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Unexpected formation of 1,4-diphenylbutylphosphinic acid from 1,4-diphenylbuta-1,3-diene and elemental phosphorus *via* the Trofimov–Gusarova reaction

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General remarks

The reaction was carried out under inert atmosphere (argon blanket). *trans*,*trans*-1,4-Diphenylbuta-1,3-diene, red phosphorus, $KOH \cdot 0.5H_2O$ and DMSO (0.5-1% of H_2O) were used as purchased (Alfa Aesar, Ksan Sea). ^{1}H, ^{13}C, ^{31}P NMR and 2D spectra were recorded with an AV-400 Bruker BioSpin spectrometer. The ^{1}H NMR chemical shifts are measured with respect to residual protons in $CDCl_3$ (7.27 ppm), which served as an internal standard. The ^{13}C NMR shifts are measured with respect to the $CDCl_3$ (77.1 ppm). 85% H_3PO_4 was used as external standard for ^{31}P NMR. FT-IR spectrum was recorded on a Bruker Vertex 70 spectrometer. The C, H microanalyses were performed on a Flash EA 1112 analyzer, while the content of P was determined by combustion method. Melting point (uncorrected) was measured on a Kofler micro hot stage.

EI mass spectrum of acid **2** (**Figure S12**) was recorded on Shimadzu GCeMS QP-5050A mass spectrometer at 70 eV with the source temperature fixed at 200 ^{o}C. The compound was introduced through a direct insertion probe (DI-50) heated at the minimum temperature necessary to obtain reproducible ion abundances.

Chromatographic separation of organic residue (**Figure S3**) was carried out on Agilent 6890N gas chromatograph coupled with the mass spectrometer (injector and interface temperature 250 °C, ion source 250 °C; HP-5MS column, 30 m × 0.25 mm, film thickness 0.25 μm; helium as a carrier gas).

Figure S1 Typical ^{31}P NMR spectrum of the reaction mixture.

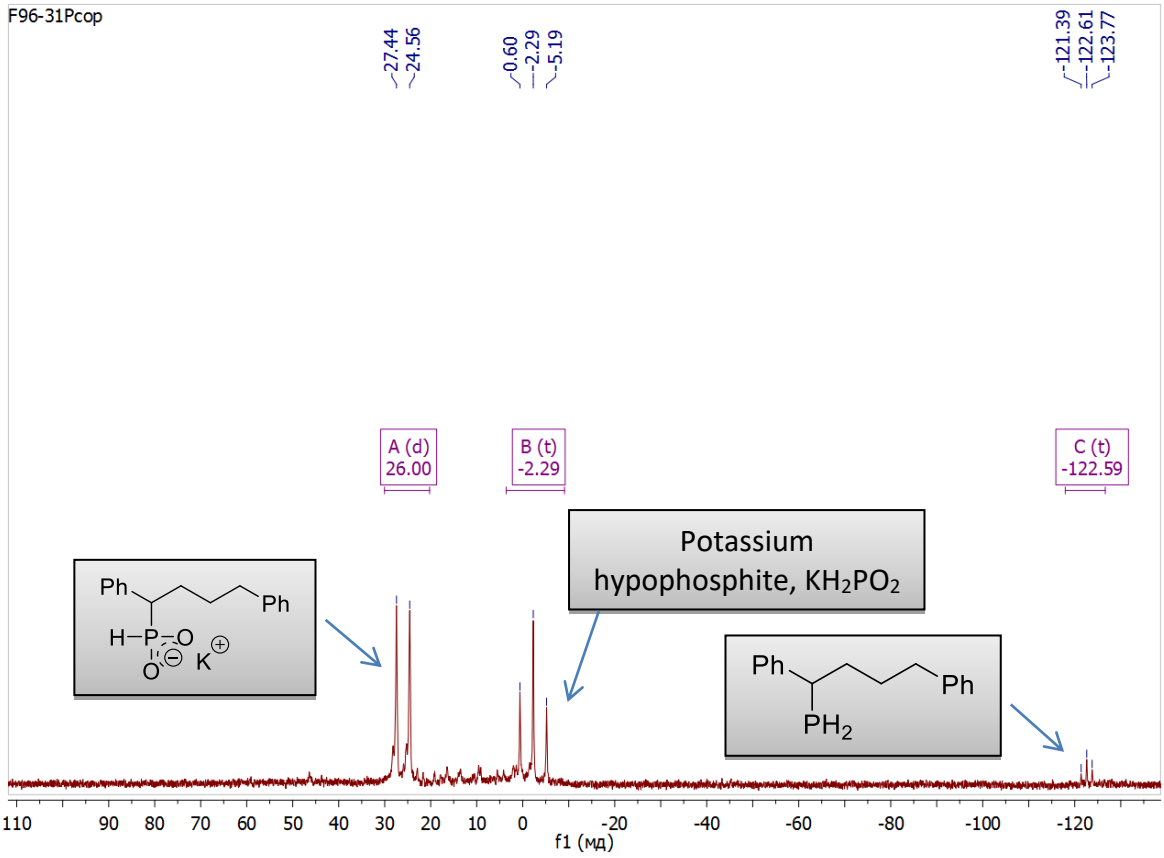


Figure S2 ^{13}C NMR spectrum of aqueous layer of the reaction mixture.

