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Microwave-assisted catalyst-free addition of secondary phosphines to fullerene C_{60}

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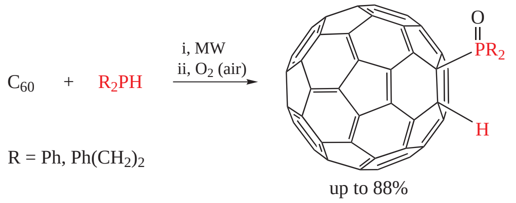
Secondary phosphines, R_2PH ($R = Ph, PhCH_2CH_2$), react with fullerene C_{60} under microwave irradiation at 200–270 °C for 1–2 h in the medium of substituted naphthalenes (1-Cl, 1-Me, 1-MeO) or without solvent to afford mono-adducts, $HC_{60}PR_2$, whose air oxidation gives the corresponding phosphine oxides, $HC_{60}P(O)R_2$, in up to 88% yield.



Over the last decades, functionalized fullerenes became a subject of sustainable interest due to their multifaceted potential for application in hi-tech materials,[1] and especially in nanotechnologies.[2,3] Despite the ever-growing stream of publications in this area, the papers devoted to phosphorus-containing derivatives of fullerenes occupy the modest place.[3,4] Among the pioneering syntheses of fullerenes bearing phosphorus-containing functionalities are those dealing with 1,3-cycloaddition reactions of phosphorylated nitrile oxides, azides,[5] and diazo compounds[6] to fullerene. The first synthesis of fullerenes with the C–P bond comprised addition of lithiated phosphine-borane ($Ph_2PLi\cdot BH_3$) to C_{60} followed by treatment with HCl and then with DABCO.[7] Addition of secondary phosphine oxides to fullerene in 20% DMSO–PhCl mixture at room temperature gives the adducts in 31–60% yield. Of particular interest was that DMSO played a role of the specific catalyst of this reaction, since in neat PhCl the reaction did not occur at all. The radical reaction of C_{60} with phosphonates or $Ph_2P(O)H$ mediated by manganese(III) acetate afforded diverse C–P phosphorylated fullerenes.[9] As for secondary phosphines, to the best of our knowledge, no unambiguous data related to the addition to C_{60} were published. Moreover, it was noted[7] that Ph_2PH did not react with C_{60} under heating in boiling toluene. This communication is the concise report of our investigation concerning the microwave-assisted (MW) addition of secondary phosphines to fullerene C_{60}.

In our experiments, secondary phosphines **1a**,**b** react with C_{60} under MW irradiation (200–270 °C, 1–2 h, in the medium of 1-chloro-, 1-methyl-, 1-methoxynaphthalenes or without solvent) to deliver tertiary phosphines **2a**,**b** (^{31}P NMR data, Figure S1, see Online Supplementary Materials), which are easily oxidized in the presence of air to the corresponding phosphine oxides **3a**,**b** (Scheme 1, Table 1).† The highest yield (88%) of adduct **3a** was achieved, when the initial reagents (C_{60} and phosphine **1a** in a molar ratio of 1 : 1.7) were MW-irradiated at 270 °C for 1 h in 1-methoxynaphthalene (1-MeONp); C_{60} conversion was 64% (Table 1, entry 1). Without MW-assistance, efficiency of this reaction was significantly lower (entry 2). The yield of phosphine oxide **3a** and C_{60} conversion were also reduced, when 1-chloronaphthalene (1-ClNp) and 1-methylnaphthalene (1-MeNp) were used as solvents as well as at lower temperatures (entries 3–6). Note that our attempt to implement phosphination of C_{60} with secondary phosphine **1b** under the best conditions of adduct **3a** synthesis (entry 1) failed. In this case, no expected mono-adduct **3b** was formed (entry 7). However, the latter was obtained in 18% yield, when fullerene C_{60} was MW-irradiated in the medium of phosphine **1b** (molar ratio C_{60} : **1b** = 1 : 64). In this experiment, apart from phosphine oxide **3b**, a mixture of poly-adducts was also formed (entry 8).‡

The products **3a**,**b** were characterized by ^1H, ^{13}C, ^{31}P NMR, UV-VIS, FT-IR spectroscopy and MALDI-TOF mass spectro-

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† Fullerene C_{60} (> 99%) was purchased from 'High-tech industry' (St. Petersburg, Russia) and used without further purification. 1-Methylnaphthalene, 1-chloronaphthalene (Alfa Aesar) and 1-methoxynaphthalene were distilled, deaerated *in vacuo* and saturated with Ar prior to use. All reactions were performed under dry argon atmosphere.

