ISSN 1070-3632, Russian Journal of General Chemistry, 2015, Vol. 85, No. 9, pp. 2058–2064. © Pleiades Publishing, Ltd., 2015.
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*To the 85th Anniversary of birthday of late Yu.G. Gololobov*

# Synthesis of Carboxylate Arsenobetaines Based on (Carboxyalkyl)triphenylarsonium Halides

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Received August 10, 2015

**Abstract**—It has been found that the reaction of triphenylarsine with unsaturated carboxylic acids (acrylic, maleic, and itaconic ones) supposed to yield the arsenobetaines does not occur, in contrast to similar reactions of carboxylic acids with tertiary phosphines. However, the interaction of tertiary arsines with the halogenated carboxylic acids has resulted in the corresponding tertiary arsonium salts, dehydrohalogenation of the latter affording the target carboxylate betaines in the quantitative yield; the products structure has been elucidated using a set of chemical, physical, and physico-chemical methods. Antibacterial activity of the prepared compounds has been studied.

**Keywords:** arsenobetaine, unsaturated carboxylic acid, quaternary arsonium salt

**DOI:** 10.1134/S1070363215090078

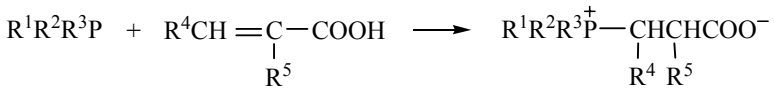
The simplest arsenic-containing carboxylate betaine, 2-(trimethylarsonio)acetate, has been recognized as the major organoarsenic component of various biological objects, especially marine biota, constituting 95% of the natural arsenic; it has been isolated from a number of organisms: algae, invertebrates, marine and freshwater shellfishes and fishes [1–3] as well as certain types of fungi and lichens [4, 5]. Importantly, the natural arsenobetaine found in the seafood exhibits low toxicity (10 g/kg) [3].

The first reports on synthesis of arsenic and phosphorus betaines date back to the end XIXth and the early XXth century [6, 7]. The present work aimed at preparation of novel arsenic-based biologically active compounds and structural study of novel arsenobetaines and arsonium salts taking advantage of modern chemical, physical, and physico-chemical methods.

We have earlier described the methods of preparation various phosphabetaines based on tertiary phosphines and unsaturated mono- and dicarboxylic acids and reported on their structure and reactivity [8–17] (Scheme 1).

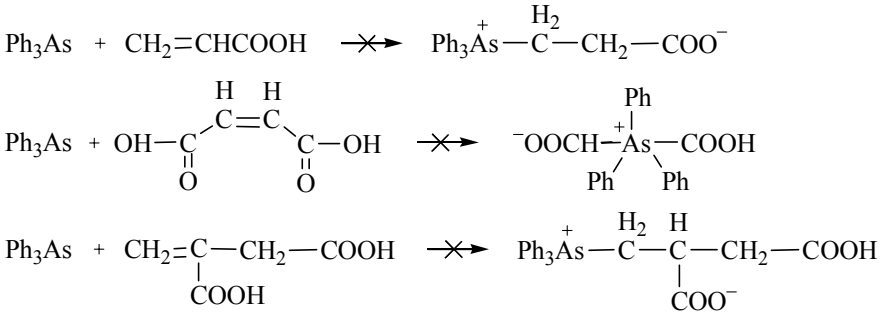
Extending those studies, we turned to arsenic, the closest analog of phosphorus. In view of that aim, we attempted to introduce triphenylarsine (the arsenic analog of triphenylphosphine earlier studied in detail by our group [8–17]) into the reaction with a series of unsaturated carboxylic acids. The choice of the acids (maleic, acrylic, and itaconic ones) was based on their excellent reactivity towards triphenylphosphine having allowed for isolation and detailed studies of the corresponding phosphabetaines [8]. We expected that similar reactions would occur smoothly in the case of triphenylarsine as well (Scheme 2).

**Scheme 1.**

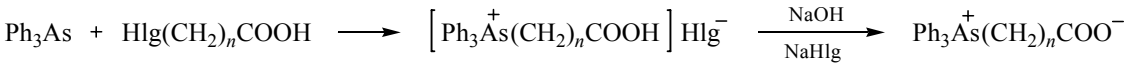


$R^1 = R^2 = R^3 = Ph, Bu, C_6H_{11}$; $R^4 = H, CH_3, Ph, COOH$; $R^5 = H, CH_3$.

**Scheme 2.**



**Scheme 3.**



$n$ = 1, 2, 3, 4; Hlg = Cl, Br.

