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

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Table of contents:

General. ^{1}H and ^{13}C NMR spectra of the studied compounds were recorded in $CS_2/CDCl_3$ (1:1) on Bruker DPX-400 spectrometer (400.13 and 100.62 MHz, respectively) at room temperature. IR spectra were measured on a Bruker Vertex-70 instrument in KBr pellets.
UV/Vis spectra were measured on a Perkin-Elmer Lambda 35 spectrometer at room temperature (cyclohexane, d = 1.0 cm). Mass spectra MALDI were obtained in reflectron mode, MS/MS spectra were obtained in lift mode on a Bruker ultrafleXtreme TOF/TOF instrument, a nitrogen laser operating at 337 nm. A saturated solution of sulfur (Alfa Aesar) in toluene was used as a matrix. Elemental analyses (EDX) were performed with a FLASH EA 1112 Series instrument. C_{60} (>99%) was purchased from ("High-tech industry" St. Petersburg, Russia) and used without further purification. 1-Methylnaphthalene, 1-methoxynaphthalene, 1-chloronaphthalene (85%) were degassed in vacuum and saturated with Ar. All reactions were performed under dry argon atmosphere. Diphenylphosphine and solvents were commercial reagents, bis(2-phenylethyl)phosphine was prepared as described.[i] The syntheses were carried out in MW reactor, heavywalled glass vials (10 mL) equipped with a magnetic stirrer and sealed with Teflon septa. Irradiation was carried out in a monomode microwave cavity Anton Paar 'Monowave 300' (magnetron frequency 2455 MHz, power up to 850 W). The temperature was monitored by internal IR sensor. The reaction was monitored with ^{31}P NMR spectroscopy by disappearance of the secondary phosphine signals at (-40 ppm for **1a** and -70 ppm for **1b**) and appearance new signals for tertiary phosphines (-14 ppm for **2a** and -29 ppm for **2b**) Figure S1.

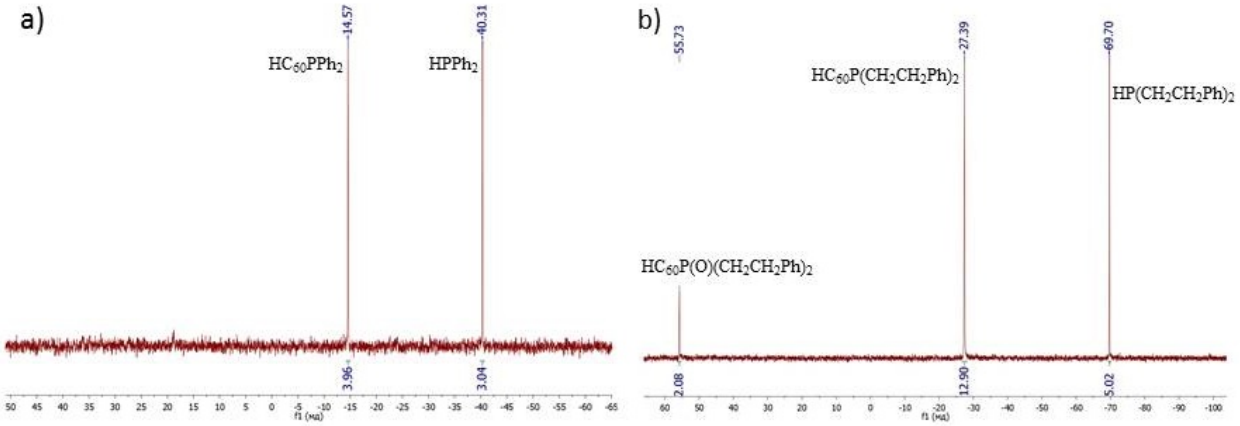

Figure S1 (a) ^{31}P NMR of typical reaction mixture C_{60} and **1a** after 1 h irradiation in 1-MeNp; (b) ^{31}P NMR of typical reaction mixture C_{60} and **1b** after 2 h irradiation in 1-MeNp.

