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Sulfite esters as products of the interaction in sulfur dioxide – alkanol – tris(hydroxymethyl)aminomethane systems

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ABSTRACT

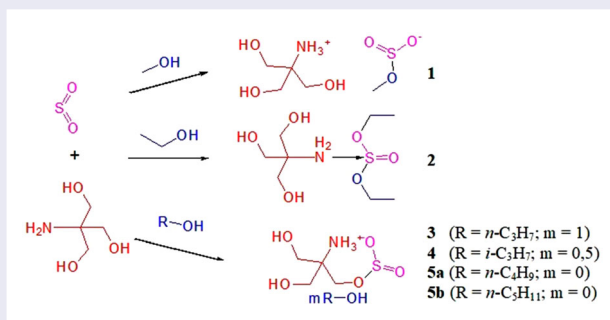
The reaction products formed in the $\text{SO}_2 - \text{ROH} - (\text{HOCH}_2)_3\text{CNH}_2$ (where $\text{R} = \text{CH}_3, \text{C}_2\text{H}_5, n\text{-C}_3\text{H}_7, i\text{-C}_3\text{H}_7, n\text{-C}_4\text{H}_9$ and $n\text{-C}_5\text{H}_{11}$) systems were isolated and identified as: binary salt – ammonium *O*-methylsulfite $[(\text{HOCH}_2)_3\text{CNH}_3]^+ [\text{CH}_3\text{OSO}_2]^-$ (**1**); 1/1 charge transfer complex between diethyl sulfite (**2**) and tris(hydroxymethyl)aminomethane by S-N binding; polymorphs of zwitterionic internal salt – *O*-sulfite tris(hydroxymethyl)methylammonium (at $\text{R} = n\text{-C}_4\text{H}_9$ and $n\text{-C}_5\text{H}_{11}$); and its solvates with *n*-propanol 1/1 (**3**) and isopropanol 2/1. The structures of all synthesized products were confirmed by FT-IR, Raman, ^1H NMR and ^{13}C NMR spectrometry, and the structure of **1** and **3** was characterized by crystal X-ray diffraction studies as well.

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Sulfur dioxide; alkanol; tris(hydroxymethyl)amino-methane; sulfite esters; zwitterionic internal salt



1. Introduction

Organic *O*-sulfites are known as effective primary [1] and secondary [2] antioxidants; hydrocarbons and polymers thermo-oxidative decomposition inhibitors [3]; analytical

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reagents in the Karl Fischer reaction [4]; insecticides [5] and biocides [6]; precursors in organic synthesis [7, 8]. Ionic liquids-based sulfite esters are perspective carbon dioxides absorbents [9]. Dialkylsulfites solutions of electrolyte have found their use in lithium-ion cells [10–12].

O-alkylsulfites synthesis is carried out by the reactions of alkanols with thionyl chloride in an organic base (pyridine) medium [13]. Instead of thionyl chloride, a solution of sulfur dioxide in pyridine in the presence of Br₂ or I₂ can be used. Sulfur dioxide and 1-hexanol were found [14] to chemically react with organic amines (Am: 1,8-diazabicyclo[5.4.0]undec-7-ene, 1,1,3,3-tetramethylguanidine and *N*-ethyl-*N,N*-diisopropylamine) to form binary SO₂-binding organic ionic liquids – ammonium *O*-hexylsulfite salts [AmH]⁺ [C₆H₁₃OSO₂]⁻. Alkanolamine (*N,N*-dibutylundecanolamine) reversibly reacts with SO₂ to form organic zwitterionic liquid – estersalt (C₄H₉)₂N⁺H(CH₂)₁₁OSO₂⁻ [15].

In this paper, we describe method of synthesis, structural study, spectral properties of products resulting from the reaction in the SO₂ – ROH – (HOCH₂)₃CNH₂ (where R – CH₃, C₂H₅, *n*-C₃H₇, *i*-C₃H₇, *n*-C₄H₉ and *n*-C₅H₁₁) systems: [(HOCH₂)₃CNH₃]⁺[CH₃OSO₂]⁻ (**1**), (C₂H₅O)₂SO·(HOCH₂)₃CNH₂ (**2**), H₃N⁺C(CH₂OH)₂CH₂OSO₂⁻·*n*-C₃H₇OH (**3**), H₃N⁺C(CH₂OH)₂CH₂OSO₂⁻·0,5*i*-C₃H₇OH (**4**), H₃N⁺C(CH₂OH)₂CH₂OSO₂⁻ polymorphic forms **I** (**5a**), and **II** (**5b**), respectively.

2. Results and discussions

2.1. Synthesis and characterization

We started our investigation with a one-pot, three-component reaction of sulfur dioxide, tris(hydroxymethyl)aminomethane (Tris), and methanol. The corresponding ammonium *O*-alkylsulfite **1** was obtained in ~100% isolated yield. Next, alkanol screening of the model reaction was performed (Scheme 1). It was found that an internal zwitterionic salt is formed with the most lipophilic *n*-C₄H₉OH and *n*-C₅H₁₁OH in the series of alkanols studied – *O*-alkylsulfite **5** (polymorphic forms **I** (**5a**) and **II** (**5b**), correspondingly). In case of *n*-C₃H₇OH – solvate of *O*-alkylsulfite **5** with 1:1 molecular ratio (**3**), with *i*-C₃H₇OH – solvate with 2:1 (**4**) molecular ratio. In the reaction medium SO₂ – Tris – C₂H₅OH a charge-transfer complex (CTC) **2** is formed with composition (C₂H₅O)₂SO·NH₂C(CH₂OH)₃ on account of S←N interaction. Methanol in a similar case forms a binary salt – ammonium *O*-alkyl sulfite **1** with the formula [(HOCH₂)₃CNH₃]⁺ [CH₃OSO₂]⁻.

The compounds obtained above have been structurally characterized including FT-IR, Raman, ¹H, ¹³C NMR spectra (Figure S1–S18) together with element analysis.

The assignment of stretching vibrations was carried out using the data [16, 17]. FT-IR and Raman technique has been used to differentiate polymorphs of ampholytes [18, 19]. The presence of the band ν(OH) was observed in the FT-IR spectra of ammonium *O*-alkyl sulfite **1** (Figure S1), solvates **3** (Figure S3) and **4** (Figure S4), with maxima at 3430, 3360 and 3359 cm⁻¹, respectively, unlike CTC **2** (Figure S2) and internal salt **5b** (Figure S6); the band ν(OH) has high-frequency shift (by 60 cm⁻¹) comparing spectrum of Tris [17] with spectrum of **1** and low frequency shift (by ~10 cm⁻¹) – comparing with spectra of **3** and **4**, due to the redistribution of H-bond systems.

