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Aminomethanesulfonic Acids as Reaction Products in SO_2–NH_2Alk–CH_2O–H_2O Systems: Synthesis and Structure

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Abstract—An original procedure was proposed for the synthesis of a series of aminomethanesulfonic acids $AlkNHCH_2SO_3$ (Alk = *n*-Bu, *n*-Hept, *n*-Oct, Bn) and *N*-tris(hydroxymethyl)methylammonium hydroxymethanesulfonate. The structure of the compounds synthesized was examined by elemental analysis, X-ray diffraction, IR spectroscopy, and mass spectrometry.

Keywords: aminoalkanesulfonic acids, sulfur(IV) oxide, paraformaldehyde, alkylamine, condensation

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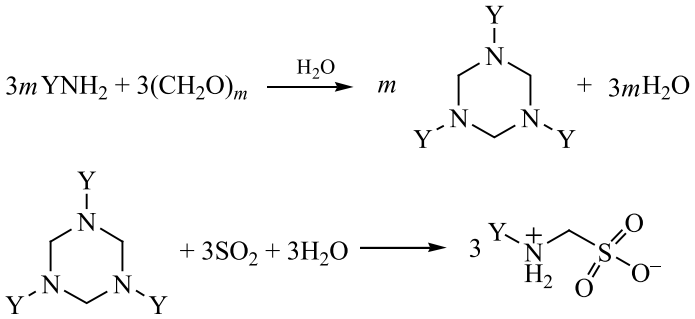
Aminoalkanesulfonic acids, in particular aminomethanesulfonic acid (AMSA) and its N-alkylated derivatives (YAMSA), possess specific physicochemical properties, display broad-spectrum biological activity [1–4], and have low toxicity [5]. This makes them promising drug candidates and buffer components for biomedical research [6–11].

Earlier, we synthesized by the original technique and spectrally characterized a number of new derivatives of aminomethanesulfonic acid [5, 12–14] (Scheme 1). *N*-Methyl [15], *N*-2-hydroxyethyl [13], *N*-*n*-propyl [5], and *N*-*tert*-butyl [14] derivatives of AMSA were structurally characterized in contrast to the *N*-benzyl analog [12].

As a continuation of our previous studies [5, 12–14], we report herein on the synthesis, structure, and spectral features of *N*-(butyl)- (**1**), *N*-(heptyl)- (**2**), *N*-(octyl)- (**3**), and *N*-benzylaminomethanesulfonic acids (**4**) and *N*-tris(hydroxymethyl)methylammonium hydroxymethanesulfonate (**5**) formed as the reaction

Scheme 1.



Y = CH_3 (MeAMSA), $HOCH_2CH_2$ (HEAMSA), *n*-Pr (*n*-PrAMSA), *t*-Bu (*t*-BuAMSA) and Bn (BnAMSA).

Table 1. Crystallographic data and structure refinement results for compounds **1**–**5**