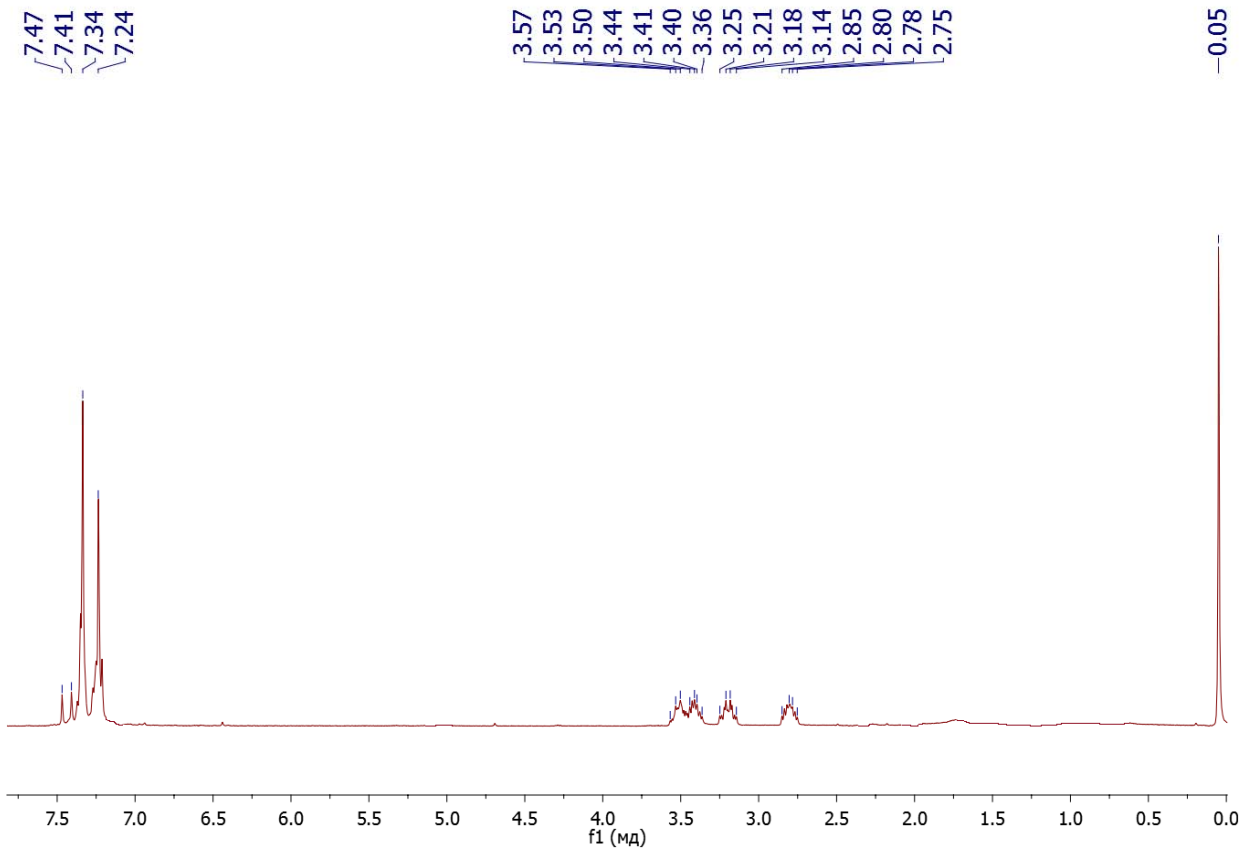

Figure S2 ^{1}H NMR ($CDCl_3$:CS_2) spectrum of compound **3b**.