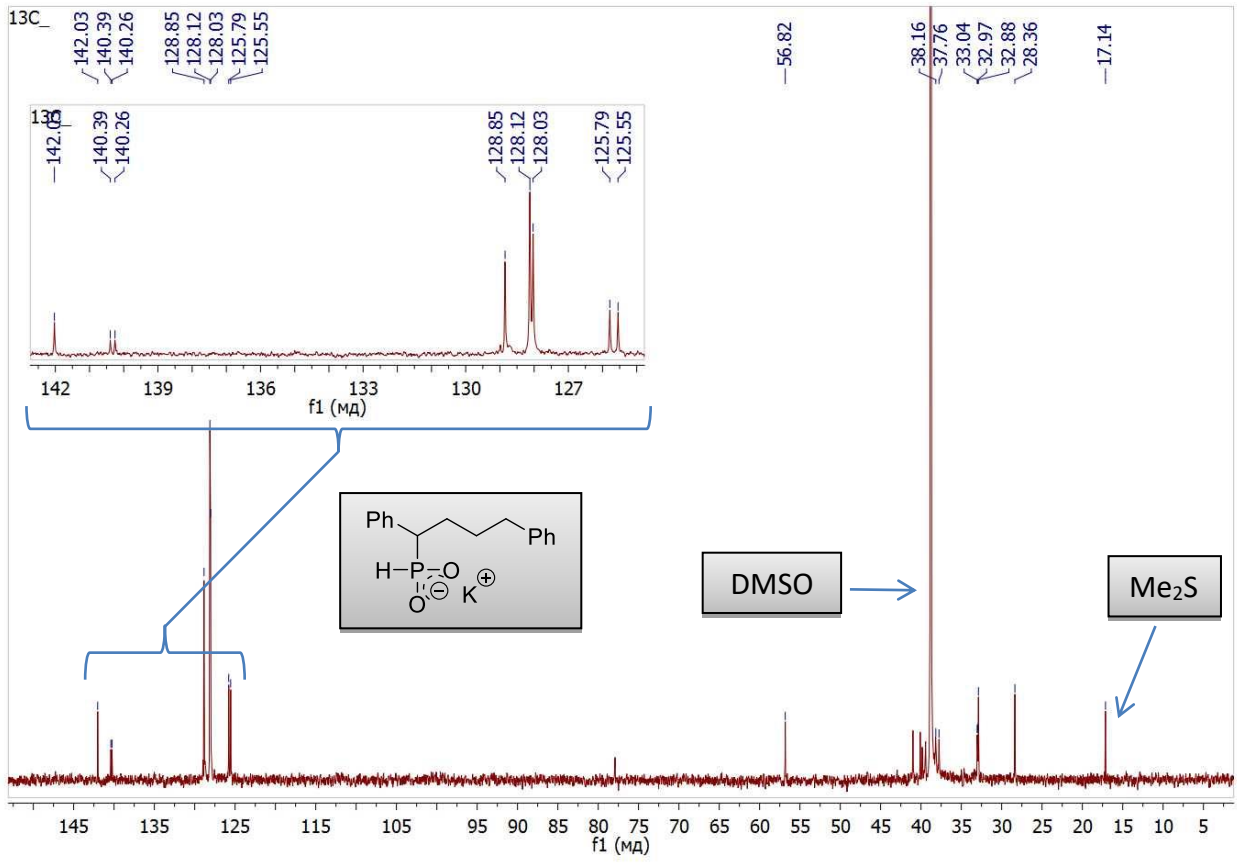


Figure S3 GC-MS analysis data for organic residue.