Diphenyl(C_{60}H)phosphine oxide **3a** (*general procedure*). The mixture of Ph_2PH and the solution of C_{60} (100 mg, 139 μmol) in degassed solvent was MW-irradiated (Monowave 300W Anton Paar reactor, mean value 30–38 W power of irradiation) for a specified period (Table 1) under Ar atmosphere in a closed vial (10 ml). After irradiation, the mixture was concentrated *in vacuo*, put onto a silica gel column (Alfa Aesar, 70–230 mesh), and eluted with CS_2–toluene (1 : 9) for C_{60} recovery, then with EtOAc–PhMe (1 : 9 up to 1 : 1) to afford product **3a** as a brown powder, yield 72 mg (56%). ^1H NMR (400.1 MHz, CS_2–$CDCl_3$) δ: 7.10 (d, 1H, HC_{60}, ^3J 25.7 Hz), 7.63–7.71 (m, 6 H), 8.50–8.55 (m, 4 H). ^{13}C NMR (100.6 MHz, CS_2–$CDCl_3$) δ: 57.03 [d, 1C, $HC(C_{60})$, ^2J 2.4 Hz], 70.56 (d, 1C, PC_{60}, ^1J 67.2 Hz), 128.82 (d, 4 C, Ph, ^2J 12.0 Hz), 129.03 (d, 2 C, Ph, ^1J 98.6 Hz), 132.94 (d, 2 C, Ph, ^4J 2.0 Hz), 133.11 (d, 4 C, Ph, ^3J 8.4 Hz); fullerene moiety: 135.55 (C_{60}), 137.11 (d, C_{60}, ^3J 4.9 Hz), 139.59, 140.40, 141.36, 141.40, 141.42, 141.66, 141.77, 141.99, 142.02, 142.47, 142.62, 143.20, 144.22, 144.66, 145.34, 145.39, 145.51, 145.72, 146.19, 146.26, 146.33, 146.42, 146.79, 147.12, 147.22 (all C_{60}, 2 C), 149.34 (d, 2 C, C_{60}, ^2J 6.9 Hz), 152.33 (d, 2 C, C_{60}, ^3J 4.6 Hz). ^{31}P NMR (161.98 MHz, CS_2–$CDCl_3$) δ: 34.18 (s). IR (ν/cm^{-1}): 530, 556, 619, 696, 729, 751, 848, 1030, 1041, 1071, 1098, 1117, 1129, 1182, 1197, 1287, 1382, 1437, 1456, 1597, 1704, 1724, 2855, 2925, 2955, 3058. UV (hexane, λ/nm): 224, 256, 326, 404, 431. Found (%): C, 93.64; P, 3.28. Calc. for $C_{72}H_{11}OP$ (%): C, 93.71; P, 3.36. In some cases, soluble oligo-adducts could be isolated (eluent 20% MeOH in $CHCl_3$).

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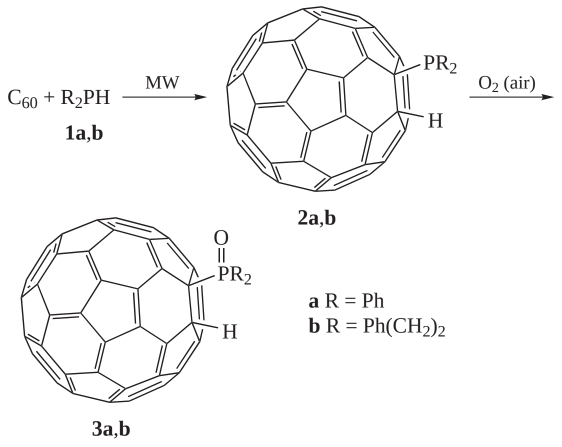


Scheme 1

Table 1 Phosphination of C_{60} with secondary phosphines under MW.[a]

Entry	R_2PH	Molar ratio C_{60} : **1**	Solvent (volume/ml)	T/°C	t/h	Conversion of C_{60} (%)	Product	Yield (%)[b]
1	**1a**	1 : 1.7	1-MeONp (5.0)	270	1	64	**3a**	56 (88)
2[c]	**1a**	1 : 2	1-MeONp (5.0)	270	1	14	**3a**	7 (50)
3	**1a**	1 : 1.17	1-ClNp (4.0)	270	1	40	**3a**	27 (66)
4	**1a**	1 : 1	1-ClNp (2.5)	260	1	25	**3a**	20 (80)
5	**1a**	1 : 1	1-MeNp (5.0)	245	2	99	**3a**	17 (17)
6	**1a**	1 : 1	1-MeNp (5.0)	200	2	10	**3a**	4 (42)
7	**1b**	1 : 1.7	1-MeONp (2.5)	270	1	36	–[d]	
8[e]	**1b**[e]	1 : 64	not used	270	2	100	**3b**	18 (18)[f]

