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PHYSICAL METHODS OF INVESTIGATION

Onium Sulfates and Hydrogen Sulfates: Products of Reactions of Sulfur(IV) Oxide with Aqueous Solutions of Alkylamines and Aniline

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Abstract—The reaction products formed in the SO_2–L–H_2O–O_2 systems (L is *n*-propylamine, *n*-butylamine, *tert*-butylamine, *n*-heptylamine, *n*-octylamine, aniline) were isolated and identified as "onium" salts $[n\text{-}C_3H_7NH_3]_2SO_4$, $[n\text{-}C_4H_9NH_3]_2SO_4$, $[t\text{-}C_4H_9NH_3]_2SO_4$, $[n\text{-}C_7H_{15}NH_3]_3SO_4(HSO_4)$, $[n\text{-}C_8H_{17}NH_3]_3SO_4(HSO_4)$, and $[C_6H_5NH_3]_2SO_4$. The products were characterized by elemental analysis, IR and Raman spectroscopy, mass spectrometry, and thermogravimetry.

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The non-catalyzed auto-oxidation reactions of sulfurous compounds, most often, sulfur(IV) oxide, are of obvious interest for the theory and practice of chemical engineering processes [1–3]. For example, scavenging of sulfur(IV) oxide from gas–air mixtures by chemisorbents based on organic nitrogen bases in the presence of O_2 is accompanied by S(IV) → S(VI) oxidation [4]. The reaction products thus formed (depending on the chemisorption conditions) may suppress the absorption capacity during sulfur(IV) oxide sorption–desorption cycles and complicate the thermal regeneration of the sorbents [5, 6]. The sulfur oxidation can also considerably affect the ion exchange efficiency [7].

Previously [8–10], we isolate onium sulfites from the SO_2–L–H_2O–O_2 reaction systems (L = ethanolamines, aminoguanidine); in the case of analogous systems in which L is methylamine, *tert*-butylamine (*t*-BA), benzylamines, tris(hydroxymethyl)aminomethane (TRIS), or hexamethylenediamine, onium sulfates are formed under similar conditions [11–14]. This paper presents a method of synthesis, spectral characteristics, and thermal stability data for the products of reactions of SO_2 with aqueous solutions of *n*-propylamine, *n*-butylamine, *t*-BA, *n*-heptylamine, *n*-octylamine, and aniline (compounds **I–VI,** respectively) in the presence of air oxygen.

EXPERIMENTAL

***n*-Propylammonium sulfate (I).** A solution of *n*-propylamine (0.10 mol) in 25 mL of water was poured into a temperature-controlled cell, and at 0°C, gaseous SO_2 was bubbled though the solution at a 50 mL min^{-1} rate until pH < 1.0 was attained. The solution with the precipitate was subjected to isothermal evaporation at room temperature in air until water was completely removed. The isolated white-colored crystalline product **I** (10.28 g, 95.0% yield based on *n*-propylamine) was not additionally purified. FAB MS: $[M_L + H]^+$ (m/z 60, I, 100%); $[M_L - H]^+$ (m/z 58, I, 5%); m/z 43, I, 13%; $[M_L - NH_3]^+$ (m/z 42, I, 96%); $[M_L - NH_3 + H]^+$ (m/z 41, I, 15%).

For $C_6H_{20}N_2O_4S$ anal. calcd. (%): C, 33.32; H, 9.32; N, 12.95; S, 14.82. FW = 216.30.

Found (%): C, 33.89; H, 9.54; N, 12.42; S, 15.31.

***n*-Butylammonium sulfate (II)**. A similar sequence of procedures with the use of an aqueous solution of *n*-butylamine (0.1 mol of the amine in 25 mL of H_2O) afforded a gel-like pale yellow product **II** (11.95 g isolated; 97.8% yield based on *n*-butylamine). FAB MS: m/z 245, I, 27%; m/z 194, I, 7%; m/z 75, I, 7%; $[M_L + H]^+$ (m/z 74, I, 100%); m/z 69, I, 6%; $[M_L - NH_3 + H]^+$ (m/z 57, I, 9%); m/z 55, I, 9%.