Like ammonium salts $(\text{TrisH})_2\text{SO}_4$ [17] and $(\text{TrisH})_2\text{SO}_3$ [20], broad structured bands of stretching vibrations of NH_3^+ -groups involved in H-bonding are observed in the FT-IR spectra of compounds **1**, **3–5** (Figure S1, S3–S5) in the region of $3290\text{--}2345\text{ cm}^{-1}$; vibrations $\nu_{\text{as,s}}(\text{NH}_2)$ appear in the spectrum of CTC **2** (Figure S2) as bands of medium and low intensity at $2933\text{--}2325\text{ cm}^{-1}$.

In the FT-IR spectra of compounds **1**, **3–5**, the $\delta_{\text{as,s}}(\text{NH}_3^+)$ vibrations appear as: two comparatively intense bands at 1632 and 1552 cm^{-1} (**1**); one band of average intensity at 1629 cm^{-1} (**3**); two low and medium intense bands at 1620 and 1602 cm^{-1} (**4**); two medium and low intense bands at 1631 and 1607 cm^{-1} (**5a**); two medium and low intense bands at 1635 and 1613 cm^{-1} (**5b**). Vibrations $\delta_{\text{as,s}}(\text{NH}_2)$ – two bands of medium intensity at 1612 and 1520 cm^{-1} in the spectrum of CTC **2**.

In the Raman spectra of compounds **1**, **3–5**, the $\nu(\text{NH}_3^+)$ vibrations correspond to the bands in the range $3236\text{--}2675\text{ cm}^{-1}$ (Figure S7, S9–S12); $\nu(\text{NH}_2)$ vibrations in spectrum **2** (Figure S8), the $\nu_{\text{as,s}}(\text{NH}_2)$ vibrations appear as three bands of strong and weak intensity in the region of $2930\text{--}2753\text{ cm}^{-1}$. Vibrations $\delta_{\text{as,s}}(\text{NH}_3^+)$, which are inactive in the Raman spectra **3–5a**, are fixed in the form of lines of low intensity in the region $1633\text{--}1601\text{ cm}^{-1}$; vibrations $\delta_{\text{as,s}}(\text{NH}_2)$ in spectrum **2** are low intense bands at 1617 cm^{-1} .

Vibration bands $\nu(\text{CN})$ of ammonium cations in FT-IR and Raman spectra **1**, **3–5** were identified in the range of $1068\text{--}1030$ and $1097\text{--}1023\text{ cm}^{-1}$, respectively; the low-frequency shift of this band during the transition from the Tris spectrum [17] to spectrum **2** by 50 cm^{-1} confirms the redistribution of the electron density in the amine molecule and the weakening of the C–N bond due to the formation of a new $\text{S}\leftarrow\text{N}$.

The absorption bands with maxima in the region $1245\text{--}1218\text{ cm}^{-1}$ (in the FT-IR spectra of salts **1**, **3–5**), $1247\text{--}1220\text{ cm}^{-1}$ (in the Raman spectra **2**, **4**, **5b**) are attributed to stretching vibrations $\nu(\text{S}=\text{O})$. Stretching vibrations $\nu(\text{S}=\text{O})$ appear in the spectra of compounds **1–5** at $672\text{--}554\text{ cm}^{-1}$ (FT-IR), $650\text{--}554\text{ cm}^{-1}$ (Raman); vibrating $\gamma(\text{S}=\text{O})$ – $714\text{--}682\text{ cm}^{-1}$ (FT-IR spectra **1**, **2**, **4**, **5a**, **5b**); $757\text{--}656\text{ cm}^{-1}$ (Raman **1–3**, **5a**, **5b**). In the FT-IR and Raman spectra of compounds **1–5**, bending vibrations $\delta(\text{OSO})$ and $\delta(\text{S}=\text{O})$ appear in the region $432\text{--}403$ and $427\text{--}404\text{ cm}^{-1}$, correspondingly.

2.2. X-ray crystal structures

The main crystal data and structure refinement results are presented in Table 1.

Structure **1** (Figure 1) contains tris(hydroxymethyl)methylammonium ions as cations and monomethylsulfite anions. This anion is structurally characterized firstly in the present work. The bond lengths and angles in the structure **1** are given in Table 2. It can be seen that these quantities have the usual values for such compounds. The crystal packing formed by this compound (Figure 2) is layered, the layers occupy the (001) planes. The characteristics of hydrogen bonds which are formed in the layer are given in Table 3. It is interesting to note that the N(1) atom of the cation forms four hydrogen bonds, the direction of three of which coincides with the directions of the N–H bonds, and the fourth hydrogen bond is almost collinear with the C(1)–N(1) bond (Table 3). No hydrogen bonds are observed between the layers.

Structure **3** contains neutral molecules in zwitterionic state which is formed due to intramolecular proton jump from $-\text{SO}_2\text{H}$ to $-\text{NH}_2$ group. Similarly to the structure **1** information on the molecular geometry in **3** is given in Tables 4, 5 and

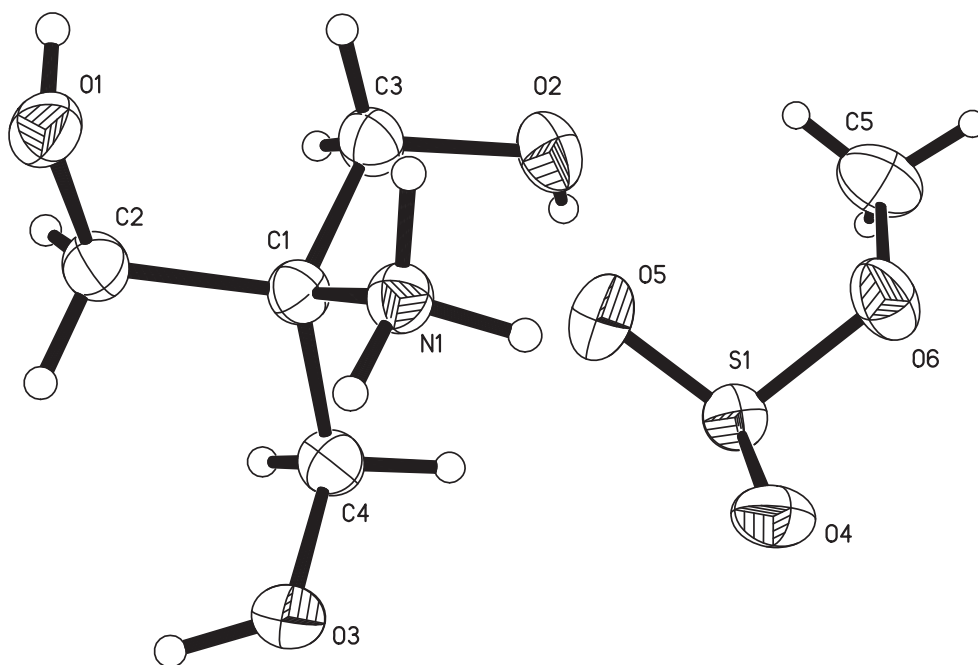


Figure 1. Atomic numbering scheme and the thermal ellipsoids (probability level of 50%) for the compound **1**.

