ISSN 1070-4280, Russian Journal of Organic Chemistry, 2006, Vol. 42, No. 7, pp. 981–985. © Pleiades Publishing, Inc., 2006.
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# Synthesis of *N*-Arylsulfonyl-2-aryl(hetaryl)aminoacetic Acids

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Received September 7, 2005

**Abstract**—Hydrolytic transformations of 4-[2,2,2-trichloro-1-(arylsulfonylamino)- and -(ethoxycarbonylamino)ethyl]phenyloxy(or sulfanyl)acetic acids under microwave irradiation in alkaline medium involve both trichloromethyl group and ester fragment to give *N*-arylsulfonyl-2-[4-carboxymethyloxy(or sulfanyl)phenyl]-2-aminoacetic acids in good yields. Hydrolysis of methyl 4-[2,2,2-trichloro-1-(arylsulfonylamino)ethyl]phenyloxy(or sulfanyl)acetates without microwave activation occurs only at the ester group with quantitative formation of 4-[2,2,2-trichloro-1-(arylsulfonylamino)ethyl]phenyloxy(or sulfanyl) acetic acids. *N*-[2,2,2-Trichloro-1-(1-naphthyl, 2-furyl, and 1-methylindol-3-yl)ethyl]-4-chlorobenzenesulfonamides in alkaline medium under microwave irradiation were converted in 10–15 min into the corresponding *N*-(4-chlorophenylsulfonyl)-2-aryl-2-aminoacetic acids in preparative yields.

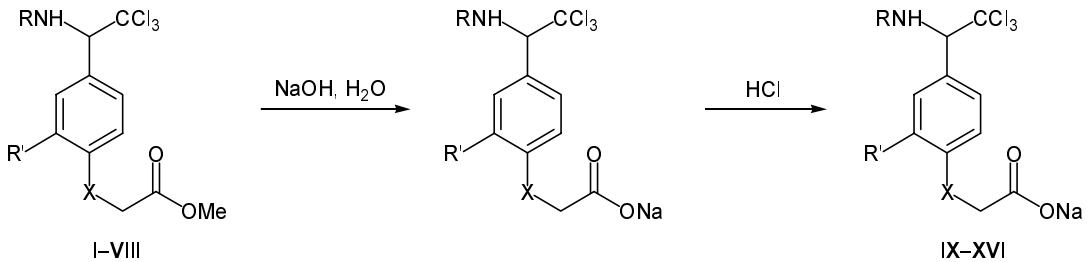
**DOI:** 10.1134/S1070428006070098

Hydrolytic transformation of the trichloromethyl group to carboxy in 1-arylsulfonylamino-2,2,2-trichloroethyl-substituted aromatic and heterocyclic compounds synthesized by us previously [1–9] opens a convenient synthetic route to new α-aryl(hetaryl)-glycines protected at the amino group, which attract interest as potential biologically active substances and promising intermediate products for the preparation of polypeptides and other amino acid derivatives. We demonstrated in [9–11] the possibility for synthesizing protected amino acids by selective hydrolysis of a number of (1-arylsulfonylamino-2,2,2-trichloroethyl)arenes, -thiophenes, and -indoles.

With the goal of developing preparative procedures for the synthesis of functionalized phenyl-, naphthyl-, and hetarylglycines protected at the amino group, in the present work we examined the behavior of 1-arylsulfanylamino-2,2,2-trichloroethyl derivatives of functionally substituted aromatic compounds, aryloxy(or sulfanyl)acetic acid esters [5], as well as of naphthalene [4], *N*-methylindole [8], and furan [2], under conditions of alkaline hydrolysis. We planned to estimate the efficiency of microwave irradiation in the transformation of the trichloromethyl group in methyl 4-(1-arylsulfonylamino-2,2,2-trichloroethyl)phenyloxy(or sulfanyl)acetates and 1-arylsulfonylamino-2,2,2-trichloroethyl-substituted arenes and hetarenes.