Parameter	**1**	**2**	**3**	**4**	**5**
CCDC	2040821	2040822	2040824	2040837	2040838
Formula	$C_5H_{13}NO_3S$	$C_8H_{19}NO_3S$	$C_9H_{21}NO_3S$	$C_8H_{11}NO_3S$	$C_5H_{15}NO_7S$
M_r	167.22	209.30	223.33	201,24	233,24
T, K	293(2)	293(2)	293(2)	293(2)	293(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	$C2/c$	$P2_1/n$	$P2_1/c$	$P2_1$	$P2_1/c$
a, Å	24.608(13)	5.4164(3)	17.748(4)	5.2699(5)	8.1634(6)
b, Å	7.9776(19)	30.8675(15)	5.4192(7)	28.105(3)	6.3494(4)
c, Å	17.811(7)	6.5500(4)	12.9508(16)	6.4061(8)	18.4863(12)
β, deg	94.63(4)	98.850(7)	93.355(15)	93.606(10)	96.132(5)
V, Å^3	3485(2)	1082.06(11)	1243.5(4)	946.93(17)	952.71(11)
Z	16	4	4	4	4
d_{calc}, g/cm^3	1.275	1.285	1.193	1.412	1.626
μ(MoK_α), mm^{-1}	0.328	0.278	0.246	0.316	0.355
θ range, deg	3.49–26.00	3.22–26.00	3.42–25.99	3.50–26.00	3.17–29.00
Crystal dimensions, mm	0.60×0.02×0.015	0.60×0.03×0.02	0.50×0.20×0.03	0.25×0.20×0.03	0.45×0.25×0.20
F_{000}	1440	456	455	424	496
Transmission, T_{min}/T_{max}	0.827/0.995	0.851/0.995	0.887/0.993	0.925/0.991	0.857/0.932
Number of measured	8838	6662	5395	5908	6560
independent	3696	2092	2323	3135	2174
observed c $I_{hkl} > 2\sigma(I)$	801	1612	1461	2388	1815
R_{int}	0.2553	0.0552	0.0746	0.0564	0.0250
Completeness, %	95.6	98.3	95.1	98.4	98.9
Number of refined parameters	202	126	135	237	151
R_F/wR^2 on observed reflections	0.0769/0.1162	0.0373/0.0865	0.0685/0.1557	0.0504/0.0646	0.0324/0.0880
R_F/wR^2 on independent reflections	0.3365/0.2035	0.0563/0.0940	0.1060/0.1782	0.0799/0.0714	0.0414/0.0940
S	0.950	0.943	0.981	0.987	0.979
$\Delta\rho_{min}/\Delta\rho_{max}$, e/Å^3	–0.218/0.239	–0.262/0.304	–0.216/0.051	–0.251/0.437	–0.351/0.314

products in the sulfur(IV) oxide–primary alkylamine–formaldehyde–water systems.

The structure of compounds **1**–**5** was proved by X-ray diffraction analysis. Tables 1 and 2 present the main crystallographic data and structure refinement parameters for these compounds. The atomic coordinates, structure factors, and all the refinement results for **1**–**5** were deposited with the Cambridge Crystallographic Data Center.

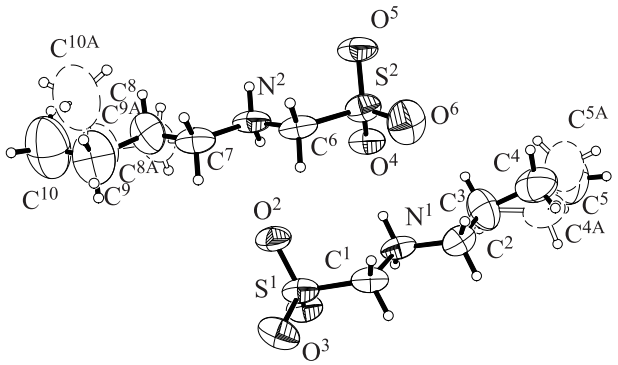


Fig. 1. General view of a molecule of compound **1** in the crystal.

The basic structural unit in compounds **1**–**4** is the $(Alk)N^+H_2CH_2SO_3^-$ zwitterion. In the independent part of the unit cell of **1** and **4** there are two zwitterions, and in those of **2** and **3**, one zwitterion. Structural features shared by aminomethanesulfonic acids **1**–**4** are hydrogen bonding between the nitrogen atoms and oxygen atoms of the sulfonic groups from the neighboring molecules and packing of the molecules such that their polar parts form layers having nonpolar parts on the outside (although there are some differences in arrangement of hydrogen bonds in the layers).

Figure 1 shows the structure of aminomethanesulfonic acid **1**. Both basic molecules exhibit disorder of terminal atoms in the nonpolar part, while the polar parts of the

