Crystal structure of new carboxylate phosphabetaines and phosphonium salts conjugated with them*

Yu. V. Bakhtiyarova,[a] A. F. Aksunova,[a] I. V. Galkina,[a] V. I. Galkin,[a] O. A. Lodochnikova,[b] and O. N. Kataeva[b]*

[a]*A. M. Butlerov Chemical Institute of Kazan (Volga Region) Federal University, 18 ul. Kremlevskaya, 420008 Kazan, Russian Federation. Fax: +7 (843 2) 238 7901. E-mail: vig54@mail.ru*

[b]*A. E. Arbuzov Institute of Organic and Physical Chemistry, Kazan Scientific Center of the Russian Academy of Sciences, 8 ul. Akad. Arbuzova, 420088 Kazan, Russian Federation*

Earlier unknown crystalline forms of three carboxylate phosphabetaines and conjugated with them phosphonium salts differing by β substituent with respect to the carboxylate group were studied. The structure of studied compounds in crystal is determined by intermolecular electrostatic interactions. This leads to the *trans* arrangement of the carboxylate and the phosphonium groups.

Key words: carboxylate phosphabetaine, single crystal X-ray diffraction, intermolecular interactions.

The study of the structure of betaines and conjugated with them phosphonium salts is substantiated by a possibility of their use in medicine,[1—11] as well as by the fact that their many biological functions are determined by the coordination properties. The ability of phosphabetaines to bind metal ions is due to the presence of such functional groups as phosphate, sulfate, carboxylate. Apart from the anionic group, which is responsible for the coordination properties of compounds, a cationic group is no less important. It is known that a cationic group in surface-active compounds determines their interaction with substrates, critical micelle concentration, *etc*. Its significant role can be observed, for example, in gene therapy. It was found that the substitution of an ammonium group most frequently encountered in biologically active systems with the phosphonium or arsonium ones led to an enhancement of transfection activity and a decrease in the cationic carrier cytotoxicity.[12—16] Apart from that, arsonium and phosphonium compounds are more stable than ammonium derivatives.[17—20]

Recent studies demonstrated broad possibilities in the use of triphenylphosphonium derivatives in the area of biotechnologies due to their lipophilic head group with the delocalized positive charge.[21—23]

Thus, a combination of a phosphonium cationic group and a carboxylate functional group in one molecule is promising for the development of biologically active compounds, whereas the studies of the structure of phosphabetaines, their crystal structure, intramolecular and intermolecular interactions in crystal are of special importance for the interpretation of properties of these compounds.

In the present work, we report the earlier unknown crystalline forms of carboxylate phosphabetaines **1**—**3** with the ethylene bridge connecting the phosphonium and the carboxylate groups and having different substituents at β-position. Earlier, we have studied complexation properties of a number of carboxylate phosphabetaines,[24] carried out quantum chemical modeling and calculations of the most stable conformation. The DFT calculations showed that a *gauch*-conformation of the molecule is characteristic of the free state of phosphabetaines under consideration, which provides the closest arrangement of the oppositely charged phosphonium and carboxylate groups.[24]

Results and Discussion

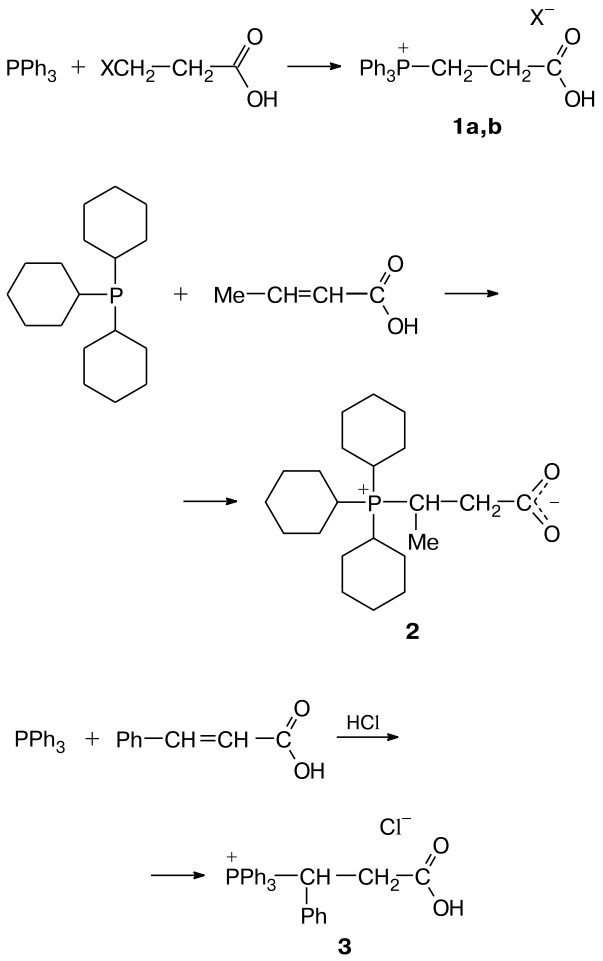
In order to study the influence of intra- and intermolecular interactions, as well as substituents on the structure of carboxylate phosphabetaines and their hydrohalides in crystal, in particular on their conformational behavior in crystals, we synthesized the corresponding betaines both in the free form and as hydrohalides of their phosphonium salts, and then we studied the crystal structures of four compounds: **1a**, **1b**, **2**, and **3** (Scheme 1).