We have found that the trichloromethyl group in acetates **I**–**VIII** does not undergo hydrolysis to carboxy group on heating in a boiling dilute solution of

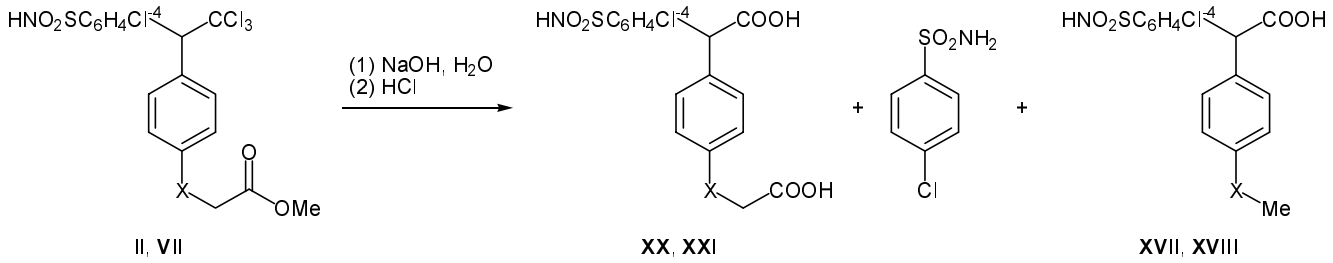
**Scheme 1.**




I–VIII IX–XVI

**I**, **XII**, R = $PhSO_2$, R' = H, X = O; **II**, **IX**, R = 4-$ClC_6H_4SO_2$, R' = H, X = O; **III**, **XI**, R = EtOC(O), R' = H, X = O; **IV**, **XIII**, R = $PhSO_2$, R' = Me, X = O; **V**, **XIV**, R = $PhSO_2$, R' = Cl, X = O; **VI**, **XV**, R = $PhSO_2$, R' = H, X = S; **VII**, **X**, R = 4-$ClC_6H_4SO_2$, R' = H, X = S; **VIII**, **XVI**, R = 4-$ClC_6H_4SO_2$, R' = Me, X = S.

**Scheme 2.**




II, VII XX, XXI XVII, XVIII

**XVII**, **XX**, X = O; **XVIII**, **XXI**, X = S.

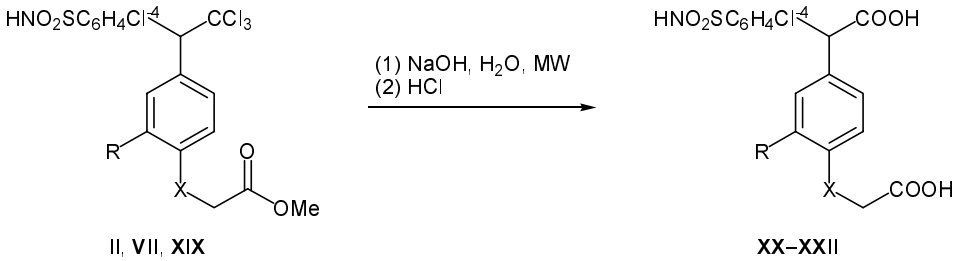
sodium hydroxide (4 equiv) over a period of 30 min {these conditions were successfully used previously in the synthesis of aryl(or thienyl)glycine derivatives [9–11]}. By heating esters **I**–**VIII** in 2–5% aqueous alkali for 20–30 min at 100°C and subsequent acidification we obtained the corresponding 4-(1-arylsulfonylamino-2,2,2-trichloroethyl)phenyloxy(sulfanyl)-acetic acids **IX**–**XVI** in up to 99% yield via hydrolysis of the ester moiety (Scheme 1). Prolonged heating of the reaction mixture (1–8 h) using 8 equiv of alkali with a concentration of 20% also gave acids **IX**–**XVI**, but the reaction was accompanied by hydrolysis of the trichloromethyl group. However, we failed to isolate the desired α-arylglycine derivatives in preparative yield, for the target compounds were strongly contaminated with by-products resulting from cleavage of the C–N bond (arenesulfonamide or ethyl carbamate in the hydrolysis of ester **III**) and decarboxylation of dicarboxylic acids **XX** and **XXI** {to 2-arylsulfonylamino-2-[4-methoxy(or sulfanyl)phenyl]acetic acids **XVII** and **XVIII**; Scheme 2}. Also, a water-soluble residue containing unidentified products was obtained. Analysis of the hydrolysis products obtained from methyl esters **II** and **VII** under the above conditions showed that the concentration of glycine derivatives **XX** and **XXI** in the product mixtures (which were difficult to separate) did not exceed 40%.

When the hydrolysis of compounds **II**, **VII**, and **XIX** with excess 3–5% aqueous alkali was performed under microwave irradiation, we isolated highly pure arylglycine derivatives **XX**–**XXII** (Scheme 3) in good yields (up to 89%) within in a much shorter time (10 min). The reaction was not accompanied by side decarboxylation process. Moreover, hydrolytic cleavage of the C–N bond was suppressed either completely or to a considerable extent; as a result, either no formation of 4-chlorobenzenesulfonamide was observed or its yield did not exceed 20%.

