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1,3-Diammonio-2-propanol Sulphate Monohydrate

BY RAIKKO KIVEKÄS

University of Helsinki, Department of Inorganic Chemistry, Vuorikatu 20, *SF*-00100 *Helsinki* 10, *Finland*

AND JUSSI VALKONEN

Helsinki University of Technology, Department of Chemistry, SF-02150 *Espoo* 15, *Finland*

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Abstract. $[NH_3CH_2CH(OH)CH_2NH_3]SO_4.H_2O$, $C_3H_{12}N_2O^{2+}.SO_4^{2-}.H_2O$, monoclinic, $P2_1/c$, a = 8·460 (3), b = 5·750 (2), c = 19·780 (6) Å, β = 113·61 (2)°, V = 881·7 (5) Å^3, Z = 4, D_m = 1·53 (flotation), D_c = 1·55 Mg m^{-3}, μ(Mo $K\alpha$) = 0·349 mm^{-1}, R = 3·6% for 1247 reflections. The structure contains divalent aminoalcohol cations, sulphate anions and water molecules joined together by hydrogen bonds.

Introduction. Several X-ray analyses of complexes formed by 1,3-diamino-2-propanol and Cu^{II} salts have been reported in recent years (Bertrand, Marabella & Vanderveer, 1977; Pajunen & Kivekäs, 1979; Kivekäs, 1977, and references therein). In these complexes the diaminoalcohol molecule is coordinated in several different ways to the central atom, or it may be present in the structure as a non-coordinated divalent cation. Extending our studies on 1,3-diamino-2-propanol in solids we have determined the crystal structure of the title compound.

The present compound was prepared by adding dropwise 0·1 mol 1,3-diamino-2-propanol in ethanol to 30 ml of solution containing 0·2 mol sulphuric acid. During this operation the solution was cooled in ice (273 K) and stirred vigorously. The precipitate was separated and washed with ethanol. The recrystallization was carried out in a water–ethanol (1:3) solution.

Preliminary photographs indicated monoclinic symmetry and systematically absent reflections showed the space group to be $P2_1/c$. A computer-controlled Syntex $P2_1$ (Fortran version) four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation was used to determine cell parameters and collect intensities. The crystal size was 0·2 × 0·3 × 0·3 mm and the temperature was 298 K. Cell dimensions were calculated on the basis of 25 reflections in the range $3 < 2\theta < 21°$ and refined by least squares. Intensities were collected in the interval $5 < 2\theta < 50°$ with the ω technique and scan speed 2–20° min^{-1}. Of the 1596 unique reflections measured, 1247 had $I > 3\sigma(I)$. The data were corrected for Lorentz and polarization effects, but not for absorption.

The position of the S atom was determined from Harker sections and the positions of the other non-hydrogen atoms from a subsequent Fourier synthesis. After anisotropic least-squares refinement R was 0·05 and the difference map calculated at this point revealed the positions of all H atoms. In the final block-diagonal refinement with anisotropic thermal parameters for the non-hydrogen atoms and isotropic for H, R decreased to 0·036 (R_w = 0·043).* The function minimized was $w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2_{F_o}$.

Scattering factors for S, O, N and C were from Cromer & Mann (1968) and those for H from Stewart, Davidson & Simpson (1965). The calculations were carried out on a Univac 1108 computer with XRAY 76 (Stewart, 1976).

Discussion. Positional parameters are listed in Tables 1 and 2, bond lengths and angles in Tables 3 and 4. The contents of one unit cell are shown in Fig. 1.