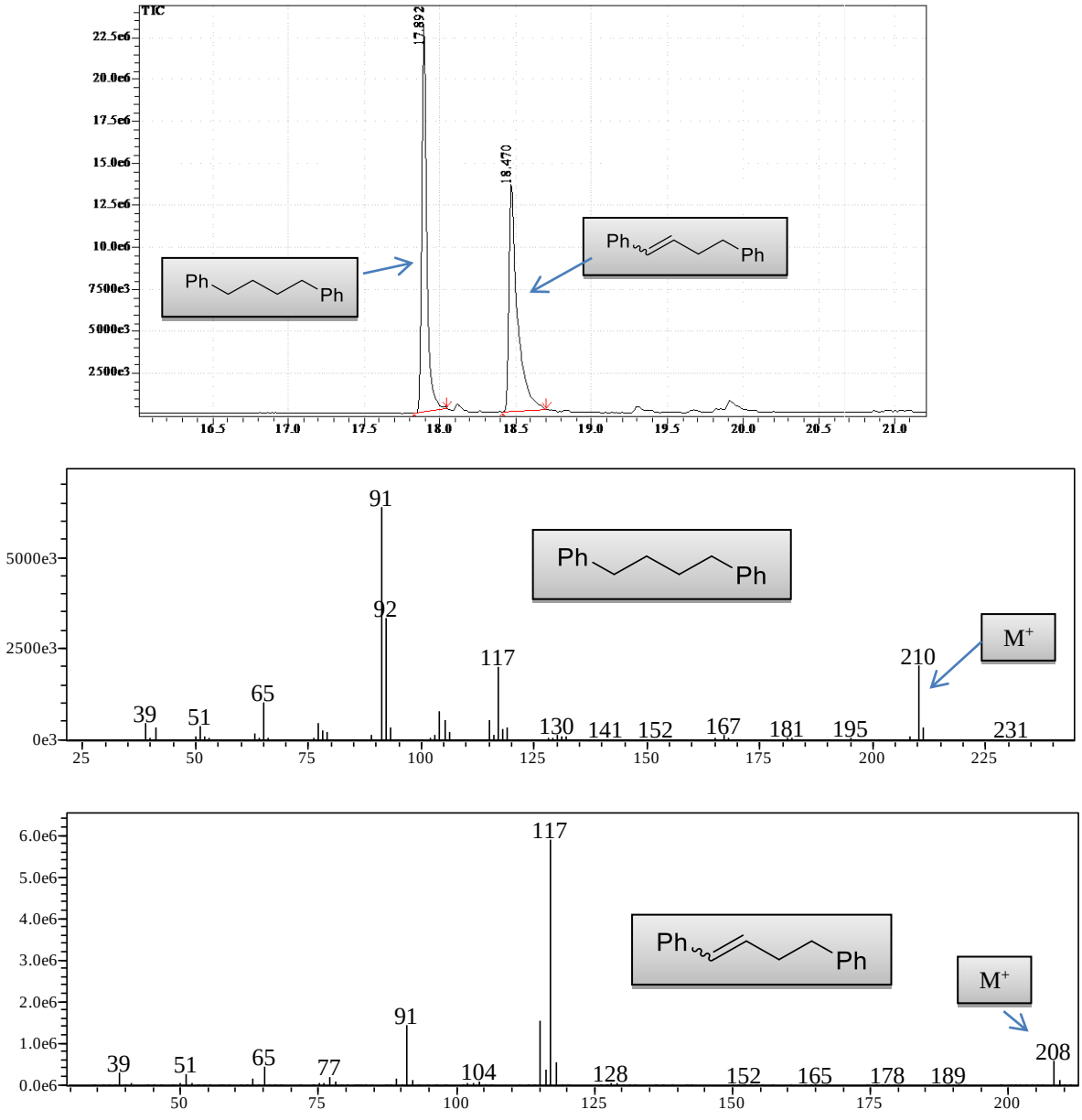


Figure S4 ^{1}H NMR spectrum of acid **2** ($CDCl_3$).

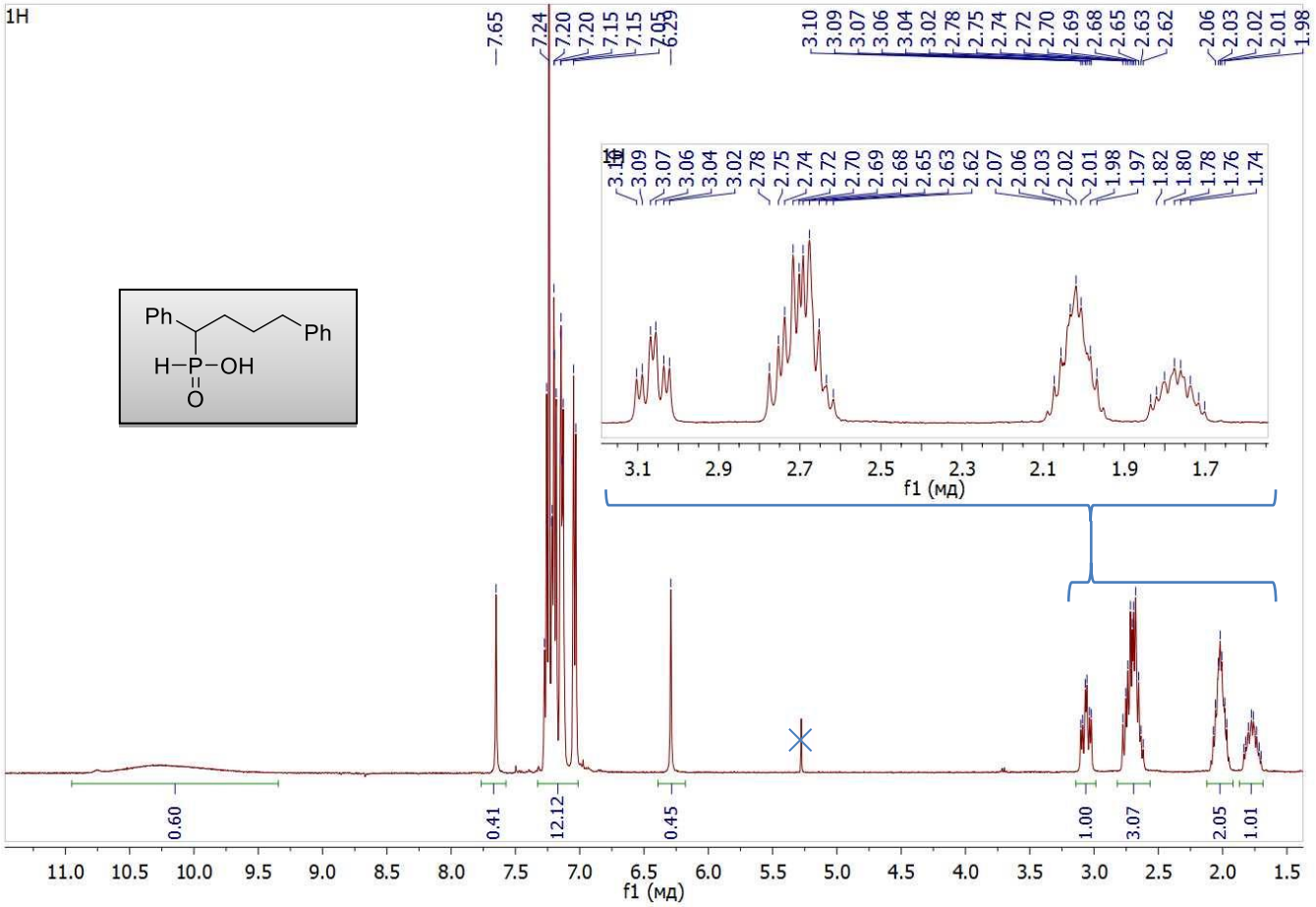


Figure S5 ^{13}C NMR spectrum of acid **2** ($CDCl_3$).