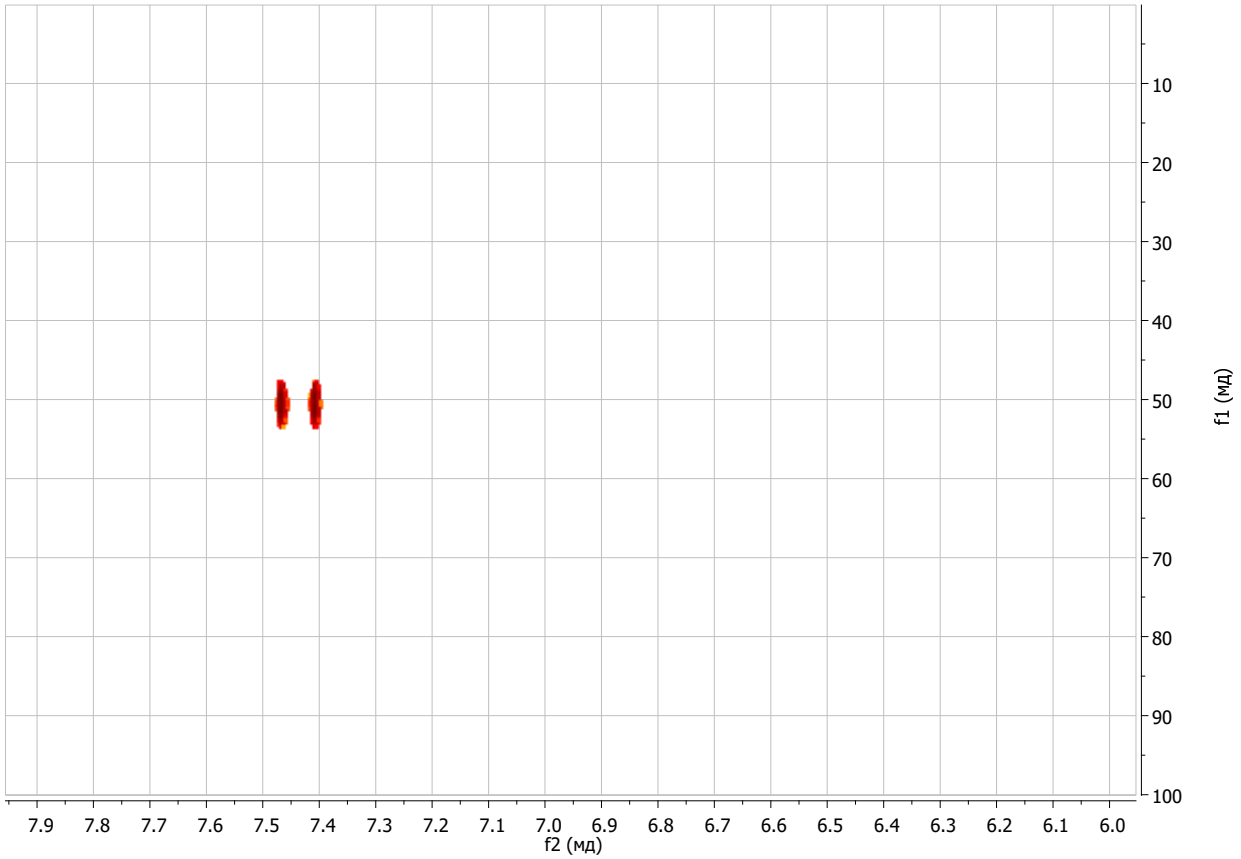

Figure S3 Hmbc spectrum of **3b** ($CDCl_3$:CS_2).