We also examined the effect of microwave irradiation on the alkaline hydrolysis of 1-arylsulfanylamino-2,2,2-trichloroethyl-substituted furan [3], naphthalene [4], and *N*-methylindole [5] derivatives which were synthesized by us previously. The hydrolysis of compounds **XXIII**–**XXV** in dilute aqueous alkali under microwave irradiation gave *N*-(4-chlorophenylsulfonyl)-substituted 2-furyl-, 1-naphthyl-, and 1-methylindol-3-yl(amino)acetic acids **XXVI**–**XXVIII** in preparative yields (Scheme 4), and the optimal reaction time was 10–15 min. Furan derivative **XXIII** failed to undergo hydrolysis of the trichloromethyl group in the absence of microwave activation, while naphthyl- and indolylglycines **XXVII** and **XXVIII** were obtained in poor yields (in the reaction with **XXIV**, an inseparable mixture of products was obtained).

The structure of compounds **IX**–**XVI**, **XX**–**XXII**, and **XXVI**–**XXVIII** was confirmed by the spectral and analytical data (see Experimental). The data for compounds **IX**–**XI** and **XXVIII** coincided with those

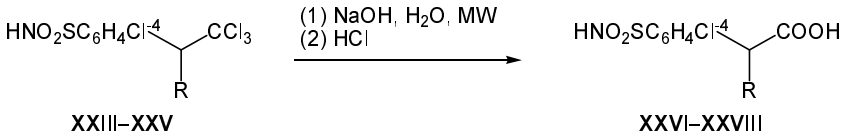
**Scheme 3.**




II, VII, XIX XX–XXII

**XIX**, **XXII**, R = Me, X = O.

**Scheme 4.**




**XXIII**–**XXV** **XXVI**–**XXVIII**

**XXIII**, **XXVI**, R = 2-furyl; **XXIV**, **XXVII**, R = 1-naphthyl; **XXV**, **XXVIII**, R = 1-methyl-1*H*-indol-3-yl.

reported in [6, 11]. 4-(1-Arylsulfonylamino-2,2,2-trichloroethyl)phenyloxy(sulfanyl)acetic acids **XII**–**XVI** and glycine derivatives **XX**–**XXII**, and **XXVI**–**XXVIII** are slightly colored solids which are poorly soluble in water, chloroform, and diethyl ether and readily soluble in alcohol, acetone, DMSO, and aqueous alkali. The IR spectra of **XII**–**XVI** contain strong absorption bands due to NH, $SO_2$, and carboxy groups, aromatic C=C and C–H bonds, and aliphatic C–H bonds. In the $^1$H NMR spectra of **XII**–**XVI**, the NH and CH protons are coupled with a vicinal coupling constant $^3J$ of 10–11 Hz, the $XCH_2$ protons appeared as singlets, and aromatic proton signals were present.

Like initial esters **II**, **VII**, and **XIX**, acids **XX**–**XXII** showed in the $^1$H NMR spectra a singlet from the $XCH_2$ group. However, the signals of the latter appeared in a weaker field, and no coupling between protons in the NHCH fragment was observed: the corresponding signals appeared as singlets. The $^1$H NMR spectra of **XXVI** and **XXVII** also contained signals from the NH and CH protons, protons in the benzene ring, and protons in the furyl or naphthyl substituent.

Thus we have shown that microwave-assisted alkaline hydrolysis of methyl 4-(1-arylsulfonylamino-2,2,2-trichloroethyl)phenyloxy(sulfanyl)acetates occurs at both trichloromethyl and ester groups to give new α-arylglycine derivatives with protected amino group in a good yield within a short time. In the absence of microwave irradiation, only the ester moiety undergoes hydrolysis with quantitative formation of 4-(1-arylsulfonylamino-2,2,2-trichloroethyl)phenyloxy(sulfanyl)acetic acids.

## EXPERIMENTAL

The IR spectra were recorded in KBr on a Specord 75IR spectrophotometer. The $^1$H NMR spectra were measured on a Bruker DPX-400 instrument operating at 400.13 MHz; HMDS was used as internal reference. Microwave-assisted reactions were carried out in a Samsung microwave furnace (ν 2450 MHz, 800 W).