The indicated compounds were synthesized according to the procedures developed by us earlier for other carboxylate phosphabetaines[25—29] based on the reactions of

* 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. 1313—1318, May, 2016.

Scheme 1



X = Cl (**a**), Br (**b**)

tertiary phosphines with unsaturated carboxylic acids (see Scheme 1).

The crystals of compounds **1a**,**b** and **3** were studied as phosphonium halides stabilized by hydrogen bonds O—H···Cl^- (**1a**, **3**) and O—H···Br^- (**1b**), as well as the crystals of compound **2**, in which phosphabetaine is co-crystallized with crotonic acid. The crystals of betaine **1** and its derivatives considered earlier in the literature indicate the formation of different forms of phosphonium salts: zwitterionic with water and acetonitrile solvent molecules in crystals and protonated with molecules of fumaric acid anion.[25—29] The structure of β-substituted betaines **2** and **3** was not virtually studied: only one structure of the crystal of compound **3** was reported, in which it is in the protonated form with the chloride anion and a chloroform solvent molecule.[30] The structure of compound **2** was not studied earlier by X-ray diffraction.

The crystal of **1a** is a 1 : 1 crystal solvate with acetonitrile. In contrast to the solvate of betaine **1** with the same solvent studied earlier,[31] in this case compound **1** is

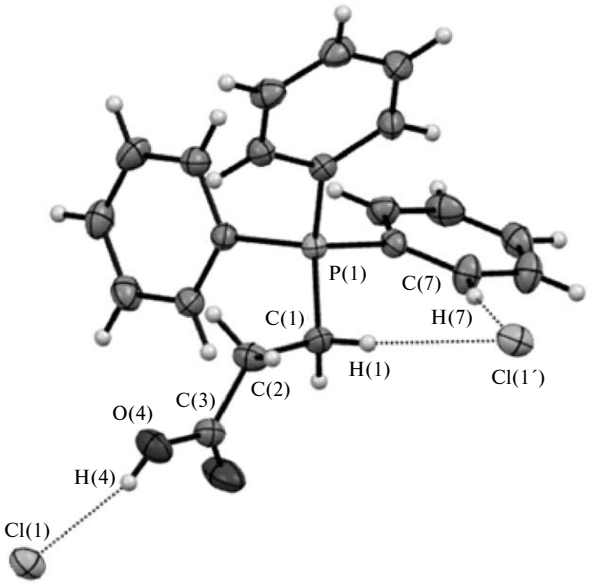


Fig. 1. The structure of phosphonium salt **1a**, hydrogen bonds are shown by dotted lines.

in the protonated form, rather than in zwitterionic, therefore, the crystal structure of **1a** contains a chloride anion (Fig. 1).

The phosphorus atom in crystal **1a** is in the usual tetrahedral surrounding with the P—C bond distances from 1.789(2) to 1.802(2) Å and the bond angle values at the phosphorus atom from 107.7(1) to 110.6(1)°. The C—O bond distances in the carboxy group (1.194(3) Å and 1.316(3) Å), as well as the presence of a hydrogen atom at the carboxy group unambiguously indicate that the molecule exists in the protonated form $[Ph_3P(CH_2)_2CO_2H]^+$, whereas the chlorine atom acts as a counterion. In the crystal, a hydrogen bond is observed between the hydrogen atom of the carboxy group and the chloride anion (see Fig. 1, Table 1).