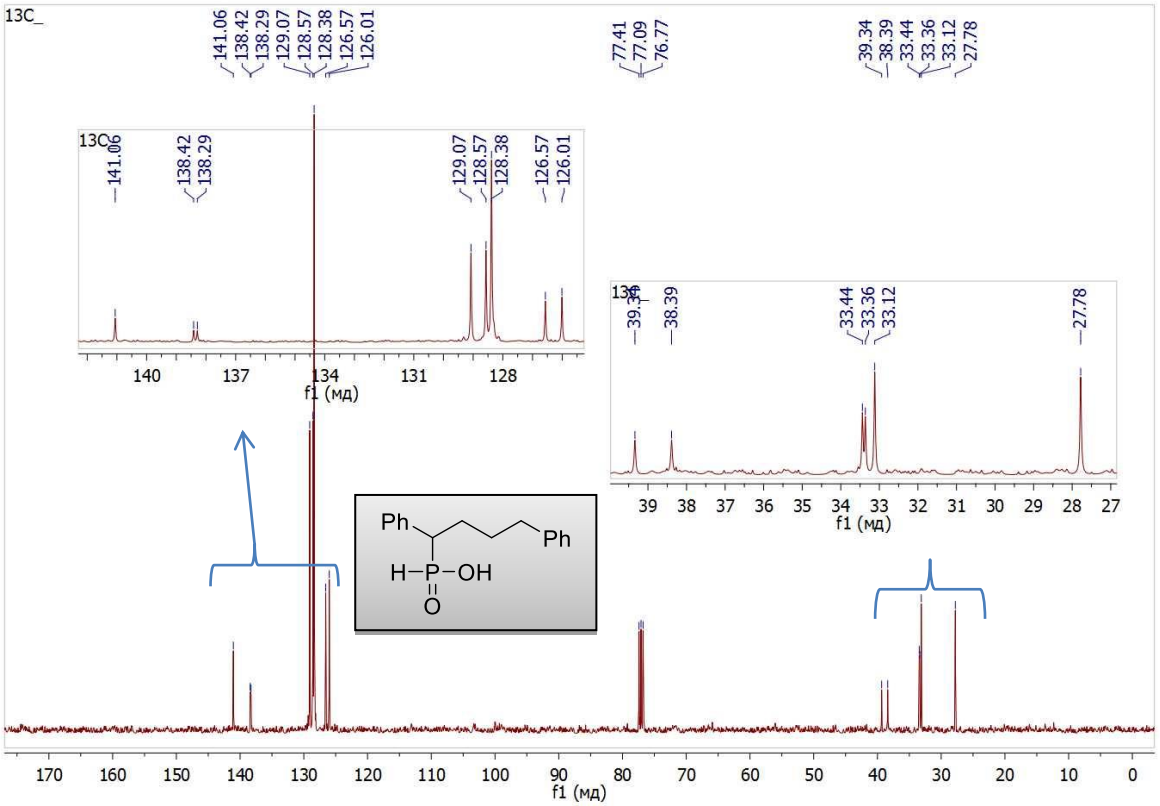


Figure S6 ^{13}C JMOD spectrum of acid **2** ($CDCl_3$).

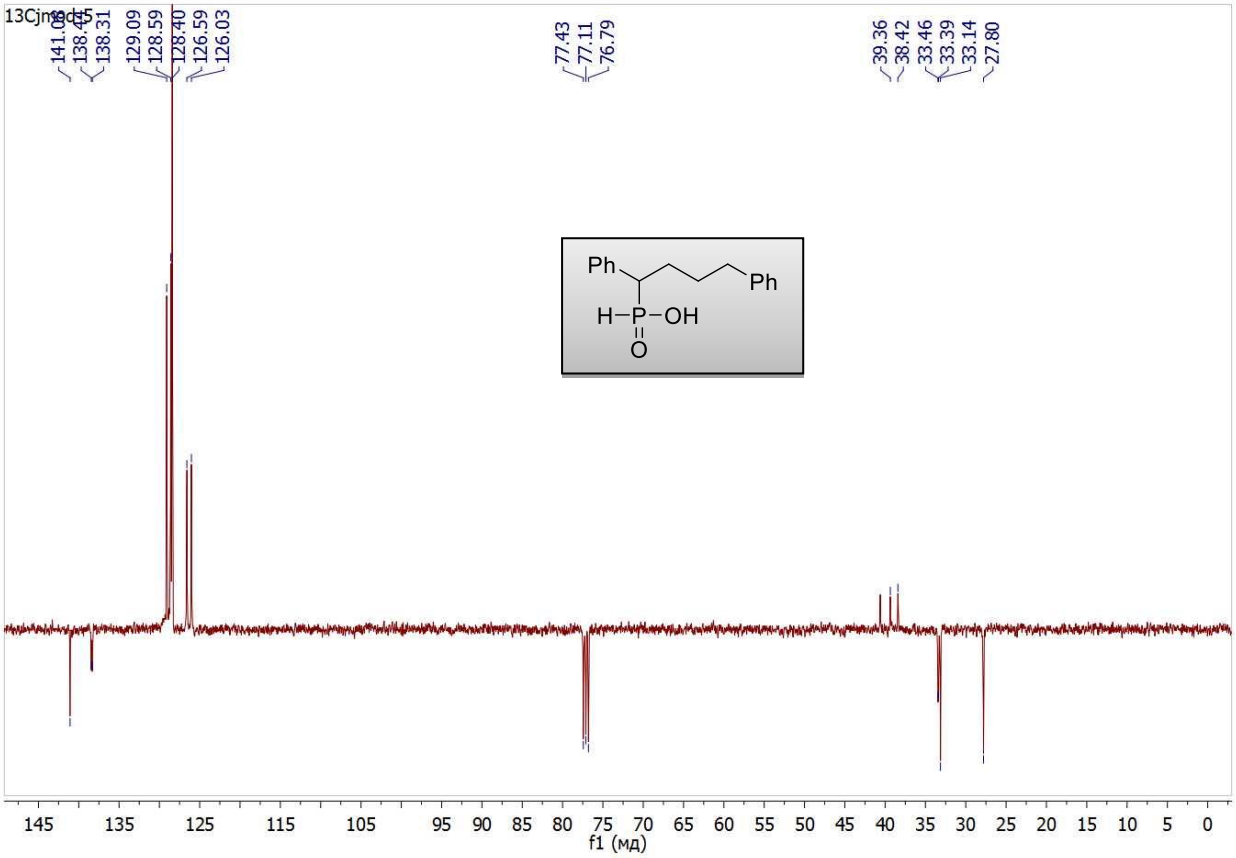


Figure S7 $^{31}P\{^{1}H\}$ NMR spectrum of acid **2** ($CDCl_3$).