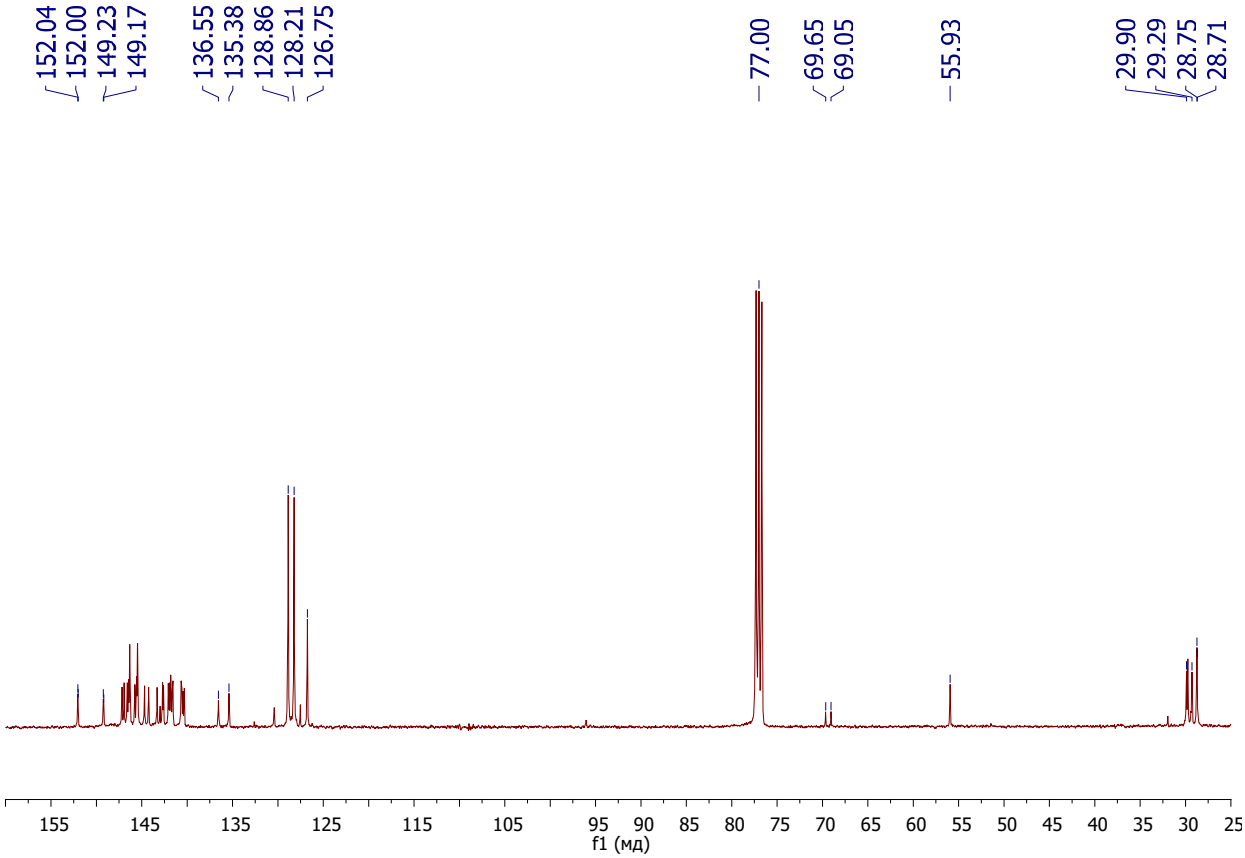

Figure S4 ^{13}C NMR ($CDCl_3$:CS_2) spectrum of compound **3b**.