The space between the triphenylphosphonium groups of neighboring cations of salt **1a** is occupied by the acetonitrile solvent molecules, which fill the channels in the crystal (Fig. 2).

The crystal **1b**, in contrast to **1a** considered above, does not contain the solvent molecule and includes only molecule **1** in the protonated form and a bromide anion (Fig. 3). The geometry of phosphonium salt considerably differs for two crystalline forms of its hydrohalide derivatives. Thus, if crystal **1a** has a traditional and most often encountered *syn*-conformation of the carboxy group, in crystal **1b** it is in the rare *anti*-conformation described earlier in the review on crystallization of carboxylic acids.[32] The energy of *anti*-conformation is lower than the energy of *syn*-conformation by about 2—4 kcal mol^{-1} and, therefore, *anti*-conformation is usually observed in the case of the presence of some intra- or intermolecular interactions. Earlier, we have found such a conformation in the

Table 1. Parameters of hydrogen bonds in crystals **1—3**

Crystal	D—H···A	D—H	H···A	D···A	Angle D—H···A /deg
		Å			
1a	O4)—H(4)···Cl(1)	0.77(4)	2.22(4)	2.966(2)	164(4)
	C(1)—H(1)···Cl(1)	0.97	2.75	3.712(2)	173
	C(21)—H(21)···Cl(1)	0.93	2.65	3.571(3)	170
1b	O(4)—H(4)···Br(1)	0.82(3)	2.31(3)	3.128(2)	179(4)
	C(2)—H(2)···Br(1)	0.99	2.72	3.624(2)	152
2	O(26)—H(26)···O(4)	1.07(4)	1.39(4)	2.457(3)	172(4)
3	O(4)—H(4)···Cl(1)	0.95(3)	2.09 (3)	3.033(2)	171(3)
	C(1)—H(1)···Cl(1)	0.98	2.49	3.427(2)	161

crystal of the representative of keto acids,[33] where it was stabilized by an intramolecular hydrogen bond. In crystal **1b**, apparently, the *anti*-conformation of the carboxy group is realized due to the tendency of the bromine atom to the formation of not only classic hydrogen bond O—H...Br, but also nonclassic interaction C—H...Br (see Fig. 3 and Table 1).

Apart from that, the conformation of the central fragment P(1)C(1)C(2)C(3) changes from the *gauch* (in **1a**) to the *trans* (in crystal **1b**, the torsion angle is 152.6(1)°). This leads to more dense molecular packing and the absence of solvate molecules in the crystal.

The crystal **2** is a 1 : 1 molecular complex of β-methyl-substituted betaine and crotonic acid. In the crystal **2**, the geometry of the phosphorus atom (Fig. 4) is close to that in the crystal of compound **1a**: the bond angles at the phosphorus atom are virtually identical (from 108.2(1) to 111.8(1)°). However, in this compound the replacement of a triphenylphosphonium group with the tricyclohexylphosphonium one leads to the insignificant elongation of the P—C bonds. The C—O bond distances in the carboxylate group of betaine in crystal **2** (1.223(3) and 1.283(3) Å), as well as the geometrical parameters of the carboxy group of crotonic acid, in which the hydrogen atom is found and refined in isotropic approximation (O(26)—H(26) = = 1.07(4) Å; O(4)...H(26) = 1.39(4) Å), indicate that in this case the betaine exists in the zwitterionic form.

The crystal **3** is a phosphonium salt, in which the carboxy moiety is protonated, with chloride anion being a counterion. In this structure, like in the crystal **1b** considered above, the carboxy group is in the *anti*-conformation, that also facilitates additional interactions C—H...Cl^- (Fig. 5, Table 1). The tetrahedral geometry of phosphorus atom is close to the geometry of compound **1a**, that is reflected in similar bond distances and bond angles. The C—O bond distances of the carboxy group (1.194(2) and