For $C_8H_{24}N_2O_4S$ anal. calcd. (%): C, 39.32; H, 9.90; N, 11.46; S, 13.12. FW = 244.36.

Found (%): C, 39.82; H, 9.38; N, 12.78; S, 12.80.

***tert*-Butylammonium sulfate (III)** was synthesized using analogous procedures [11]. EI MS: $[SO_3]^{+\bullet}$ (*m/z* 80, *I*, 32%); $[SO_2]^{+\bullet}$ or $[S_2]^{+\bullet}$ (*m/z* 64, *I*, 13%); $[M_L - NH_3 + 2H]^{+\bullet}$ (*m/z* 58, *I*, 100%); $[SO]^+$ (*m/z* 48, *I*, 10%); *m/z* 43, *I*, 10%; *m/z* 41, *I*, 22%; *m/z* 40, *I*, 12%; $[S]^+$ (*m/z* 32, *I*, 10%). Raman lines in the spectrum of **III** (cm^{-1}): 3230 w, 3159 m [$\nu(NH_3^+)$]; 2988 s, 2900 m [$\nu_{as}(CH_3)$, $\nu(NH_3^+)$]; 2885 m, 2775 w, 2745 w, 2650 w [$\nu_s(CH_3)$, $\nu(NH_3^+)$]; 1316 w [$\nu(CN)$, $\nu(CC)$]; 1220 w [$\nu(CN)$]; 1125 m [$\nu_3 \equiv \nu_{as}(SO)$]; 1010 m, 975 s, 941 m [$r(CH_3)$, $r(NH_3^+)$, $\nu_1 \equiv \nu_s(SO)$] (recording the spectra below 700 cm^{-1} is impossible because of strong luminescence of the sample).

***n*-Heptylammonium sulfate-hydrogen sulfate (IV).** A similar sequence of procedures with the use of an aqueous solution of *n*-heptylamine (0.05 mol of the amine in 25 mL of H_2O) gave a white-colored waxy product **IV** (8.74 g isolated, 96.8% yield based on *n*-heptylamine). FAB MS: $[M_L + H]^+$ (*m/z* 116, *I*, 79%); *m/z* 57, *I*, 38%; *m/z* 56, *I*, 8%; *m/z* 55, *I*, 49%; *m/z* 53, *I*, 8%; *m/z*, 43, *I*, 16%; *m/z* 42, *I*, 100%; *m/z* 41, *I*, 14%.

For $C_{21}H_{55}N_3O_8S_2$ anal. calcd. (%): C, 46.55; H, 10.23; N, 7.76; S, 11.84. FW = 541.82.

Found (%): C, 45.74; H, 10.11; N, 7.68; S, 12.32.

***n*-Octylammonium sulfate-hydrogen sulfate (V).** A similar sequence of procedures with the use of an aqueous solution of *n*-octylamine (0.05 mol of the amine in 25 mL of H_2O) gave a gray-yellow waxy product **V** (9.47 g isolated, 97.3% yield based on *n*-heptylamine). FAB MS: $[M_L + H]^+$ (*m/z* 130, *I*, 32%); *m/z* 56, *I*, 16%; *m/z* 55, *I*, 36%; *m/z* 53, *I*, 8%; *m/z* 42, *I*, 100%; *m/z* 41, *I*, 8%.

For $C_{22}H_{61}N_3O_8S_2$ anal. calcd. (%): C, 49.37; H, 10.53, N 7.20; S, 10.98. FW = 583.89.

Found (%): C, 49.12; H, 10.68; N, 7.66; S, 11.78.