Table 1. Crystallographic data and structure refinement results for compound **1, 3**.

Identification code	1	3
Deposition code	CCDC 2255568	CCDC 2255569
Empirical formula	C ₅ H ₁₅ NO ₆ S	C ₄ H ₁₁ NO ₅ S
Formula weight	217.24	185.20
Temperature (K)		293(2)
Wavelength MoK α		0.71073
Crystal system	Monoclinic	Monoclinic
Space group	P2 ₁ /c	P2 ₁ /c
Unit cell dimensions: <i>a</i> (Å)	8.2170(5)	6.9493(6)
<i>b</i> (Å)	6.2840(5)	9.8955(9)
<i>c</i> (Å)	18.4669(9)	11.1160(10)
α (°)	90	90
β (°)	95.299(5)	93.604(8)
γ (°)	90	90
Volume <i>V</i> (Å ³)	949.47(11)	762.90(12)
<i>Z</i>	4	4
<i>D</i> _{calc} (g cm ⁻³)	1.520	1.612
μ (mm ⁻¹)	0.343	0.402
<i>F</i> ₀₀₀	464	392
Crystal size (mm)	0.400 × 0.030 × 0.005	0.230 × 0.070 × 0.060
θ range (°)	3.69–31.78	3.587–28.388
Transmission <i>T</i> _{min} / <i>T</i> _{max}	0.875/0.998	0.913/0.976
Completeness, %	98.6	98.9
Reflections collected/unique	5296/2874 (<i>R</i> _{int} = 0.0565)	5117/3137 (<i>R</i> _{int} = 0.0796)
Data / restraints / parameters	2874/0/139	3137/2/137
Goodness-of-fit on <i>F</i> ²	0.971	0.937
<i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0524, <i>wR</i> ² = 0.0926	<i>R</i> ₁ = 0.0644, <i>wR</i> ² = 0.1790
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0872, <i>wR</i> ² = 0.1104	<i>R</i> ₁ = 0.0891, <i>wR</i> ² = 0.1901
$\Delta\rho_{\min}/\rho_{\max}$, e Å ⁻³	−0.376/0.367	−0.423/0.436

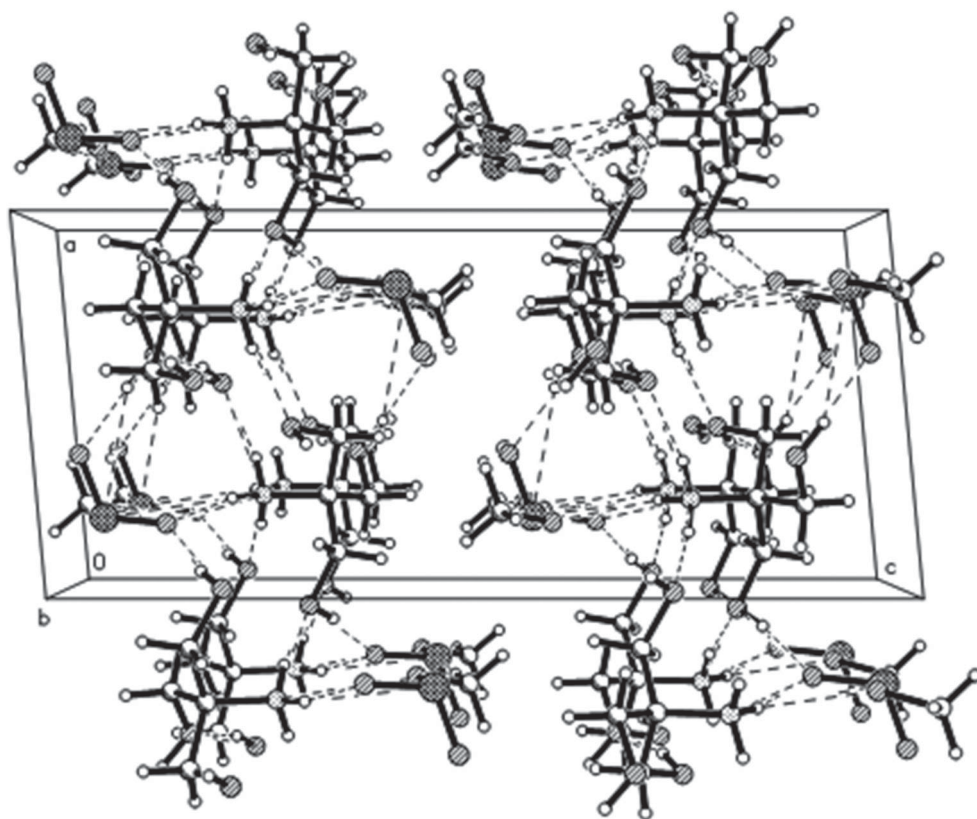


Figure 2. Crystal packing and hydrogen bonds in the compound **1**.

Table 2. Selected bond lengths and angles in the structure **1**.

S(1)–O(5)	1.4604(14)	O(2)–C(3)	1.413(2)	C(1)–C(4)	1.522(3)
S(1)–O(4)	1.4848(14)	O(3)–C(4)	1.422(2)	C(1)–C(3)	1.529(3)
S(1)–O(6)	1.6524(16)	O(6)–C(5)	1.428(2)	C(1)–C(2)	1.527(3)
O(1)–C(2)	1.418(2)	N(1)–C(1)	1.507(2)		
O(5)–S(1)–O(4)	110.37(9)	N(1)–C(1)–C(2)	108.15(15)		
O(5)–S(1)–O(6)	103.77(9)	C(4)–C(1)–C(2)	110.52(15)		
O(4)–S(1)–O(6)	96.54(8)	C(3)–C(1)–C(2)	110.98(14)		
C(5)–O(6)–S(1)	115.20(14)	O(1)–C(2)–C(1)	113.89(15)		
N(1)–C(1)–C(4)	107.98(13)	O(2)–C(3)–C(1)	112.65(14)		
N(1)–C(1)–C(3)	108.03(15)	O(3)–C(4)–C(1)	111.24(16)		
C(4)–C(1)–C(3)	111.05(15)				

Figure 3 and 4. As opposed to the structure **1** the H-bonds network, which is observed in **3**, is three-dimensional. According to the X-ray diffraction data, the structure of $\text{H}_3\text{N}^+\text{C}(\text{CH}_2\text{OH})_2\text{CH}_2\text{OSO}_2^-$ in compound **3**; obviously, the *n*-C₃H₇OH solvate molecules therein are X-ray amorphous.

