electronic reprint

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368
Editors: **W. Clegg and D. G. Watson**

A low-temperature determination of butyramide

Thomas C. Lewis and Derek A. Tocher

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A low-temperature determination of butyramide

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 σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.111
Data-to-parameter ratio = 13.7

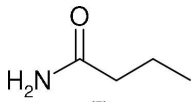
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 May 2005
Accepted 27 May 2005
Online 10 June 2005

The low-temperature structure determination of butyramide, C_4H_9NO, obtained as part of a experimental polymorph screen on adenine, is reported here. Each molecule takes part in four hydrogen bonds to form a three-dimensional ribbon structure.

Comment

The title compound, (I), is one of the *n*-aliphatic amides and has recently been studied as a possible agent for growth inhibition of human neuroblastoma cell lines (Rocchi *et al.*, 1998) and inhibitory effects on DNA synthesis on hepatoma cells (Lea *et al.*, 1993).



(I)

The powder diffractogram data for (I) were reported in 1950 (Matthews *et al.*, 1950), as part of a study on derivatives of fatty acids, and the unit cell was determined five years later (Turner & Lingafelter, 1955) using Weissenberg photographs, to give a = 9.94 Å, b = 5.79 Å, c = 10.02 Å and β = 100.9°. Examination of the systematic absences showed the space group to be $P2_1/a$; however, no atomic coordinates were published. We have solved and refined the crystal structure of butyramide at 150 K, to give a final R value of 0.041. There is a 12° difference in the β angle between the two determinations. In (I), the bond lengths and angles are within expected values (Allen *et al.*, 1987), with the C—C bond lengths in the range 1.5057 (18)–1.515 (2) Å and with N1—C1 and O2—C1 bond lengths of 1.3257 (15) and 1.2395 (13) Å, respectively. There is a relative twist of the carbon chain from planarity, with torsion angles C1—C2—C3—C4 and N1—C1—C2—C3 of 177.41 (21) and 151.62 (12)°, respectively. The packing consists of

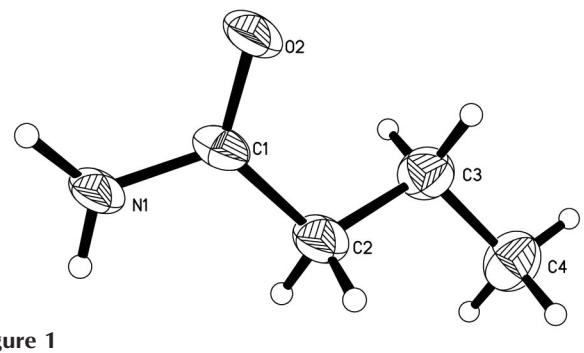


Figure 1
View of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

centrosymmetric dimers, linked through a pair of N—H···O hydrogen bonds [2.9470 (15) Å]. The other amine H atom is used to hydrogen bond to an adjacent dimer unit which is approximately perpendicular (73°), through an N—H···O hydrogen bond [2.8496 (14) Å], resulting in the formation of a three-dimensional criss-crossed ribbon structure (Fig. 2).

Experimental

As part of an experimental polymorph screen on adenine, (I) was obtained from a 0.3 *M* aqueous solution of (I), to which approximately 0.15 g of adenine was added, and which was stirred on a hotplate at 303 K for 3 d. This solution was filtered, then evaporated at room temperature (10 ml solution, in 75 × 25 mm vessels) in an attempt to crystallize adenine, as it has been found that the solubility of purine and pyrimidine bases increases in aqueous amide solutions (Herskovits & Bowen, 1974). Colourless block-like crystals of (I) were formed after a number of days.

Crystal data

C_4H_9NO
$M_r = 87.12$
Monoclinic, $P2_1/c$
$a = 9.814$ (3) Å
$b = 5.9232$ (17) Å
$c = 9.701$ (3) Å
$\beta = 112.070$ (4)°
$V = 522.6$ (3) Å^3
$Z = 4$
$D_x = 1.107$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1237 reflections
$\theta = 2.2$–$25.4°$
$\mu = 0.08$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.38 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.971$, $T_{max} = 0.987$
4321 measured reflections
1244 independent reflections
993 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 28.3°$
$h = -13 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.111$
$S = 1.01$
1244 reflections
91 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.0651P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.92 (2)	2.03 (2)	2.9470 (15)	176 (1)
N1—H2···O2^{ii}	0.89 (2)	1.98 (2)	2.8496 (14)	168 (1)

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

H atoms were refined independently with an isotropic model.

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

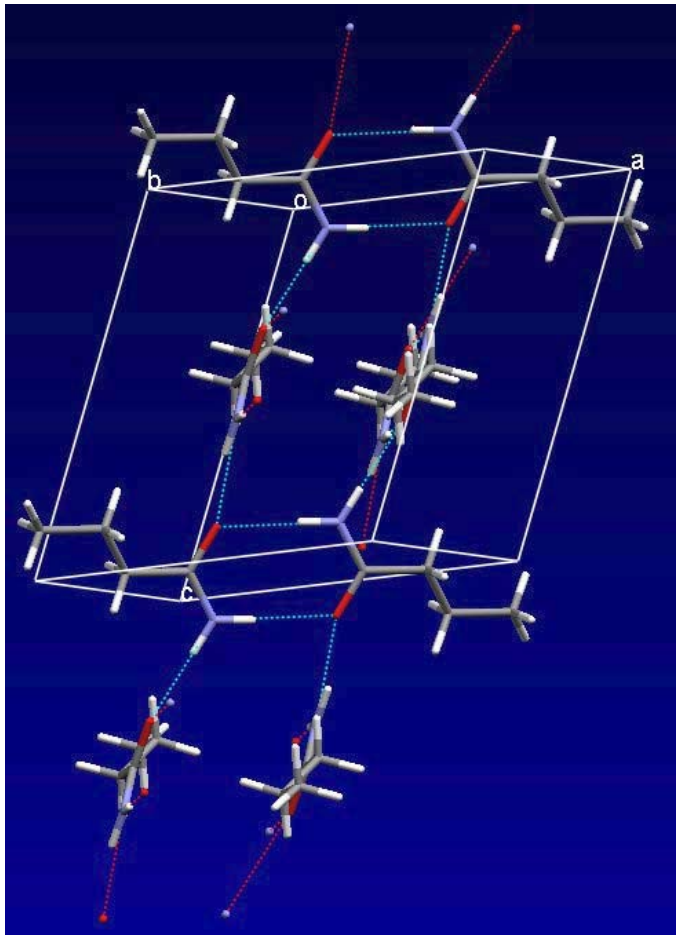


Figure 2
The packing in (I), showing the butyramide dimer unit which forms a hydrogen-bonded (dashed lines) criss-cross ribbon motif.

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 more information on this work, please visit http://www.cposs.org.uk.

References

Allen, F. H., Kennard, O. & Watson, D. G. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–18.
Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M. K., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Herskovits, T. T. & Bowen, J. J. (1974). *Biochemistry*, **13**, 5474–5483.
Lea, M. A., Xiao, Q., Sadhukhan, A., Sharma, S. & Newmark, H. L. (1993). *Anticancer Res.* **13**, 145–150.
Matthews, F. W., Warren, G. G. & Michell, J. H. (1950). *Anal. Chem.* **22**, 514–519.
Rocchi, P., Ferreri, A. M., Magrini, E. & Perocco, P. (1998). *Anticancer Res.* **18**, 1099–1103.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Turner, J. D. & Lingafelter, E. C. (1955). *Acta Cryst.* **8**, 549–550.