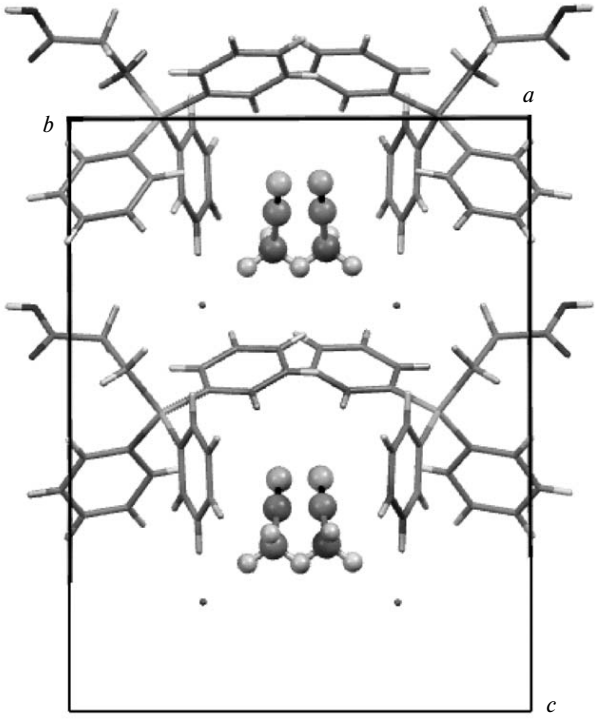


Fig. 2. A fragment of the crystal packing of phosphonium salt **1a**.

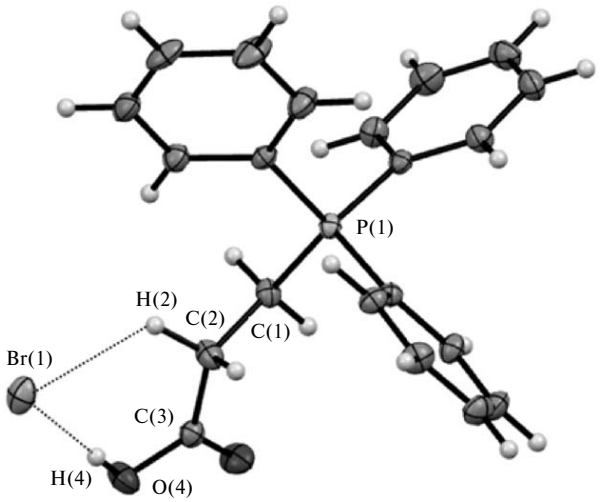


Fig. 3. The structure of phosphonium salt **1b**, intermolecular interactions are shown by dotted lines.

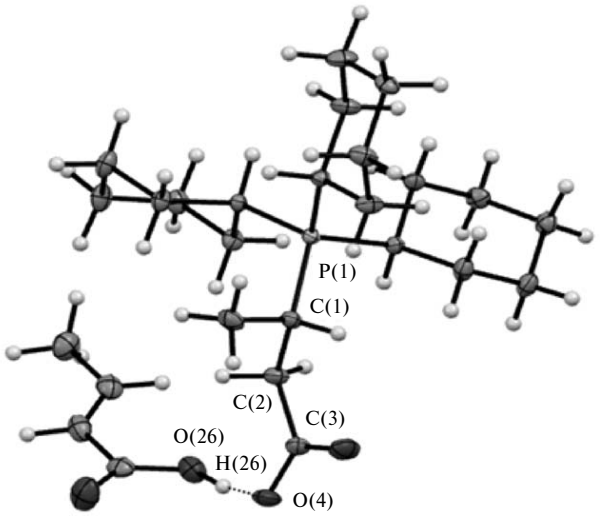


Fig. 4. The structure of phosphabetaine **2**.

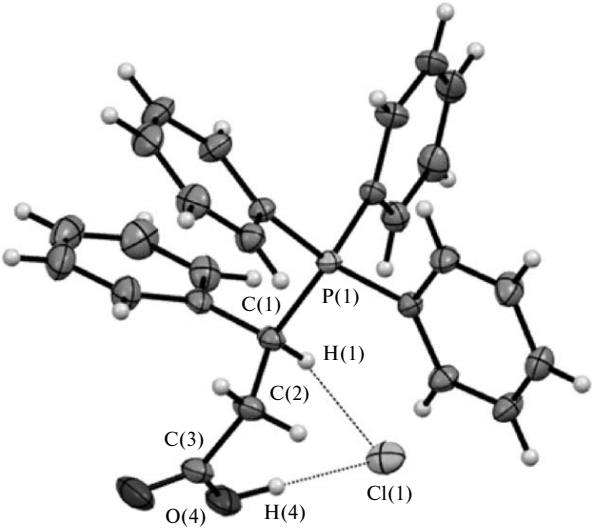


Fig. 5. The structure of phosphonium salt **3**.

1.315(2) Å) indicate that the betaine molecule is represented in the crystal by a protonated form.