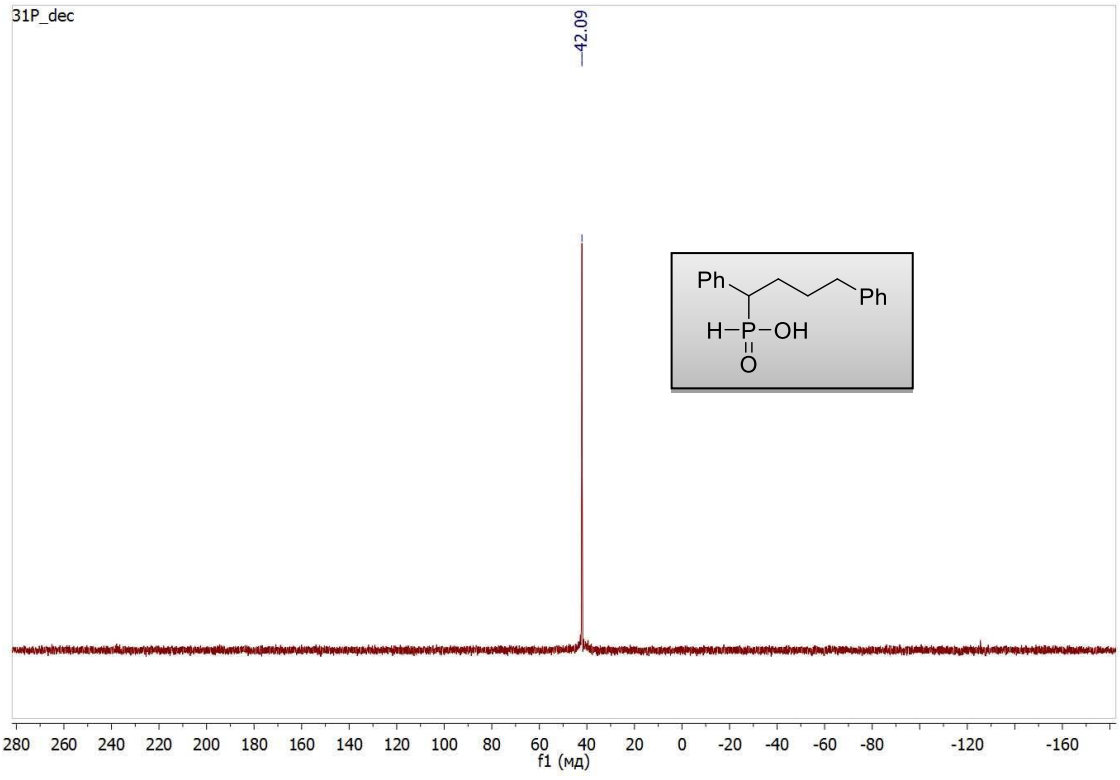


Figure S8 ^{31}P NMR spectrum of acid **2** ($CDCl_3$).

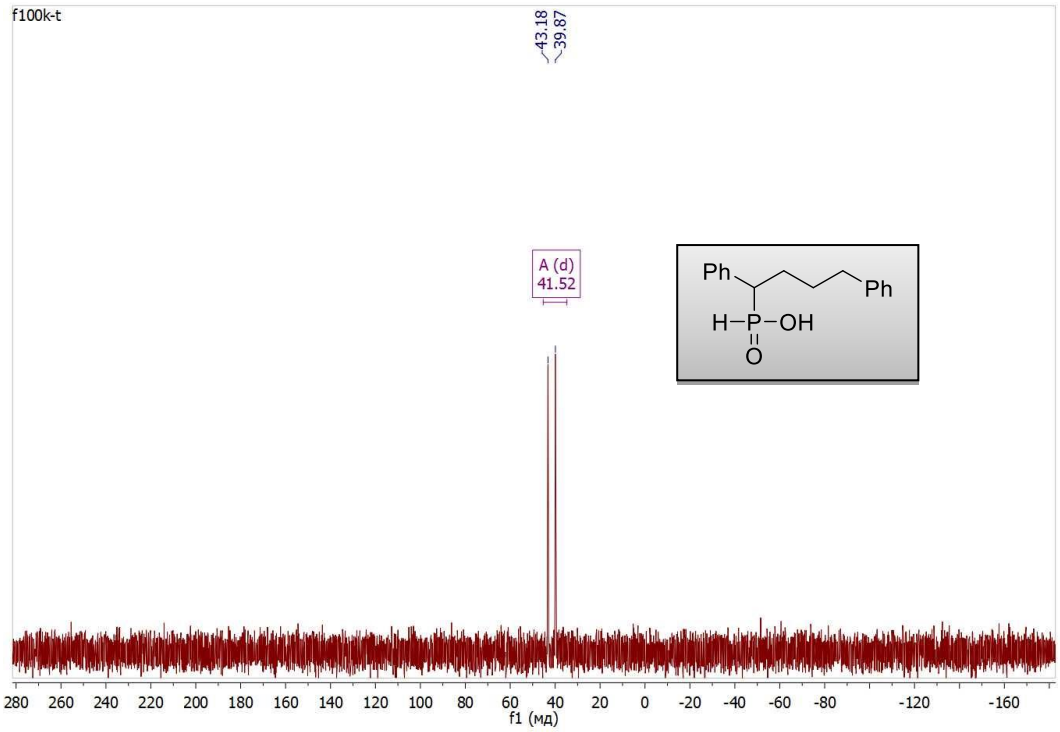


Figure S9 ^{1}H-^{1}H COSY spectrum of acid **2** ($CDCl_3$).