Rietveld analyses of powder diffraction patterns of **1** and **3**, in which the above described structures of these compounds were used as initial data for the Rietveld calculations have showed that both compounds are the single phases with no impurity lines (Figure 5).

Table 3. Parameters of hydrogen bonds D–H···A for the compound **1**.

Bond D–H···A	Distances, Å				Symmetry transformation for the atom A
	d(D–H)	d(H···A)	d(D···A)	<DHA,°	
O(1)–H(1)···O(4)	0.82(3)	1.88(3)	2.6998(19)	171(3)	$x - 1, y, z$
O(2)–H(2)···O(5)	0.92(3)	1.77(3)	2.685(2)	178(2)	
O(3)–H(3)···O(2)	0.94(3)	1.75(3)	2.670(2)	165(3)	$x, y + 1, z$
N(1)–H(1A)···O(3)	0.94(3)	1.93(2)	2.8423(19)	163(2)	$-x + 1, y - 1/2, -z + 1/2$
N(1)–H(1B)···O(4)	0.98(3)	1.91(3)	2.879(2)	167(2)	$-x + 1, y + 1/2, -z + 1/2$
N(1)–H(1B)···O(6)	0.98(3)	2.49(3)	2.974(2)	110.4(19)	$-x + 1, y + 1/2, -z + 1/2$
N(1)–H(1C)···O(1)	0.92(3)	2.03(3)	2.937(2)	169(2)	$-x, y - 1/2, -z + 1/2$

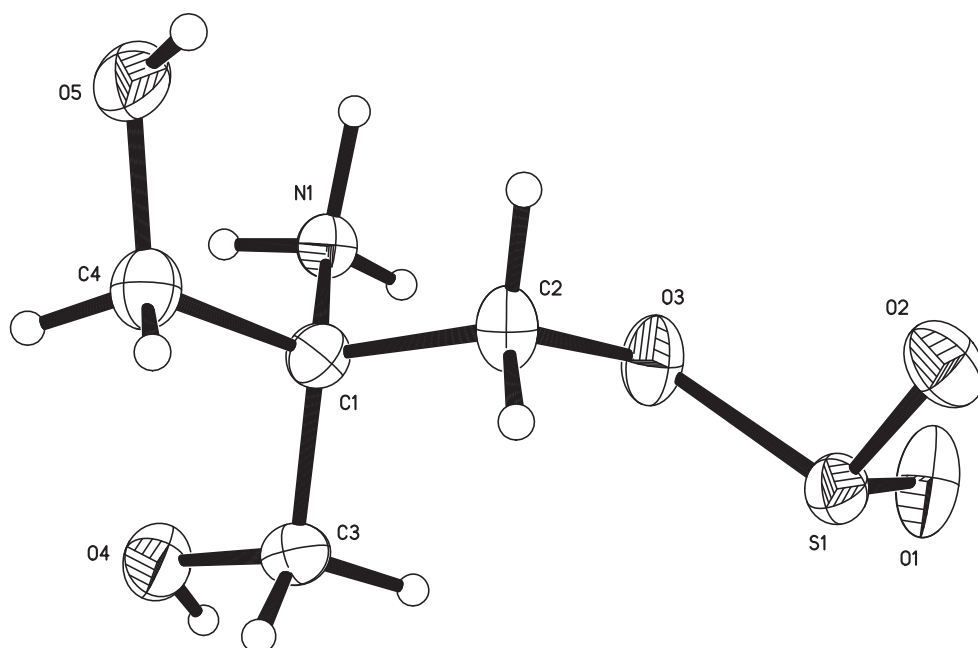
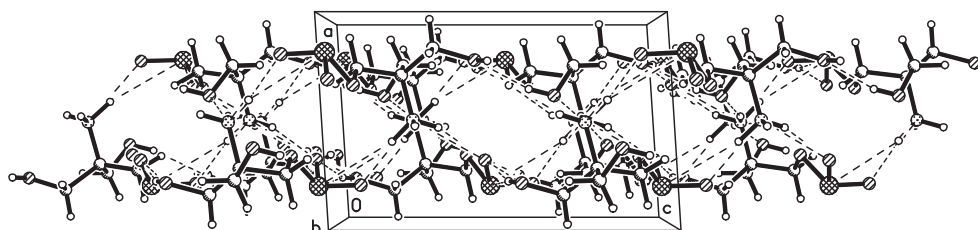
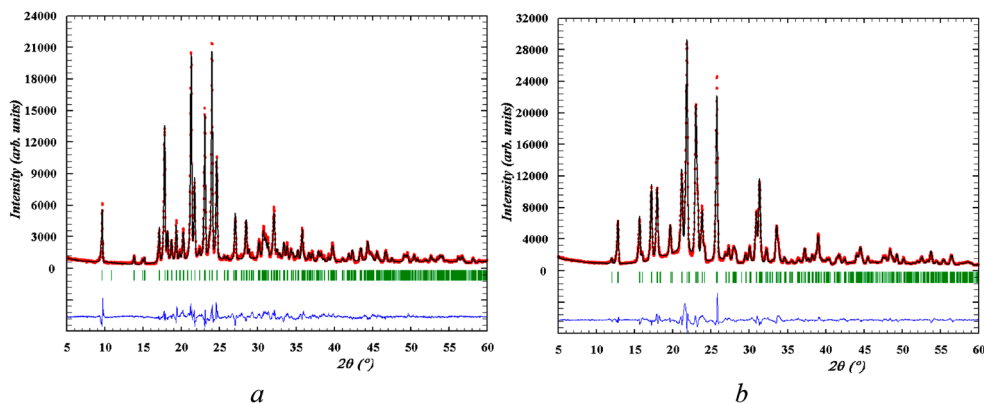
**Figure 3.** Atomic numbering scheme and the thermal ellipsoids (probability level of 50%) for the compound **3**.**Figure 4.** Crystal packing and hydrogen bonds in the compound **3**.

Table 4. Selected bond lengths and angles in the structure **3**.