Table 2. Characteristics of the D–H···A hydrogen bonds in compounds **1**–**5**

D–H···A	Distance, Å			DHA angle, deg	Transformation for atom A
	d(D–H), Å	*d*(H···A), Å	*d*(D···A), Å		
			1		
N^1–H^{1A}···O^4	0.90	1.90	2.762(7)	159.5	
N^1–H^{1B}···O^1	0.90	2.14	2.957(8)	150.5	–*x*+1, *y*, –*z*+1/2
N^1–H^{1B}···O^5	0.90	2.34	2.932(7)	123.2	*x*, *y*+1, *z*
N^2–H^{2C}···O^4	0.90	2.14	2.933(8)	147.2	–*x*+1, *y*, –*z*+1/2
N^2–H^{2C}···O^2	0.90	2.42	2.957(7)	118.8	
N^2–H^{2D}···O^1	0.90	2.02	2.840(7)	150.7	*x*, *y*–1, *z*
			2		
N^1–H^{1A}···O^2	0.844(16)	2.018(16)	2.8252(15)	159.8(15)	*x*+1/2, –*y*+1/2, *z*+1/2
N^1–H^{1B}···O^1	0.823(15)	2.329(15)	2.8971(15)	126.7(14)	*x*–1/2, –*y*+1/2, *z*–1/2
N^1–H^{1B}···O^2	0.823(15)	2.487(14)	2.9222(15)	114.1(12)	
			3		
N^1–H^{1N}···O^2	0.839(5)	2.046(7)	2.881(3)	173(3)	–*x*+1, *y*+1/2, –*z*+3/2
N^1–H^{2N}···O^1	0.840(5)	2.075(15)	2.838(3)	151(2)	–*x*+1, *y*–1/2, –*z*+3/2
			4		
N^1–H^{1A}···O^4	0.89	2.57	3.141(4)	122.6	
N^1–H^{1A}···O^5	0.89	2.53	3.165(4)	129.3	*x*, *y*, *z*–1
N^1–H^{1B}···O^4	0.89	2.59	3.033(4)	111.5	*x*+1, *y*, *z*
N^1–H^{1B}···O^6	0.89	2.04	2.847(4)	150.8	
N^2–H^{2A}···O^1	0.89	2.04	2.848(4)	149.5	
N^2–H^{2A}···O^2	0.89	2.59	3.038(4)	111.8	*x*–1, *y*, *z*
N^2–H^{2B}···O^2	0.89	2.52	3.106(4)	123.6	
N^2–H^{2B}···O^3	0.89	2.55	3.175(4)	127.8	*x*, *y*, z+1
			5		
O^1–H^1···O^6	0.818(17)	1.913(17)	2.7219(12)	169.4(17)	*x*+1, *y*, *z*
O^2–H^2···O^3	0.821(19)	1.882(19)	2.6945(13)	170.1(18)	*x*, *y*–1, *z*
O^3–H^3···O^5	0.81(2)	1.94(2)	2.7491(14)	170.8(18)	
O^7–H^7···O^5	0.886(19)	2.020(19)	2.8517(16)	155.8(17)	–*x*+1, –*y*+1, –*z*+1
N^1–H^{1A}···O^6	0.886(15)	1.994(15)	2.8781(14)	174.7(14)	–*x*+1, *y*–1/2, –*z*+1/2
N^1–H^{1B}···O^2	0.856(16)	1.976(16)	2.8208(13)	168.9(14)	–*x*+1, *y*+1/2, –*z*+1/2
N^1–H^{1C}···O^1	0.852(16)	2.108(16)	2.9415(13)	165.8(14)	–*x*+2, *y*+1/2, –*z*+1/2

molecules are interconnected by hydrogen bonds and tightly packed. Each molecule has a close to planar backbone chain (sulfur, nitrogen, and carbon atoms). These chains are located in $y = 0$ and $y = 0.5$ planes and form layers interconnected by a two-dimensional network of H-bonds (Fig. 2) elongated along the b-axis; among them, N^1–H^{1B}···O^1, N^1–H^{1B}···O^5, N^2–H^{2C}···O^4, N^2–H^{2C}···O^2 are forked bonds.

The structure of the basic unit of *n*-$C_7H_{15}N^+H_2CH_2SO_3^-$ (**2**) is shown in Fig. 3. The N^1 and C^1–C^8 atoms are coplanar within 0.018 Å; the deviations of the sulfo group atoms from this plane are –0.223(2), 1.050(2), –1.252(3), and –0.650(2) Å for S^1, O^1, O^2, and O^3 atoms, respectively. In a crystal, the H-bonded molecules (Table 2) are packed into layers in the (101) planes. Both the molecules in the layers and the layers are crosslinked by hydrogen bonds. Like in the case of compound **1**, N^1–H^{1B}···O^1 and N^1–H^{1B}···O^2 are forked bonds. As a result, a two-dimensional network of H-bonds is formed in the $z = 0.25$ plane (Fig. 4).

