Crystal structure of hexaaquamagnesium sulfate bis(*N*,*N*,*N*-trimethylaminoethanoic acid) trihydrate, $[Mg(H_2O)_6]SO_4 \cdot 2(CH_3)_3NCH_2COO \cdot 3H_2O$

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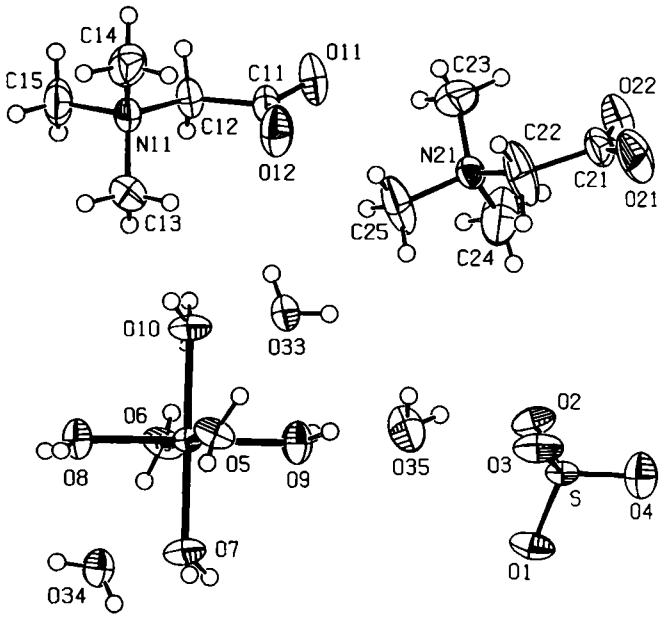

Abstract

$C_{10}H_{40}MgN_2O_{17}S$, monoclinic, $P12_1/c1$ (no. 14), $a = 6.3729(8)$ Å, $b = 15.905(1)$ Å, $c = 24.994(2)$ Å, $\beta = 97.521(7)°$, $V = 2511.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.123$, $T = 293$ K.

Source of material

Small block-shaped colorless crystals were obtained after a few months of slow evaporation from an aqueous solution containing betaine and magnesium sulfate in the ratio 2:1. A suitable crystal was separated and its quality was checked by photographic methods before data collection.

Experimental details

All water molecules H atoms were located on a Fourier difference map and refined isotropically with thermal parameters constrained to be proportional to those of the parent atom. The hydrogen atoms involved in C—H bonds were refined as riding using appropriate AFIX instructions with SHELXL-97 defaults [1]. Examination of the crystal structure with PLATON [2] showed that there are no solvent-accessible voids in the crystal lattice.

Discussion

Betaine compounds are of importance in biological systems as components of complex lipids and as transmethylating agents. Pure betaine is an inner salt (zwitterion) where the proton of the carboxylic group has been transferred to the amino group. It may be combined with a variety of acids to form 1:1 and 2:1 complexes. These undergo a variety of transitions exhibiting antiferroelectric and ferroelastic behaviour as well as phases with commensurate and incommensurate superstructures [3-6]. The present work represents an attempt to find more betaine compounds with interesting physical properties. This is the first crystal structure being reported of a co-crystal of betaine with a magnesium salt.

The neutral zwitterionic states of the two betaine molecules were confirmed from the bond distances within the carboxylate groups. The betaine molecules have a dipolar form with a mono-positive amino group and a mono-negative carboxylate group. The structure contains a sulfate ion counter balancing the $[Mg(H_2O)_6]^{2+}$ complex. The conformation of the neutral betaine molecules are similar to zwitterionic forms found in other betaine compounds. The main carboxy skeleton is planar within 0.05 Å and 0.03 Å, for O11-O12-C11-C12-N11 and O21-O22-C21-C22-N21, respectively. Magnesium ions show a high ability to coordinate to small molecules and in all reported structures water molecules are found in the coordination sphere of the cation [7]. In the title compound the magnesium ion is coordinated to six independent water molecules with Mg—O distances from 2.043(1) Å to 2.080(1) Å. The environment of the hexaaquamagnesium cation is almost perfectly octahedral. The betaine molecules do not coordinate directly to the cation. The sulfate anion has a slightly distorted tetrahedral geometry with one of the S—O distances smaller than the others. Moreover, one of the O—S—O angles is more deviated from the ideal value (108.49(8)° - 109.59(9)°, 111.18(9)°). The present crystal structure is a network with a large number of strong hydrogen bonds between inorganic layers of hexaaquamagnesium, sulfate and water molecules and alternating double layers of betaine and free water molecules parallel to the (001) planes. The water molecules mediate all the intermolecular interactions shielding the magnesium ion from direct electrostatic interactions.

Table 1. Data collection and handling.