Note that the differences in the intermolecular interactions lead to conformational differences in the series of compounds **1**—**3**: the *gauch*-conformation exists in compound **1a** (the torsion angle P(1)—C(1)—C(2)—C(3) = = 126.7(1)°), the *trans*-conformation in compounds **1b** (the torsion angle P(1)—C(1)—C(2)—C(3) = 152.6(1)°), **2** (the torsion angle P(1)—C(1)—C(2)—C(3) = 152.0(1)°), and **3** (the torsion angle P(1)—C(1)—C(2)—C(3) = = 176.5(1)°). Interestingly that in the earlier studied crystals of compound **1** stabilized by water molecules,[32] fumaric acid anion,[28] fumaric acid anion and water,[28] as well as acetonitrile molecule,[32] a *trans*-conformation was realized with the torsion angles P(1)—C(1)—C(2)—C(3) lying within the range 156.1(2)—172.9(1)°. In the case of compound **1a**, the *gauch*-conformation becomes more favorable, apparently, due to the weak intermolecular interactions C—H···Cl (see Fig. 1 and Table 1).

In conclusion, the structure in crystal of phosphabetaines under study is mainly determined by intermolecular electrostatic interactions. This leads to the conversion of the *gauch*-conformation typical of the gas phases to the predominantly transoid. Such a structural lability of carboxylate phosphabetaines should improve their complexation properties, since it allows the ligands to adopt a conformation optimal for the metal coordination.

Experimental

^{1}H and ^{31}P NMR spectra were recorded on a Bruker Avance-400 spectrometer. IR spectra were recorded on a IR Prestige-21 spectrometer in the range 400—3700 cm^{-1} in Nujol or liquid film between KBr plates.

Synthesis of (2-carboxyethyl)triphenylphosphonium chloride (1a). A mixture of triphenylphosphine (1 g, 0.0038 mol) and ω-chloropropionic acid (0.43 g, 0.0038 mol) was melted on a water bath at a temperature 100 °C for 21 h. The melt was treated with water, a part of the solid compound was transferred into solution, an insoluble in water white precipitate of unreacted triphenylphosphine and formed in the course of the reaction triphenylphosphine oxide was filtered off. The aqueous filtrate was concentrated, a white precipitate formed was washed with diethyl ether. The yield was 0.95 g (57.2%), m.p. 198 °C (from acetonitrile). The Beilstein test was positive. The substance is well soluble in water and chloroform, insoluble in diethyl ether. IR (KBr pellets), ν/cm^{-1}: 1160 s (C—O); 1223 m (COH); 1710 (COOH); 2945 m (OH). ^{1}H NMR (D_2O), δ: 2.47 (m, 2 H, $PCH_2C\underline{H}_2$); 3.44 (m, 2 H, PCH_2); 7.35—7.85 (m, 15 H, Ph). ^{13}C NMR (D_2O), δ: 17.36 (d, PCH_2, $^1J_{P,C}$ = 56.1 Hz); 26.54 (s, $PCH_2\underline{C}H_2$); 117.25 (d, C_{ipso}, $^1J_{P,C}$ = 87.4 Hz); 130.08 (d, C_o, $^2J_{P,C}$ = 12.8 Hz); 133.46 (d, C_m, $^3J_{P,C}$ = 10.2 Hz); 135.17 (d, C_p, $^4J_{P,C}$ = 2.6 Hz); 174.17 (d, COOH, $J_{P,C}$ = 15.1 Hz). ^{31}P NMR (D_2O), δ: 24.83. Found (%): C, 67.91; H, 5.35; Cl, 9.80; P, 8.34. $C_{21}H_{20}ClO_2P$. Calculated (%): C, 68.04; H, 5.40; Cl, 9.56; P, 8.36.

