1308 *Russian Chemical Bulletin, International Edition, Vol. 65, No. 5, pp. 1308—1312, May, 2016*

New di- and tricarboxylate phosphabetaines*

Yu. V. Bakhtiyarova, A. F. Aksunova, R. R. Minnullin, I. V. Galkina,★ and V. I. Galkin

Kazan (Volga Region) Federal University,
18 ul. Kremlevskaya, 420008 Kazan, Russian Federation.
Fax: +7 (843) 238 7901. E-mail: vig54@mail.ru

New stable tricarboxylate phosphabetaines were synthesized based on 3-(diphenylphosphino)propionic acid and unsaturated dicarboxylic acids (maleic and itaconic). A new dicarboxylate phosphabetaine was synthesized based on 1,3-bis(diphenylphosphino)propane, which did not contain any proton-donor reagents in its crystal lattice.

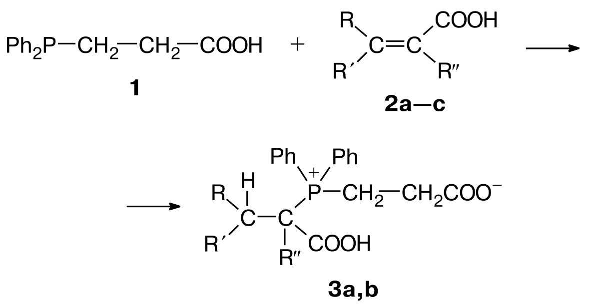
Key words: carboxylate phosphabetaines, unsaturated dicarboxylic acids, phosphonium salts.

Earlier, we have studied synthesis, structure, reactivity, and biological activity of carboxylate phosphabetaines based on various tertiary phosphines and unsaturated carboxylic acids.[1—11]

Recently, we have studied phosphorylation reactions of 3-(diphenylphosphino)propionic acid (**1**) with unsaturated monocarboxylic acids such as acrylic, crotonic, methacrylic, and cinnamic to obtain stable dicarboxylate phosphabetaines, the structure of which was established by a combination of physicochemical methods.[12]

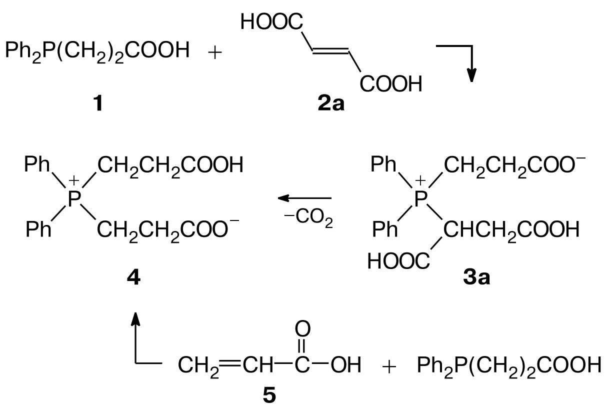
In the present work, we study reactions of acid **1** with unsaturated dicarboxylic acids, namely, fumaric (**2a**), maleic (**2b**), and itaconic (**2c**) (Scheme 1). It was supposed that the final product would contain three carboxy groups.

Scheme 1



2	R	R′	R″	Product
a	H	COOH	H	3a
b	COOH	H	H	3a
c	H	H	CH$_2$COOH	3b

Scheme 2



The reaction of fumaric acid (**2a**) with acid **1** proceeds similarly to already known reactions of tertiary phosphines.[13—16] These reactions are accompanied by decarboxylation, however, we detected the formation of tricarboxylate phosphabetaine **3a** by ^{31}P NMR spectroscopy. Initially, the ^{31}P NMR spectra of the reaction mixture exhibited two signals for the phosphorus atoms at δ_P 27 (**3a**) and 28.5 (**4**) (Scheme 2), with time, the intensity of the second signal increased, while that of the second signal decreased. The isolated crystalline product contains the only signal at δ_P 28.5 (**4**). The IR and NMR spectra, as well as melting points of the compound completely correspond to product **4** obtained in the studied earlier[12] reaction of 3-(diphenylphosphino)propionic (**1**) and acrylic acids (**5**) (see Scheme 2). It is obvious that the reaction course is accompanied by decarboxylation process.