Anilinium sulfate (VI). A similar sequence of procedures with the use of an aqueous solution of aniline (0.05 mol of the amine in 25 mL of H_2O) gave a colorless crystalline product **V** (6.65 g isolated, 93.6% yield based on aniline). EI MS: $[M_L]^{+\bullet}$ (*m/z* 93, *I*, 100%); $[SO_3]^{+\bullet}$ *m/z* 80, *I*, 46%; $[M_L - HCN]^{+\bullet}$ (*m/z* 66, *I*, 16%); $[M_L - HCN{-}H]^{+\bullet}$ (*m/z* 65, *I*, 10%); $[SO_2]^{+\bullet}$ (*m/z* 64, *I*, 15%); $[SO]^{+\bullet}$ *m/z* 48, *I*, 24%; $[S]^+$ *m/z* 32, *I*, 10%.

For $C_{12}H_{16}N_2O_4S$ anal. calcd. (%): C, 50.69; H, 5.67; N, 9.85; S, 11.28. FW = 284.33.

Found (%): C, 51.27; H, 5.48, N, 9.27; S, 11.76.

Analysis for carbon, hydrogen, and nitrogen was carried out on an elemental CHN analyzer; sulfur was quantified by the Sheniger method [15]. IR absorption spectra were recorded on a Spectrum BX II FT-IR System spectrophotometer (Perkin-Elmer) (4000–350 cm^{-1} range; the samples were prepared as KBr pellets); Raman spectra were measured on a DFS-24 laser spectrometer with a semiconductor laser excitation (emission wavelength of 532 nm; interference monochromator; a 90° illumination scheme was used); EI mass spectra were run on an MX-1321 instrument (direct sample injection to the source; ionizing electron energy of 70 eV); FAB mass spectra were recorded on a VG 7070 instrument (ion desorption from the liquid matrix was accomplished by a beam of argon atoms with an energy of 8 keV; *m*-nitrobenzyl alcohol was used as the matrix).

The thermochemical transformations of compounds were studied on a Q-1500 D Paulik-Paulik-Erdey derivatograph in air (platinum crucibles, temperature range of 20–1000°C, heating rate of 10°C/min, DTA and DTG sensitivity of 1/5 of the maximum, and Al_2O_3 as the reference).

RESULTS AND DISCUSSION

The mass spectra of *n*-alkylammonium salts **I**, **II**, **IV**, and **V** exhibit an intense peak of the $[M_L + H]^+$ ion (the maximum intensity is observed for compound **II**). The characteristics of *t*-BA fragmentation products in the mass spectra of onium salt **III** are in good agreement with those in the tabulated mass spectrum of *t*-BA [16]; the same is true for aniline [17] and its onium salt **VI**. The mass spectrum of **VI** shows the fragmentation of $[M_L]^+$ ion [18] involving the expulsion of HCN and H_2CN, which is typical of arylamines.

Table 1 presents the results of analysis of the IR spectra of compounds **I**–**VI**. The IR absorption bands of sulfates **I**, **II**, and **VI** and mixed sulfates–hydrogen sulfates **IV** and **V** were assigned using published data [19–25]; the IR and Raman absorption bands (lines) of salt **III** were assigned taking account of the known experimental and theoretical data [20].

In the IR spectra of **I**–**VI**, the $\nu_{as,\,s}(NH_3^+)$ stretching modes occur at 3550–3400 and 3050–3020 cm^{-1}, respectively, and the $\delta_{as,s}(NH_3^+)$ bending modes occur at 1690–1500 cm^{-1}. In the Raman spectra of salt **III**, the $\nu(NH_3^+)$ modes mainly give rise to low-intensity lines at 3230–2775 cm^{-1}; and the $\delta(NH_3^+)$ vibrations show low activity in the Raman spectra and have not been considered.

It is known [19] that the vibrational spectra of the T_d-symmetric isolated SO_4^{2-} anion exhibit four normal modes at 983 cm^{-1} (ν_1, Raman-active), 450 cm^{-1} (ν_2,

Table 1. Wave numbers (cm^{-1}) of the principal absorption peaks in the IR spectra of **I–VI***