S(1)–O(2)	1.464(3)	O(4)–C(3)	1.424(5)	C(1)–C(4)	1.506(5)
S(1)–O(1)	1.475(3)	O(5)–C(4)	1.417(5)	C(1)–C(3)	1.524(5)
S(1)–O(3)	1.672(3)	N(1)–C(1)	1.498(5)	C(1)–C(2)	1.525(5)
O(3)–C(2)	1.442(5)				
O(2)–S(1)–O(1)	110.19(17)	N(1)–C(1)–C(2)	107.9(3)		
O(2)–S(1)–O(3)	103.32(15)	C(4)–C(1)–C(2)	110.7(3)		
O(1)–S(1)–O(3)	97.25(16)	C(3)–C(1)–C(2)	110.4(3)		
C(2)–O(3)–S(1)	113.2(2)	O(3)–C(2)–C(1)	110.1(3)		
N(1)–C(1)–C(4)	108.6(3)	O(4)–C(3)–C(1)	110.2(3)		
N(1)–C(1)–C(3)	108.4(3)	O(5)–C(4)–C(1)	113.0(3)		
C(4)–C(1)–C(3)	110.8(3)				

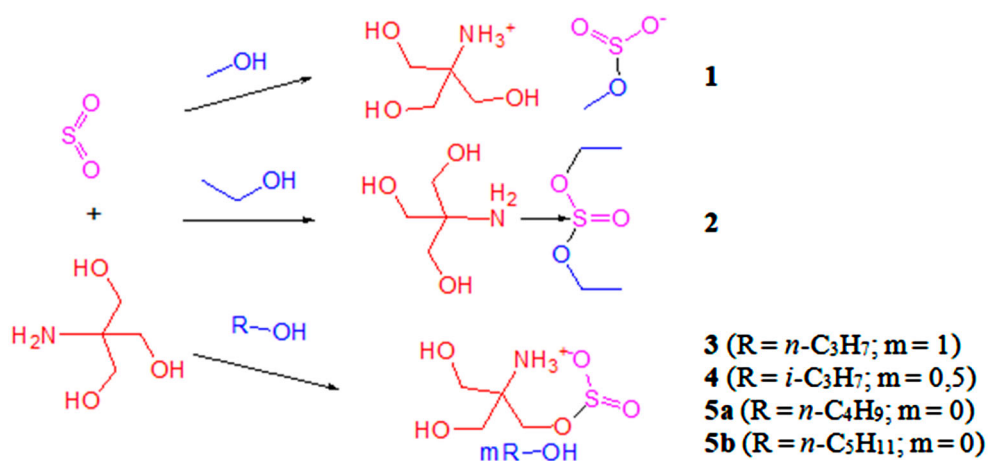
Table 5. Parameters of hydrogen bonds D–H···A for the compound **3**.

Bond D–H···A	Distances, Å			\angle DHA, °	Symmetry transformation for the atom A
	d(D–H)	d(H···A)	D(D···A)		
O(4)–H(4)···O(1)	0.847(13)	1.95(2)	2.770(4)	163(5)	$x, -y + 1/2, z + 1/2$
O(5)–H(5)···O(4)	0.847(13)	2.17(2)	2.963(4)	157(5)	$x, -y + 3/2, z - 1/2$
N(1)–H(1A)···O(2)	1.05(4)	1.77(4)	2.792(4)	165(3)	$-x + 1, -y + 1, -z + 1$
N(1)–H(1B)···O(1)	0.94(4)	1.89(4)	2.786(4)	158(3)	$-x + 1, y + 1/2, -z + 3/2$
N(1)–H(1C)···O(5)	0.83(4)	2.02(4)	2.823(4)	161(4)	$-x + 1, y - 1/2, -z + 3/2$
N(1)–H(1A)···S(1)	1.05(4)	2.91(4)	3.937(3)	165(3)	$-x + 1, -y + 1, -z + 1$

**Figure 5.** Rietveld refinement results for **1** (a) and **3** (b). Experimental pattern is shown as red circles; calculated pattern is shown as black line; vertical bars show the Bragg position for lines; bottom line shows the difference between experimental and calculated intensities in every point.

3. Conclusion

In summary, an efficient and fast procedure has been described in this study for the synthesis of novel *O*-alkylsulfites through one-pot three-component assembly of sulfur dioxide, Tris and alkanol under oxygen- and water-free conditions. In the proposed synthesis scheme, alkanols are both the reaction medium and the reagents: the nature (lipophilicity) of the used alkanol has a differentiating effect on the composition and structure of the reaction products (Scheme 1). There is a change in the composition of the reaction product from ammonium methylsulfite (solvent – methanol) to a complex with charge transfer



Scheme 1. Synthesis of sulfite esters 1–5.

between diethylsulfite and Tris (solvent – ethanol), alcohol solvates of various compositions of zwitterionic salts of tris(hydroxymethyl)methylammonium *O*-sulfite (solvent – *n*-propanol, isopropanol) and unsolvated tris(hydroxymethyl)methylammonium *O*-sulfite zwitterionic polymorphs (solvent – *n*-butanol, *n*-pentanol). Fast synthetic procedure, fast reaction times, carrying out the reactions without any hazardous reagents are the main advantages of presented research.

4. Experimental

4.1. Materials and methods

Tris and alkanols were obtained from Merck. All the alkanols were dried and degassed before the use. Solvents were transferred by a plastic syringe. We used commercial sulfur dioxide after preliminary purification and drying according to the procedure described in [21]. All reactions were run under an atmosphere of argon. Flasks were oven-dried and cooled under a stream of argon.

Elemental analysis for C, H and N were performed using a Carlo Erba Elemental Analyzer Model 1106; sulfur content, by Schöniger's method. FT-IR spectra were measured on a Spectrum BX II FT-IR System (PerkinElmer) instrument in the 4000–350 cm^{-1} range. Raman spectra were recorded on a DXR Raman Microscope «Thermo Scientific». ^1H and ^{13}C NMR spectra were obtained on Mercury 400 (Varian) spectrometer (^1H NMR at 400 MHz and ^{13}C NMR at 100 MHz) in DMSO-d_6 using TMS as internal standard. Chemical shifts (δ) are given in ppm.

4.2. Typical procedure for the synthesis

Tris(hydroxymethyl)methylammonium methylsulphite (1). A degassed with argon Tris solution (0.05 mol) in 30 ml of methanol was poured into a cell filled (Figure S19), and SO_2 was passed through it in the bubbling mode at a rate of 50 $\text{ml}\cdot\text{min}^{-1}$ for 45 min ($t = 20^\circ\text{C}$). The resulting solution was kept for 1 d at room temperature in a hermetically