In the structure of compound **3** (Figs. 5 and 6) only the carbon backbone of the alkyl moiety is planar within 0.046 Å; the ammoniomethanesulfonyl (AMS) moiety rotates around the C^2–C^3 bond and deviates significantly from the backbone plane [the torsion angle $N^1C^2C^3C^4$ is –65.3(5)° against 179.61(12)° in the molecule of **2**]. The AMS moieties in the crystal are packed in the $x = 0.5$ plane (Fig. 6) and are interconnected by zigzag chains of H-bonds, aligned along the [010] axis.

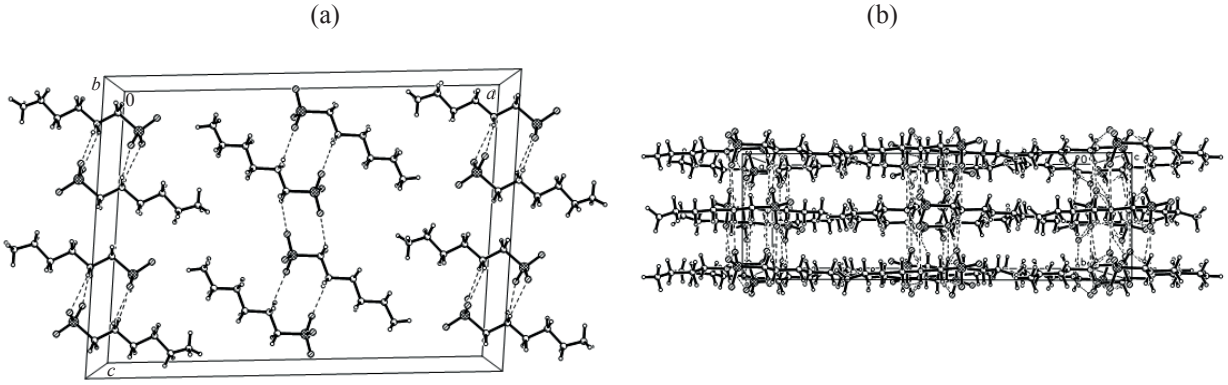


Fig. 2. Crystal packing in compound **1**: (a) location of the molecules in the layer and (b) mutual arrangement of the layers and the hydrogen bonds network. Hydrogen bonds are depicted by dashed lines. Disorder of the terminal atoms is omitted.

Compound **4** (Figs. 7 and 8) has two basic zwitterions n-$C_6H_5CH_2N^+H_2CH_2SO_3^-$ paired via H-bonds; the neighboring pairs are linked by hydrogen bonds into one-dimensional zigzag chains aligned along the [010] axis, like in the molecule of compound **3**.

By contrast to aminomethanesulfonic acids **1**–**4**, in compound **5** the basic structural units are *N*-tris(hydroxymethyl)methylammonium ion $[(HOCH_2)_3CNH_3]^+$ and hydroxymethanesulfonate ion $HOCH_2SO_3^-$ (see Fig. 9). Not only the ammonium group and the oxygen atoms of the sulfo group but also all the hydroxo groups are involved in hydrogen bonding (Table 2), which results in a three-dimensional network of H-bonds (Fig. 10).

Thermal decomposition of aminomethanesulfonic acids **1**–**4** under mass-spectrometric conditions (EI, FAB) leads to degradation of the products, accompanied by the elimination of SO_3 (**1**–**3**, similarly to MeAMSA, HEAMSA, and *t*-BuAMSA [12–14]). The decomposition

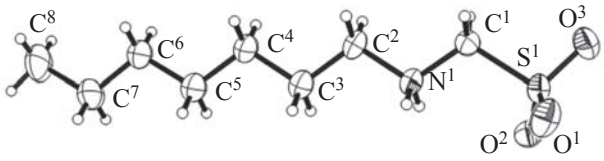


Fig. 3. General view of a molecule of compound **2** in the crystal.