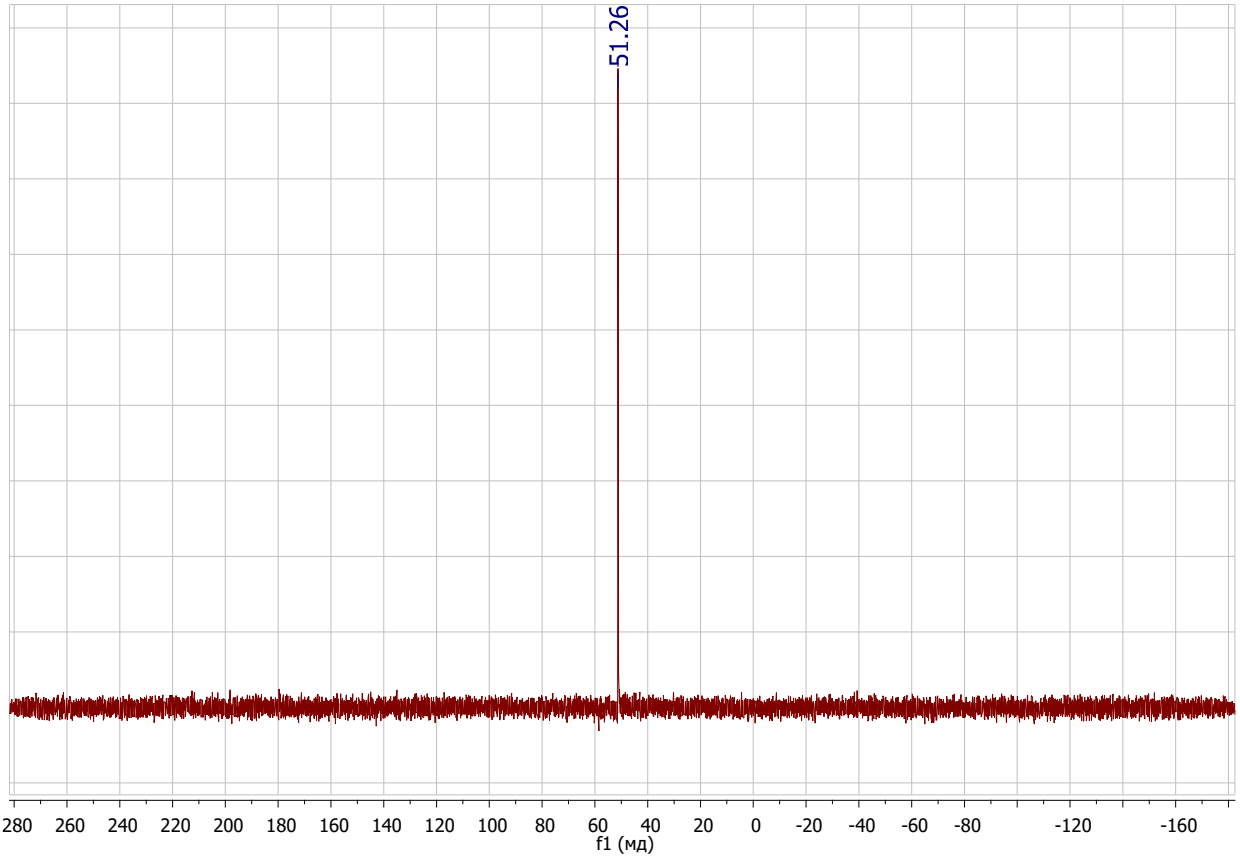

Figure S5 ^{31}P NMR ($CDCl_3$:CS_2) spectrum of compound **3b**.