Such a behavior of fumaric acid (**2a**) in the reactions with tertiary phosphines accompanied by decarboxylation of the intermediate product is quite expected.[13—16] Nonetheless, in this case the ^{31}P NMR spectrum of the reaction mixture for the first time exhibited two signals (tri- and

---

* Dedicated to Academician of the Russian Academy of Sciences O. G. Sinyashin on the occasion of his 60th birthday.

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 1308—1312, May, 2016.

dicarboxylate betaines), which confirmed the fact of the intermediate formation of structure **3a**.

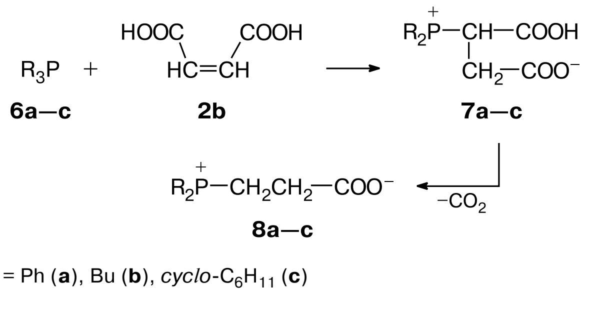
The reaction of acid **1** with maleic acid (**2b**) proceeds within several minutes with the formation of a white precipitate. The ^{31}P NMR spectrum contains one signal for the phosphorus atom at δ_P 28.7. Product **3a** with m.p. 80 °C is well soluble in water and ethyl alcohol and poorly in acetonitrile. Based on the ^{13}C NMR data, it can be inferred that the synthesized betaine **3a** contains three carboxy groups, the doublet signals (spin-spin interaction with the phosphorus atom nucleus) of which are found in the indicative region: δ_C 169.5, 173.6, and 174.2 (see Experimental section). Elemental analysis data also correspond to the tricarboxylate phosphabetaine.

It is necessary to note that upon prolonged storage for several months, product **3a** shows signs of decarboxylation. Apart from that, if the reaction of acid **1** with maleic acid (**2b**) is carried out under influence of heat, the decarboxylation occurs already in the course of this reaction to yield dicarboxylate phosphabetaine **4** (see Scheme 2).

In this connection, we carried out additional studies of phosphabetaine **3a** by a combined method of thermogravimetry, differential scanning calorimetry, and mass spectrometry TG-DSC-MS, which showed not very high thermal stability of the synthesized betaine. The sample was heated in the temperature range from 30 to 200 °C. No mass loss was observed in the range of 30—80 °C, whereas from 80 to 130 °C its mass changed by 13%, with simultaneous liberation of carbon dioxide and water. Note that three peaks are observed during heating the sample to 200 °C, which correspond to the liberation of carbon dioxide, that also indicates the presence of three carboxy groups in phosphabetaine **3a**.

Apart from that, the reactions of tertiary phosphines **6a—c** with maleic acid (**2b**) studied earlier[13—16] showed that the first step led to the formation of dicarboxylate phosphabetaines **7a—c**, insoluble in organic solvents, which were relatively stable under the layer of a solvent, but with time anyway underwent decarboxylation giving betaines **8a—c** (Scheme 3).

Scheme 3

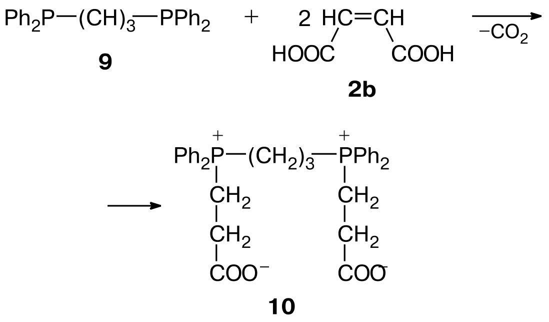


R = Ph (**a**), Bu (**b**), *cyclo*-C_6H_{11} (**c**)

In the present work, we additionally investigated betaine **7a** by ^{1}H and ^{31}P NMR spectroscopy. These studies were carried out in D_2O, since this compound is insoluble in organic solvents and can be stored only under the layer of diethyl ether. The chemical shift of the phosphorus nucleus was found in the region indicative of phosphabetaines with δ_P 27.6. The ^{1}H NMR spectrum exhibited signals for all the protons corresponding to this structure.