**4-[2,2,2-Trichloro-1-(4-chlorophenylsulfonylamino)ethyl]phenoxyacetic acid (IX).** Methyl 4-[2,2,2-trichloro-1-(4-chlorophenylsulfonylamino)ethyl]phenoxyacetate (**II**), 4.87 g (0.01 mol), was added to a solution of 3.2 g (0.08 mol) of sodium hydroxide in 20–30 ml of water. The mixture was heated to the boiling point and stirred for 20–30 min at that temperature; during the process, water was added to maintain the initial volume. When the reaction was complete, the mixture was cooled to 30°C and acidified to pH 1 with 10% hydrochloric acid. A tarry material separated and was left to stand for 3 h for crystallization, washed with water until neutral, and dried in air. Yield 5.05 g (98%), mp 166–170°C; published data [6]: mp 165–171°C. IR spectrum, ν, cm$^{-1}$: 1170, 1340 ($SO_2$); 1740 (C=O); 3250 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 4.68 s (2H, $OCH_2$), 5.22 d (1H, CH, $^3J$ = 10.6 Hz), 7.45 d and 6.77 d (2H each, *AA'BB'*, $C_6H_4O$), 7.57 d and 7.44 d (2H each, *AA'BB'*, $ClC_6H_4$), 7.92 d (1H, NH, $^3J$ = 10.6 Hz).

Compounds **X**–**XVI** were synthesized in a similar way.

**4-[2,2,2-Trichloro-1-(4-chlorophenylsulfonylamino)ethyl]phenylsulfanylacetic acid (X).** Yield 4.67 g (96%), mp 150–156°C; published data [6]: mp 150–154°C. IR spectrum, ν, cm$^{-1}$: 1170, 1350 ($SO_2$); 1710 (C=O); 3250 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 3.78 s (2H, $XCH_2$), 5.24 d (1H, CH, $^3J$ = 10.7 Hz), 7.22 d and 7.46 d (2H each, *AA'BB'*, $C_6H_4S$), 7.58 d and 7.33 d (2H each, *AA'BB'*, $ClC_6H_4$), 7.98 d (1H, NH, $^3J$ = 10.7 Hz).

**4-[2,2,2-Trichloro-1-(ethoxycarbonylamino)ethyl]phenoxyacetic acid (XI).** Yield 3.53 g (96%), mp 120–124°C; published data [6]: mp 120–122°C. IR spectrum, ν, cm$^{-1}$: 1170, 1240 ($SO_2$); 1700 (C=O); 3410 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 1.17 t and 4.07 q (5H, $C_2H_5$), 4.72 s (2H, $OCH_2$), 5.62 d (1H, CH, $^3J$ = 10.4 Hz), 7.67 d and 6.97 d (2H each, *AA'BB'*, $C_6H_4O$), 7.59 d (1H, NH, $^3J$ = 10.4 Hz).

**4-[2,2,2-Trichloro-1-(phenylsulfonylamino)ethyl]phenoxyacetic acid (XII).** Yield 4.33 g (99%), mp 161–165°C. IR spectrum, ν, cm$^{-1}$: 1120, 1350 ($SO_2$); 1710 (C=O); 3290 (NH). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 4.61 s (2H, $OCH_2$), 4.90 d (1H, CH, $^3J$ = 9.8 Hz), 6.82 d and 7.22 d (2H each, *AA'BB'*,

$C_6H_4O$), 7.54 t (2H, *m*-H), 7.65 t (1H, *p*-H), 7.81 d (2H, *o*-H), 8.3 d (1H, NH, $^3J$ = 9.8 Hz). Found, %: C 43.84; H 3.18; Cl 24.11; N 3.15; S 7.22. $C_{16}H_{14}Cl_3NO_5S$. Calculated, %: C 43.80; H 3.22; Cl 24.24; N 3.19; S 7.31.

**2-Methyl-4-[2,2,2-trichloro-1-(phenylsulfonylamino)ethyl]phenoxyacetic acid (XIII).** Yield 4.38 g (97%), mp 184–188°C. IR spectrum, ν, $cm^{-1}$: 1130, 1350 ($SO_2$); 1710 (C=O); 3250 (NH). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 2.01 s (3H, $CH_3$), 4.62 s (2H, $OCH_2$), 5.1 d (1H, CH, $^3J$ = 10.6 Hz), 6.57 d (1H, 6-H), 7.15 s (1H, 3-H), 7.27 d (1H, 5-H), 7.29 t (2H, *m*-H), 7.44 t (1H, *p*-H), 7.58 d (2H, *o*-H), 9.02 d (1H, NH, $^3J$ = 10.6 Hz). Found, %: C 45.19; H 3.58; Cl 23.48; N 3.12; S 7.21. $C_{17}H_{16}Cl_3NO_5S$. Calculated, %: C 45.10; H 3.56; Cl 23.49; N 3.09; S 7.08.