Contrary to the expectations, no distinct sign of the reaction between the mentioned acids and triphenylarsine was observed after 3–4 months, even though the reactions with triphenylphosphine under the same conditions occurred within several minutes to several hours. That could be owing to either thermodynamic instability of the arsenobetaines as compared to the corresponding phosphabetaines, or the substantially higher activation barrier of the arsenobetaines formation.

According to the reference X-ray diffraction data, the CPC and CAsC bond angles are noticeably different [18–20]; in particular, the CPC angle in triphenylphosphine is close to 103° (Fig. 1) [19, 20], and the CAsC angle in triphenylarsine is of 99° (Fig. 2) [18]. Hence, the phosphorus lone-electron pair in tertiary phosphines exhibits the state close to the tetrahedral $sp^3$ hybridization (108°), and the s orbital contribution to that of the trivalent arsenic is about 50% (with the purely s character of the lone-electron pair meaning the $p^3$ hybridization the bond angles would show up the 90° angle).

Since the configuration of both phosphorus and arsenic in the betaine structures is purely tetrahedral ($sp^3$, 108°), it is obvious that energy of re-hybridization of the orbitals in the course of the reaction should be lower in the case of phosphorus.

Apparently, the observed inactivity of triphenylarsine was not due to thermodynamic impossibility of the corresponding reactions but resulted from the higher activation energy (and, hence, the much lower reaction rate).

The study on preparation, structure, and reactivity of halide salts of carboxylate triphenylarsenobetaines was reported two decades ago [21]. In this work we took advantage of the same synthesis method (Scheme 3).

ω-Chloro- and bromocarboxylic acids were introduced in the reactions with triphenylarsine (Scheme 4).

The reactions were carried out via melt-fusion of the starting compounds in bulk at 100°C during 25 to 50 h. The product yields were substantially different depending on the substrate nature. In detail, the reaction of triphenylarsine with bromoacetic acid

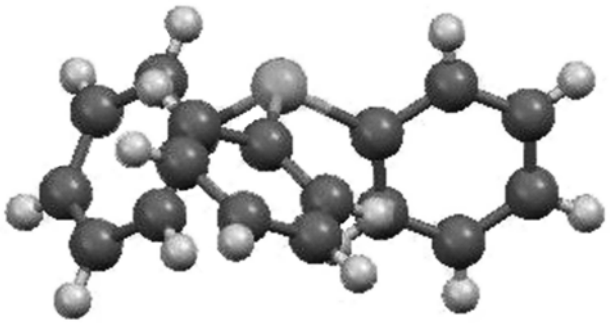


**Fig. 1.** Molecular structure of triphenylphosphine.

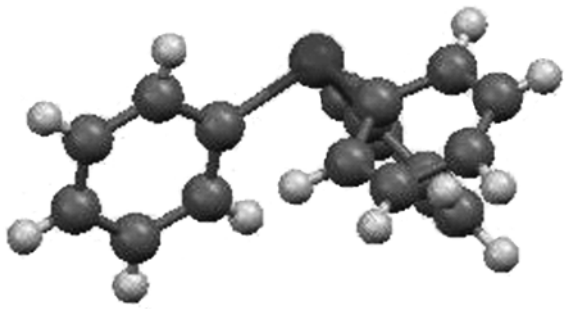


**Fig. 2.** Molecular structure of triphenylarsine.

**Scheme 4.**

$$Ph_3As + Hlg(CH_2)_nCOOH \longrightarrow [Ph_3\overset{+}{A}s(CH_2)_nCOOH]Hlg^-$$

**1**–**7**

$n$ = 1 (**1, 5**); 2 (**2, 6**); 3 (**3, 7**); 4 (**4**); Hlg = Cl (**1**–**4**), Br (**5**–**7**).

**Scheme 5.**

$$\left[Ph_3\overset{+}{A}s(CH_2)_2COOH\right]Cl^- \xrightarrow[NaCl]{NaOH} Ph_3\overset{+}{A}sCH_2COO^- \cdot H_2O \xleftarrow[NaBr]{NaOH} \left[Ph_3\overset{+}{A}s(CH_2)_2COOH\right]Br^-$$

**1** **8** **5**

$$\left[Ph_3\overset{+}{A}s(CH_2)_3COOH\right]Cl^- \xrightarrow[NaCl]{NaOH} Ph_3\overset{+}{A}s(CH_2)_2COO^- \cdot H_2O \xleftarrow[NaBr]{NaOH} \left[Ph_3\overset{+}{A}s(CH_2)_3COOH\right]Br^-$$

**2** **9** **6**

$$\left[Ph_3\overset{+}{A}s(CH_2)_4COOH\right]Cl^- \xrightarrow[NaCl]{NaOH} Ph_3\overset{+}{A}s(CH_2)_3COO^- \cdot H_2O \xleftarrow[NaBr]{NaOH} \left[Ph_3\overset{+}{A}s(CH_2)_4COOH\right]Br^-$$

**3** **10** **7**

afforded the arsonium salt **5** with yield of 89%, whereas the salts yield was of 20–40% in the majority of the studied reactions. Noteworthily, the reactions with ω-chlorocarboxylic acids afforded the products **1**–**4** in the noticeably lower yields than those with ω-bromocarboxylic acids. Increasing the reaction duration up to 50 h did not affect the yield; the reaction temperature marginally influenced the yield as well.