Table 1. *Positional parameters* (×10^4) *for the non-hydrogen atoms*

The e.s.d.'s of the last figures are given in parentheses.

	x	y	z
S	4022 (1)	1611 (1)	1044 (1)
O(1)	2718 (3)	3027 (4)	467 (1)
O(2)	5760 (3)	2170 (4)	1102 (2)
O(3)	3684 (3)	−904 (4)	859 (1)
O(4)	3893 (3)	2030 (4)	1757 (1)
O(5)	−109 (3)	4205 (4)	1651 (1)
O(6)	−616 (3)	1810 (5)	444 (1)
N(1)	−3332 (3)	6714 (5)	847 (1)
N(2)	3014 (3)	6728 (5)	1973 (1)
C(1)	−1773 (4)	7748 (6)	1448 (2)
C(2)	−150 (4)	6585 (6)	1452 (2)
C(3)	1445 (4)	7768 (6)	2021 (2)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34930 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Positional parameters* (×10³) *for the* H *atoms*

The e.s.d.'s of the last figures are given in parentheses.

	x	y	z
H(1)	−324 (4)	670 (7)	37 (2)
H(2)	−441 (5)	764 (7)	75 (2)
H(3)	−360 (5)	521 (7)	88 (2)
H(4)	−195 (4)	752 (6)	200 (2)
H(5)	−178 (4)	934 (6)	138 (2)
H(6)	−7 (3)	673 (5)	93 (1)
H(7)	−38 (5)	353 (7)	124 (2)
H(8)	144 (4)	752 (6)	253 (2)
H(9)	151 (4)	960 (6)	196 (2)
H(10)	411 (6)	679 (9)	248 (2)
H(11)	333 (4)	732 (7)	159 (2)
H(12)	289 (4)	530 (6)	188 (2)
H(13)	−150 (5)	159 (7)	10 (2)
H(14)	3 (5)	212 (7)	36 (2)

Table 3. *Bond lengths* (Å) *and angles* (°) *in the sulphate group and the water molecules*

The e.s.d.'s of the last figures are given in parentheses.

S–O(1)	1·475 (2)	O(1)–S–O(2)	110·8 (2)
S–O(2)	1·465 (3)	O(1)–S–O(3)	109·6 (1)
S–O(3)	1·491 (2)	O(1)–S–O(4)	109·3 (1)
S–O(4)	1·478 (3)	O(2)–S–O(3)	108·7 (2)
		O(2)–S–O(4)	110·2 (1)
O(6)–H(13)	0·79 (3)	O(3)–S–O(4)	108·2 (1)
O(6)–H(14)	0·65 (5)		
		H(13)–O(6)–H(14)	116 (4)

Table 4. *Bond lengths* (Å) *and angles* (°) *in the* 1,3-*diammonio*-2-*propanol cation*

The e.s.d.'s of the last figures are given in parentheses.

C(1)–C(2)	1·527 (5)	N(2)–H(10)	1·06 (4)
C(2)–C(3)	1·528 (4)	N(2)–H(11)	0·95 (4)
C(1)–N(1)	1·502 (4)	N(2)–H(12)	0·84 (3)
C(3)–N(2)	1·493 (5)	C(1)–H(4)	1·16 (4)
C(2)–O(5)	1·420 (4)	C(1)–H(5)	0·92 (4)
N(1)–H(1)	0·97 (4)	C(2)–H(6)	1·06 (3)
N(1)–H(2)	1·00 (4)	C(3)–H(8)	1·01 (3)
N(1)–H(3)	0·90 (4)	C(3)–H(9)	1·06 (3)
		O(5)–H(7)	0·84 (4)
N(1)–C(1)–C(2)	109·3 (3)		
O(5)–C(2)–C(1)	110·4 (3)		
O(5)–C(2)–C(3)	108·0 (2)		
C(1)–C(2)–C(3)	109·5 (3)		
N(2)–C(3)–C(2)	108·7 (3)		

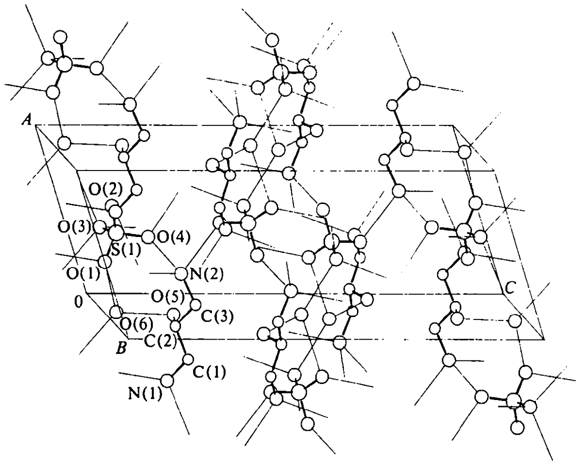

Fig. 1. The contents of the unit cell. Thin lines indicate hydrogen bonds.