Another approach to the synthesis of dicarboxylate phosphabetaines used in the present work became the studies of the reaction of 1,3-bis(diphenylphosphino)propane (**9**) with maleic acid (**2b**) (Scheme 4).

Scheme 4

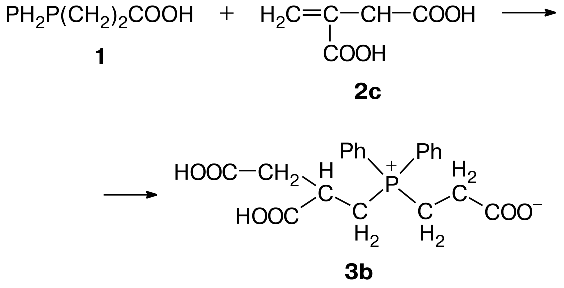


The reaction of bisphosphine **9** with maleic acid (**2b**) in acetonitrile proceeds rapidly, it is also accompanied by decarboxylation, and gives rise to colorless stable crystals with m.p. 234 °C. Elemental analysis of synthesized dicarboxylate diphosphabetaine **10** shows that it does not contain any foreign molecules in the crystal lattice, though earlier we stated many times that the stabilization of carboxylate phosphabetaines with water molecules and other proton-donor reagents is their distinguishing feature.[1—11] The structure of product **10** was confirmed by IR spectroscopy and ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy. The IR spectrum exhibited one characteristic absorption band of carboxylate anion in the region of 1580 cm^{-1}. ^{13}C NMR spectrum showed the presence of only one signal for carbon nuclei of both carboxylate groups, that indicates their magnetic equivalence. The spectrum also shows the presence of signals for all the other carbon atoms.

Such a dicarboxylate diphosphabetaine structure based on 1,2-bis(diphenylphosphino)ethane was described earlier.[17] It was shown that it contains four water molecules per one betaine molecule. Apart from that, ethylene-bis(diphenylphosphonio)propionate and propylene-bis-(diphenylphosphonio)propionate were used as ligands in different complexes both with metals and organic compounds.[18,19]

Earlier, we have studied the formation of stable dicarboxylate phosphabetaines in the reactions of tertiary phosphines with itaconic acid (**2c**).[12—16,20] In the present work, we carried out the reaction of 3-(diphenylphosphino)-propionic acid (**1**) with itaconic acid (**2c**) to prepare tricarboxylate phosphabetaine **3b** (Scheme 5).

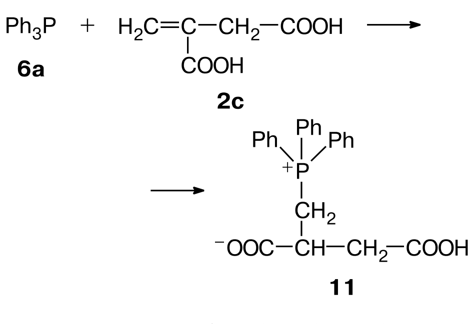
Scheme 5



The reaction proceeded rapidly in ethyl acetate or acetonitrile with the formation of the only crystalline product **3b** with m.p. 122 °C, δ_P 27. The IR spectrum exhibited two absorption bands of carboxy groups in the region of 1700 cm^{-1}, as well as that of carboxylate anion in the region of 1600 cm^{-1}. The signals for the carbon nuclei of three carboxy groups were found in the ^{13}C NMR spectrum, as well as the signals for all the other carbon atoms. Elemental analysis correspond to the composition of product **3b**. Like the dicarboxylate betaine **11** based on triphenylphosphine and itaconic acid (**2c**) (Scheme 6), betaine **3b** is stable and does not undergo decarboxylation with time.