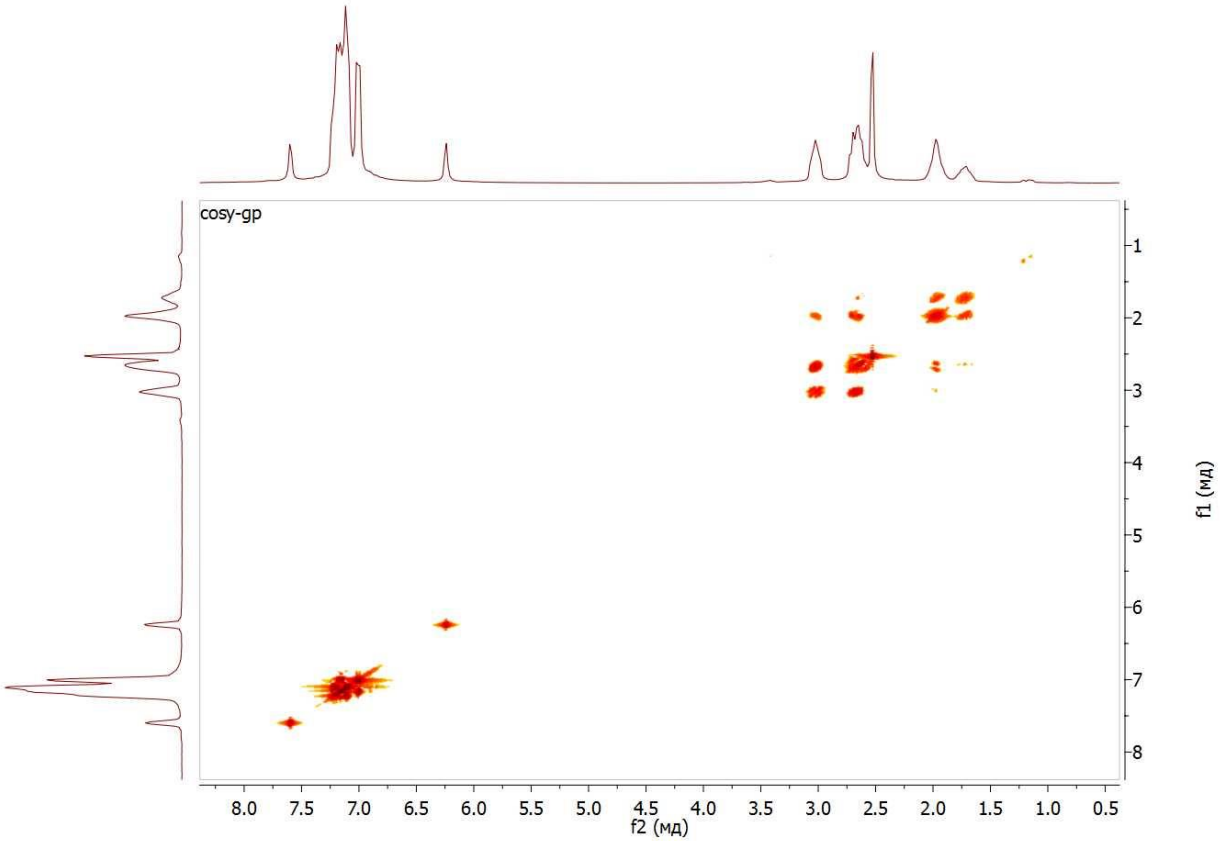


Figure S10 $^{1}H–^{13}C$ HSQC spectrum of acid **2** ($CDCl_3$).