Structure of all the quaternary arsonium salts was confirmed using a set of spectral methods (IR, $^1$H and $^{13}$C NMR spectroscopy). Table 1 lists the selected properties of the arsonium salts **1**–**7**. All the arsonium salts revealed the halogen presence (the positive Beilstein test).

The prepared arsonium salts were further used for synthesis of the corresponding arsenobetaines **8**–**10**. To do so, the chlorine (**1**–**3**) or bromine (**5**–**7**) arsonium salts were treated with 1 mol/L NaOH solution; the reactions afforded the corresponding carboxylate arsenobetaines with high yields (Scheme 5). The products parameters are collected in Table 2.

Structure of the products was confirmed by IR, $^1$H NMR, and $^{13}$C NMR spectroscopy. IR spectra of arsenobetaines **8**–**10** did not contain the carboxylic group absorption band (1700–1710 cm$^{-1}$); instead, the carboxylate absorption band appeared at 1570–1610 cm$^{-1}$. A broad band assigned to hydroxyl group absorption was observed at 2800–3500 cm$^{-1}$, pointing at the presence of water in the crystal structure.

Noteworthily, the arsenobetaines prepared from different halide salts (chloride or bromide) revealed the slightly different (by 2°C) melting points; that was

**Table 1.** Yields, melting points, and IR spectral data for arsonium salts **1**–**7**

| Comp. no. | mp, °C | Synthesis time, h | ν, cm$^{-1}$ | | | Yield, % |
|---|---|---|---|---|---|---|
| | | | ν(C=O) | ν(C–O) | β(COH) | |
| **1** | 134 | 25 | 1700 | 1170 | 1270 | 27 |
| **2** | 158 | 48 | 1710 | 1160 | 1260 | 29 |
| **3** | 156 | 41 | 1700 | 1170 | 1270 | 33 |
| **4** | Oil | 31 | 1705 | 1150 | 1260 | 20 |
| **5** | 177 | 36 | 1730 | 1170 | 1255 | 89 |
| **6** | 172 | 31 | 1700 | 1180 | 1220 | 39 |
| **7** | 185 | 30 | 1705 | 1130 | 1310 | 40 |

likely owing to the minor changes of the water content in the crystal lattice.

The presence of water in the crystal structure of the prepared carboxylate arsenobetaines **8–10** was unambiguously confirmed by the data of combined DSC–TGA–MS studies. For example, the thermogram of arsenobetaine **9a** (Fig. 3) contained a pronounced endothermic melting peak with the maximum at 131°C and the assigned mass loss of 6%; that corresponded to elimination of one water molecule. The presence of water in the arsenobetaine crystal lattice coincided with the earlier data from our [10–12] and other research groups [21] having stated that carboxylate betaines were inclined to incorporation of proton-donating compounds within the crystal lattice. That was additionally confirmed by IR spectra of the arsenobetaines **8–10**. Hence, the melting point of compound **9a** with maximum at 131°C corresponded to melting of the crystal hydrate rather than the betaine itself. The same was true for the other arsenobetaines.

The prepared compounds **1–10** were tested for biological activity against the pathogenic and conditionally pathogenic microflora of human and animals: *Staphylococcus aureus*, *Bacillus cereus, Pseudomonas aeruginosa, Escherihia coli, Proteus mirabilis, Aspergillus niger*, and *Candida albicans*. The highly selective antibacterial activity of compounds **7** and **8** with respect to the Bacillus cereus and Pseudomonas aeruginosa, respectively, was revealed. Those compounds could be recommended for deeper investigation as potential drugs.

**Table 2.** Yields, melting points, and IR spectral data for arsenobetaines **8–10**

| Comp. no. | mp, °C | $\nu_s(COO^-)$, $cm^{-1}$ | $\nu_{as}(COO^-)$, $cm^{-1}$ | $\nu(OH)$, $cm^{-1}$ | Yield, % |
|---|---|---|---|---|---|
| **8a** | 121 | 1570 | 1360 | 3200 | 80 |
| **8b** | 119 | 1610 | 1380 | 3200 | 85 |
| **9a** | 131 | 1580 | 1370 | 3200 | 83 |
| **9b** | 129 | 1620 | 1390 | 3200 | 91 |
| **10a** | 211 | 1605 | 1380 | 3300 | 60 |
| **10b** | 209 | 1600 | 1370 | 3200 | 65 |

To conclude, the studied arsenation reactions have allowed introduction of various substituents at tertiary arsenic atom to prepare the corresponding arsonium salts and carboxylate betaines. This opens a range of possibilities to synthesize novel biologically active compounds.