To increase the yield of the target product, in the present work we continued the studies of the reaction of triphenylphosphine (**6a**) with itaconic acid (**2c**) already started earlier.[13,16] We changed conditions of the synthesis of betaine **11**, namely, we used diethyl ether as the solvent for acid **2c** (see Experimental section). This resulted in the preparation of the stable dicarboxylate phosphabetaine **11** with m.p. 162 °C (see Scheme 6). Its structure was confirmed by IR spectroscopy and ^{1}H and ^{31}P NMR spectroscopy. Elemental analysis confirmed the composition of compound **11**.

Scheme 6



The characteristics of carboxylate phosphabetaines obtained in the present work are given in the Experimental section.

In conclusion, new stable di- and tricarboxylate phosphabetaines were synthesized in the course of these studies, their composition and structure were confirmed by a combination of physicochemical methods.

Experimental

IR spectra were recorded on a Perkin—Elmer Spectrum Two IR Fourier-transform spectrometer. NMR spectra were recorded on a Bruker Avance III 400 Nanobay NMR spectrometer in D_2O, if another solvent is not indicated. Derivatograms TG-DSC were recorded on an STA 449 F1 Jupiter instrument of TG-DTA/DSC synchronous thermal analysis conjugated with a Netzsch QMS 403 D Aeolos mass spectrometer.

3-[(1,2-Dicarboxyethyl)diphenylphosphonio]propanoate (3a). The reaction proceeded similarly in both acetonitrile and ethyl acetate. A solution of maleic acid (**2b**) (0.14 g, 0.0012 mol) in ethyl acetate (3 mL) was added dropwise to a solution of 3-tridiphenylphosphinopropanoic acid (**1**) (0.3 g, 0.0012 mol) in EtOAc (5 mL) with continuous stirring. The reaction reached completion within several minutes to form a white precipitate, which was filtered on a Shott funnel, washed with diethyl ether, and dried *in vacuo*. The product **3a** is well soluble in water and ethanol with heating, insoluble in acetonitrile, m.p. 80 °C (from ethanol). The yield was 0.381 g (86%). IR (Nujol), ν/cm^{-1}: 1600 (COO$^-$); 1700 (COOH). ^{1}H NMR, δ: 2.43—2.57 (m, 2 H, $PCH_2C\underline{H}_2$; 1 H, $C\underline{H}CH_2$); 2.58—2.78 (m, 2 H, $CHC\underline{H}_2$); 2.43—2.57 (m, 2 H, $PC\underline{H}_2$); 7.53—7.80 (m, 10 H, Ar). ^{13}C (δ: 17.39 (d, PCH_2, $^1J_{P,C}$ = 54.2 Hz); 26.0 (d, $PCH\underline{C}H_2$, $^2J_{P,C}$ = 1.3 Hz); 26.45 (d, $PCH_2\underline{C}H_2$, $^2J_{P,C}$ = 1.3 Hz); 31.30 (d, PCH, $^2J_{P,C}$ = = 46.6 Hz); 114.60 (d, C_{ipso}, $^1J_{P,C}$ = 83.4 Hz); 130.05 (d, C_o, $^2J_{P,C}$ = 29.75 Hz); 133.57 (d, C_m, $^3J_{P,C}$ = 9.4 Hz); 135.30 (d, C_p, $^4J_{P,C}$ = 2.5 Hz); 169.51 (d, C(O)O, $^3J_{P,C}$ = 1.5 Hz); 173.58 (d, C(O)O, $^3J_{P,C}$ = 14.3 Hz); 174.09 (d, C(O)O, $^2J_{P,C}$ = 17.1 Hz). ^{31}P NMR, δ: 28.7. Found (%): C, 57.69; H, 4.69; P, 7.12. $C_{18}H_{19}O_4P$. Calculated (%): C, 58.30; H, 4.86; P, 7.19. Shows signs of decarboxylation upon prolonged storage (several months).

