Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A synchrotron redetermination of 2-(morpholinium-4-yl)ethanesulfonate monohydrate, including a disordered water molecule

Maciej Kubicki,[a]* Dorota A. Adamiak,[b] Wojciech R. Rypniewski[b] and Agnieszka Olejniczak[c]

[a]Department of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland, [b]Institute of Bioorganic Chemistry, Polish Academy of Sciences, Noskowskiego 12/14, 61-704 Poznań, Poland, and [c]Center for Medical Biology, Laboratory of Medical Virology and Biological Chemistry, 93-232 Łódź, Poland

Correspondence e-mail: mkubicki@amu.edu.pl

Received 21 February 2007
Accepted 15 April 2007

Key indicators

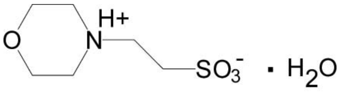
Single-crystal synchrotron study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.127
Data-to-parameter ratio = 10.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_6H_{13}NO_4S\cdot H_2O$ or $OC_4H_8NHCH_2CH_2SO_3\cdot H_2O$, was redetermined at 100 (1) K using synchrotron radiation. The water molecule, in contrast to the results of the prior room-temperature study [Christensen, Haxell, Lehmann & Nielsen (1993). *Acta Chem. Scand.* **47**, 753–756], was found to be disordered over two positions. The disordered model is more consistent with the observed hydrogen-bond network than the earlier suggested ordered structure.

Comment

The 2-(*N*-morpholino)ethanesulfonic acid (MES) buffer is commonly used in crystallization of proteins, nucleic acids and their derivatives. Crystals of MES monohydrate, (I), were obtained fortuitously during our crystallization trials of modified nucleosides (Olejniczak *et al.*, 2007).



(I)

The crystal structure determination of MES monohydrate reported earlier (Christensen *et al.*, 1993) is of relatively poor quality (room temperature, H atoms not refined, R > 7%). Most importantly, the solvent water molecule is described in that study as occupying a single position with relatively high displacement parameters. The results of our study at 100 K show that this water molecule can and, in fact, should be described as disordered over two positions. As the precise structural data might be important, for instance for modelling

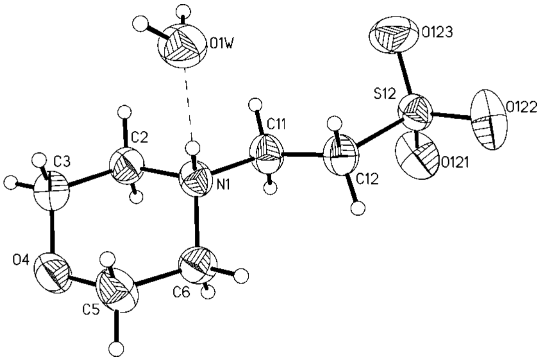


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level; H atoms are depicted as spheres of arbitrary radius. Only the water molecule in the higher occupancy position is shown and the hydrogen bond is drawn as a dashed line.

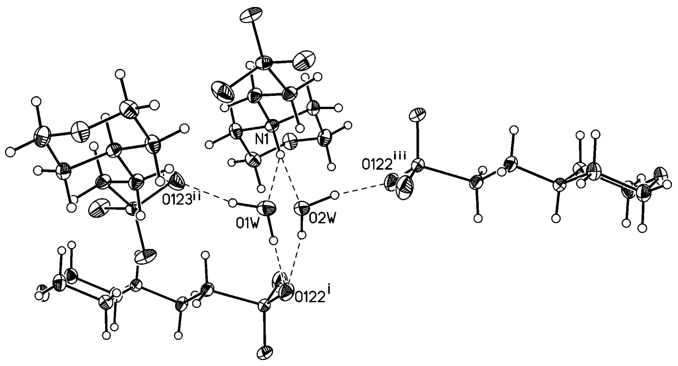


Figure 2
The two positions of the disordered water molecule and the hydrogen bonds (drawn as dashed lines). [Symmetry codes: (i) $2 - x, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $2 - x, -y, -z$; (iii) $x, -\frac{1}{2} - y, z + \frac{1}{2}$.]

purposes, we have decided to complete the full crystal structure determination.

The MES molecule exists in a zwitterionic form (Fig. 1). This is confirmed by successful refinement of atom H1 bonded to the morpholine atom N1, as well as by the approximately equal S—O bonds in the SO_3 group (Table 1). The bond length and angle pattern is similar to that found in related compounds (*cf* for instance Christensen *et al.*, 1993; Deschamps *et al.*, 2002; Milić *et al.*, 2005); the morpholine ring is in a chair conformation [maximum values of the asymmetry parameters (Duax & Norton, 1975) are 4.7° for twofold axes and 5.9° for mirror planes]. The ethanesulfonate substituent occupies an equatorial position with respect to the morpholine ring [C3—C2—N1—C11 = −179.7 (2)° and C5—C6—N1—C11 = −177.6 (2)°] and has an extended zigzag-like conformation [N1—C11—C12—S12 = −171.0 (1)°].