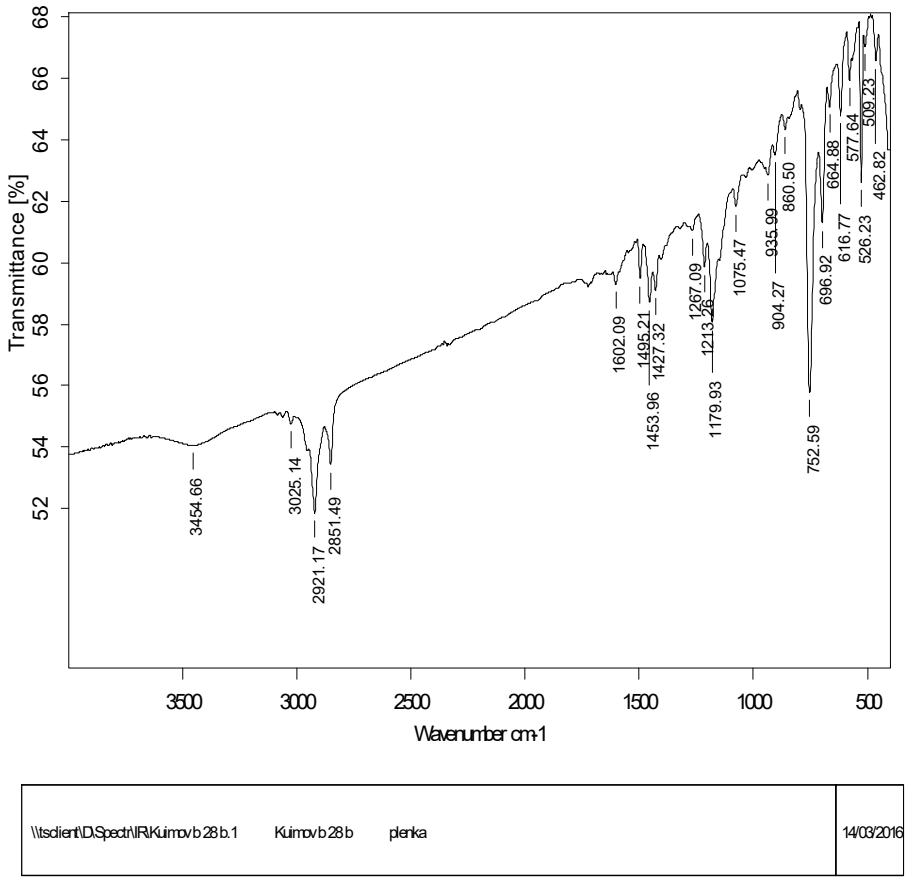

Figure S6 FT-IR of compound **3b**.

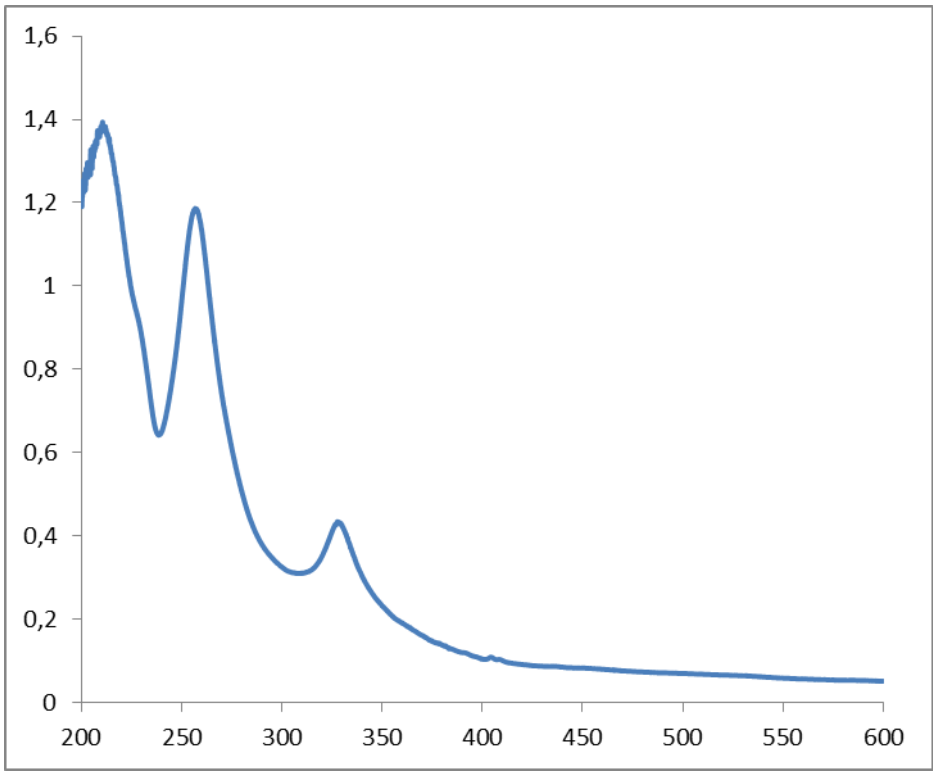

Figure S7 UV-VIS spectrum of **3b** (cyclohexane).

[i] B.A. Trofimov, L. Brandsma, S.N. Arbuzova, S.F. Malysheva and N.K. Gusarova, *Tetrahedron Lett.*, 1994, **35**, 7647.