3-[(2,3-Dicarboxypropyl)diphenylphosphonio]propanoate (3b). A solution of itaconic acid (**2c**) (0.15 g, 0.0012 mol) in ethyl acetate (3 mL) was added dropwise to a solution of acid **1** (0.3 g, 0.0012 mol) in ethyl acetate (5 mL) with continuous stirring. The reaction reached completion within several minutes to form a white precipitate of **3b**. The precipitate was filtered on a Shott funnel, washed with diethyl ether, and dried *in vacuo*. The product **3b** is well soluble in water and ethanol, poorly soluble in acetonitrile, m.p. 122 °C (from acetonitrile). The yield was 0.379 g (84.2%). IR (Nujol), ν/cm^{-1}: 1550 (COO$^-$); 1700 (COOH). ^{1}H NMR (D_2O), δ: 2.43—2.57 (m, 2 H, $PCH_2C\underline{H}_2$; 1 H, $C\underline{H}CH_2$); 2.58—2.78 (m, 2 H, $CHC\underline{H}_2$); 2.43—2.57 (m, 2 H, $PC\underline{H}_2$); 7.53—7.80 (m, 10 H, Ar). ^{13}C NMR, δ: 17.18 (d, $P\underline{C}H_2CH_2$, $^1J_{P,C}$ = 54.3 Hz); 22.7 (d, $P\underline{C}H_2CH$, $^1J_{P,C}$ = 52.3 Hz); 26.7 (d, $PCH_2\underline{C}H_2$, $^2J_{P,C}$ = 1.3 Hz); 37.07 (s, $PCH_2CH\underline{C}H_2$); 37.98 (d, $PCH_2\underline{C}H$, $^2J_{P,C}$ = 12.8 Hz); 116.23 (d, C_{ipso}, $^1J_{P,C}$ = = 84.7 Hz); 130.05 (d, C_o, $^2J_{P,C}$ = 12.4 Hz); 133.57 (d, C_m, $^3J_{P,C}$ = 9.6 Hz); 135.30 (d, C_p, $^4J_{P,C}$ = 2.5 Hz); 175.05—175.35 (m, 2 C(O)O); 176.78 (d, C(O)O, $^4J_{P,C}$ = 3.1 Hz). ^{31}P NMR, δ: 27.0. Found (%): C, 60.99; H, 5.70; P, 7.98. $C_{20}H_{21}O_6P$. Calculated (%): C, 61.86; H, 5.41; P, 7.99.

3-[(1,2-Dicarboxyethyl)diphenylphosphonio]propanoate (3a) and 3-[(2-carboxyethyl)diphenylphosphonio]propanoate (4). A solution of fumaric acid (**1a**) (0.22 g, 0.0019 mol) in hot distilled water (5 mL) was added dropwise to a solution of acid **1** (0.5 g, 0.0019 mol) in acetonitrile (5 mL) with continuous stirring. The reaction mixture was allowed to stand for 1 days. ^{31}P NMR spectrum of the reaction mixture (D_2O, δ: 26.6 (**3a**), 28.8 (**4**) (both s). The second signal increased with time. Anhydrous diethyl ether was poured to the reaction mixture, a precipitate formed was filtered on a Shott funnel, washed with diethyl ether, and dried *in vacuo*. The product **4** is well soluble in water and ethanol with heating, insoluble in acetonitrile, m.p. 236 °C (from ethanol). The yield was 0.591 g (82.08%). IR (Nujol), ν/cm^{-1}: 1680 (COO^-). ^{1}H NMR (D_2O)), δ: 2.38—2.45 (m, 4 H, $CH_2C(O)$); 3.04—3.11 (m, 4 H, PCH_2); 7.57—7.76 (m, 10 H, Ar). ^{13}C NMR, δ: 16.73 (d, PCH_2, $^{1}J_{\mathrm{P,C}}$ = 53.0 Hz); 27.28 (d, $PCH_2\underline{C}H_2$, $^{2}J_{\mathrm{P,C}}$ = 3.0 Hz); 116.51 (d, C_{ipso}, $^{1}J_{\mathrm{P,C}}$ = 85.0 Hz); 129.99 (d, C_o, $^{2}J_{\mathrm{P,C}}$ = 13.0 Hz); 132.86 (d, C_m, $^{3}J_{\mathrm{P,C}}$ = 10.0 Hz); 135.03 (d, C_p, $^{4}J_{\mathrm{P,C}}$ = 3.0 Hz); 175.87 (d, C(O)O, $^{3}J_{\mathrm{P,C}}$ = 14.5 Hz). ^{31}P NMR, δ: 28.8. Found (%): C, 66.27; H, 5.50; P, 9.09. $C_{18}H_{19}O_4P$. Calculated (%): C, 65.45; H, 5.75; P, 9.39.