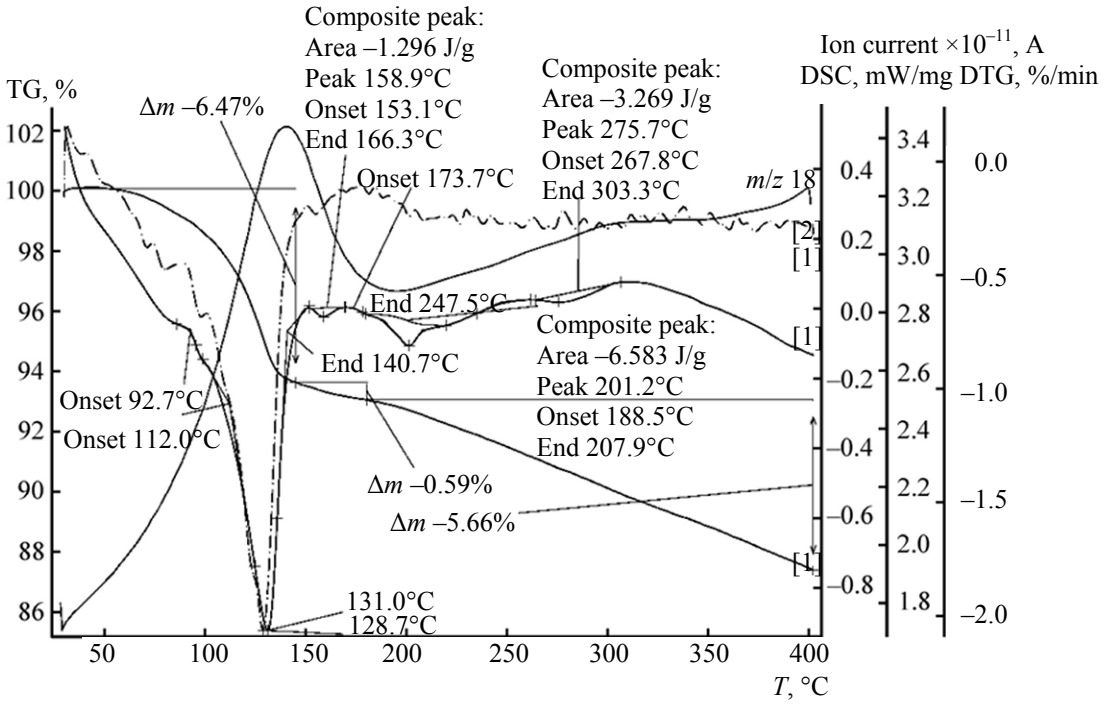



**Fig. 3.** DSC–TG curves for arsenobetaine **9a**.

## EXPERIMENTAL

$^1$H and $^{13}$C NMR spectra ($D_2O$ or $CDCl_3$) were registered using a Bruker Avance-400 spectrometer. IR spectra (Vaseline oil suspension or thin film between KBr plates) were recorded using an IR Prestige-21 spectrometer at 400–3700 cm$^{-1}$. Thermal stability was studied using a STA 449C Jupiter synchronous microthermoanalyzer (Netzsch, Germany) coupled with a QMS 403C Aeolos quadrupole mass spectrometer (Netzsch, Germany) at heating rate of 10 deg/min under argon atmosphere.

**(2-Carboxyethyl)triphenylarsonium chloride (1).** A mixture of 1.5 g (0.0049 mol) of triphenylarsine and 0.47 g (0.0049 mol) of chloroacetic acid was melt-fused at 100°C during 25 h. The reaction mixture was then treated with water, and the unreacted triphenylarsine was filtered off. The white crystalline product obtained after distillation the solvent off in vacuum was washed several times with diethyl ether, recrystallized from a mixture of anhydrous ethanol and diethyl ether, and dried in vacuum. Yield 0.54 g (27.6%), white powder, mp 134°C. Positive Beilstein test. IR spectrum, ν, cm$^{-1}$: 1170 s (C–O), 1270 m (COH), 1700 s (COOH), 3500 m (OH). $^1$H NMR spectrum ($D_2O$), δ, ppm: 4.06 s (2H, $CH_2$), 7.45–7.64 m (15H, Ph). $^{13}$C NMR spectrum ($D_2O$), $\delta_C$, ppm: 59.26 ($CH_2$), 121.75, 130.32, 132.49, 133.88 (Ph), 176.42 (COOH). Found, %: C 59.27; H 4.53; Cl 9.19. $C_{20}H_{18}AsClO_2$. Calculated, %: C 59.92; H 4.49; Cl 8.86.