Synthesis of (2-carboxyethyl)triphenylphosphonium bromide (1b). The product **1b** was obtained similarly to **1a** by melting triphenylphosphine (0.5 g, 0.0019 mol) and ω-bromopropionic acid (0.29 g, 0.0019 mol) at 100 °C for 23 h. The yield was 0.618 g (78.4%), a white powder, m.p. 195 °C (from acetonitrile). Well soluble in water, chloroform. Insoluble in diethyl ether. IR (KBr pellets), ν/cm^{-1}: 1200 s (C—O); 1225 m (COH); 1720 (COOH); 2945 m (OH). ^{1}H NMR (D_2O), δ: 2.45 (m, 2 H, $PCH_2C\underline{H}_2$); 3.49 (m, 2 H, PCH_2); 7.35—7.85 (m, 15 H, Ph). ^{13}C NMR (D_2O), δ: 17.35 (d, PCH_2, $^1J_{P,C}$ = 56.1 Hz); 26.54 (d, $PCH_2\underline{C}H_2$, $^2J_{P,C}$ = 1.9 Hz); 117.25 (d, C_{ipso}, $^1J_{P,C}$ = 87.4 Hz); 130.06 (d, C_o, $^2J_{P,C}$ = 12.8 Hz); 133.48 (d, C_m, $^3J_{P,C}$ = 10.2 Hz); 135.14 (d, C_p, $^4J_{P,C}$ = 2.6 Hz); 174.18 (d, COOH, $J_{P,C}$ = 15.3 Hz). ^{31}P NMR (D_2O), δ: 23.41. According to the elemental analysis data, the salt is stabilized by acetonitrile molecule. Found (%): C, 61.23; H, 4.20; Br, 17.40; P, 7.40. $C_{23}H_{23}BrNO_2P$. Calculated (%): C, 60.53; H, 5.04; Br, 17.53; P, 6.80.

Synthesis of 3-(tricyclohexylphosphonio)butanoate (2). A solution of crotonic acid (0.112 g, 0.0013 mol) in acetonitrile (5 mL)

Table 2. Parameters of X-ray diffraction studies of carboxylate phosphabetaines

Parameter	**1a**	**1b**	**2**	**3**
Molecular formula	$C_{21}H_{20}ClO_2P \cdot C_2H_3N$	$C_{21}H_{20}BrO_2P$	$C_{22}H_{39}O_2P \cdot C_4H_6O_2$	$C_{27}H_{24}ClO_2P$
Molecular weight/g mol^{-1}	411.84	415.25	452.59	446.88
Z	4	4	2	4
Crystal system	Orthorhombic	Monoclinic	Triclinic	Monoclinic
Calculated density, ρ/g cm^{-3}	1.240	1.444	1.202	1.321
Absorption coefficient, μ/cm^{-1}	2.63	22.47	1.39	2.63
T/K	296(2)	198(2)	198(2)	296(2)
a/Å	9.2404(5)	9.5369(5)	9.528(1)	9.851(1)
b/Å	13.6091(8)	14.3626(7)	10.629(1)	13.716(2)
c/Å	17.540(1)	13.9618(7)	12.757(1)	16.632(2)
α/deg	90	90	101.736(1)	90
β/deg	90	92.698(1)	93.269(1)	91.237(4)
γ/deg	90	90	97.232(1)	90
V/Å^3	2205.7(2)	1910.3(2)	1250.3(2)	2246.7(5)
Space group	$Pca2_1$	$P2_1/n$	$P\bar{1}$	$P2_1/n$
Number of reflections				
measured	17685	19238	5930	29618
independent with $I > 2\sigma(I)$	4377	3460	4257	3747
R_{int}	0.0347	0.0333	0.0607	0.0375
Range of masurements θ_{min}—θ_{max}/deg	2.32 — 27.99	2.035 — 27.99	1.636 — 28.00	1.925 — 25.997
Completeness of data	0.99	1.000	0.982	1.000
R_1 ($I > 2\sigma$)	0.0314	0.0321	0.0572	0.0339
wR_2 (for all reflections)	0.0750	0.0764	0.1274	0.0987
GOOF	0.958	1.045	1.029	1.043
Number of parameters	258	230	297	284
Extrema of residual electron density/e Å^{-3}	0.206/−0.150	0.298/−0.493	0.454/−0.340	0.339/−0.220

was added dropwise to a solution of tricyclohexylphosphine (0.364 g, 0.0013 mol) in benzene (5 mL) with continuous stirring. The reaction mixture was allowed to stand for one week at room temperature. The solvent was removed *in vacuo*. After addition of diethyl ether, a precipitate was formed as a white powder, which was washed with diethyl ether, filtered, and dried *in vacuo*. Product **2** is well soluble in chloroform, acetone, and water, m.p. 154 °C (from pentane). The yield was 0.231 g (48.53%). IR (Nujol), ν/cm^{-1}: 1600 (COO^-). ^{31}P NMR (CD_3CN), δ: 34.8. Found (%): C, 67.94; H, 9.77; P, 6.68. $C_{26}H_{45}O_4P$. Calculated (%): C, 68.87; H, 10.15; P, 6.84.