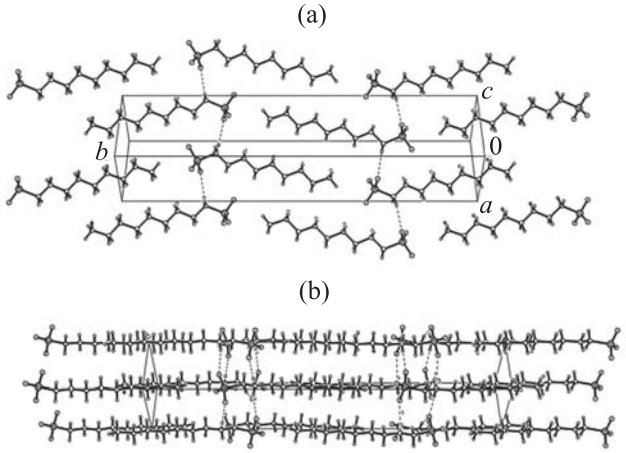


Fig. 4. Crystal packing in compound **2**: (a) location of the molecules in the layer and (b) mutual arrangement of the layers and the hydrogen bonds network. Hydrogen bonds are depicted by dashed lines.

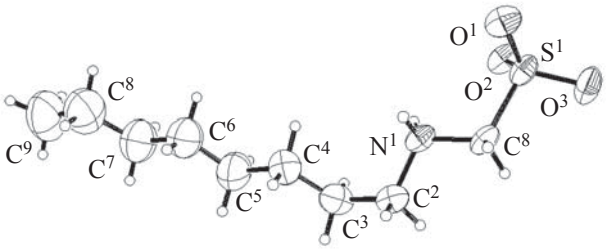


Fig. 5. General view of a molecule of compound **3** in the crystal.

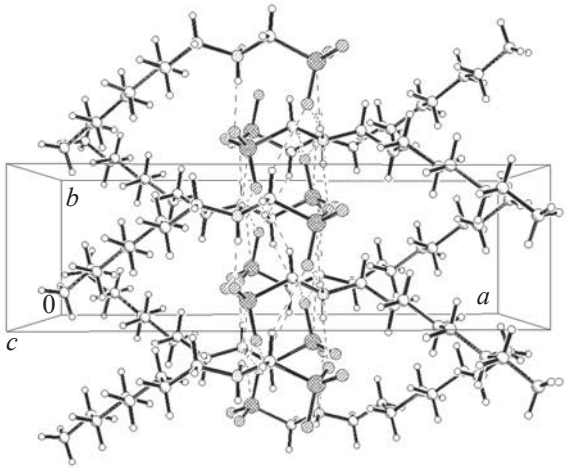


Fig. 6. Crystal packing of compound **3**. Hydrogen bonds are depicted by dashed lines.

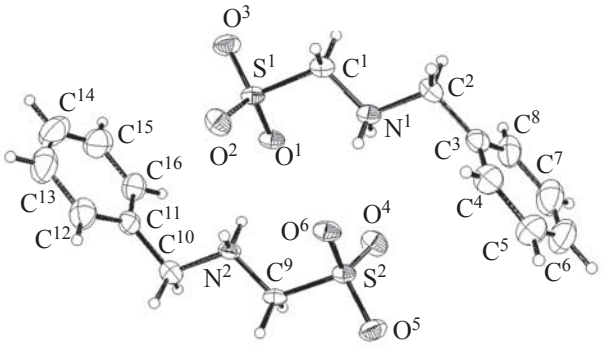


Fig. 7. General view of a molecule of compound **4** in the crystal.

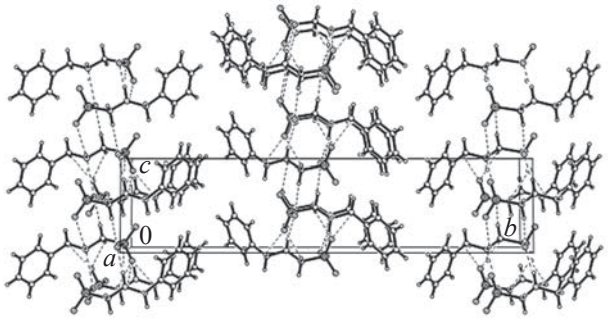


Fig. 8. Crystal packing and the hydrogen bonds network in compound **4**. Hydrogen bonds are depicted by dashed lines.

