to a heated solution (78 °C) of compound **2** (1.8 g, 0.005 mol) in benzene (10 ml). The resulting reaction mixture was refluxed for 3 h (78 °C), cooled to 20 °C and treated with water, hydrochloric acid solution (5 ml in 20 ml of water) to pH 4.0 and water. The aqueous and organic layers were separated and the organic layer was dried *in vacuo* (12 Torr, 80 °C). The resulting viscous oil was twice extracted with diethyl ether. The ether extract was separated. The residue was crystallized from chloroform. The crystalline precipitate that formed was filtered off and dried *in vacuo* (12 Torr, 100 °C). The yield of compound **5** (as a solvate with chloroform and water, 1:1:1) was 0.83 g (42%), mp 125–128 °C. ^1H NMR (400 MHz, $CDCl_3$, 25 °C) δ: 6.32 [br. d, H(3), $^2J_{PCH}$ 15.8 Hz], 6.92 [br. d, H(5), $^4J_{H(7)CCCH(5)}$ 2.3 Hz], 7.23 [br. dd, 1H, H(7), $^3J_{H(8)CCH(7)}$ 8.9 Hz, $^4J_{H(5)CCCH(7)}$ 2.3 Hz], 7.05 [br. d, H(8), $^3J_{H(7)CCH(8)}$ 8.9 Hz], 7.29, 7.35 (2m, Ph), 7.38 (s, $CHCl_3$), 1.92, 1.84, 1.73, 1.54, 1.36, 1.24–1.25 (6br. m, C_6H_{11}). ^{31}P-{^1H} NMR (36.5 MHz, [$^2H_6$]acetone) δ_P: 45.1 (br. s). IR (ν/cm^{-1}): 3380 (very br., OH), 2722 (very br.), 1588, 1568, 1495, 1413, 1330, 1281, 1219, 1115-1120, 1031, 942, 917, 890, 846, 823, 766, 723, 692, 633, 554, 517, 494, 452. MS, *m*/*z*: 774 $[M]^{+\cdot}$, 757 $[M – OH]^+$, 756 $[M - H_2O]^+$, 739 $[M – Cl]^+$, 721 $[M – H_2O – Cl]^+$, 673 $[M – H_2O – C_6H_{11}]^+$, 528 $[C_{30}H_{39}ClO_2P]^+$, 445 $[C_{24}H_{28}ClO_2P]^+$, 228 $[C_{14}H_9ClO]^+$, 212 $[C_{14}H_9Cl]^+$, 83 $[C_6H_{11}]^+$, 55 $[C_4H_7]^+$, 41 $[C_3H_5]^+$.

2,3,8,9-Bis(4-chlorobenzo)-6-cyclohexyl-6,9-diphenyl-1,7-dioxa-6-phosphaspiro[5.5]deca-1,9-diene **9**. The ethereal extract from the synthesis of compound **5** was evaporated and the resulting viscous residue was crystallized from benzene. The yield of compound **9** (as a solvate with two benzene molecules) was 0.3 g (21%), mp 75 °C. ^1H NMR (400 MHz, $CDCl_3$, 25 °C) δ: 7.39–7.41 [m, H(11), H(12)], 7.34 [dd, H(7), $^3J_{H(8)CCH(7)}$ 8.7 Hz, $^4J_{H(5)CCCH(7)}$ 2.6 Hz], 7.10 [m, H(10)], 7.07 [d, H(5), $^4J_{H(7)CCCH(5)}$ 2.6 Hz], 7.02 [d, H(8), $^3J_{H(7)CCH(8)}$ 8.7 Hz], 5.88 [d, H(3), $^2J_{PCH(3)}$ 25.2 Hz], 2.68 [dtt, H(15), $^2J_{PCH(15)}$ 12.7 Hz, $^3J_{H(16ax)CCH(15)}$ 12.3–12.5 Hz, $^3J_{H(16eq)CCH(15)}$ 2.9–3.0 Hz], 2.06 [dtdd, H(18ax), $^2J_{H(18eq)CH(18ax)}$ 12.6 Hz, $^3J_{H(17ax)CCH(18ax)}$ 12.6 Hz, $^3J_{H(17eq)CCH(18ax)}$ 4.0 Hz, $^3J_{H(19eq)CCH(18ax)}$ 4.3 Hz], 2.30, 1.91, 1.77–1.79, 1.35–1.45 [4m, H(16), H(17), H(19), H(20)]. ^{31}P-{^1H} NMR (36.5 MHz, $CDCl_3$) δ_P: –65.0 (s). For ^{13}C NMR, IR and mass spectra of **9** see Online Supplementary Materials.