Synthesis of (2-carboxy-1-phenylethyl)triphenylphosphonium chloride (3). A solution of cinnamic acid (0.28 g, 0.0019 mol) in diethyl ether (5 mL) was added dropwise to a solution of triphenylphosphine (0.5 g, 0.0019 mol) in acetonitrile (5 mL) with continuous stirring. The reaction mixture was allowed to stand for one month at room temperature. To more efficiently isolate phosphabetaine, a slight excess of an aqueous solution of hydrochloric acid was added to the reaction mixture. After removal of a precipitate of unreacted triphenylphosphine, a white crystalline product was precipitated from the filtrate, m.p. 227—229 °C (from a mixture of ethanol—diethyl ether). Product **3** is soluble in acetonitrile, in water with heating and insoluble in diethyl ether. The yield was 0.615 g (78.85%). IR (Nujol), ν/cm^{-1}: 1720 (COOH). ^{31}P NMR (D_2O), δ: 24.2. Found (%): C, 71.36; H, 5.21; P, 6.90; Cl, 8.15. $C_{27}H_{24}O_2PCl$. Calculated (%): C, 72.56; H, 5.38; P, 6.94; Cl, 7.95.

X-ray diffraction studies of crystals were carried out on a Bruker SMART Apex II diffractometer (graphite monochromator, λ-Mo-$K\alpha$ 0.71073 Å). Crystallographic data and parameters of structure refinement are given in Table 2. Semi-empirical correction for absorption was carried out using the SADABS[33] program. The structures were solved by direct method using the SHELXS[34] program. Nonhydrogen atoms were refined in isotropic and then in anisotropic approximation using the SHELXL-2014[34] program. Hydrogen atoms at the carbon atoms were placed in calculated positions and refined using a riding model. Hydroxy hydrogen atoms were localized from the difference Fourier series, their positions were refined isotropically in the final step of refinement. all the calculations were performed using the WinGX[35] and APEX2[36] programs.

The X-ray diffraction data for the structures were deposited with the Cambridge Crystallographic Data Center, the CCDC numbers are: 1437095 (**1a**), 1437096 (**1b**), 1437097 (**2**), 1437098 (**3**).

The work was performed at the expense of subsidy allocated to the Kazan Federal University for the State Assignment in Academic Area.

References

1. T. Aniszewski, *Alkaloids — Secrets of Life: Alkaloid Chemistry, Biological Significance, Applications and Ecological Role*, 1st ed, Amsterdam, Elsevier Science, 2007, 335 pp.