sealed cell; was purged with argon and evacuated to a complete excess of sulfur dioxide and methanol. The isolated white crystalline product **1** (10.86 g) was not further purified. FT-IR (KBr) (ν , cm^{-1}): 3430 s br, 3230 s br, 3062 s br, 2951 s br, 2821, 2720 sh [$\nu(\text{OH})$, $\nu(\text{NH}_3^+)$]; 2717 m, 2571 sh, 2123 w [$\nu(\text{NH}_3^+)$, $\nu(\text{OH} \cdots \text{OSO}_2)$]; 1634, 1552 s [$\delta_{\text{as,s}}(\text{NH}_3^+)$]; 1463 m, 1407 w [$\delta(\text{CNH})$]; 1296 w [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_3^+)$, $\nu(\text{CC})$]; 1245 w [$\nu(\text{S}=\text{O})$]; 1206 w [$\omega(\text{NH}_3^+)$]; 1151 w [$\omega(\text{CH}_2)$]; 1068, 1038 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 945, 916, 897, 873 m [$\rho(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 711 w [$\gamma(\text{S}=\text{O})$]; 622, 599 w [$\nu(\text{S}-\text{O})$]; 522, 486 w [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 432, 420 w [$\delta(\text{OSO})$]; 410 w [$\delta(\text{S}=\text{O})$]. Raman (ν , cm^{-1}): 3005 m, 2970, 2948, 2937 s, 2899 m [$\nu(\text{OH})$, $\nu(\text{NH}_3^+)$]; 2828, 2767 w [$\nu(\text{NH}_3^+)$, $\nu(\text{OH} \cdots \text{OSO}_2)$]; 1462 s, [$\delta(\text{CNH})$]; 1311, 1296 w [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_3^+)$, $\nu(\text{CC})$]; 1189, 1154 w [$\omega(\text{CH}_2)$]; 1097, 1068, 1054, 1040 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 917, 904 m [$\rho(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 760, 656 s [$\gamma(\text{S}=\text{O})$]; 605 m, 557 s [$\nu(\text{S}-\text{O})$]; 458 m [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 424 s [$\delta(\text{OSO})$]. ^1H NMR (400 MHz, DMSO- d_6 , 2.5 ppm): δ 7.73 (2.61H, br s, NH_3), 5.28 (2.46H, br s, OH), 3.50(6H, s, CH_2 of Tris), 3.13 (3H, s, CH_3 of Me), ppm. For methanol – 3.165 in pure DMSO- d_6 , ppm. ^{13}C NMR (100 MHz, DMSO- d_6 39.5 ppm): δ 60.70 (C of Tris), 58.75 (CH_2 of Tris), 48.01 (CH_3 of Me), ppm. For methanol 48.76 in pure DMSO- d_6 , ppm. Anal. Calcd. for $\text{C}_8\text{H}_{24}\text{N}_2\text{O}_9\text{S}$: C, 27.64; H, 6.96; N, 6.45; S, 14.76. Found: C, 27.91; H, 7.08; N, 6.53; S, 14.87.

Diethylsulfite – tris(hydroxymethyl)aminomethane 1/1 (2) was prepared similarly from 0.05 mol of Tris in 30 ml of ethanol. Yield 13.01 g, colorless liquid. FT-IR (ν , cm^{-1}): 2933, 2881 m [$\nu(\text{OH})$, $\nu(\text{NH}_2)$]; 2348, 2325 w [$\nu(\text{NH}_2)$, $\nu(\text{OH} \cdots \text{OSO}_2)$]; 1612, 1522 m [$\delta_{\text{as,s}}(\text{NH}_2)$]; 1464 m [$\delta(\text{CNH})$]; 1382 m, 1287 w [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_2)$, $\nu(\text{CC})$]; 1192 w [$\omega(\text{NH}_2)$]; 1041 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 1001 s [$\nu(\text{CO})$]; 881 s [$\rho(\text{NH}_2)$, $\rho(\text{CH}_2)$]; 682 s [$\gamma(\text{S}-\text{O})$]; 620 s [$\nu(\text{S}-\text{O})$]; 530 m [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 427 m [$\delta(\text{OSO})$]; 408 m [$\delta(\text{S}=\text{O})$]. Raman (ν , cm^{-1}): 2930, 2889 s [$\nu(\text{OH})$, $\nu(\text{NH}_2)$]; 2753 w [$\nu(\text{NH}_2)$, $\nu(\text{OH} \cdots \text{OSO}_2)$]; 1617 w [$\delta_{\text{as,s}}(\text{NH}_2)$]; 1464 m [$\delta(\text{CNH})$]; 1389, 1278 w [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_2)$, $\nu(\text{CC})$]; 1247 [$\nu(\text{S}=\text{O})$]; 1054 m [$\nu(\text{CO})$, $\nu(\text{CN})$]; 1006 m [$\nu(\text{CO})$]; 882 m [$\rho(\text{NH}_2)$, $\rho(\text{CH}_2)$]; 684 w [$\gamma(\text{S}-\text{O})$]; 594 sh, 574 w [$\nu(\text{S}-\text{O})$]; 420 w [$\delta(\text{OSO})$]. ^1H NMR (400 MHz, DMSO- d_6): δ 6.01 (6H, br s, NH_2 , OH), 3.5 (6H, s, CH_2 of Tris), 3.43 (4H, m, CH_2 of Et), 0.999 (6H, t, $^3J_{\text{HH}} = 7.4$ Hz, CH_3); for ethanol: CH_2 – 3.44, CH_3 1.045 in pure DMSO- d_6 ppm. ^{13}C NMR (100 MHz, DMSO- d_6 , 39.5 ppm): δ 61.76 (C of Tris), 59.95 (CH_2 of Tris), 56.82 (CH_2 of Et), 18.45 (CH_3), ppm; for ethanol: CH_2 –56.53, CH_3 –18.55 in pure DMSO- d_6 , ppm. Anal. Calcd. for $\text{C}_8\text{H}_{22}\text{NO}_6\text{S}$: C 36.91; H 8.52; N 5.38; S 12.32. Found: C 36.83; H 8.31; N 5.49; S 12.56.

Solvate O-sulfite tris(hydroxymethyl)methylammonium – *n*-propanol 1/1 (3) was prepared similarly from 0.5 g of Tris in 20 ml of *n*-propanol. Yield 1.01 g, white crystals. FT-IR (KBr) (ν , cm^{-1}): 3230–3195 w br, 3083 m, 2963 sh, 2932, 2877 s [$\nu(\text{OH})$, $\nu(\text{NH}_3^+)$]; 2598 sh. [$\nu(\text{NH}_3^+)$, $\nu(\text{OH} \cdots \text{OSO}_2)$]; 1629 m, 1617, 1611 sh [$\delta_{\text{as,s}}(\text{NH}_3^+)$]; 1524 m, 1503 sh, 1460 m, 1421, 1385 w [$\delta(\text{CNH})$]; 1296 w [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_3^+)$, $\nu(\text{CC})$]; 1235 w [$\nu(\text{S}=\text{O})$]; 1200 w [$\omega(\text{NH}_3^+)$]; 1043 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 985 sh [$\nu(\text{CC})$]; 949 s, 906 m [$\rho(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 783 w [$\delta(\text{COH})$, $\delta(\text{CCC})$, $t(\text{CO})$]; 672, 617 sh, 597 s [$\nu(\text{S}-\text{O})$]; 521 w, 480 s [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 427 w [$\delta(\text{OSO})$]; 413 w [$\delta(\text{S}=\text{O})$]. Raman (ν , cm^{-1}): 2938, 2913

