

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Editors: **W. Clegg** and **D. G. Watson**

5,5-Dihydroxybarbituric acid monohydrate (alloxan dihydrate)

Thomas C. Lewis and Derek A. Tocher

Copyright © International Union of Crystallography

Author(s) of this paper may load this reprint on their own web site provided that this cover page is retained. Republication of this article or its storage in electronic databases or the like is not permitted without prior permission in writing from the IUCr.

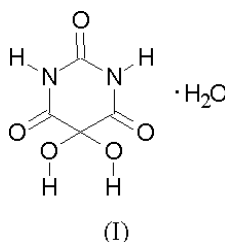
5,5-Dihydroxybarbituric acid monohydrate
(alloxan dihydrate)Thomas C. Lewis and
Derek A. Tocher*Department of Chemistry, University College
London, 20 Gordon Street, London WC1H 0AJ,
England

Correspondence e-mail: d.a.tocher@ucl.ac.uk

Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.043
 wR factor = 0.102
Data-to-parameter ratio = 11.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_4\text{H}_4\text{N}_2\text{O}_5 \cdot \text{H}_2\text{O}$, was crystallized from both tetrahydrofuran and 1,4-dioxane solutions of alloxan as part of an experimental polymorph screen on alloxan.Received 25 August 2004
Accepted 1 September 2004
Online 11 September 2004

Comment

It has previously been reported that alloxan has two hydrates, *viz.* 5,5-dihydroxybarbituric acid (Singh, 1965; Harrowfield *et al.*, 1989) and 5,5-dihydroxybarbituric acid trihydrate (Mootz & Jeffrey, 1965). The crystal structure of a new hydrate of alloxan, namely 5,5-dihydroxybarbituric acid monohydrate, (I), has one organic molecule and one water molecule in the asymmetric unit (Fig. 1). The heterocyclic ring has an envelope conformation with the flap at C5, with the angle between the mean C4/N3/C2/N1/C6 and C4/C5/C6 planes being $20.1(2)^\circ$. The C—N bond lengths are in the range $1.360(2)$ – $1.378(2)$ Å, with the bond lengths associated with the sp^3 -hybridized carbon being $1.536(2)$ and $1.527(2)$ Å for C4—C5 and C5—C6, respectively.The crystal packing (Fig. 2) consists of a series of ribbon motifs arranged in an overall sheet structure. Water molecules lie in the sheets and between the ribbons. Each water molecule acts as a hydrogen-bond donor to a carbonyl group in the same sheet and to a hydroxyl group on a molecule in the adjacent sheet. Each water molecule also acts as a hydrogen-bond acceptor for a hydroxyl group on a molecule in the same sheet. The axial hydroxyl group on each molecule acts as a hydrogen-bond donor to the unique carbonyl of a molecule in an adjacent sheet. The $D \cdots A$ distances within the sheets are in the range $2.6380(19)$ – $2.9516(19)$ Å, whilst the distances between the sheets are $2.6958(17)$ and $2.9973(19)$ Å. All potential hydrogen-bond acceptors and donors participate in the hydrogen bonding.

Experimental

5,5-Dihydroxybarbituric acid monohydrate was crystallized over a number of weeks by slow evaporation of tetrahydrofuran and 1,4-dioxane solutions of alloxan (0.002 – 0.03 mol dm^{-3}) at room temperature, forming colourless plate crystals.

Crystal data

$C_4H_4N_2O_5 \cdot H_2O$
 $M_r = 178.11$
 Triclinic, $P\bar{1}$
 $a = 6.6730$ (11) Å
 $b = 7.5834$ (13) Å
 $c = 7.6157$ (13) Å
 $\alpha = 105.401$ (3)°
 $\beta = 93.134$ (3)°
 $\gamma = 115.089$ (2)°
 $V = 330.26$ (10) Å³

$Z = 2$
 $D_x = 1.791$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 712 reflections
 $\theta = 2.8$ – 25.0 °
 $\mu = 0.17$ mm⁻¹
 $T = 150$ (2) K
 Plate, colourless
 $0.23 \times 0.11 \times 0.07$ mm

Data collection

Bruker SMART APEX diffractometer
 Narrow-frame ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.962$, $T_{max} = 0.988$
 2972 measured reflections