**(3-Carboxypropyl)triphenylarsonium chloride (2)** was prepared similarly via melt-fusion of 0.5 g (0.0016 mol) of triphenylarsine and 0.18 g (0.0016 mol) of ω-chloropropionic acid at 100°C during 48 h. Yield 0.197 g (29.8%), white powder, mp 158°C. Positive Beilstein test. IR spectrum, ν, cm$^{-1}$: 1160 s (C–O), 1260 m (COH), 1710 s (COOH), 3500 m (OH). $^1$H NMR spectrum ($D_2O$), δ, ppm: 2.87 t (2H, $AsCH_2$, *J* 6.8 Hz), 3.51 t (2H, $CH_2COOH$, *J* 6.8 Hz) 7.56–7.75 m (15H, Ph). $^{13}$C NMR spectrum ($D_2O$), $\delta_C$, ppm: 20.06 ($AsCH_2$), 27.82 ($CH_2COOH$), 121.16, 130.42, 132.69, 133.98 (Ph), 176.47 (COOH). Found, %: C 59.94; H 4.55; Cl 8.39. $C_{21}H_{20}AsClO_2$. Calculated, %: C 60.80; H 4.83; Cl 8.56.

**(4-Carboxybutyl)triphenylarsonium chloride (3)** was prepared similarly via melt-fusion of 0.5 g (0.0016 mol) of triphenylarsine and 0.2 g (0.0016 mol) of ω-chlorobutanoic acid at 100°C during 41 h. Yield 0.235 g (33.6%), white powder, mp 156°C. Positive Beilstein test. IR spectrum, ν, cm$^{-1}$: 1170 s (C–O), 1270 m (COH), 1700 s (COOH), 3500 m (OH). $^1$H NMR spectrum ($D_2O$), δ, ppm: 1.80 m (2H, $AsCH_2\underline{CH_2}$), 2.31 t (2H, $AsCH_2$, *J* 6.8 Hz) 3.48 t (2H, $CH_2COOH$, *J* 6.8 Hz), 7.37–7.54 m (15H, Ph). $^{13}$C NMR spectrum ($D_2O$), $\delta_C$, ppm: 21.86 ($AsCH_2\underline{CH_2}$), 32.43 ($AsCH_2$), 42.43 ($CH_2COOH$), 127.98, 128.53, 132.34, 139.79 (Ph), 178.35 (COOH). Found, %: C 61.04; H 4.95; Cl 8.21. $C_{22}H_{22}AsClO_2$. Calculated, %: C 61.61; H 5.13; Cl 8.28.

**(5-Carboxypentyl)triphenylarsonium chloride (4)** was prepared similarly via melt-fusion of 0.5 g (0.0016 mol) of triphenylarsine and 0.22 g (0.0016 mol) of ω-chloropentanoic acid at 100°C during 42 h. Yield 0.197 g (33.7%), yellow oil. Positive Beilstein test. IR spectrum, ν, cm$^{-1}$: 1150 s (C–O), 1260 m (COH), 1705 s (COOH), 3500 m (OH). $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 1.72–1.88 m (4H, $AsCH_2\underline{CH_2}\underline{CH_2}$), 2.37 t (2H, $AsCH_2$, *J* 7.0 Hz), 3.51 t (2H, $CH_2COOH$, *J* 7.0 Hz), 7.25–7.45 m (15H, Ph), br.s 11.10 (OH). $^{13}$C NMR spectrum ($CDCl_3$), $\delta_C$, ppm: 22.00 ($AsCH_2\underline{CH_2}$), 31.74 ($\underline{CH_2}CH_2COOH$), 33.23 ($CH_2COOH$), 44.43 ($AsCH_2$), 128.53, 128.73, 1332.79, 139.69 (Ph), 179.74 (COOH). Found, %: C 61.04; H 4.95; Cl 8.21. $C_{23}H_{24}AsClO_2$. Calculated, %: C 62.13; H 5.40; Cl 7.99.

**(2-Carboxyethyl)triphenylarsonium bromide (5).** A mixture of 0.5 g (0.0016 mol) of triphenylarsine and 0.23 g (0.0016 mol) of bromoacetic acid was melt-fused at 100°C during 36 h. The reaction mixture was treated with diethyl ether, and the formed beige precipitate was recrystallized from a mixture of acetonitrile and diethyl ether. Yield 0.649 g (88.9%), mp 177°C. Positive Beilstein test. IR spectrum, ν, cm$^{-1}$: 1170 s (C–O), 1255 m (COH), 1700 s (COOH), 3500 m (OH). $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 5.08 s (2H, $CH_2$), 7.55–7.70 m (15H, Ph), br.s 9.08 (OH). $^{13}$C NMR spectrum ($CDCl_3$), $\delta_C$, ppm: 34.46 ($CH_2$), 121.21, 130.90, 132.90, 134.32 (Ph), 165.64 (COOH). Found, %: C 53.90; H 3.95; Cl 18.37. $C_{23}H_{24}AsBrO_2$. Calculated, %: 53.93; H 4.04; Br 17.98.