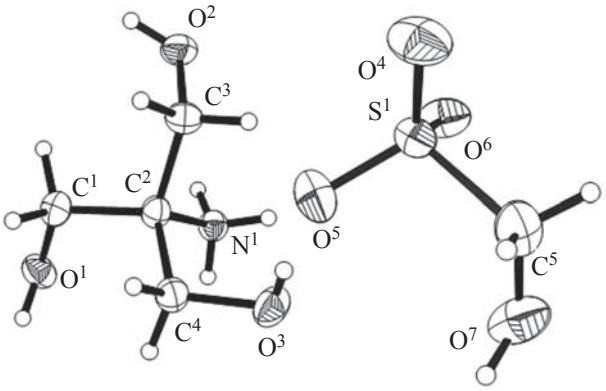


Fig. 9. General view of a molecule of compound **5** in the crystal.

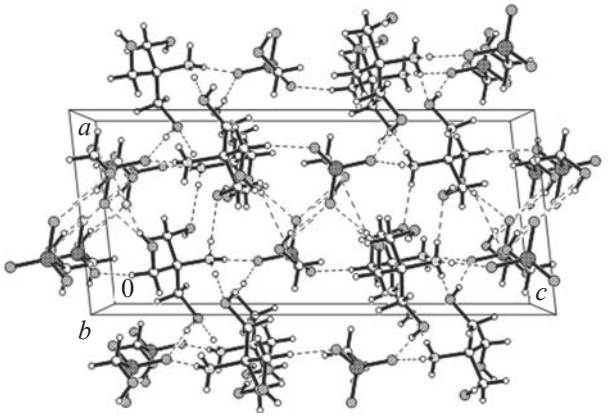


Fig. 10. Crystal packing and the hydrogen bonds network in compound **5**. Hydrogen bonds are depicted by dashed lines.

of compound **1**, like that in the case of *n*-PrAMSA and *t*-BuAMSA [5, 14], is accompanied by the elimination of NH_3.

In the mass spectrum of salt **5** the most intense peak is due to the $[M_{TRIS} - CH_2OH]^{+\cdot}$ ion, a characteristic product of ethanolamine fragmentation [16].

Table 3 presents the results of analysis of the IR spectra of compounds **1**–**5**. The bands were assigned using the data from [17, 18]. Stretching vibrations of the OH groups in the IR spectrum of compound **5** gave rise to a doublet with maxima at 3440 and 3230 cm^{-1}. The ν(NH) vibrations of the hydrogen-bonded amino groups are observed in the 3470–3020 cm^{-1} region for all the compounds synthesized. For the N-derivatives of AMSA **1**–**4** and salt **5** the $\nu_{as}(SO_2)$ vibrations occur in the 1270–1130 cm^{-1} region, and the $\nu_s(SO_2)$ vibrations, in the 1183–1023 cm^{-1} region; the ν(S–O) vibrations gave rise to bands of strong (**4** and **5**), medium (**1** and **2**), and weak (**3**) intensity in the 590–525 cm^{-1} region.

Like for the previously studied AMSA [19] and its derivatives [5, 12–14], no major shifting of the ν(NH) band was observed in the IR spectra of compounds **1**–**4**, which evidences the preservation of their zwitterionic structure.

It can be stated that the interaction in the SO_2–YNH_2–CH_2O–H_2O systems [where Y = Alk, except for $(HOCH_2)_3C$] involves condensation as accompanied

Scheme 2.

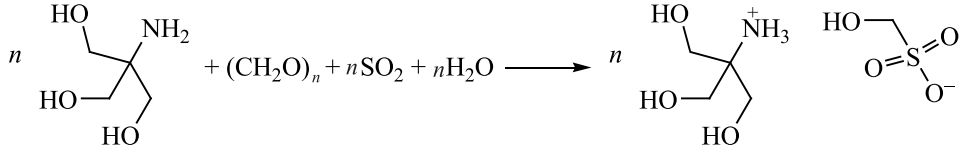


Table 3. Wavenumbers, cm^{-1}, of the IR absorption maxima in the spectra of compounds **1**–**5**