**2-Chloro-4-[2,2,2-trichloro-1-(phenylsulfonylamino)ethyl]phenoxyacetic acid (XIV).** Yield 4.63 g (98%), mp 181–185°C. IR spectrum, ν, $cm^{-1}$: 1140, 1180, 1340 ($SO_2$); 1700 (C=O); 3430 (NH). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 4.80 s (2H, $OCH_2$), 4.98 d (1H, CH, $^3J$ = 10.6 Hz), 6.96 d (1H, 6-H), 7.22 d (1H, 3-H), 7.36 d (1H, 5-H), 7.57 t (2H, *m*-H), 7.67 t (1H, *p*-H), 7.83 d (2H, *o*-H), 9.80 d (1H, NH, $^3J$ = 10.6 Hz). Found, %: C 40.52; H 2.71; Cl 29.71; N 2.81; S 6.79. $C_{16}H_{13}Cl_4NO_5S$. Calculated, %: C 40.62; H 2.77; Cl 29.97; N 2.96; S 6.78.

**4-[2,2,2-Trichloro-1-(phenylsulfonylamino)-ethyl]phenylsulfanylacetic acid (XV).** Yield 4.31 g (95%), mp 153–156°C. IR spectrum, ν, $cm^{-1}$: 1140, 1350 ($SO_2$); 1700 (C=O); 3250 (NH). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 3.65 s (2H, $SCH_2$), 5.12 d (1H, CH, $^3J$ = 10.2 Hz), 7.02 d and 7.26 d (2H each, *AA'BB'*, $C_6H_4S$), 7.40 t (2H, *m*-H), 7.64 t (1H, *p*-H), 7.78 d (2H, *o*-H), 9.06 d (1H, NH, $^3J$ = 10.2 Hz). Found, %: C 42.18; H 3.11; Cl 23.99; N 3.18; S 14.14. $C_{16}H_{14}Cl_3NO_4S_2$. Calculated, %: C 42.26; H 3.10; Cl 23.39; N 3.08; S 14.10.

**2-Methyl-4-[2,2,2-trichloro-1-(4-chlorophenylsulfonylamino)ethyl]phenylsulfanylacetic acid (XVI).** Yield 4.87 g (97%), mp 149–153°C. IR spectrum, ν, $cm^{-1}$: 1160, 1340 ($SO_2$); 1700 (C=O); 3240 (NH). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 2.08 s (3H, $CH_3$), 3.76 s (2H, $SCH_2$), 5.09 d (1H, CH, $^3J$ = 10.2 Hz), 7.03 d (1H, 6-H), 7.11 s (1H, 3-H), 7.84 d (1H, 5-H), 7.31 d and 7.50 d (2H each, *AA'BB'*, $ClC_6H_4$), 9.11 d (1H, NH, $^3J$ = 10.2 Hz). Found, %: C 40.59; H 3.08; Cl 28.67; N 2.64; S 12.91. $C_{16}H_{13}Cl_4NO_5S$. Calculated, %: C 40.57; H 3.00; Cl 28.18; N 2.78; S 12.74.

**2-(4-Carboxymethoxyphenyl)-2-(4-chlorophenylsulfonylamino)acetic acid (XX).** Compound **II**, 4.87 g (0.01 mol), was added to a solution of 4.00 g (0.1 mol) of sodium hydroxide in 200 ml of water. The mixture was placed in a microwave oven and was irradiated for 5×2 min (800 W), water being added to maintain the initial volume. The mixture was then cooled to 30°C and acidified to pH 1 with 10% hydrochloric acid. A tarry material separated and was left to stand for 3 h for crystallization, washed with water until neutral, and dried in air. Yield 3.55 g (89%), mp 85–88°C. IR spectrum, ν, $cm^{-1}$: 1170, 1350 ($SO_2$); 1700 (C=O); 3200 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 4.70 s (2H, $OCH_2$), 5.09 s (1H, CH), 6.86 d and 7.29 d (2H each, *AA'BB'*, $ClC_6H_4$), 7.64 d and 7.96 d (2H each, *AA'BB'*, $C_6H_4O$), 7.58 s (1H, NH). Found, %: C 48.67; H 3.81; Cl 9.36; N 3.78; S 8.56. $C_{16}H_{14}ClNO_7S$. Calculated, %: C 48.07; H 3.53; Cl 8.87; N 3.50; S 8.02.