The water molecule is disordered over two positions O1*W* and O2*W* with site occupancy factors of 0.581 (6) and 0.419 (6), respectively. Interestingly, the water molecule in each of the two positions is involved in two similar hydrogen bonds, *viz.* as an acceptor for the morpholine N1/H1 group and as a donor to atom O122^{i}. In both positions it forms one more bond as a donor; however, for one of the positions atom O123^{ii} serves as an acceptor, whereas for the second position the role of the hydrogen-bond acceptor is played by atom O122^{iii} (Fig. 2; see the caption to Fig. 2 for the equivalent positions corresponding to the symmetry codes). As a result, one of the water positions gives rise to centrosymmetric dimers, whereas the alternative position produces chains along the [001] direction. One would thus expect the crystal structure to feature chain fragments which are arbitrarily interrupted by centrosymmetric dimers. Using graph-set notation (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the latter can be described as $R_4^4(16)$, while the former is (second order) $C_2^2(8)$. Additionally, relatively strong C—H···O hydrogen bonds strengthen the structure (*cf* Table 2).

Experimental

The crystals were grown by the method of sitting-drop vapour diffusion (Weber, 1997). The reservoir solution contained 0.1 *M* potassium chloride, 0.1 *M* magnesium chloride, 0.05 *M* MES buffer pH 6.0 and 10% *w*/*v* polyethyleneglycol (PEG 400) as precipitating agent. The solution used in the reservoir was from tube #9 of the Natrix crystallization kit purchased from Hampton Research (http://www.hamptonresearch.com). The crystallization drop initially contained equal proportions of the above solution and a solution of 6-*N*-{5-[3-cobalt-bis(1,2-dicarbollide)-8-yl]-3-oxa-pentoxy}-2′-*O*-deoxyadenosine at 10 mg ml^{-1}. Small crystals appeared within two weeks. At first we thought that the crystals were of the nucleoside, but analysis of the crystals revealed that they contained the MES monohydrate.

Crystal data

$C_6H_{13}NO_4S \cdot H_2O$
$M_r = 213.25$
Monoclinic, $P2_1/c$
$a = 8.609$ (1) Å
$b = 9.959$ (2) Å
$c = 11.144$ (2) Å
$\beta = 93.79$ (3)°
$V = 953.4$ (3) Å^3
$Z = 4$
Synchrotron radiation
$\lambda = 0.8156$ Å
$\mu = 0.33$ mm^{-1}
$T = 100$ (1) K
0.4 × 0.2 × 0.2 mm

Data collection

MAR CCD 165mm diffractometer
Absorption correction: none
6095 measured reflections
1893 independent reflections
1679 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.127$
$S = 1.06$
1893 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1*W*	0.96 (3)	1.80 (3)	2.683 (3)	151 (2)
N1—H1···O2*W*	0.96 (3)	1.83 (3)	2.783 (4)	175 (2)
O1*W*—H1*W*2···O122^{i}	0.85	1.98	2.790 (3)	159
O2*W*—H2*W*1···O122^{i}	0.85	1.97	2.727 (4)	147
O1*W*—H1*W*1···O123^{ii}	0.85	1.98	2.775 (4)	156
C2—H2*A*···O123^{ii}	0.97 (3)	2.52 (3)	3.366 (3)	145 (2)
C11—H11*B*···O123^{ii}	0.98 (3)	2.52 (3)	3.362 (3)	145 (2)
O2*W*—H2*W*2···O122^{iii}	0.85	2.01	2.834 (5)	162
C12—H12*A*···O4^{iv}	0.96 (3)	2.52 (3)	3.351 (3)	145 (2)
C6—H6*B*···O121^{v}	0.98 (2)	2.38 (2)	3.293 (3)	154.3 (17)

Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 2, -y, -z$; (iii) $x, -y - \frac{1}{2}, z + \frac{1}{2}$; (iv) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (v) $-x + 1, -y, -z$.

The water molecule was found in two alternative positions. Site occupation factors were refined at 0.581 (6) and 0.419 (6). O atoms were refined anisotropically without any constraints; the positions of water H atoms were calculated on the basis of the potential hydrogen-bond scheme with an O—H distance of 0.85 Å, and refined as riding with $U_{\rm iso}$ calculated as 1.2 times $U_{\rm eq}$ for the O atoms. All other H atoms were refined isotropically [N—H = 0.96 (3) Å; C—H 0.94 (3)–1.06 (3) Å]. The refinement without splitting the water molecule was significantly worse; the *R* factor was 0.078 and the largest component of the displacement tensor of the O atom was as high as 0.5.

Data collection: *MARCCD* (MarResearch, 2002); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure:

Acta Cryst. (2007). E**63**, o2604–o2606 Kubicki *et al.* • $C_6H_{13}NO_4S \cdot H_2O$ **o2605**

SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

This work was supported by the European Community Research Infrastructure Action under the FP6 'Structuring the European Research Area Programme' (contract number RII3/CT/2004/5060008).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1565.

Christensen, A. N., Hazell, R. G., Lehmann, M. S. & Nielsen, M. (1993). *Acta Chem. Scand.* **47**, 753–756.

Deschamps, J. R., Flippen-Anderson, J. L. & George, C. (2002). *Acta Cryst.* E**58**, m167–m168.

Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1, pp. 462-473. New York: Plenum.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.

MarResearch (2002). *MARCCD*. MarResearch GmbH, Norderstedt, Germany.

Milić, D., Renić, M. & Matković-Čalogović, D. (2005). *Acta Cryst.* E**61**, m757–m758.

Olejniczak, A. B., Plesek, J. & Lesnikowski, Z. J. (2007). *Chem. Eur. J.* **13**, 311–318.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Siemens (1989). *Stereochemical Workstation Operation Manual*. Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Weber, P. C. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 13–22. New York: Academic Press.

o2606 Kubicki *et al.* • $C_6H_{13}NO_4S{\cdot}H_2O$ *Acta Cryst.* (2007). E**63**, o2604–o2606