Table 5. *Hydrogen bonds*

The e.s.d.'s of the last figures are given in parentheses.

D–H···A	D–A (Å)	D–H (Å)	H···A (Å)	∠D–H···A (°)
O(5)–H(7)···O(6)	2·638 (4)	0·84 (4)	1·80 (4)	170 (4)
O(6)–H(14)···O(1)	2·888 (4)	0·65 (5)	2·26 (4)	162 (4)
O(6)–H(13)···O(3)$^{\text{I}}$	2·880 (3)	0·79 (3)	2·09 (3)	174 (5)
N(1)–H(1)···O(1)$^{\text{II}}$	2·851 (4)	0·97 (4)	1·89 (4)	170 (3)
N(1)–H(2)···O(3)$^{\text{III}}$	2·881 (4)	1·00 (4)	1·91 (4)	163 (3)
N(1)–H(3)···O(2)$^{\text{IV}}$	2·825 (4)	0·90 (4)	1·93 (4)	170 (4)
N(2)–H(10)···O(4)$^{\text{V}}$	2·814 (3)	1·06 (4)	1·77 (4)	171 (5)
N(2)–H(11)···O(3)$^{\text{VI}}$	2·834 (4)	0·95 (4)	1·90 (4)	167 (4)
N(2)–H(12)···O(4)	2·879 (4)	0·84 (3)	2·12 (3)	151 (3)

Equivalent positions

(I)	$-x,-y,-z$	(IV)	$-1+x,y,z$
(II)	$-x,1-y,-z$	(V)	$1-x,\frac{1}{2}+y,\frac{1}{2}-z$
(III)	$-1+x,1+y,z$	(VI)	$x,1+y,z$

The dimensions of the sulphate ion are as expected. The S—O distances vary between 1·465 and 1·491 Å and the O—S—O angles of 108·2–110·8° are near the tetrahedral value.

The aminoalcohol molecule $NH_2CH_2CH(OH)CH_2NH_2$ acts as a proton acceptor through the two N atoms and exists as the divalent 1,3-diammonio-2-propanol cation $NH_3^+CH_2CH(OH)CH_2NH_3^+$. The bond lengths between non-hydrogen atoms do not deviate significantly from the corresponding single-bond values: C—C 1·541 (3), C—N 1·479 (5) and C—O 1·43 (1) Å (*International Tables for X-ray Crystallography*, 1968). All bond angles around the C atoms agree well with the tetrahedral value. The angles vary between 104·8 and 113·1° for C(1), between 107·4 and 111·5° for C(2) and between 105·8 and 115·4° for C(3). The angles of non-hydrogen atoms are listed in Table 4. These results are in good agreement with values found in 1,3-diammonio-2-propanol bis(oxalato)cuprate(II) trihydrate (Kivekäs & Pajunen, 1977).

The 1,3-diammonio-2-propanol cation, the sulphate anion and the water molecule are joined together by a hydrogen-bond network. The H atoms of the ammonium groups, the hydroxyl group and the water molecule are capable of forming hydrogen bonds and all O atoms of the sulphate ion and water molecule take part as acceptors (Table 5). Ammonium groups are connected only to the sulphate ions by hydrogen bonds and the water molecule lies between the sulphate ions and the hydroxyl group of the 1,3-diammonio-2-propanol cation. The role of the hydrogen bonds has been found to be similar in the bis(oxalato)cuprate(II) complex.