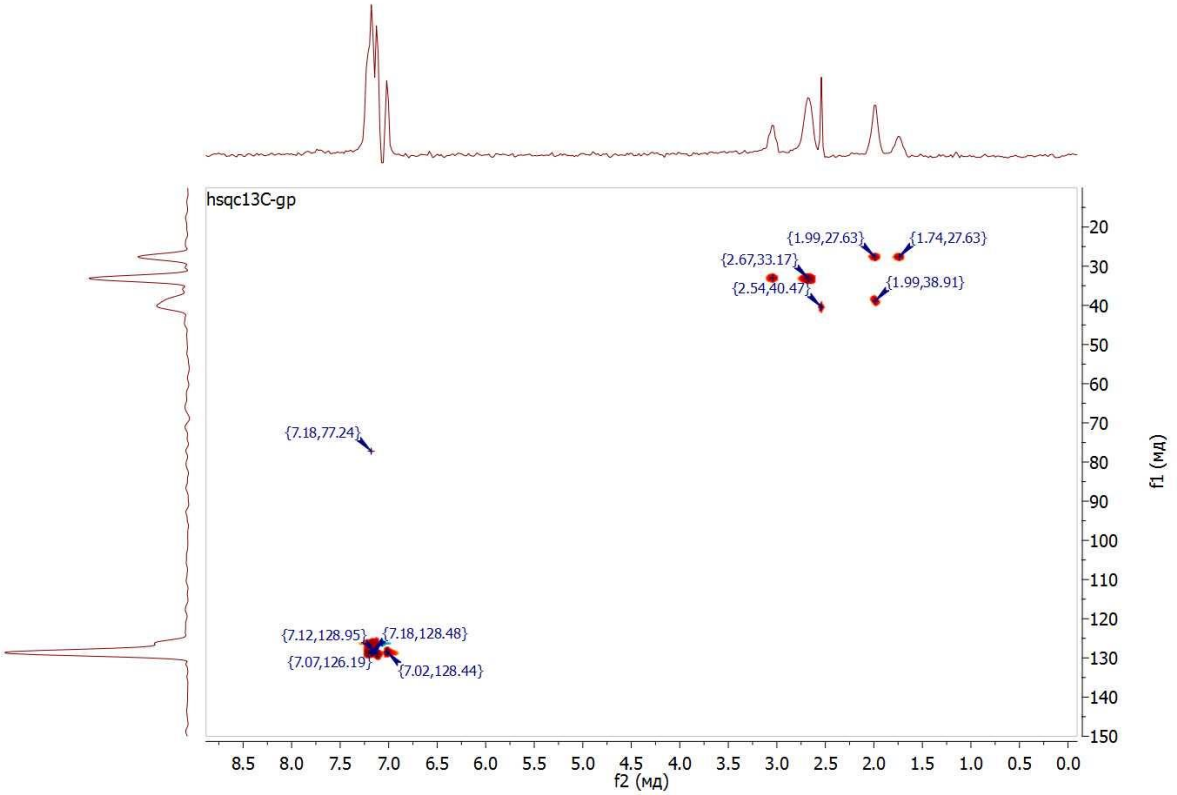


Figure S11 $^{1}H–^{31}P$ HMBS spectrum of acid **2** ($CDCl_3$).

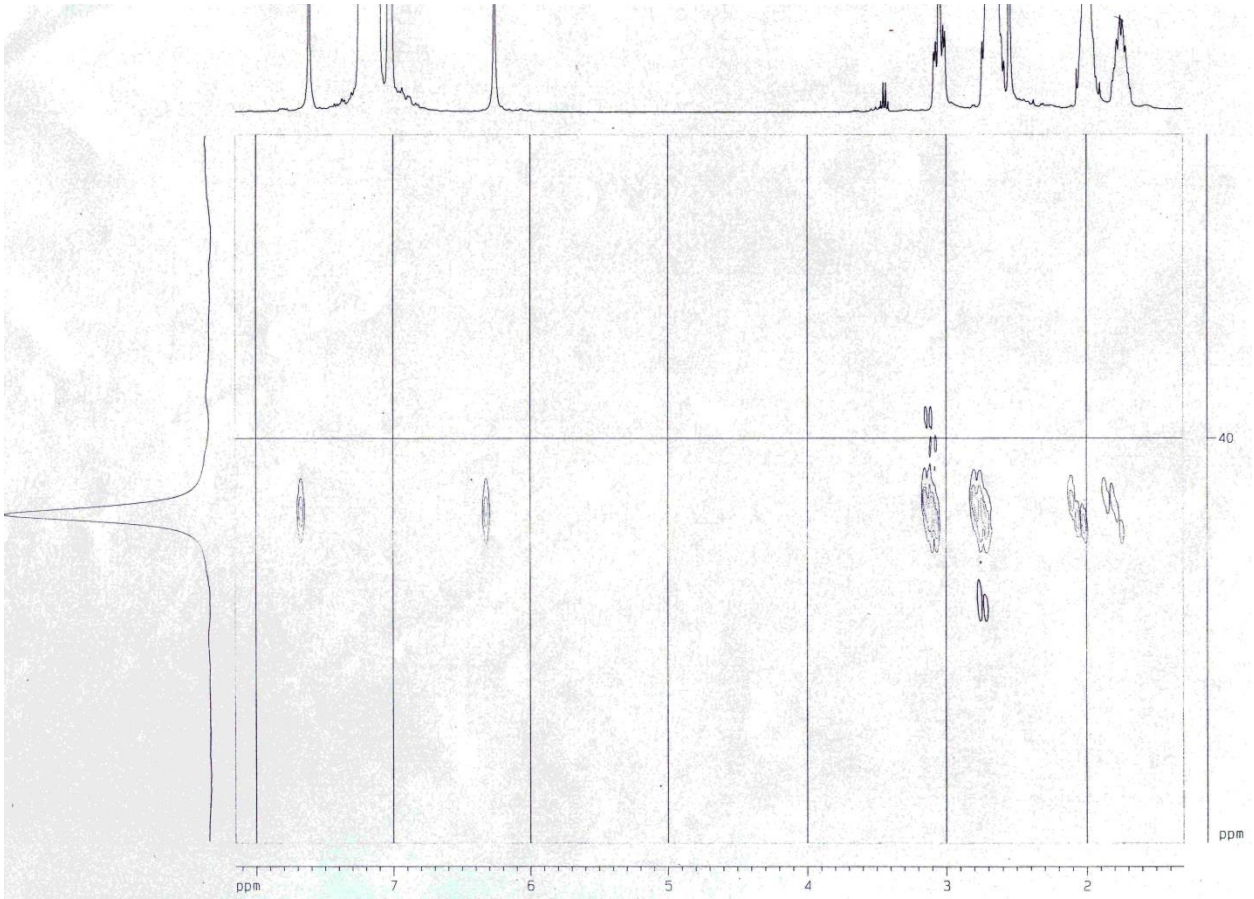


Figure S12 EI-MS spectrum of acid **2**.