2. G. A. Cordell, *The Alkaloids*, 64, Elsevier Science, 2007, 248 pp.
3. Z. Wang, E. Klipfell, B. J. Bennett, R. Koeth, B. S. Levison, B. DuGar, A. E. Feldstein, E. B. Britt, X. Fu, Y.-M. Chung, Y. Wu, P. Schauer, J. D. Smith, H. Allayee, W. H. Wilson Tang, J. A. DiDonato, A. J. Lusis, S. L. Hazen, *Nature*, 2011, **472**, 57.
4. E. K. Perttu, A. G. Kohli, F. C. Szoka, *J. Am. Chem. Soc.*, 2012, **134**, 4485.
5. X. Yu, J. Zheng, *J. Mol. Biol.*, 2012, **421**, 561.
6. F. Mollinedo, C. Gajate, S. Marthn-Santamaria, F. Gago, *Curr. Med. Chem.*, 2004, **11**, 3163.
7. S. Azzouz, M. Maache, R. G. Garcia, A. Osuna, *Basic and Clinical Pharmacol. and Toxicology*, 2005, **96**, 60.
8. S. B. Kondapaka, S. S. Singh, G. P. Dasmahapatra, E. A. Sausville, K. K. Roy, *Mol. Cancer Ther.*, 2003, **2**, 1093.
9. T. P. Dorlo, P. P. Van Thiel, A. D. Huitema, R. J. Keizer, H. J. deVries, J. H. Beijnen, P. J. deVries, *Antimicrob. Agents Chemother*, 2008, **52**, 2855.
10. F. Widmer, L. Wright, D. Obando, R. Handke, R. Ganendren, D. Ellis, T. Sorrell, *Antimicrob. Agents Chemother*, 2006, **50**, 414.
11. K. Barhwal, S. K. Hota, V. Jain, D. Prasad, S. B. Singh, G. Ilavazhagan, *Neuroscience*, 2009, **161**, 501.
12. V. V. Floch, S. Loisel, E. Guénin, A. C. Hervé, J. C. Clément, J. J. Yaouanc, H. des Abbayes, C. Férec, *J. Med. Chem.*, 2000, **43**, 4617.
13. E. Guйnin, A. C. Hervé, V. V. Floch, S. Loisel, J. J. Yaouanc, J. C. Clйment, C. Fйrec, H. des Abbayes, *Angew. Chem., Int. Ed.*, 2000, **39**, 629.
14. A. Fraix, T. Montier, T. L. Gall, C. M. Sevrain, N. Carmoy, M. F. Lindberg, P. Lehnb, P.-A. Jaffrès, *Org. Biomol. Chem.*, 2012, **10**, 2051.
15. Yu. Strobykina, M. G. Belenok, M. N. Semenova, V. V. Semenov, V. M. Babaev, I. Kh. Rizvanov, V. F. Mironov, V. E. Kataev, *J. Natur. Prod.*, 2015, **78**, 1300.
16. E. A. Chugunova, R. E. Mukhamatdinova, A. R. Burilov, *Russ. J. Gen. Chem.* (*Engl. Transl.*), 2015, **85**, 1201 [*Zh. Obshch. Khim.*, 2015, **85**, 872].
17. T. Sakurai, K. Fujiwara, *Br. J. Pharmacol.*, 2001, **132**, 143.
18. J. S. Edmonds, K. A. Francesconi, *Appl. Organometal. Chem.*, 1988, **2**, 297.
19. C. Kojima, T. Sakurai, M. Ochiai, H. Kumata, W. Qu, M. P. Waalkes, K. Fujiwara, *Appl. Organometal. Chem.*, 2002, **16**, 421.
20. T. Rezanka, K. Sigler, *Phytochemistry*, 2008, **69**, 585.
21. A. Mattarei, L. Biasutto, E. Marotta, U. De Marchi, N. Sassi, S. Garbisa, M. Zoratti, C. Paradisi, *ChemBioChem.*, 2008, **9**, 2633.
22. S. Rodriguez-Cuenca, H. M. Cochemé, A. Logan, I. Abakumova, T. A. Prime, C. Rose, A. Vidal-Puig, A. C. Smith, D. C. Rubinsztein, I. M. Fearnley, B. A. Jones, S. Pope, S. J. R. Heales, B. Y. H. Lam, S. Guha Neogi, I. McFarlane, A. M. James, R. A. J. Smith, M. P. Murphy, *Free Radical Biol. Med.*, 2010, **48**, 161.
23. J. Ripcke, K. Zarse, M. Ristow, M. Birringer, *ChemBioChem.*, 2009, **10**, 1689.
24. I. V. Galkina, A. I. Tufatullin, D. B. Krivolapov, Y. V. Bakhtiyarova, D. R. Chubukaeva, V. V. Stakheev, V. I. Galkin, R. A. Cherkasov, B. Büchner, O. N. Kataeva, *Cryst. Eng. Comm.*, 2014, **16**, 9010.
25. 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].
26. 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.
27. 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].
28. V. I. Galkin, Yu. V. Bakhtiyarova, R. I. Sagdieva, 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].
29. 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. Gen. Chem.* (*Engl. Transl.*), 2007, **43**, 207 [*Zh. Obshch. Khim.*, 2007, **43**, 215].
30. S.-L. Li, T.C.W. Mak, *J. Mol. Struct.*, 1996, **384**, 135.
31. L. Leiserowitz, *Acta Cryst.*, 1976, **B32**, 775.
32. E. V. Mironova, M. S. Dzyurkevich, O. A. Lodochnikova, D. B. Krivolapov, I. A. Litvinov, V. V. Plemenkov, *Russ. J. Struct. Chem.* (*Engl. Transl.*), 2012, **53**, 361 [*Zh. Struktur. Khim.*, 2012, **53**, 360].
33. G. M. Sheldrick, *SADABS*, University of Göttingen, Germany, 2004.
34. G. M. Sheldrick, *Acta Crystallogr., Sect. A*, 2008, **64**, 112.
35. L. J. Farrugia, *J. Appl. Cryst.*, 1999, **32**, 837.
36. APEX2 (Version 2.1), *SAINTPlus: Data Reduction and Correction Program (Version 7.31A), Bruker Advanced X-Ray Solutions*, *Bruker AXS Inc.*, Madison, Wisconsin, USA, 2006.

Received November 16, 2015;
in revised form February 5, 2016