sh, 2879 vs $[\nu(\text{OH}), \nu(\text{NH}_3^+)]$; 2741 w $[\nu(\text{NH}_3^+), \nu(\text{OH} \cdots \text{OSO}_2)]$; 1633 w $[\delta_{\text{as,s}}(\text{NH}_3^+)]$; 1457 s $[\delta(\text{CNH})]$; 1298 m, 1275 w $[\delta(\text{COH}), \text{t}(\text{CH}_2), \rho(\text{NH}_3^+), \nu(\text{CC})]$; 1248 w $[\nu(\text{S}=\text{O})]$; 1200 w $[\omega(\text{NH}_3^+)]$; 1056, 1035 sh $[\nu(\text{CO}), \nu(\text{CN})]$; 972 m $[\nu(\text{CC})]$; 880 m, 860 s $[\rho(\text{NH}_3^+), \rho(\text{CH}_2)]$; 763 s $[\delta(\text{COH}), \delta(\text{CCC}), \text{t}(\text{CO})]$; 702 w $[\gamma(\text{S}=\text{O})]$; 608, 573 w $[\nu(\text{S}-\text{O})]$; 463 w $[\delta(\text{NCC}), \delta(\text{CCO})]$; 403 w $[\delta(\text{S}=\text{O})]$. ^1H NMR (400 MHz, DMSO- d_6): δ 7.67 (2.1H, br s, NH_3), 5.15 (2.6H, br s, OH), 3.5 (6H, s, CH_2 of Tris), 3.32 (2H, t, $^3J_{\text{HH}} = 6.7$ Hz, CH_2 -OH of *n*-PrOH), 1.38 (2H, m, CH_2 of PrOH), 0.79 (3H, t, $^3J_{\text{HH}} = 7.5$ Hz, CH_3) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ 62.5 (CH_2 -OH of *n*-PrOH), 60.97 (C of Tris), 59.00 (CH_2 of Tris), 25.59 (CH_2 of *n*-PrOH), 10.65 (CH_3). Anal. Calcd. for $\text{C}_7\text{H}_{19}\text{NO}_6\text{S}$: C 34.28, H 7.81, N 5.71, S 13.07. Found: C 34.16, H 7.94, N 5.77, S 13.22.

Solvate O-sulfite tris(hydroxymethyl)methylammonium – isopropanol 2/1 (4) was prepared similarly from 0.5 g of Tris in 20 ml of isopropanol. Yield 0.88 g, white crystals. FT-IR (KBr) (ν , cm^{-1}): 3360 s, 3283 m, 3240 w, 3178 sh, 3032, 2974 w, 2918 sh, 2891 s, 2834 m, $[\nu(\text{OH}), \nu(\text{NH}_3^+)]$; 2162 w $[\nu(\text{NH}_3^+), \nu(\text{OH} \cdots \text{OSO}_2)]$; 1620 w, 1602 m $[\delta_{\text{as,s}}(\text{NH}_3^+)]$; 1539 sh, 1504, 1471 s, 1419 m $[\delta(\text{CNH})]$; 1340 m, 1280 w $[\delta(\text{COH}), \text{t}(\text{CH}_2), \rho(\text{NH}_3^+), \nu(\text{CC})]$; 1238, 1218 w $[\nu(\text{S}=\text{O})]$; 1172, 1143 w $[\omega(\text{CH}_2)]$; 1066 s, 1051 w, 1029 s, 974 vs $[\nu(\text{CO}), \nu(\text{CN})]$; 937 s, 898 m $[\rho(\text{NH}_3^+), \rho(\text{CH}_2)]$; 797 m, 700 vs $[\gamma(\text{S}=\text{O})]$; 630 s, 572 vs $[\nu(\text{S}-\text{O})]$; 527, 491 s $[\delta(\text{NCC}), \delta(\text{CCO})]$; 426 w $[\delta(\text{OSO})]$; 410 s $[\delta(\text{S}=\text{O})]$. Raman (ν , cm^{-1}): 2984, 2962, 2951, 2938, 2892 s $[\nu(\text{OH}), \nu(\text{NH}_3^+)]$; 2791, 2771, 2675 w $[\nu(\text{NH}_3^+), \nu(\text{OH} \cdots \text{OSO}_2)]$; 1622, 1601 w $[\delta_{\text{as,s}}(\text{NH}_3^+)]$; 1513 w, 1472 m, 1427, 1374 w $[\delta(\text{CNH})]$; 1330, 1278 w $[\delta(\text{COH}), \text{t}(\text{CH}_2), \rho(\text{NH}_3^+), \nu(\text{CC})]$; 1237 w $[\nu(\text{S}=\text{O})]$; 1200 $[\omega(\text{NH}_3^+)]$; 1075 s, 1056 m, 1038 vs $[\nu(\text{CO}), \nu(\text{CN})]$; 936 m, 895 s $[\rho(\text{NH}_3^+), \rho(\text{CH}_2)]$; 797 s $[\gamma(\text{S}=\text{O})]$; 635 w, 570 m $[\nu(\text{S}-\text{O})]$; 528 w, 498 s $[\delta(\text{NCC}), \delta(\text{CCO})]$; 421 m $[\delta(\text{OSO})]$; 370 m $[\delta(\text{S}=\text{O})]$. ^1H NMR (400 MHz, DMSO- d_6): δ 7.72 (2.0H, br s, NH_3), 5.45 (2.5H, br s, OH), 3.75 (0.5H, m, CH of *i*-PrOH), 3.49 (6H, s, CH_2 of Tris), 1.00 (3H, d, $^3J_{\text{HH}} = 6.2$ Hz, CH_3 of *i*-PrOH) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ 62.30 (CH of *i*-PrOH), 60.99 (C of Tris), 59.07 (CH_2 of Tris), 25.55 (CH_3 of *i*-PrOH) ppm. Anal. Calcd. for $\text{C}_{11}\text{H}_{30}\text{N}_2\text{O}_{11}\text{S}_2$: C 30.69, H 7.02, N 6.51, S 14.90. Found: C 30.78, H 7.12, N 6.58, S 14.81.