2-[(Carboxymethyl)diphenylphosphonio]acetate (7a). A solution of maleic acid (**2b**) (0.23 g, 0.0019 mol) in acetonitrile (3 mL) was added dropwise to a solution of triphenylphosphine **6a** (0.5 g, 0.0019 mol) in acetonitrile (5 mL) with continuous stirring. The reaction reached completion within several minutes to form a white precipitate of **7a**, which was insoluble in organic solvents. Attempted isolation of crystalline product **7a** led to its decarboxylation. The precipitate remained stable under the layer of diethyl ether. ^{1}H NMR (D_2O), δ: 2.71—2.78 (m, 1 H, PCH); 2.89 (dd, 2 H, $C\underline{H}_2$, *J* = 51.8 Hz, *J* = 17.1 Hz); 2.43—2.57 (m, 2 H, $PC\underline{H}_2$); 7.54—7.85 (m, 15 H, Ar). ^{31}P NMR, δ: 25.0. The decarboxylation product **8a** was characterized by IR spectra, ^{1}H, ^{13}C, and ^{31}P NMR spectra, and X-ray diffraction.[5]

3,3´-[Propane-1,3-diylbis(diphenylphosphonionediyl)]dipropanoate (10). A solution of maleic acid (**2b**) (0.28 g, 0.0024 mol) in acetonitrile (5 mL) was added dropwise to a solution of 1,3-bis(diphenylphosphino)propane (**9** (0.5 g, 0.0012 mol)) in acetonitrile (5 mL) with continuous stirring. The reaction mixture was allowed to stand for 1 days at room temperature, showing signs of decarboxylation. The solvent was removed *in vacuo*. A precipitate formed was filtered on a Shott funnel, washed with diethyl ether, and dried *in vacuo*, which resulted in the formation of colorless crystals with m.p. 234 °C (from acetonitrile). The yield was 0.510 g (65.4%). IR (Nujol), ν/cm^{-1}: 1580 (COO^-). ^{1}H NMR (D_2O), δ: 1.38—1.52 (m, 2 H, $PCH_2C\underline{H}_2CH_2$); 2.14—2.27 (m, 4 H, $CH_2C(O)$); 2.83—3.01 (m, 8 H, PCH_2); 7.44—7.78 (m, 20 H, Ar). ^{13}C NMR, δ: 17.59 (s, $CH_2\underline{C}H_2CH_2$); 19.92 (d, $P\underline{C}H_2CH_2C(O)O$, $^{1}J_{\mathrm{P,C}}$ = 52.2 Hz); 23.71 (dd, $P\underline{C}H_2CH_2CH_2$, $^{1}J_{\mathrm{P,C}}$ = 52.3 Hz, $^{1}J_{\mathrm{P,C}}$ = 16.9 Hz); 31.04 (s, $\underline{C}H_2C(O)O$); 118.72 (d, C_{ipso}, $^{1}J_{\mathrm{P,C}}$ = 85.0 Hz); 132.61 (d, C_o, $^{2}J_{\mathrm{P,C}}$ = 6.3 Hz); 135.18 (d, C_m, $^{3}J_{\mathrm{P,C}}$ = 4.3 Hz); 137.70 (s, C_p); 179.89 (d, C(O)O, $^{3}J_{\mathrm{P,C}}$ = 12.3 Hz). ^{31}P NMR, δ: 27.8. Found (%): C, 70.73; H, 5.90; P, 11.00. $C_{33}H_{34}O_4P_2$. Calculated (%): C, 71.22; H, 6.11; P, 11.15.