**(3-Carboxypropyl)triphenylarsonium bromide (6).** A mixture of 0.7 g (0.0023 mol) of triphenylarsine and 0.36 g (0.0023 mol) of ω-bromopropionic acid was melt-fused at 100°C during 36 h. The reaction mixture was treated with water, the unreacted triphenylarsine was separated off, and the solvent was distilled off in vacuum. The orange oil formed after elimination of the solvent was washed with diethyl ether to give the

crystalline product. The crystals were washed with diethyl ether several times, recrystallized from a mixture of anhydrous ethanol and diethyl ether, and dried in vacuum. Yield 0.42 g (29.8%), beige crystals, mp 172°C. Positive Beilstein test. IR spectrum, ν, $cm^{-1}$: 1180 s (C–O), 1220 m (COH), 1700 s (COOH), 3500 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 2.85 t (2H, $AsCH_2$, *J* 7.1 Hz), 3.50 t (2H, $CH_2COOH$, *J* 7.1 Hz) 7.54–7.73 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 20.14 ($AsCH_2$), 27.88 ($CH_2COOH$), 121.37, 130.44, 132.69, 134.32 (Ph), 174.55 (COOH). Found, %: C 54.19; H 4.55; Br 18.39. $C_{21}H_{20}AsBrO_2$. calculated, %: C 54.90; H 4.58; Br 17.43.

**(4-Carboxybutyl)triphenylarsonium bromide (7).** A mixture of 0.7 g (0.0023 mol) of triphenylarsine and 0.39 g (0.0023 mol) of ω-bromobutanoic acid was melt-fused at 100°C during 32 h. The reaction mixture was treated with water, the unreacted triphenylarsine was separated off, and the solvent was distilled off in vacuum. The orange oil formed after elimination of the solvent was washed with diethyl ether to give the crystalline product. The crystals were washed with diethyl ether several times, recrystallized from a mixture of anhydrous ethanol and diethyl ether, and dried in vacuum. Yield 0.44 g (40%), beige crystals, mp 185°C. Positive Beilstein test. IR spectrum, ν, $cm^{-1}$: 1130 s (C–O), 1310 m (COH), 1705 s (COOH), 3500 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 1.99 m (2H, $AsCH_2\underline{CH_2}$), 2.51 t (2H, $AsCH_2$, *J* 7.1 Hz), 3.34 t (2H, $CH_2COOH$, *J* 7.1 Hz), 7.55–7.78 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 18.29 ($AsCH_2\underline{CH_2}$), 23.04 ($AsCH_2$), 33.77 ($CH_2COOH$), 120.62, 130.51, 132.67, 133.79 (Ph), 176.44 (COOH). Found, %: C 55.17; H 4.54; Br 16.18. $C_{22}H_{22}AsBrO_2$. Calculated, %: C 55.81; H 4.65; Br 16.91.

**2-(Triphenylarsonio)acetate (8a).** 1.1 mL of 1 mol/L alcoholic solution of NaOH was added to a solution of 0.45 g (0.0011 mol) of compound **1** in 3 mL of acetonitrile. The formed precipitate of NaCl was filtered off, and the solvent was distilled off in vacuum. The formed reaction product was recrystallized from a mixture of ethanol and diethyl ether. Yield 0.33 g (80%), white powder, mp 121°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1150 s (C–O), 1360 m ($COO^-$), 1570 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 2.64 s (2H, $CH_2$), 7.30–7.60 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 34.55 ($CH_2$), 121.94, 130.66, 131.92, 134.17 (Ph), 174.22 (COOH). Found, %: C 62.17; H 4.67. $C_{20}H_{17}AsO_2\cdot H_2O$. Calculated, %: C 62.83; H 4.97.

**2-(Triphenylarsonio)acetate (8b)** was prepared similarly from 0.35 g (0.0008 mol) of compound **5** and 0.8 mL of 1 mol/L alcoholic solution of NaOH. Yield 0.24 g (85%), white powder, mp 119°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1140 s (C–O), 1380 m ($COO^-$), 1610 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 2.68 s (2H, $CH_2$), 7.30–7.60 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 34.51 ($CH_2$), 122.44, 130.28, 133.50, 133.71 (Ph), 175.01 (COOH). Found, %: C 62.04; H 4.58. $C_{20}H_{17}AsO_2\cdot H_2O$. Calculated, %: C 62.83; H 4.97.