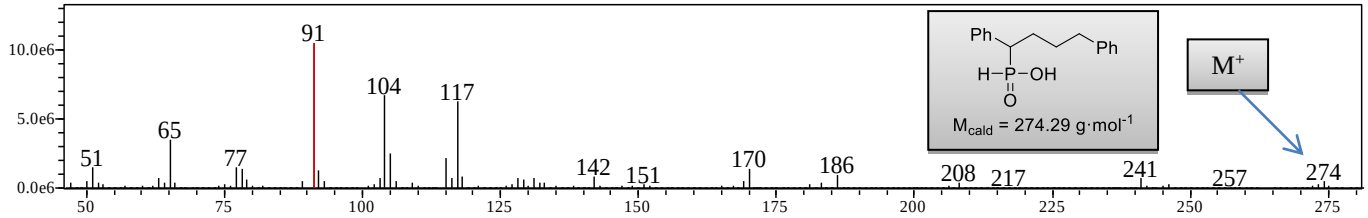
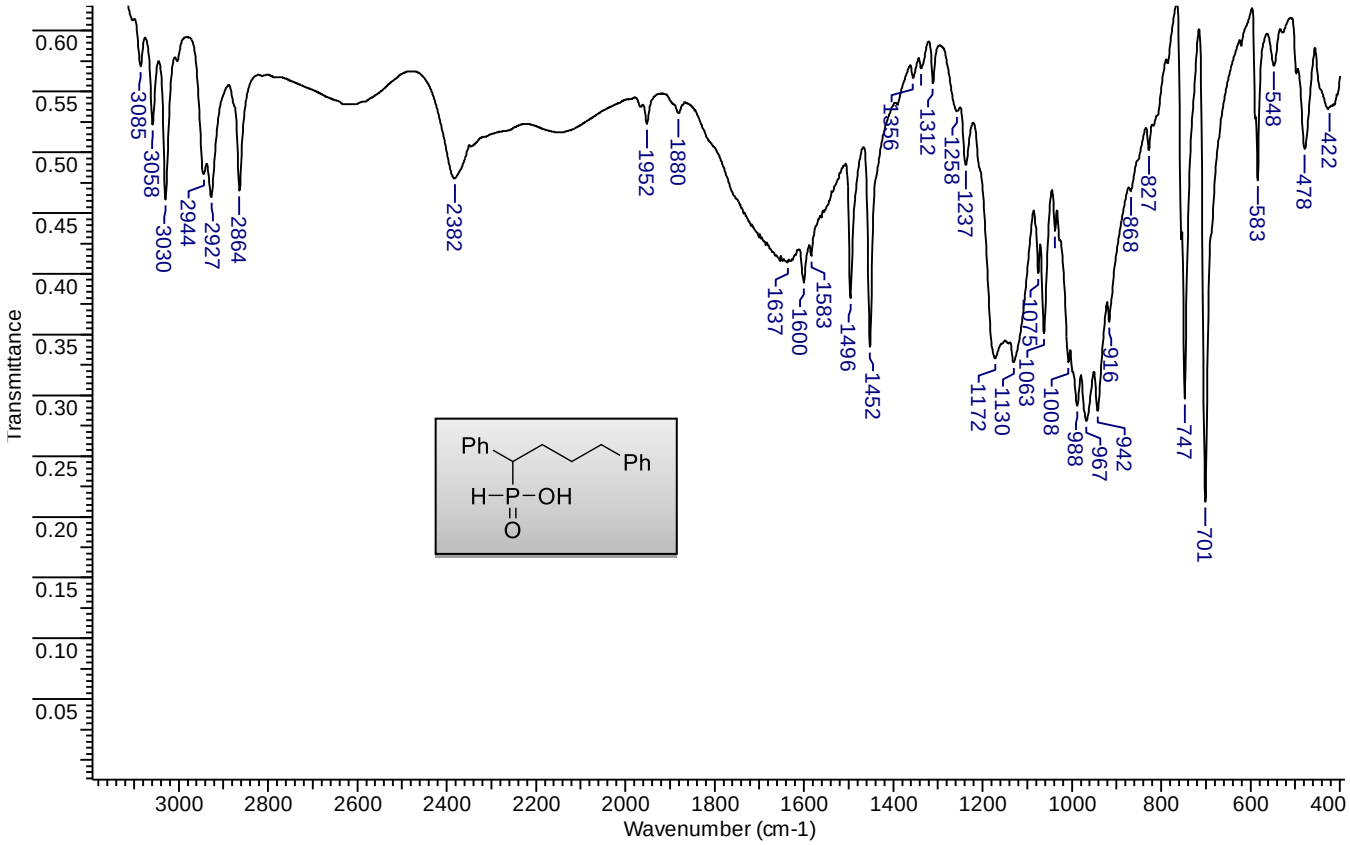
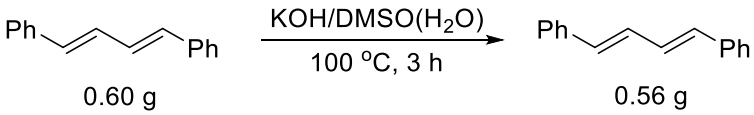


Figure S13 FT-IR spectrum of acid **2** (KBr).



FT-IR (KBr, cm^{-1}): 422 (w), 478 (m), 548 (w), 583 (m), 701 (vs), 747 (s), 827 (m), 868 (m), 916 (s), 942 (s), 967 (s), 988 (s), 1008 (s), 1038 (m), 1063 (s), 1075 (m), 1130 (s), 1172 (s), 1237 (m), 1258 (w), 1312 (w), 1338 (w), 1356 (w), 1452 (s), 1496 (m), 1583 (m), 1600 (m), 1637 (m), 1880 (w), 1952 (w), 2382 (m), 2864 (m), 2927 (m), 2944 (m), 3030 (m), 3058 (w), 3085 (w).

Additional experiments



A mixture of *trans*,*trans*-1,4-diphenylbuta-1,3-diene (**1**) (0.600 g), KOH·0.5H_2O (1.60 g), DMSO (7 ml) and H_2O (0.2 ml) was stirred at 100 °C for 3 h. The mixture was then cooled to r.t., diluted with water (150 ml) and extracted with $CHCl_3$ (3 × 10 ml). The extract was washed with water (3 × 100 ml) and dried over K_2CO_3. The solvent was distillated off and the residue was dried in vacuum to give 0.56 g of **1** (7% conversion) as slightly yellowish powder. Its ^1H NMR spectrum is in agreement with that for pure sample of **1**.