[a] Reaction conditions: C_{60} (100 mg, 139 µmol), argon. [b] Isolated yield after purification by column chromatography. That in parentheses was based on consumed C_{60}. [c] The experiment was carried out without MW-irradiation. [d] No mono-adduct **3b** was formed. In the reaction mixture, bis(2-phenylphosphine) oxide, bis(2-phenyl)phosphonic acid and poly-adducts of **2b** with fullerene C_{60} were identified (^{1}H, ^{31}P NMR, elemental analysis). [e] The solubility of C_{60} in the phosphine at 200 °C is ~50 mg ml^{-1}. [f] A mixture of mono- and poly-adducts of phosphine **2b** with fullerene C_{60} was formed (^{1}H, ^{31}P NMR, FT-IR, elemental analysis).

metry, which proved the addition of the phosphine to the 6,6-ring junction in the synthesized compounds. Spectral characteristics of the phosphine oxide **3a** were identical to those previously described in the literature.[7]

The ^{1}H NMR spectrum of **3b** (Figure S2) reveals the multiplet signals corresponding to the ethane-1,2-diyl protons (~2.75–3.57 ppm), whereas the H atom at C_{60} core appears as a doublet at δ 7.44 ppm (J_{PH} 23.9 Hz) *vs.* those in **3a** at 7.10 ppm (J 25.7 Hz). This assignment for **3b** has been proved by 2D H-P HMBC spectrum (Figure S3). The high-field region of the ^{13}C NMR spectrum of **3b** (Figure S4) demonstrates two signals at 28.73 (J 3.2 Hz) and 29.59 ppm (J_{CP} 61.4 Hz) for the CH_2CH_2 group and two sp^3 signals in the C_{60} core [55.93 and 69.35 ppm (J_{PC} 59.9 Hz)]. The low-field part of the spectrum shows 34 sp^2 signals in the C_{60} core (135.38–152.04 ppm) and three signals for Ph rings (126.75, 128.21 and 128.86 ppm). The FT-IR spectrum of **3b** contains a strong absorption at 1170 cm^{-1} belonging to the P=O group (Figure S6). The bands at 527, 579, 1180 and 1428 cm^{-1} are attributed to C_{60} skeleton absorption peaks. The aromatic ring stretching vibration bands are present at 1377, 1406, 1454 and 1496 cm^{-1}. The stretching vibration bands of the CH, CH_2 moieties are observed at 2850, 2921 and 2955 cm^{-1}.

The MALDI-TOF mass spectrum of monoadduct **3b** ($C_{76}H_{19}OP$) displays a peak at m/z 979 as well as one for C_{60} at m/z 720, which arise from loss of $C_{16}H_{18}OP$ fragment. There are three peaks higher than m/z 720, *i.e.* at m/z 979 (monoadduct), 1237 (diadduct), and 1495 (triadduct) (Figure 1). The UV-VIS adsorptions are typical of the 1 : 1 adduct at the 6,6-bond. The electronic absorption spectra of the monoadduct in cyclohexane are similar to that of C_{60}. Thus, three strong absorption bands at 211, 257 and 329 nm are observed. However, they are different from weak adsorption bands of the UV and visible region. New band with a shoulder at 229 nm is detected, while two new weak adsorptions at 405 and 409 nm almost disappear (Figure S7). A C_{60}-mono-adduct absorption peak is also present at 430 nm.

According to the known data,[8,10] the following mechanism for the reaction of C_{60} with R_2PH can be proposed (Scheme 2). The first stage involves one-electron transfer from the phosphine **1** to C_{60} molecule to give ion-radical pair. Then recombination of phosphine cation-radical and C_{60} anion-radical occurs. The proton transfer from phosphorus to C_{60} carbon atom affords neutral adduct **2**, which is readily oxidized in the presence of air to the relative phosphine oxide **3**. The study of the scope of this reaction is underway.

In conclusion, the catalyst-free, convenient and atom-economic method for synthesis of diorganyl(C_{60}H)phosphines (isolated as the corresponding phosphine oxides) has been developed. The method is based on the mono-addition of secondary phosphines to