References

BERTRAND, J. A., MARABELLA, C. P. & VANDERVEER, D. G. (1977). *Inorg. Chim. Acta*, **25**, L69–L70.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

International Tables for X-ray Crystallography (1968). Vol. III. Birmingham: Kynoch Press.

KIVEKÄS, R. (1977). *Ann. Acad. Sci. Fenn. Ser. A*2, No. 185.

KIVEKÄS, R. & PAJUNEN, A. (1977). *Cryst. Struct. Commun.* **6**, 477–483.

PAJUNEN, A. & KIVEKÄS, R. (1979). *Cryst. Struct. Commun.* **8**, 385–391.

STEWART, J. M. (1976). The XRAY system – version of 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure of 3-[4,5-Bis(methoxycarbonyl)isoxazol-3-yl]-4,6-dioxo-*syn*-*cis*-*syn*-1,3,5-triphenylperhydrothieno[3,4-*c*]pyrrole-1-carbonitrile

BY I. UEDA

College of General Education, Kyushu University, Ropponmatsu, Chuo-ku, Fukuoka 810, *Japan*

AND T. TAKATA AND O. TSUGE

Research Institute of Industrial Science, Kyushu University, Hakozaki, Higashi-ku, Fukuoka 812, *Japan*

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Abstract. $C_{32}H_{23}N_3O_7S$, monoclinic, $P2_1/c$, $Z = 4$, a = 12·764 (6), b = 22·90 (1), c = 10·647 (4) Å, β = 110·41 (3)°, D_m = 1·351, D_x = 1·334 Mg m^{-3}. The structure was solved by the symbolic-addition procedure; block-diagonal least-squares refinement led to a final R of 0·0425 for 3471 observed reflexions. Molecules are held together by van der Waals interactions. The condensed perhydrothiophene–pyrrolidine ring has a chair conformation. A partial selective ester-exchange reaction occurred during the recrystallization from ethanol at the more protruding methoxycarbonyl group.

Introduction. In contrast to the adduct of 1,3-diphenylthieno[3,4-*c*][1,2,5]thiadiazole and *N*-phenylmaleimide (Tsuge, Takata & Ueda, 1979), neither the *endo* nor the *exo* adduct of 1,3-diphenylthieno[3,4-*c*]-[1,2,5]oxadiazole and the imide underwent a thermal retro-cycloaddition reaction, but reacted with dimethyl acetylenedicarboxylate (DMAD) to yield the corresponding 1:1 adduct (Tsuge, Takata & Noguchi, 1977). The product was assumed from spectroscopy to be a 1,3-cycloadduct of the nitrile oxide generated *in situ* from a ring opening of the oxadiazole moiety of the initial reactant, and DMAD (Tsuge, Takata & Ueda, 1979). Therefore, the X-ray analysis of the title compound was carried out to provide conclusive evidence for the chemical structure of the product obtained from the reaction of the *endo* adduct with DMAD.

The title compound (Fig. 1) was recrystallized at 313 K from an ethanol solution as colorless prisms. Preliminary Weissenberg photographs showed the space group to be $P2_1/c$. The unit-cell parameters were refined by a least-squares procedure using the 2θ values of 15 reflexions measured on a Syntex $P\bar{1}$ four-circle diffractometer. The density was measured by flotation in an aqueous KI solution. A cylindrical crystal (length 0·45 mm, diameter 0·39 mm) was used for the data collection. The intensities of 5746 reflexions within $2\theta \leq 50°$ were measured by the θ–2θ scan technique (monochromated Mo $K\alpha$ radiation), of which 3471 independent reflexions with $I > 2{\cdot}33\sigma(I)$ were used for the analysis. Corrections for Lorentz and polarization effects and for fluctuation of the monitored intensity were applied, but not for absorption (μ = 0·166 mm^{-1} for Mo $K\alpha$).

The structure was solved by the direct method using the program *MULTAN* (Germain, Main & Woolfson, 1971) and refined by a block-matrix least-squares

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