Compounds **XXI**, **XXII**, and **XXVI**–**XXVIII** were obtained in a similar way.

**2-(4-Carboxymethylsulfanylphenyl)-2-(4-chlorophenylsulfonylamino)acetic acid (XXI).** Yield 2.98 g (72%), mp 110–112°C. IR spectrum, ν, $cm^{-1}$: 1160, 1340 ($SO_2$); 1700 (C=O); 3250 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 3.77 s (2H, $SCH_2$), 5.13 s (1H, CH), 7.16 d and 7.58 d (2H each, *AA'BB'*, $C_6H_4S$), 7.62 d and 7.94 d (2H each, *AA'BB'*, $ClC_6H_4$), 7.84 s (1H, NH). Found, %: C 46.70; H 3.30; Cl 9.53; N 3.30; S 15.37. $C_{16}H_{14}ClNO_6S_2$. Calculated, %: C 46.21; H 3.39; Cl 8.53; N 3.37; S 15.42.

**2-(4-Carboxymethoxy-3-methylphenyl)-2-(4-chlorophenylsulfonylamino)acetic acid (XXII).** Yield 3.09 g (75%), mp 66–70°C. IR spectrum, ν, $cm^{-1}$: 1120, 1150, 1350 ($SO_2$); 1710 (C=O); 3250 (NH). $^1$H NMR spectrum (acetone-$d_6$), δ, ppm: 2.15 s (3H, $CH_3$), 4.67 s (2H, $OCH_2$), 5.05 s (1H, CH), 6.73 d (1H, 6-H), 7.06 s (1H, 3-H), 7.11 d (1H, 5-H), 7.56 d and 7.94 d (2H each, *AA'BB'*, $ClC_6H_4$), 7.86 s (1H, NH). Found, %: C 49.70; H 3.94; Cl 7.51; N 3.45; S 7.38. $C_{17}H_{16}ClNO_7S$. Calculated, %: C 49.34; H 3.90; Cl 8.57; N 3.38; S 7.75.

**2-(4-Chlorophenylsulfonylamino)-2-(2-furyl)-acetic acid (XXVI).** Yield 0.34 g (53%), mp 145–149°C. IR spectrum, ν, $cm^{-1}$: 1170, 1310 ($SO_2$); 1450, 3060 (furyl); 1710 (C=O); 3250 (NH). $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 5.31 (1H, CH), 6.50 d and 6.21 d (3H, furyl), 7.40 d and 7.88 d (2H each, *AA'BB'*, $ClC_6H_4$). Found, %: C 45.01; H 3.15; Cl 10.58; N 4.40;

S 10.25. $C_{12}H_{10}ClNO_5S$. Calculated, %: C 45.65; H 3.19; Cl 11.23; N 4.44; S 10.15.

**2-(4-Chlorophenylsulfonylamino)-2-(1-naphthyl)-acetic acid (XXVII).** Yield 0.33 g (45%), mp 100–102°C. IR spectrum, ν, $cm^{-1}$: 1150, 1310 ($SO_2$); 1710 (C=O); 3360 (NH). $^1H$ NMR spectrum (acetone-$d_6$), δ, ppm: 5.83 s (1H, CH), 7.38–8.14 m (11H, $C_6H_4$, $C_{10}H_7$), 10.74 s (1H, OH). Found, %: C 57.59; H 3.65; Cl 9.00; N 3.75; S 8.61. $C_{18}H_{14}ClNO_4S$. Calculated, %: C 57.53; H 3.75; Cl 9.43; N 3.73; S 8.53.

**2-(4-Chlorophenylsulfonylamino)-2-(1-methyl-1*H*-indol-3-yl)acetic acid (XXVIII).** Yield 0.9 g (49%), mp 152–160°C; published data [11]: mp 155–160°C. IR spectrum, ν, $cm^{-1}$: 1120, 1320 ($SO_2$); 1700 (C=O); 3230 (NH). $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 3.91 s (3H, $NCH_3$), 3.38 s (1H, CH), 7.33–8.22 m (9H, $H_{arom}$), 8.47 s (1H, NH).

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