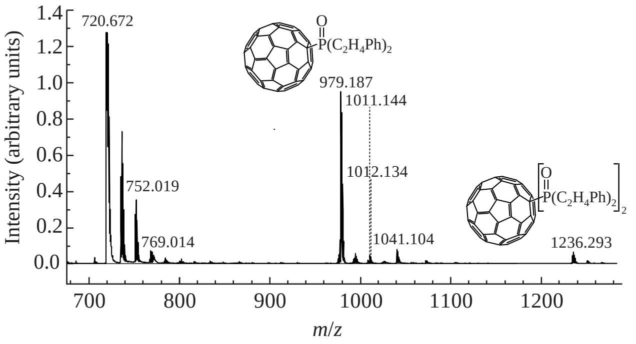
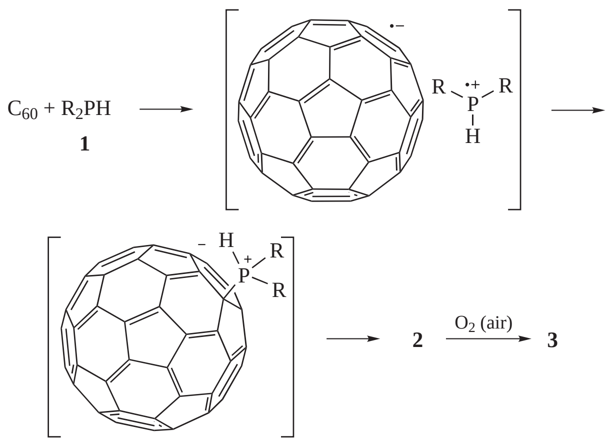


Figure 1 MALDI-TOF/TOF mass spectrum of **3b** (positive ion, reflectron mode, matrix S8, toluene as solution).

[‡] *Bis(2-phenylethyl)(C_{60}H)phosphine oxide* **3b**. A mixture of C_{60} (100 mg, 139 µmol) and bis(2-phenylethyl)phosphine (2147 mg, 8.861 mmol) was stirred and MW-irradiated under Ar atmosphere in a closed vial (10 ml) at 270 °C for 2 h. During the heating C_{60} was dissolved in hot bis(2-phenylethyl)phosphine (the solubility of C_{60} in the phosphine at 270 °C is ~50 mg ml^{-1}). In 2 h, the reaction mixture represented an orange suspension with orange-brown residue. In addition, along with the mono-adducts, unsolved residue, likely a mixture of poly-adducts, was formed. The residue was diluted with $CHCl_3$ (1 ml) and purified by column chromatography using silica gel (Alfa Aesar, 70–230 mesh, eluent Et_2O) to give unreacted bis(2-phenylethyl)phosphine (505 mg). Further elution with $CHCl_3$ gave bis(2-phenylethyl)phosphine oxide (1146 mg), then elution with 10–20% EtOAc in toluene gave mono-adduct **3b** (24 mg). Elution with MeOH afforded bis(2-phenylethyl)phosphinic acid (382 mg). The fraction containing mixture of poly-adducts remained at the top of the column.

3b: brown powder, isolated yield 24 mg (18%). ^{1}H NMR (400.1 MHz, CS_2–$CDCl_3$) δ: 2.75–2.85 (m, 2H, CH_2P), 3.14–3.25 (m, 2H, CH_2P), 3.36–3.46 and 3.47–3.57 (m, 4H, CH_2Ph), 7.11–7.37 (m, 10H, Ph), 7.44 (d, 1H, HC_{60}, ^3J 23.9 Hz). ^{13}C NMR (100.6 MHz, CS_2–$CDCl_3$) δ: 28.73 (d, 2C, CH_2Ph, ^2J 3.2 Hz), 29.59 (d, 2C, CH_2P, ^1J 61.4 Hz), 55.93 (1C, HC_{60}), 69.35 (d, 1C, PC_{60}, ^1J 59.9 Hz), 126.75 (2C, Ph), 128.21 (4C, Ph), 128.86 (4C, Ph); fullerene moiety: 127.53, 130.41, 135.38, 136.51, 136.55, 140.31, 140.45, 140.58, 140.66, 141.56, 141.60, 141.61, 141.75, 141.85, 141.95, 142.06, 142.59, 142.70, 142.97, 143.30, 144.23, 144.69, 145.41, 145.46, 145.57, 145.75, 146.29, 146.33, 146.45, 146.59, 146.93, 147.17, 149.20 (d, 1C, PC_{60}, ^2J 5.7 Hz), 152.02 (d, 1C, PC_{60}, ^2J 4.0 Hz). ^{31}P NMR (161.98 MHz, CS_2–$CDCl_3$) δ: 51.26 (s). IR (ν/cm^{-1}): 527, 565, 579, 753, 1142, 1170, 1180, 1214, 1428, 1453, 1495, 1602, 2850, 2920, 2955, 3025, 3061, 3085. UV (cyclohexane, λ/nm): 211, 229, 257, 329, 405, 409. MS (MALDI-TOF), m/z: 720.672 [C_{60}], 979.187 [M, $C_{76}H_{19}OP$].



Scheme 2

fullerene C_{60} under MW irradiation. The compounds synthesized are promising building blocks for drug design and advanced materials.

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Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.mencom.2017.03.031.

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