Comp. no.	ν(OH)	ν(NH^+), ν(CH)		ν(NH^+)	$\delta_s(NH_3^+)$, $\delta_s(NH_2^+)$	$\nu_{as}(SO_2)$	$\nu_s(SO_2)$	ν(S–O)
1		3320 m, 3123 sh, 3083 s, 3032 s	2963 sh, 2953 s, 2922 vs, 2870 s	2427 w, 2352 w	1679 sh, 1601 sh, 1592 s, 1501 s, 1493 s	1235 sh, 1206 d.s	1081 d.w, 1051 s, 1046 s	557 m
2		3322 s, 3205 s	3025 s, 2965 sh, 2957 vs, 2942 vs, 2926 vs, 2872 s, 2855 s	2352 w	1625 m, 1542 w, 1504 br. w, 1491 br. w, 1469 s	1241 d.s	1090 w, 1080 w, 1076 sh, 1054 s	551 m
3		3470 br. vs, 3412 br. vs	3013 br. vs, 2950 br. vs, 2878 br. vs, 2837 br. vs, 2816 br. vs	2570 m, 2492 sh	1626 s, 1565 s, 1542 sh, 1538 m, 1506 sh, 1498 br. m	1244 m. 1203 s,	1079 sh, 1056 s	580 w
4		3044 s, 2940 sh, 2847 m, 2819 m	2780 sh, 2605 m, 2316 w	1555 s	1237 s, 1217 s	1056 s, 1032 w, 1016 m	589 s	
5	3440 br.m, 3230 s	3039 sh, 2991 sh, 2942 sh, 2891 sh, 2835 m	2748 m, 2694 m, 2601 m, 2468 m, 2382 w, 2296 w	1631 s, 1552 s, 1516 w	1133 sh	1035 s	526 s	

by the S(IV)→S(VI) oxidation with the formation of N-alkylated AMSA derivatives. The yield of the target product depends substantially on the structure of the N-substituent. Specifically, in the series of N-substituents CH_3 (~100% [12])–$HOCH_2CH_2$ (~100% [13])–*n*-C_3H_7 (~100% [5])–*n*-C_4H_9 (~92.3%)–*n*-C_7H_{15} (~67.3%)–*n*-C_8H_{17} (~ 56.2%) the yield (bracketed figures) of the target product tends to decrease as the hydrocarbon substituent gets bulkier starting from C_4, which is probably due to the side reaction of hydrolysis of the AMSA derivatives [20]. In particular, for the system containing TRIS the product of hydrolysis of the target compound, N-tris(hydroxymethyl)methylammonium hydroxymethanesulfonate, was isolated, like in the previously described example (Scheme 2) [8].

Taking into account our previous results [5, 12], we can conclude that the aminomethanesulfonic acid derivatives characterized in this study may be of interest for further pharmacological studies as potential antiviral and antibacterial agents.

EXPERIMENTAL

The carbon, hydrogen, and nitrogen contents were estimated using a CHN elemental analyzer, and the sulfur content, by Schöniger's method. IR spectra (KBr pellets) were measured on a Spectrum BX II FT-IR System (PerkinElmer) instrument in the 4000–350 cm^{-1} range. Mass spectra (EI) were recorded on an MX-1321 instrument (direct sample introduction into the ion source, ionizing electron energy 70 eV). Mass spectra (FAB) were taken on a VG 7070 instrument, with 8 keV argon atoms desorbing ions from the liquid matrix (*m*-nitrobenzyl alcohol).

X-ray diffraction analysis was carried out on an Xcalibur-3 (Oxford Diffraction) instrument (MoK_α radiation, graphite monochromator, Sapphire-3 CCD detector). The structure was decoded, refined, and analyzed using SHELXT, SHELXL-16, and WinGX software suites [21–23]. Positions of H atoms were found in a difference electron-density synthesis and refined using a rider model. Hydrogen atoms involved in hydrogen bonding in the **2**, **3**, and **5** structures were refined in the isotropic approximation.

We used commercial sulfur(IV) oxide after preliminary purification and drying according to the procedure described in [24]. *n*-BuA, *n*-HpA, *n*-OcA, TRIS, benzylamine, and paraformaldehyde were commercial

reagents of pure grade and were used without further purification.