Compound	**I**	**II**	**III**	**IV**	**V**	**VI**
$\nu_{as,s}(NH_3^+)$	3433 m 3031vs	3550–3400 m.br. 3020 vs	3180 sh	3020 m	3020 m	3421m.br. 3061 w 3050 s
$\nu_{as,s}(NH_3^+)$, $\nu(CH)$			2981 m 2870 m 2775 w 2715 w 2580 w			2936 s 2877 s 2592 s
$\delta_{as,s}(NH_3^+)$	1569 s 1504 s	1590 s 1505 s	1690m 1615sh 1565 sh 1510 m	1613 w 1531 m 1506 sh	1612 w 1525 s 1507 w	1608 m 1587 m 1572 sh
$\nu_1(SO_4^{2-}) \equiv \nu_s(SO)$	954 m	965 sh	980 m	966 sh	965 m	972 sh
$\nu_1(HSO_4^-) \equiv \nu_s(SO)$	–	–	–	1048 s	1046 s	–
$\nu_2(SO_4^{2-}) \equiv \delta_s(OSO)$	448 m	440 w	510 w 455 m 425 w	442 w	441 w	490 w 477 w 444 w
$\nu_2(HSO_4^-) \equiv \gamma(S\text{-}OH)$	–	–	–			–
$\nu_3(SO_4^{2-}) \equiv \nu_{as}(SO)$	1120 vs	1119 vs	1118 vs	1117 vs	1115 vs	1133 vs 1115 sh 1088 s 1058 s
$\nu_3(HSO_4^-) \equiv \nu_{as}(SO) + \delta(OH)$	–	–	–	1209 m 1197 sh	1210 m 1197 m	–
$\nu_4(SO_4^{2-}) \equiv \delta_{as}(OSO)$	669 w 660 w 618 vs	683 w 671 w 618 s	690 sh 617 s 605 sh	683 w 671 w 617 s	683 w 671 w 618 s	617 m 606 sh
$\nu_4(HSO_4^-) \equiv \nu_s(SO) + \gamma(OH)$	–	–	–	882 w	880 w	–

* Types of vibrations: ν is stretching, δ is bending, γ is out-of-plane (libration).

Raman-active), 1105 cm^{-1} (ν_3, IR- and Raman-active), and 611 cm^{-1} (ν_4, IR- and Raman-active). In the IR spectra of **I** and **II**, all ν_1–ν_4 modes are active: the $\nu_{as,s}(SO_4^{2-})$ stretching vibrations (ν_3, ν_1) give rise to 1120, 954 cm^{-1} and 1119, 965 cm^{-1} bands, respectively, while the $\delta_{as,s}(SO_4^{2-})$ bending vibrations (ν_4, ν_2) occur as 669, 660, 618, 448 cm^{-1} and 683, 671, 618, 440 cm^{-1} bands, respectively. The observed differences in the anion characteristics in the spectra of **I** and **II** and in the tabulated spectrum [19] (in particular, the appearance of the ν_1 and ν_2 modes) reflect the decrease in the SO_4^{2-} anion symmetry in salts **I** and **II** caused by perturbing effect of the NH···O hydrogen bonds.

A similar trend can be followed in the spectra of sulfates **III** and **VI**. In the IR spectrum of salt **III**, in which the anion has C_2 symmetry according to X-ray diffraction data [22], the ν_1 stretching vibrations are manifested at 980 cm^{-1} (in the Raman spectrum, this vibration is active at 975 cm^{-1}) and the ν_3 vibrations occur at 1118 cm^{-1} (IR) and 1125 cm^{-1} (Raman). The anion bending vibrations ν_4 are manifested as a triplet at 617 cm^{-1} (a strong band), 690, and 605 cm^{-1} (shoulders). Generally, the number and positions of the identified modes of the SO_4^{2-} anion incorporated in salts **I**, **II**, **III**, and **VI** attest to a decrease in the anion symmetry as compared with the perfect T_d structure; this is in line with X-ray diffraction data [22].