**3-(Triphenylarsonio)propanoate (9a)** was prepared similarly from 0.40 g (0.0010 mol) of compound **2** and 1 mL of 1 mol/L alcoholic solution of NaOH. Yield 0.32 g (83%), white powder, mp 131°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1160 s (C–O), 1370 m ($COO^-$), 1580 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 2.57 t (2H, AsCH2, *J* 7.0 Hz), 3.44 t (2H, CH2COOH, *J* 7.0 Hz), 7.54–7.70 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 21.94 ($AsCH_2$), 30.30 ($CH_2COOH$), 122.17, 130.32, 132.69, 133.70 (Ph), 177.55 (COOH). Found, %: C 62.86; H 5.04. $C_{21}H_{19}AsO_2\cdot H_2O$. Calculated, %: C 63.63; H 5.30.

**3-(Triphenylarsonio)propanoate (9b)** 0.9 mL of 1 mol/L alcoholic solution of NaOH was added to a solution of 0.40 g (0.0009 mol) of compound **6** in 3 mL of acetonitrile. The formed NaBr precipitate was filtered off, and the solvent was distilled off in vacuum. The formed product was recrystallized from a mixture of ethanol and diethyl ether. Yield 0.315 g (91%), white powder, mp 129°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1155 s (C–O), 1390 m ($COO^-$), 1620 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 2.59 t (2H, $AsCH_2$, *J* 7.0 Hz), 3.39 t (2H, $CH_2COOH$, *J* 7.0 Hz) 7.50–7.68 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 22.07 ($AsCH_2$), 30.32 ($CH_2COOH$), 122.24, 130.22, 132.60, 133.60 (Ph), 177.84 (COOH). Found, %: C 63.17; H 5.15. $C_{21}H_{19}AsO_2\cdot H_2O$. Calculated, %: C 63.63; H 5.30.

**4-(Triphenylarsonio)butanoate (10a)** was prepared similarly from 0.40 g (0.0009 mol) of compound **2** and 0.9 mL of 1 mol/L alcoholic solution of NaOH. Yield 0.23 g (60%), white powder, mp 211°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1160 s (C–O), 1380 m ($COO^-$), 1605 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 1.84 m (2H, $AsCH_2\underline{CH_2}$), 2.55 t (2H, AsCH2, *J* 7.0 Hz), 3.40 t (2H, CH2COOH, *J* 7.0 Hz), 7.45–7.69 m (15H, Ph). $^{13}C$ NMR spectrum

($D_2O$), $\delta_C$, ppm: 17.28 ($AsCH_2\underline{CH_2}$), 23.09 ($AsCH_2$), 30.89 ($CH_2COOH$), 121.54, 130.51, 132.65, 133.79 (Ph), 176.95 (COOH). Found, %: C 64.01; H 5.54. $C_{22}H_{21}AsO_2 \cdot H_2O$. Calculated, %: C 64.39; H 5.61.

**4-(Triphenylarsonio)butanoate (10b)** was prepared similarly from 0.40 g (0.0008 mol) of compound **7** and 0.8 mL of 1 mol/L alcoholic solution of NaOH. Yield 0.225 g (65%), white powder, mp 209°C. Negative Beilstein test. IR spectrum, ν, $cm^{-1}$: 1155 s (C–O), 1370 m ($COO^-$), 1600 s ($COO^-$), 3200 m (OH). $^1H$ NMR spectrum ($D_2O$), δ, ppm: 1.87 m (2H, $AsCH_2\underline{CH_2}$), 2.54 t (2H, $AsCH_2$, *J* 7.0 Hz), 3.39 t (2H, CH2COOH, *J* 7.0 Hz), 7.50–7.70 m (15H, Ph). $^{13}C$ NMR spectrum ($D_2O$), $\delta_C$, ppm: 17.22 ($AsCH_2\underline{CH_2}$), 23.05 ($AsCH_2$), 30.82 ($CH_2COOH$), 121.48, 130.45, 132.63, 133.76 (Ph), 177.01 (COOH). Found, %: C 63.87; H 5.28. $C_{22}H_{21}AsO_2 \cdot H_2O$. Calculated, %: C 64.39; H 5.61.

## ACKNOWLEDGMENTS

This work was financially supported by the grant allocated to Kazan Federal University to fulfill the governmental task in the field of scientific activity.