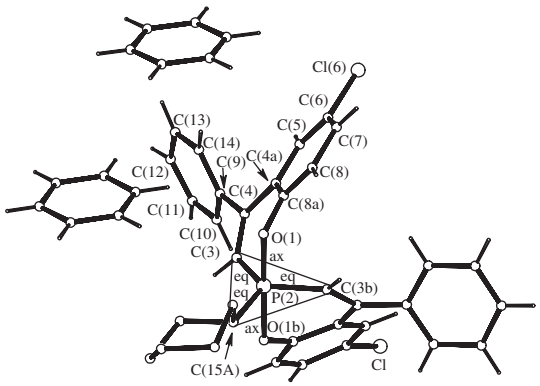


Figure 2 Molecular geometry of compound **9** in a crystal (a solvate with two benzene molecules) (the base of the trigonal bipyramid is shown by thin lines, the disordered cyclohexyl substituent is shown in one of the two possible positions only). Selected bond lengths (Å): P(2)–O(1) 1.778(2), P(2)–C(3) 1.783(3), P(2)–C(15A) 1.801(6), P(2)–O(1b) 1.778(2), P(2)–C(3b) 1.783(3), O(1)–C(8a) 1.360(4), C(3)–C(4) 1.347(4), C(4)–C(4a) 1.487(4); bond angles (°): O(1)–P(2)–C(3) 90.8(1), O(1)–P(2)–C(15A) 90.03(7), O(1)–P(2)–O(1b) 179.8(1), O(1)–P(2)–C(3b) 89.2(1), C(3)–P(2)–C(15A) 118.2(1), O(1b)–P(2)–C(3) 89.2(1), C(3)–P(2)–C(3b) 123.7(2), O(1b)–P(2)–C(15A) 90.03(7), C(3b)–P(2)–C(15A) 118.2(1), O(1b)–P(2)–C(3b) 90.8(1), P(2)–O(1)–C(8a) 118.3(2), P(2)–C(3)–C(4) 124.9(2), C(3)–C(4)–C(4a) 119.7(3); torsion angles (°): O(1)–P(2)–C(3)–C(4) 37.1(3), P(2)–O(1)–C(8a)–C(4a) 43.7(4), P(2)–O(1)–C(8a)–C(8) –136.9(3), C(3)–C(4)–C(4a)–C(8a) –22.0(5), C(3)–C(4)–C(9)–C(10) –44.5(5), C(9)–C(4)–C(4a)–C(5) –24.8(5), C(4a)–C(4)–C(9)–C(14) –46.7(4).

configuration. The base of the trigonal bipyramid lies in the C(3)C(3b)C(15A)P(2) plane, and the O(1) and O(1b) atoms occupy apical positions [the bond lengths P(2)–O(1) and P(2)–O(1b) are 1.778(2) Å]. The bond angles O(1)P(2)C(3), O(1)P(2)C(15A), O(1)P(2)C(3b), O(1b)P(2)C(3b), O(1b)P(2)-C(15A) and O(1b)P(2)C(3) are within 89.2(1)–90.03(7)°; and the O(1)P(2)O(1b) bond angle equals 179.8(1)°. The sum of the C(3)P(2)C(3b), C(3)P(2)C(15A) and C(3b)P(2)C(15A) bond angles at the base of the bipyramid amounts to 360.1(2)°. The oxaphosphinine rings have a distorted boat conformation containing two planar fragments, *viz.*, O(1)C(8a)C(4a)C(4) and C(4a)C(4)C(3)P(2) [the P(2) and C(3) atoms deviate by 1.1152(4) and 0.411(3) Å from the first plane, which is planar to within 0.008(3) Å; the O(1) and C(8a) atoms deviate by –0.985(2) and –0.545(3) Å from the second plane, which is planar to within 0.021(3) Å].

Online Supplemetary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2008.05.012.

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§ *X-ray diffraction data for compounds* **5** *and* **9** were collected at 20 °C using Bruker Kappa Apex2 (graphite-monochromator CuKα radiation for **5**) and Bruker Smart Apex2 (graphite-monochromator MoKα radiation for **9**) diffractometers. The images were indexed, integrated and scaled using the APEX2 data reduction package.[29] All raw data were corrected for absorption using the SADABS[30] program. The structures were solved by the direct method using the SHELXS[31] program and refinement was carried out with SHELXL[32] using anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were added to the structure model at calculated positions and refined as fixed atoms. The hydrogen atoms of OH groups for compound **5** were included from $\Delta\rho$ maps and refined isotropically. All calculations were performed on a PC using the WinGX[33] program. All figures were made using the PLATON program.[34]

Compound **5**: $C_{44}H_{50}Cl_2O_4P_2{\cdot}2CHCl_3{\cdot}2H_2O$, $M_r = 1050.45$ g mol^{-1}, monoclinic, space group $P2_1/c$ (molecule in a special position), $a = 13.8864(5)$, $b = 18.6026(7)$ and $c = 9.8699(4)$ Å, $\beta = 92.673(3)°$, $V = 2546.85(17)$ Å^3, $Z = 2$, $d_{calc} = 1.37$ g cm^{-3}, μ(CuKα) = 5.000 mm^{-1}, reflections: 14098 collected, 3908 unique, $R_{int} = 0.0653$, $R_1 = 0.0635$, $wR_2 = 0.1563$ for 2293 independent reflections with $F^2 \geq 4\sigma$ (GOOF = 1.018). The thichloromethane solvent in the crystal is disordered.

Compound **9**: $C_{34}H_{29}Cl_2O_2P{\cdot}2C_6H_6$, $M_r = 716.57$ g mol^{-1}, monoclinic, space group $C2/c$ (the molecule is in a special position on axis 2), $a = 15.2147(13)$, $b = 10.4610(9)$ and $c = 24.038(2)$ Å, $\beta = 92.118(1)°$, $V = 3823.3(6)$ Å^3, $Z = 4$, $d_{calc} = 1.245$ g cm^{-3}, μ(MoKα) = 0.249 mm^{-1}, reflections: 8340 collected, 4093 unique, $R_{int} = 0.0496$, $R_1 = 0.0577$, $wR_2 = 0.1519$ for 1991 independent reflections with $F^2 \geq 4\sigma$ (GOOF = 0.876). Cyclohexyl groups in molecule are disordered; they were refined isotropically.

CCDC 665576 and 665577 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, 2008.