Table 2. Results of thermogravimetric analysis of onium sulfates and sulfates–hydrogen sulfates

Compound	Decomposition stage	Temperature, °C			Mass loss, %
		T_{ons}	T_{end}	T_{max}	
$[C_3H_7NH_3]_2SO_4$ (**I**)	First (endo)	260	320	300	71.2
	Second (endo)	320	350	330	
	Third (exo)	500	560	540	93.3
$[C_4H_9NH_3]_2SO_4$ (**II**)	First (endo)	270	340	330	75.6
	Second (exo)	480	580	550	94.3
$[(CH_3)_3CNH_3]_2SO_4$ (**III**)	First (endo)	125	140	130	0
	Second (endo)	275	340	310	67.4
	Third (endo)	340	400	370	
	Fourth (exo)	500	570	520	74.1
$[C_7H_{15}NH_3]_3SO_4(HSO_4)$ (**IV**)	First (endo)	290	350	330	78.7
	Second (exo)	480	620	530	93.3
$[C_8H_{17}NH_3]_3SO_4(HSO_4)$ (**V**)	First (endo)	290	330	320	76.6
	Second (exo)	470	550	510	96.7
	Third (exo)	550	630	610	
$[C_6H_5NH_3]_2SO_4$ (**VI**)	First (endo)	210	260	250	65.3
	Second (endo)	380	415	410	
	Third (exo)	510	650	600	95.0

In comparison with the sulfate anion, an isolated hydrogen sulfate anion has an additional proton, which is bound to one hydrogen; on going from SO_4^{2-} to HSO_4^-, the symmetry changes from T_d to C_{3v} [19, 23, 25]. In the mixed salts with the $(SO_4HSO_4)^{3-}$ anions [23, 25], two tetrahedral SO_4^{2-} groups are linked by a strong hydrogen bond: in the case of symmetric H-bond, the ν_1 SO_4^{2-} vibrations give rise to a singlet absorption band, whereas asymmetric H-bond induces splitting of the ν_1 mode into two components. The doublet nature of the ν_1 band in the IR spectra of mixed salts **IV** and **V** (1048, 966 cm^{-1} and 1046, 965 cm^{-1}; the splitting is 82 and 81 cm^{-1}, respectively) attests to asymmetric H-bond in the salt crystals; by analogy with [24, 25], the lower-wavelength component of ν_1 corresponds to the hydrogen sulfate anion.

The results of thermogravimetric analysis of compounds **I**–**VI** are presented in Table 2. According to these data, the relative thermal stability of the sulfate salts decreases in the following order, depending on the nature of the onium cation:

n-$C_8H_{17}NH_3^+$ (10.65 + 3.09) ≈ n-$C_7H_{15}NH_3^+$ (10.67 + 2.57) > $(CH_3)_3CNH_3^+$ (10.68 + 0.40) > n-$C_4H_9NH_3^+$ (10.77 + 0.86) > n-$C_3H_7NH_3^+$ (10.60 + 0.48) > $C_6H_5NH_3^+$ (4.63 + 0.90).

This sequence of onium cations (except for the *tert*-butylammonium cation) correlates with the basicity and lipophilicity function (the values in parentheses are $pK_a + \log P_{ow}$ [26, 27]). Note that previously, an attempt was made to relate the solubility and melting points of compounds to their lipophilicity ($\log P_{ow}$) and pK_a [28].

The endothermic effects observed for salts **I**, **II**, and **IV**–**VI** (the first and second for sulfates **I** and **VI** and the first for sulfate **II** and hydrogen disulfates **IV** and **V**) correspond to elimination of $[RNH_3]HSO_4$ (and its decomposition products; 1 mol for sulfates and 2 mol for hydrogen disulfates) to the gas phase, which is typical of ammonium sulfate [29]. The exothermic effects in the thermograms of salts **I**–**VI** correspond to the oxidative destruction of 1 mol of the amine (and its decomposition products). Thermolysis of compound **III** is accompanied by a phase transition (endotherm at 125–140°C without mass loss) [22] followed by elimination of 1 mol of $(CH_3)_3CNH_2$ (its decomposition products), 1 mol of SO_3, and 1 mol of H_2O (100% H_2SO_4 boils with decomposition at 275°C [30]), and the products of t-BA oxidative destruction (exotherm).