Crystal:	transparent colorless block, size 0.45 × 0.48 × 0.49 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.28 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$ (profile data)
$2\theta_{max}$:	59.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7881, 7283
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4907
$N(param)_{refined}$:	340
Programs:	SHELXL-97 [1], PLATON [2], SHELXS-97 [8], ORTEP-II [9]

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Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(5A)	4e	0.837(3)	0.039(1)	0.2322(9)	0.045
H(5B)	4e	0.828(3)	0.110(1)	0.2002(9)	0.045
H(6A)	4e	0.162(3)	0.200(1)	0.2793(9)	0.047
H(6B)	4e	0.175(4)	0.126(1)	0.3076(9)	0.047
H(7A)	4e	0.509(4)	-0.044(1)	0.2716(8)	0.045
H(7B)	4e	0.310(4)	-0.025(1)	0.2648(8)	0.045
H(8A)	4e	0.610(4)	0.132(1)	0.362(1)	0.047
H(8B)	4e	0.782(4)	0.104(1)	0.3435(9)	0.047
H(9A)	4e	0.379(4)	0.102(2)	0.154(1)	0.053
H(9B)	4e	0.221(4)	0.128(1)	0.173(1)	0.053
H(10A)	4e	0.486(3)	0.283(1)	0.2400(8)	0.046
H(10B)	4e	0.696(4)	0.259(1)	0.2454(8)	0.046
H(12A)	4e	0.6711	0.3239	0.4525	0.049
H(12B)	4e	0.6806	0.4220	0.4537	0.049
H(13A)	4e	1.1144	0.4479	0.4233	0.081
H(13B)	4e	0.9110	0.4977	0.3996	0.081
H(13C)	4e	1.0421	0.4485	0.3609	0.081
H(14A)	4e	1.0364	0.2917	0.3614	0.090
H(14B)	4e	0.9070	0.2450	0.4016	0.090
H(14C)	4e	1.1129	0.2946	0.4236	0.090

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15A)	4e	0.7371	0.3734	0.3218	0.089
H(15B)	4e	0.5949	0.4204	0.3588	0.089
H(15C)	4e	0.5982	0.3219	0.3576	0.089
H(22A)	4e	0.4909	0.2699	0.0196	0.093
H(22B)	4e	0.6339	0.3488	0.0318	0.093
H(23A)	4e	0.2985	0.4754	0.0425	0.145
H(23B)	4e	0.5115	0.4654	0.0811	0.145
H(23C)	4e	0.2949	0.4591	0.1042	0.145
H(24A)	4e	0.0679	0.3375	0.0813	0.138
H(24B)	4e	0.1375	0.2704	0.0412	0.138
H(24C)	4e	0.0748	0.3613	0.0207	0.138
H(25A)	4e	0.5935	0.3336	0.1270	0.114
H(25B)	4e	0.4585	0.2534	0.1099	0.114
H(25C)	4e	0.3651	0.3245	0.1432	0.114
H(33A)	4e	0.949(3)	0.228(2)	0.1565(9)	0.046
H(33B)	4e	0.915(3)	0.170(1)	0.1189(9)	0.046
H(34A)	4e	0.067(4)	0.005(2)	0.3497(9)	0.048
H(34B)	4e	0.129(4)	0.061(1)	0.385(1)	0.048
H(35A)	4e	0.344(4)	0.097(2)	0.056(1)	0.072
H(35B)	4e	0.561(5)	0.099(2)	0.068(1)	0.072