1536 independent reflections
 1274 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.021$
 $\theta_{max} = 28.3$ °
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 9$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.102$
 $S = 1.07$
 1536 reflections
 133 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.0119P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.34$ e Å⁻³
 $\Delta\rho_{min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------|----------|-------------|-------------|---------------|
| $N1-H1\cdots O6^i$ | 0.89 (2) | 1.95 (2) | 2.8366 (18) | 170.7 (19) |
| $N3-H3\cdots O4^{ii}$ | 0.81 (2) | 2.11 (2) | 2.8736 (18) | 157 (2) |
| $O7-H7\cdots O2^{iii}$ | 0.87 (2) | 1.83 (3) | 2.6958 (17) | 173 (2) |
| $O8-H8\cdots O1W^{iii}$ | 0.80 (2) | 1.87 (2) | 2.6380 (19) | 161 (2) |
| $O1W-H1W\cdots O6^{iv}$ | 0.92 (3) | 2.04 (3) | 2.9516 (19) | 173 (2) |
| $O1W-H2W\cdots O7^{ii}$ | 0.82 (3) | 2.28 (3) | 2.9973 (19) | 147 (3) |

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

H atoms were refined freely with an isotropic model.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2000; Bruno *et al.*, 2002); software used to prepare material for publication: SHELXL97.

This research was supported by the EPSRC in funding a studentship for TCL. The authors acknowledge the Research Councils UK Basic Technology Programme for supporting 'Control and Prediction of the Organic Solid State'. For

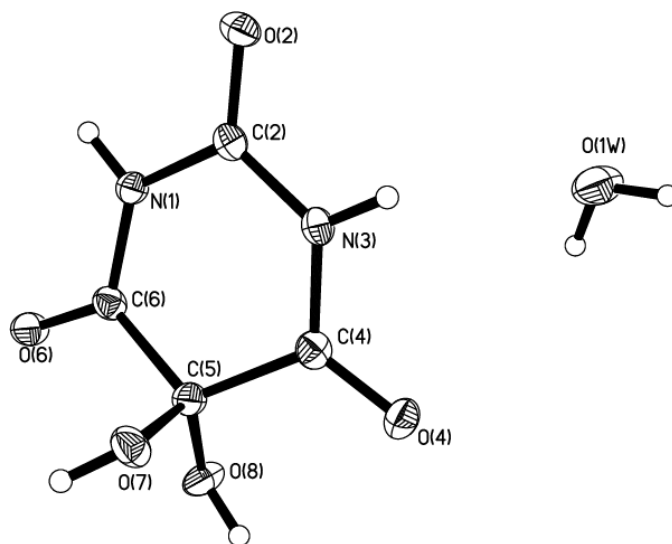


Figure 1

The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

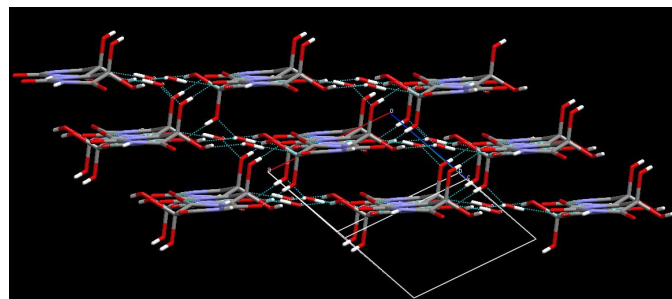


Figure 2

The crystal packing of (I), showing the N–H...O and O–H...O hydrogen-bonding interactions as dashed lines; the view is approximately on to the (011) plane.

more information on this work, please visit <http://www.chem.ucl.ac.uk/basictechorg/>.

References

Bruker (2000). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruno, Z. J., Cole, J. C., Edgington, P. R., Kessler, M. K., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). Acta Cryst. B58, 389–397.
 Harrowfield, J. M., Skelton, B. W., Soudi, A. A. & White, A. H. (1989). Aust. J. Chem. 42, 1795–1798.
 Mootz, D. & Jeffrey, G. A. (1965). Acta Cryst. 19, 717–725.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1990). Acta Cryst. A46, 467–473.
 Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
 Singh, C. (1965). Acta Cryst. 19, 759–767.