3-Carboxy-2-[(triphenylphosphonio)methyl]propanoate (11). A solution of itaconic acid (**2c**) (0.81 g, 0.0062 mol) in diethyl ether (5 mL) was added dropwise to a solution of triphenylphosphine **6a** (1.63 g, 0.0062 mol) in acetonitrile (10 mL) with continuous stirring. The reaction mixture was allowed to stand for one week at room temperature. The solvent was evaporated *in vacuo* to give a white crystalline product. The precipitate was filtered on a Shott funnel, washed with diethyl ether, and dried *in vacuo*, m.p. 162 °C (from ethanol). The yield was 1.537 g (62.99%). IR (Nujol), ν/cm^{-1}: 1590 (COO^-); 1720 (COOH); 1730 (COOH). ^{1}H NMR (D_2O), δ: 2.26—2.54 (dd, 2 H, $PC\underline{H}_2CH_2$, *J* = 51.51 Hz, *J* = 7.25 Hz); 2.85 (m, 1 H, CH); 3.11—3.63 (dd, 2 H, $PC\underline{H}_2$, *J* = 130.29 Hz, *J* = 13.83 Hz); 7.30—7.81 (m, 15 H, Ar). ^{13}C NMR, δ: 23.65 (d, $P\underline{C}H_2$, $^{1}J_{\mathrm{P,C}}$ = 53.9 Hz); 37.92 (s, $\underline{C}H_2C(O)$); 38.98 (d, $PCH_2\underline{C}H$, $^{2}J_{\mathrm{P,C}}$ = 12.4 Hz); 117.74 (d, C_{ipso}, $^{1}J_{\mathrm{P,C}}$ = 86.9 Hz); 129.91 (d, C_o, $^{2}J_{\mathrm{P,C}}$ = 12.7 Hz); 133.46 (d, C_m, $^{3}J_{\mathrm{P,C}}$ = 9.6 Hz); 134.96 (d, C_p, $^{4}J_{\mathrm{P,C}}$ = 2.0 Hz); 175.96, 176.98 (both s, COO^-, COOH)). ^{31}P NMR, δ: 21.8. ^{31}P NMR ($CDCl_3$), δ: 23.6. Found (%): C, 70.67; H, 5.37; P, 7.68. $C_{23}H_{21}O_4P$. Calculated (%): C, 70.41; H, 5.36; P, 7.91.

This work was financially supported by the Ministry of Education and Science of the Russian Federation (at the expense of subsidy allocated to the Kazan Federal University for the Project part of the State Assignment in Academic Area).