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mg	4e	0.50410(7)	0.11738(3)	0.25751(2)	0.0190(2)	0.0215(2)	0.0292(2)	0.0010(2)	0.0040(2)	0.0011(2)
O(5)	4e	0.7700(2)	0.08103(8)	0.22570(5)	0.0318(6)	0.0343(6)	0.0497(7)	0.0111(5)	0.0197(5)	0.0123(5)
O(6)	4e	0.2353(2)	0.15656(8)	0.28646(5)	0.0296(6)	0.0373(6)	0.0537(7)	0.0112(5)	0.0186(5)	0.0141(6)
O(7)	4e	0.4330(2)	-0.00533(7)	0.27557(6)	0.0261(6)	0.0228(5)	0.0647(8)	0.0000(4)	0.0086(5)	0.0022(5)
O(8)	4e	0.6698(2)	0.12781(9)	0.33435(5)	0.0335(6)	0.0546(8)	0.0288(6)	0.0086(6)	0.0024(5)	-0.0045(5)
O(9)	4e	0.3343(2)	0.1047(1)	0.18106(5)	0.0348(6)	0.0627(8)	0.0336(6)	0.0077(6)	-0.0021(5)	-0.0051(6)
O(10)	4e	0.5733(2)	0.23956(7)	0.23935(6)	0.0254(6)	0.0231(5)	0.0658(8)	0.0004(4)	0.0075(5)	0.0069(5)
O(11)	4e	0.8202(2)	0.3569(1)	0.54587(5)	0.0580(9)	0.088(1)	0.0255(6)	0.0159(8)	0.0056(6)	0.0068(6)
O(12)	4e	1.1097(2)	0.3755(1)	0.50604(6)	0.0455(8)	0.099(1)	0.0386(7)	-0.0035(8)	-0.0043(6)	0.0004(8)
N(11)	4e	0.8622(2)	0.37138(9)	0.40122(5)	0.0487(8)	0.0401(8)	0.0244(6)	-0.0006(6)	0.0032(6)	-0.0005(5)
C(11)	4e	0.9177(3)	0.3682(1)	0.50601(7)	0.047(1)	0.047(1)	0.0248(7)	0.0065(8)	-0.0008(7)	0.0002(7)
C(12)	4e	0.7661(3)	0.3716(1)	0.45308(7)	0.0401(9)	0.056(1)	0.0249(7)	0.0010(8)	0.0020(6)	0.0040(7)
C(13)	4e	0.9943(4)	0.4483(1)	0.39576(9)	0.065(1)	0.051(1)	0.046(1)	-0.010(1)	0.0088(9)	0.0085(9)
C(14)	4e	0.9912(4)	0.2937(1)	0.39651(9)	0.078(2)	0.051(1)	0.050(1)	0.014(1)	0.005(1)	-0.0124(9)
C(15)	4e	0.6817(4)	0.3718(2)	0.35573(8)	0.065(1)	0.080(2)	0.0290(9)	-0.006(1)	-0.0080(9)	0.0019(9)
O(21)	4e	0.5384(3)	0.3311(1)	-0.06894(6)	0.098(1)	0.094(1)	0.0388(8)	0.017(1)	0.0361(8)	0.0128(8)
O(22)	4e	0.2825(3)	0.4125(1)	-0.04616(6)	0.068(1)	0.086(1)	0.0365(7)	0.0128(9)	0.0037(7)	0.0184(8)
N(21)	4e	0.3628(2)	0.35600(9)	0.06391(5)	0.0396(7)	0.0380(8)	0.0270(6)	-0.0004(6)	0.0087(5)	0.0023(5)
C(21)	4e	0.4277(3)	0.3614(1)	-0.03717(7)	0.052(1)	0.058(1)	0.0279(8)	-0.0040(9)	0.0104(7)	0.0089(8)
C(22)	4e	0.4894(4)	0.3308(2)	0.02063(8)	0.081(2)	0.124(2)	0.034(1)	0.057(2)	0.030(1)	0.025(1)
C(23)	4e	0.3673(7)	0.4468(2)	0.0738(1)	0.184(4)	0.046(1)	0.061(2)	-0.028(2)	0.019(2)	-0.011(1)
C(24)	4e	0.1416(4)	0.3290(2)	0.0507(1)	0.069(2)	0.162(3)	0.047(1)	-0.060(2)	0.011(1)	-0.006(2)
C(25)	4e	0.4532(5)	0.3130(2)	0.11573(9)	0.098(2)	0.099(2)	0.033(1)	0.039(2)	0.020(1)	0.022(1)
S	4e	0.05974(5)	0.36731(2)	0.22865(2)	0.0197(2)	0.0194(1)	0.0386(2)	0.0004(1)	0.0075(1)	0.0004(1)
O(1)	4e	-0.0162(2)	0.29239(7)	0.25531(6)	0.0266(6)	0.0278(6)	0.0715(9)	-0.0016(5)	0.0099(5)	0.0132(6)
O(2)	4e	-0.0217(2)	0.44294(7)	0.25311(6)	0.0262(5)	0.0265(6)	0.0655(8)	0.0027(4)	0.0091(5)	-0.0103(5)
O(3)	4e	0.2925(2)	0.36815(7)	0.23776(7)	0.0194(5)	0.0274(6)	0.088(1)	0.0003(4)	0.0136(6)	0.0019(6)
O(4)	4e	-0.0186(3)	0.3650(1)	0.17126(6)	0.064(1)	0.072(1)	0.0370(7)	0.0048(8)	0.0058(6)	-0.0034(7)
O(33)	4e	0.9481(2)	0.17857(9)	0.15079(5)	0.0433(7)	0.0453(7)	0.0267(6)	0.0003(6)	0.0032(5)	-0.0003(5)
O(34)	4e	0.0593(2)	0.05176(9)	0.35754(5)	0.0368(7)	0.0473(7)	0.0355(6)	0.0023(6)	0.0003(5)	-0.0039(6)
O(35)	4e	0.4506(3)	0.0755(1)	0.07846(6)	0.0469(8)	0.089(1)	0.0440(8)	-0.0051(8)	0.0038(6)	0.0099(8)

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