***N*-(Butyl)aminomethanesulfonic acid (1)**. To 25 mL of an aqueous emulsion containing 0.10 mol of *n*-butylamine, an equimolar amount of paraformaldehyde was added under cooling (≤10°C) and left for 24 h, whereupon the resulting solution was purged with SO_2 to pH ≤ 1.0. The reaction mixture was kept at room temperature until complete evaporation of water. Yield 15.43 g (~92.3%), white crystals, mp 136–139°C (mp 135–137°C [20]). Mass spectrum (FAB), *m/z* (I_{rel}, %): 168 (8) $[M - NH_3 + H]^+$, 166 (7) $[M - NH_3 - H]^+$, 138 (14), 137 (43), 136 (55), 89 (12), 86 (23) $[M - NH_3 - SO_3 - H]^+$, 77 (8), 57 (7), 55 (7), 43 (11), 42 (9), 41 (7). Found, %: C 35.25; H 8.26; N 8.62; S 19.57. $C_5H_{13}NO_3S$. Calculated, %: C 35.91; H 7.84; N 8.38; S 19.17. *M* 167.23.

***N*-(Heptyl)aminomethanesulfonic acid (2)** was prepared similarly from 0.10 mol of *n*-heptylamine. The resulting white foamy mass was filtered off, and the filtrate was kept in air until the water evaporated completely to give white crystals. Yield 14.09 g (~67.3%). Mass spectrum (FAB), *m/z* (I_{rel}, %): 128 (6) $[M - SO_3 - H]^+$, 117 (5), 116 (100) $[M - SO_3 - CH_2 - H]^+$, 57 (6), 40 (9). Found, %: C 45.63; H 8.83; N 6.43; S 15.67. $C_8H_{19}NO_3S$. Calculated, %: C 45.91; H 9.15; N 6.69; S 15.32. *M* 209.31.

***N*-(Octyl)aminomethanesulfonic acid (3)** was prepared similarly from 0.10 mol of *n*-octylamine. Yield 13.11 g (~56.2%). Mass spectrum (FAB), *m/z* (I_{rel}, %): 142 (30) $[M - SO_3 - H]^+$, 137 (6), 136 (8), 131 (22), 130 (100) $[M - SO_3 - CH_2 - H]^+$, 128 (6), 71 (6), 57 (7), 42 (6), 40 (5). Found, %: C 48.11; H 9.11; N 6.49; S 14.02. $C_9H_{21}NO_3S$. Calculated, %: C 48.40; H 9.48; N 6.27; S 14.36. *M* 233.34.

***N*-Benzylaminomethanesulfonic acid (4)** was synthesized as described in [5] using 0.05 mol of benzylamine. Yield 10.00 g (~100%), white crystals, mp 144–145°C. Mass spectrum (EI), *m/z* (I_{rel}, %): 91 (100) $[C_7H_7]^{+\cdot}$, 77 (15) $[C_6H_5]^{+\cdot}$, 64 (50) $[SO_2]^{+\cdot}$, 48 (21) $[SO]^{+\cdot}$. Found, %: C 45.90; H 5.95; N 7.20; S 15.55. $C_8H_{11}NO_3S$. Calculated, %: C 47.75; H 5.51; N 6.96; S 15.93. *M* 201.25.

***N*-Tris(hydroxymethyl)methylammonium hydroxymethanesulfonate (5)** was prepared similarly to **1** using 0.05 mol of TRIS. Yield 11.66 g (~100%), white crystals, mp 82–83°C. Mass spectrum (EI), *m/z* (I_{rel}, %): 118 (12), 114 (10), 104 (14), 102 (29), 100 (36), 90 (100) $[M_{TRIS} - CH_2OH]^{+\cdot}$, 83 (21), 73 (11), 72 (11), 71 (31), 70 (35), 64 (31) $[SO_2]^{+\cdot}$, 60 (60), 56 (15), 54 (13), 48 (13) $[SO]^{+\cdot}$, 43 (12), 42 (70), 41 (20), 30 (54), 29 (20). Found, %: C 32.58; H 8.29; N 7.42; S 17.89. $C_5H_{15}NO_7S$. Calculated, %: C 32.42; H 8.16; N 7.56; S 17.31. *M* 185.07.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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