## REFERENCES

1. Kaise, N., Watanake Sh., and Jtoh, K., *Talanta,* 1985, vol. 14, no. 9, p. 1327. DOI: 10.1016/0045-6535(85)90153-5.
2. Lejkovec, Z., Bajc, Z., and Doganoc, Z., *Talanta,* 2004, vol. 62, no. 5, p. 931. DOI: 10.1016/j.talanta.2003.10.012.
3. Edmonds, J.S., Francesconi, K.A., Cannon, J.R., Raston, C.L., Skelton, B.W., and White, A.H., *Tetrahedron Lett*., 1977, vol. 18, no. 18, p. 1543. DOI: 10.1016/S0040-4039(01)93098-9.
4. Byrne, A.R., Slejkovec, Z., and Stojve, T., *Appl. Organometal. Chem*., 1995, vol. 9, no. 4, p. 305. DOI: 10.1002/aoc. 590090403.
5. Mrak, T., Šlejkovec, Z., Jeran, Z., *Talanta,* 2006, vol. 69, no. 1, p. 251. DOI: 10.1016/j.talanta.2005.10.011.
6. Michaelis, A., *Lieb. Ann*., 1902, vol. 321, p. 174. DOI: 10.1002/jlac.19023210202.
7. Michaelis, A., Soden, H.V., *Lieb. Ann*. 1885, vol. 229, p. 295. DOI: 10.1002/jlac.18852290304.
8. Bakhtiyarova, Yu.V., Sagdieva, R.I., Galkina, I.V., Galkin, V.I., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Russ. J. Org. Chem*., 2007, vol. 43, no. 2., P., 207. DOI: 10.1134/S1070428007020091.
9. Galkin, V.I., Bakhtiyarova, Yu.V., Polezhaeva, N.A., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Phosphorus, Sulfur, Silicon, Relat. Elem*., 1999, vol. 147, p. 91. DOI: 10.1080/10426509908053526.
10. Galkin, V.I., Bakhtiyarova, Yu.V., Polezhaeva, N.A., Galkina, I.V., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Russ. J. Gen. Chem*., 2002, vol. 72, no. 3, p. 376. DOI: 10.1023/A:1015435432082.
11. Galkin, V.I., Bakhtiyarova, Yu.V., Polezhaeva, N.A., Galkina, I.V., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Russ. J. Gen. Chem*., 2002, vol. 72, no. 3, p. 384. DOI: 10.1023/A:1015487416152.
12. Galkin, V.I., Bakhtiyarova, Yu.V., Sagdieva, R.I., Galkina, I.V., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Russ. J. Gen. Chem*., 2006, vol. 76, no. 3, p. 430. DOI: 10.1134/S1070363206030133.
13. Galkin, V.I., Bakhtiyarova, Yu.V., Sagdieva, R.I., Galkina, I.V., and Cherkasov, R.A., *Heteroatom. Chem*., 2006, vol. 17, no. 6, p. 557. DOI: 10.1002/hc.20276.
14. Bakhtiyarova, Yu.V., Sagdieva, R.I., Galkina, I.V., Galkin, V.I., Cherkasov, R.A., Krivolapov, D.B., Gubaidullin, A.T., and Litvinov, I.A., *Russ. J. Org. Chem*., 2007, vol. 43, no. 2, p. 207. DOI: 10.1134/S1070428007020091.
15. Galkin, V.I., Mal'tsev, D.B., Sobanov, A.A., Gorokhov, V.L., and Bakhtiyarova, Yu.V., *Uch. Zap. Kazan. Univ., Estestv. Nauki,* 2006, vol. 148, no. 4, p. 8.
16. Bakhtiyarova, Yu.V., Bondar', M.S., Galkina, I.V., and Galkin, V.I., ., *Uch. Zap. Kazan. Univ., Estestv. Nauki*, 2008, vol. 150, no. 1, p. 42.
17. Galkin, V.I., Salin, A.V., Bakhtiyarova, Yu.V., and Sobanov, A.A., *Russ. J. Gen. Chem*., 2009, vol. 79, no. 5, p. 919. DOI: 10.1134/S1070363209050090.
18. Sobolev, A.N., Belsky, V.K., Chernikova, N.Yu., and Akhmadulina, F.Yu., *J. Organomet. Chem*., 1983, vol. 244, no. 2, p. 129. DOI: 10.1016/S0022-328X(00)9893-0.
19. Dunne, B.J., Dunne, B.J., and Orpen, A.G., *Acta Crystallogr*., 1991, vol. 47, no. 12, p. 345. DOI: 0.1107/S010827019000508X.
20. Kooijman, H.A, Spek, A., Bommel, K., Verboom, W., and Reinhoudt, D., *Acta Crystallogr*., 1998, vol. 54, no. 11, p. 1695. DOI: 10.1107/S0108270198009305.
21. Gamayurova, V.S., Shulaeva, M.M., Sysoeva, M.A., and Khalitov, F.G., *Russ. J. Gen. Chem.*, 1997, vol. 67, no. 8, p. 1218.