O-sulfite tris(hydroxymethyl)methylammonium form I (5a) was prepared similarly from 0.5 g of Tris in 25 ml of *n*-butanol. Yield 1.07 g, white crystals. FT-IR (KBr) (ν , cm^{-1}): 3359, 3282 m, 3239–3216 w br, 3007 w, 2957 m, 2927, 2874 s $[\nu(\text{OH}), \nu(\text{NH}_3^+)]$; 2707, 2605, 2345 w $[\nu(\text{NH}_3^+)]$; 1631 m, 1607 w $[\delta_{\text{as,s}}(\text{NH}_3^+)]$; 1563 w, 1548, 1502, 1465, 1425 m, 1379 w $[\delta(\text{CNH})]$; 1339, 1295, 1287 w $[\delta(\text{COH}), \text{t}(\text{CH}_2), \rho(\text{NH}_3^+), \nu(\text{CC})]$; 1218 m $[\nu(\text{S}=\text{O})]$; 1196 w $[\omega(\text{NH}_3^+)]$; 1144 m $[\omega(\text{CH}_2)]$; 1064, 1050, 1031 vs $[\nu(\text{CO}), \nu(\text{CN})]$; 991, 976 sh $[\nu(\text{CC})]$; 956 sh, 941 vs, 874 s $[\rho(\text{NH}_3^+), \rho(\text{CH}_2)]$; 6796 m, 700 vs, 684 s $[\gamma(\text{S}=\text{O})]$; 621 sh, 600, 572 s $[\nu(\text{S}-\text{O})]$; 526 s, 490 vs $[\delta(\text{NCC}), \delta(\text{CCO})]$; 424 m $[\delta(\text{OSO})]$; 410 w $[\delta(\text{S}=\text{O})]$. Raman (ν , cm^{-1}): 3236 w, 2985, 2952, 2943, 2930 m, 2906, 2901, 2875 w $[\nu(\text{OH}), \nu(\text{NH}_3^+)]$; 1624, 1604 w $[\delta_{\text{as,s}}(\text{NH}_3^+)]$; 1475, 1458 sh $[\delta(\text{CNH})]$; 1343 s, 1283 w $[\delta(\text{COH}), \text{t}(\text{CH}_2), \rho(\text{NH}_3^+), \nu(\text{CC})]$; 1204, 1198 w $[\omega(\text{NH}_3^+)]$; 1080 s, 1065 m, 1042 s, 1028 m $[\nu(\text{CO}), \nu(\text{CN})]$;

967 [$\nu(\text{CC})$]; 941 w, 900 s [$\nu(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 757, 737 m [$\delta(\text{COH})$, $\delta(\text{CCC})$, $t(\text{CO})$]; 703 w [$\gamma(\text{S}=\text{O})$]; 634, 607, 595, 554 w [$\nu(\text{S}-\text{O})$]; 503 m [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 427 m [$\delta(\text{OSO})$]. ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 7.64 (2.5H, br s, NH_3), 5.25 (1.6 H, br s, OH), 3.49 (6H, s, CH_2 of Tris), 1.34 (0.02H, m, $\text{CH}_2\text{-CH}_2\text{OH}$ of BtOH), 1.23 (0.02H, m, CH_2- of BtOH), 0.79 (0.03H, m, CH_3- of BtOH). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ 60.78 (C of Tris), 59.01 (CH_2 of Tris). Anal. Calcd. for $\text{C}_4\text{H}_{11}\text{NO}_5\text{S}$: C 25.94, H 5.99, N 7.56, S 17.31. Found: C 25.78, H 5.83, N 7.63, S 17.02.

O-sulfite tris(hydroxymethyl)methylammonium form II (5b) was prepared similarly from 0.5 g of Tris in 25 ml of *n*-pentanol. Yield 0.76 g, white crystals. FT-IR (KBr) (ν , cm^{-1}): 3237, 3169, 3075, 2924, 2822 m [$\nu(\text{OH})$, $\nu(\text{NH}_3^+)$]; 2720, 2558 w [$\nu(\text{NH}_3^+)$]; 1635 m, 1613 w [$\delta_{\text{as,s}}(\text{NH}_3^+)$]; 1551 w, 1530 m, 1459, 1417 w [$\delta(\text{CNH})$]; 1296, 1287 m [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_3^+)$, $\nu(\text{CC})$]; 1218 s [$\nu(\text{S}=\text{O})$]; 1200 sh [$\omega(\text{NH}_3^+)$]; 1130 s [$\omega(\text{CH}_2)$]; 1060 vs, 1032 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 989, 984 w [$\nu(\text{CC})$]; 956 vs., 910 w, 872 m, [$\rho(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 714 sh [$\gamma(\text{S}=\text{O})$]; 650, 600, 554 s [$\nu(\text{S}-\text{O})$]; 503 s, 465 w [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 428 vs [$\delta(\text{OSO})$]; 417 s [$\delta(\text{S}=\text{O})$]. Raman (ν , cm^{-1}): 3251, 3236, 3078 w, 2985, 2952, 2943, 2930 s, 2906 m [$\nu(\text{OH})$, $\nu(\text{NH}_3^+)$]; 2763 w [$\nu(\text{NH}_3^+)$, $\nu(\text{OH}\cdots\text{OSO}_2)$]; 1463, 1450 m [$\delta(\text{CNH})$]; 1300 m [$\delta(\text{COH})$, $t(\text{CH}_2)$, $\rho(\text{NH}_3^+)$, $\nu(\text{CC})$]; 1220 w [$\nu(\text{S}=\text{O})$]; 1193 w [$\omega(\text{CH}_2)$]; 1127 w [$\omega(\text{CH}_2)$]; 1086, 1068 m, 1034 s [$\nu(\text{CO})$, $\nu(\text{CN})$]; 952 s, 909 w, 893 w [$\rho(\text{NH}_3^+)$, $\rho(\text{CH}_2)$]; 757 m, 737 w [$\gamma(\text{S}=\text{O})$]; 650 s, 607 m, 594, 554 w [$\nu(\text{S}-\text{O})$]; 523 w [$\delta(\text{NCC})$, $\delta(\text{CCO})$]; 427 vs [$\delta(\text{OSO})$]. ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 7.74 (2.5H, br s, NH_3), 5.32 (1.36 H, br s, OH), 3.5 (6H, s, CH_2 of Tris). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ 60.27 (C of Tris); 58.61 (CH_2 of Tris) ppm. Anal. Calcd. for $\text{C}_4\text{H}_{11}\text{NO}_5\text{S}$: C 25.94, H 5.99, N 7.56, S 17.31. Found: C 25.78, H 5.83, N 7.63, S 17.02.

4.3. X-ray crystal structure determination and powder diffractometry

The structure of compounds **1**, **3** was determined by single-crystal X-ray diffraction analysis («Xcalibur-3» diffractometer (Oxford Diffraction Ltd.), graphite monochromator, CCD detector «Sapphire-3»). The programs SHELXT, SHELXL and WinGX were used for the structure solution, refinement and analysis of **1**, **3** [22–24]. H-atoms were located in the Fourier D-maps and refined using the riding model (H-atoms involved in the H-bonding were refined isotropically).

X-ray powder study was carried out with «Siemens D500» diffractometer ($\text{CuK}\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$), Bragg–Brentano $\theta-2\theta$ goniometer, graphite monochromator on the counter arm, $5^\circ \leq 2\theta \leq 60^\circ$, $\Delta(2\theta) = 0.02^\circ$). Obtained powder patterns were developed using FullProf program [25].

Disclosure statement

No potential conflict of interest was reported by the author(s).

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