References

1. V. I. Galkin, Yu. V. Bakhtiyarova, N. A. Polezhaeva, R. A. Cherkasov, D. B. Krivolapov, A. T. Gubaidullin, I. A. Litvinov, *Phosphorus, Sulfur, Silicon Relat. Elem.*, 1999, **147**, 91.
2. V. I. Galkin, Yu. V. Bakhtiyarova, N. A. Polezhaeva, I. V. Galkina, R. A. Cherkasov, D. B. Krivolapov, A. T. Gubaydullin, I. A. Litvinov, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2002, **72**, 376 [*Zh. Obshch. Khim.*, 2002, **72**, 404].
3. V. I. Galkin, Yu. V. Bakhtiyarova, N. A. Polezhaeva, I. V. Galkina, R. A. Cherkasov, D. B. Krivolapov, A. T. Gubaydullin, I. A. Litvinov, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2002, **72**, 384 [*Zh. Obshch. Khim.*, 2002, **72**, 412].
4. V. I. Galkin, Yu. V. Bakhtiyarova, N. A. Polezhaeva, I. V. Galkina, R. A. Cherkasov, D. B. Krivolapov, A. T. Gubaydullin, I. A. Litvinov, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2006, **76**, 430 [*Zh. Obshch. Khim.*, 2006, **76**, 452].
5. V. I. Galkin, A. V. Salin, Yu. V. Bakhtiyarova, A. A. Sobanov, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2009, **79**, 919 [*Zh. Obshch. Khim.*, 2009, **79**, 747].
6. I. V. Galkina, N. B. Mel´nikova, Yu. V. Bakhtiyarova, V. V. Stakheev, V. I. Galkin, O. E. Zhil´tsova, O. V. Zhukova, S. N. Egorova, *Farmatsiya* [*Pharmacy*], 2010, **3**, C. 47 (in Russian).
7. A. V. Salin, A. A. Sobanov, Yu. V. Bakhtiyarova, A. A. Khabibullin, V. I. Galkin, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2010, **80**, 1736 [*Zh. Obshch. Khim.*, 2010, **80**, 1418].
8. A.V. Salin, A.A. Sobanov, Yu. V. Bakhtiyarova, A. A. Khabibullin, V. I. Galkin, R. A. Cherkasov, *Phosphorus*, *Sulfur*, *Silicon Relat. Elem.*, 2011, **186**, 854.
9. A. V. Salin, A. A. Sobanov, Yu. V. Bakhtiyarova, A. A. Khabibullin, V. I. Galkin, R. A. Cherkasov, *Phosphorus*, *Sulfur*, *Silicon Relat. Elem.*, 2011, **186**, 857.
10. A. V. Salin, A. A. Sobanov, Yu. V. Bakhtiyarova, A. A. Khabibullin, V. I. Galkin, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2011, **81**, 824 [*Zh. Obshch. Khim.*, 2011, **81**, 737].
11. I. V. Galkina, Yu. V. Bakhtiarova, M. P. Shulaeva, O. K. Pozdeev, S. N. Egorova, R. A. Cherkasov, V. I. Galkin, *J. Chem.*, 2013, http://dx.doi.org/10.1155/2013/302937.
12. Yu. V. Bakhtiyarova, R. R. Minnullin, I. V. Galkina, R. A. Cherkasov, V. I. Galkin, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2015, **85**, 2037 [*Zh. Obshch. Khim.*, 2015, **85**, 1453].
13. V. I. Galkin, Yu. V. Bakhtiyarova, I. V. Galkina, R. A. Cherkasov, A. N. Pudovik, D. B. Krivolapov, A. T. Gubaidullin, I. A. Litvinov, *Phosphorus*, *Sulfur*, *Silicon Relat. Elem.*, 2002, **177**, 2063.

14. V. I. Galkin, Yu. V. Bakhtiyarova, R. I. Sagdieva, I. V. Galkina, R. A. Cherkasov, *Heteroatom Chem.*, 2006, **17**, 557.
15. Yu. V. Bakhtiyarova, M. S. Bondar´, I. V. Galkina, V. I. Galkin, *Uchenye zapiski Kaz. un-ta. Estestvennye nauki* [*Sci. Notes Kaz. Univ. Natural Sci.*], 2008, **150**, No. 1, 42 (in Russian).
16. Yu. V. Bakhtiyarova, R. I. Sagdieva, I. V. Galkina, V. I. Galkin, R. A. Cherkasov, D. B. Krivolapov, A. T. Gubaidullin, I. A. Litvinov, *Russ. J. Org. Chem.* (*Engl. Transl.*), 2007, **43**, 207 [*Zh. Org. Khim.*, 2007, **43**, 215].
17. Aly Aref A. M., H. Schmidbaur, *Zeitschr. Naturforsch.*, *B: Chem. Sci.*, 1991, **46**, 775.
18. I. Galkina, A. Tufatullin, D. Krivolapov, Yu. Bakhtiyarova, D. Chubukaeva, V. Stakheev, V. Galkin, R. Cherkasov, B. Buchnerc, O. Kataeva, *CrystEngComm*, 2014, **16**, 9010.
19. Li Song-Lin, T. C. W. Mak, *J. Mol. Struct.*, 1996, **384**, 135.
20. V. I. Galkin, A. V. Salin, Yu. V. Bakhtiyarova, *Uchenye zapiski Kaz. un-ta. Estestvennye nauki* [*Sci. Notes Kaz. Univ. Natural Sci.*], 2008, **150**, No. 3, 54 (in Russian).

Received November 9, 2015