As has already been noted, the reactions in SO_2–RNH_2–H_2O–O_2 solutions (R = CH_3 [14], $(HOCH_2)_3C$ [12], $C_6H_5CH_2$ [13], $C_6H_5C(CH_3)H$ [13], $(CH_3)_3C$ [11], and C_6H_5) give the corresponding onium sulfates, which are stabilized, according to X-ray diffraction data, by NH···O type hydrogen bond systems [11–14, 21, 22]. Evidently, the onium sulfates $[n\text{-}C_3H_7NH_3]_2SO_4$ and $[n\text{-}C_4H_9NH_3]_2SO_4$ characterized in this study have a similar structure.

Thus, the transformation in the given reaction systems can be generally represented by the scheme

$$2SO_2 + 4RNH_2 + 2H_2O + O_2 \rightarrow 2(RNH_3)_2SO_4, \quad (1)$$

and this redox transformation obviously proceeds when the reaction products are kept in an unprotected atmosphere.

Apparently, the first stage of sulfur(IV) oxide chemisorption by aqueous solutions of alkylamines and aniline, like in the case of ethanolamines [31], affords onium sulfites (reaction (2)), which are then converted to hydrogen sulfites (reaction (3)) and metabisulfites (reaction (4)):

$$SO_2 + H_2O + 2RNH_2 \rightarrow (RNH_3)_2SO_3, \quad (2)$$

$$SO_2 + (RNH_3)_2SO_3 + H_2O \rightleftarrows 2(RNH_3)HSO_3, \quad (3)$$

$$SO_2 + (RNH_3)_2SO_3 \rightleftarrows (RNH_3)_2S_2O_5. \quad (4)$$

Evidently, during keeping the reaction mixture SO_2–L–H_2O in air, the onium sulfites are oxidized to sulfates:

$$(RNH_3)_2SO_3 + O_2 \rightarrow 2(RNH_3)_2SO_4. \quad (5)$$

The oxidation processes for methylammonium, *n*-propylammonium, *n*-butylammonium, *tert*-butylammonium, and aniline hydrogen sulfites (metabisulfites) are described by the following equations:

$$\begin{gathered} 4(RNH_3)HSO_3 + O_2 \\ \rightarrow 2(RNH_3)_2SO_4 + 2SO_2\uparrow + 2H_2O, \end{gathered} \quad (6)$$

$$\begin{gathered} 2(RNH_3)_2S_2O_5 + O_2 \\ \rightarrow 2(RNH_3)_2SO_4 + 2SO_2\uparrow . \end{gathered} \quad (7)$$

In the case of reaction systems SO_2–L–H_2O–O_2 involving $n\text{-}C_7H_{15}NH_2$ and $n\text{-}C_8H_{17}NH_2$, which are considerably more lipophilic than other alkylamines, the autooxidation proceeds according to the equation

$$\begin{gathered} 3(RNH_3)HSO_3 + O_2 \\ \rightarrow (RNH_3)_3SO_4(HSO_4) + SO_2\uparrow + H_2O, \end{gathered} \quad (8)$$

i.e., *n*-heptylammonium and *n*-octylammonium cations stabilize the hydrogen disulfates $[(SO_4)H(SO_4)]^{3-}$ and, hence, reaction (8) occurs instead of reactions (6) and (7).

Considering the results obtained in this study, together with published data, one can state that the reactions in the SO_2–L–H_2O–O_2 systems lead to the formation of the salts of sulfur oxoanions in the case of organic bases L with the following basicity constants: aniline ($pK_a = 4.63$), ethanolamines [8, 9, 32] ($7.76 \le pK_a \le 9.85$), benzylamines [13] ($8.52 \le pK_a \le 9.84$), alkylamines ($10.60 \le pK_a \le 10.77$), aminoguanidine [10] ($pK_a = 11.04$). In view of the fact that the reactions with less basic 2,2'-bipyridine ($pK_a = 4.34$) give van der Waals clathrates [33], the value $pK_a = 4.63$ can be taken, in the first approximation, as the lower boundary of the ligand basicity to provide the formation of salts in these reaction systems.

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