

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

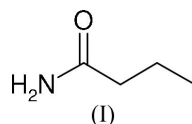
Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.041
 wR factor = 0.111
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.A low-temperature determination of
butyramide

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

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

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).



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 $\beta = 100.9^\circ$. 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

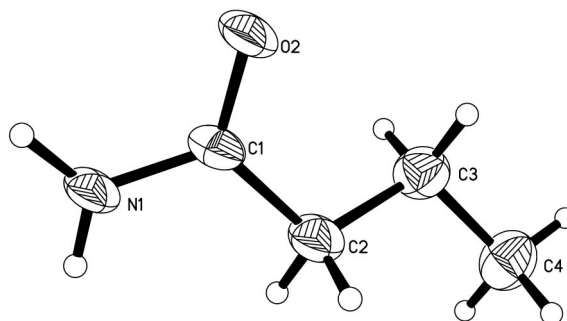


Figure 1
View of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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 ₄ H ₉ NO	$D_x = 1.107 \text{ Mg m}^{-3}$
$M_r = 87.12$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1237 reflections
$a = 9.814 (3) \text{ \AA}$	$\theta = 2.2\text{--}25.4^\circ$
$b = 5.9232 (17) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 9.701 (3) \text{ \AA}$	$T = 150 (2) \text{ K}$
$\beta = 112.070 (4)^\circ$	Block, colourless
$V = 522.6 (3) \text{ \AA}^3$	$0.38 \times 0.20 \times 0.16 \text{ mm}$
$Z = 4$	

Data collection

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

Refinement

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

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.

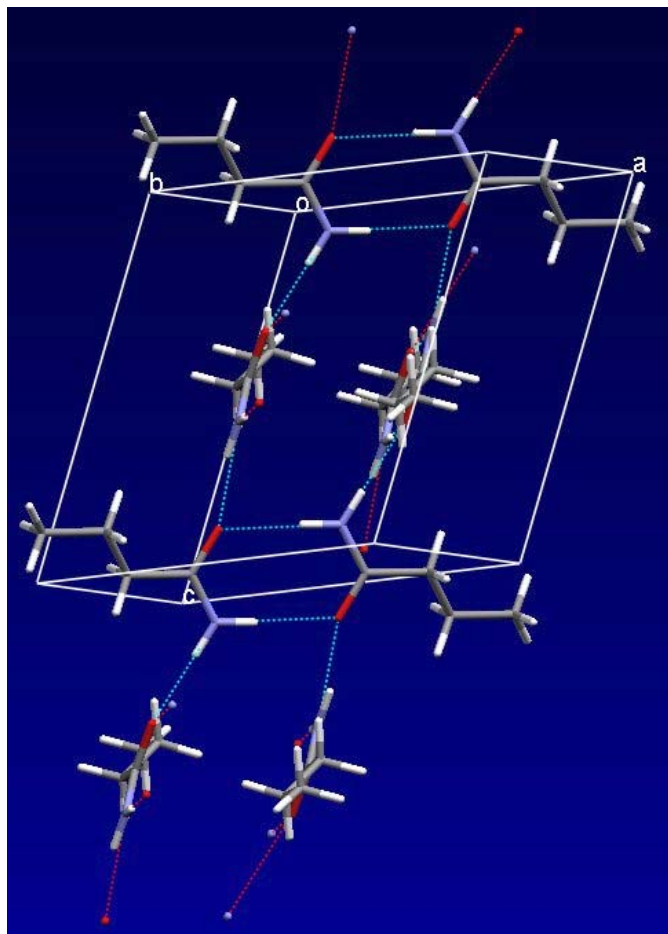


Figure 2

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. B58*, 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. A46*, 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.

supporting information

Acta Cryst. (2005). E61, o1985–o1986 [https://doi.org/10.1107/S1600536805017022]

A low-temperature determination of butyramide

Thomas C. Lewis and Derek A. Tocher

Butyramide

Crystal data

C_4H_9NO	$F(000) = 192$
$M_r = 87.12$	$D_x = 1.107 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 9.814 (3) \text{ \AA}$	Cell parameters from 1237 reflections
$b = 5.9232 (17) \text{ \AA}$	$\theta = 2.2\text{--}25.4^\circ$
$c = 9.701 (3) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\beta = 112.070 (4)^\circ$	$T = 150 \text{ K}$
$V = 522.6 (3) \text{ \AA}^3$	Plate, colourless
$Z = 4$	$0.38 \times 0.20 \times 0.16 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer	4321 measured reflections
Radiation source: fine-focus sealed tube	1244 independent reflections
Graphite monochromator	993 reflections with $I > 2\sigma(I)$
ω rotation with narrow frames scans	$R_{\text{int}} = 0.021$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.2^\circ$
$T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.987$	$h = -13 \rightarrow 12$
	$k = -7 \rightarrow 7$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	All H-atom parameters refined
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.0651P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1244 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
91 parameters	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.62317 (10)	0.21606 (15)	0.61392 (8)	0.0388 (3)
N1	0.58606 (12)	0.13501 (19)	0.37696 (10)	0.0327 (3)
C1	0.65020 (13)	0.25241 (19)	0.50094 (11)	0.0300 (3)
C2	0.76164 (15)	0.4276 (2)	0.50031 (14)	0.0370 (3)
C3	0.77563 (15)	0.6237 (2)	0.60348 (16)	0.0387 (3)
C4	0.89354 (17)	0.7892 (3)	0.60401 (19)	0.0457 (4)
H1	0.5216 (15)	0.022 (3)	0.3760 (14)	0.042 (4)*
H2	0.6058 (15)	0.164 (2)	0.2966 (16)	0.037 (3)*
H3	0.7428 (17)	0.475 (3)	0.4012 (17)	0.055 (4)*
H4	0.860 (2)	0.344 (3)	0.5341 (19)	0.061 (5)*
H5	0.680 (2)	0.701 (3)	0.5663 (18)	0.054 (4)*
H6	0.7966 (16)	0.564 (2)	0.7050 (18)	0.050 (4)*
H7	0.8744 (19)	0.845 (3)	0.506 (2)	0.063 (5)*
H8	0.898 (2)	0.914 (3)	0.664 (2)	0.070 (5)*
H9	0.9914 (18)	0.716 (3)	0.6376 (16)	0.049 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0579 (6)	0.0422 (5)	0.0254 (4)	-0.0114 (4)	0.0263 (4)	-0.0053 (3)
N1	0.0456 (6)	0.0368 (6)	0.0222 (5)	-0.0047 (5)	0.0202 (4)	-0.0012 (4)
C1	0.0398 (6)	0.0317 (6)	0.0240 (5)	0.0024 (5)	0.0183 (5)	0.0006 (4)
C2	0.0475 (7)	0.0403 (7)	0.0297 (6)	-0.0074 (6)	0.0218 (5)	-0.0019 (5)
C3	0.0383 (7)	0.0353 (7)	0.0453 (8)	0.0003 (5)	0.0190 (6)	-0.0028 (5)
C4	0.0435 (8)	0.0382 (8)	0.0549 (9)	-0.0031 (6)	0.0179 (7)	-0.0013 (7)

Geometric parameters (\AA , $^\circ$)

O2—C1	1.2395 (13)	C2—H4	1.022 (18)
N1—C1	1.3257 (15)	C3—C4	1.515 (2)
N1—H1	0.919 (16)	C3—H5	0.985 (18)
N1—H2	0.887 (15)	C3—H6	0.993 (16)
C1—C2	1.5091 (17)	C4—H7	0.954 (18)
C2—C3	1.5057 (18)	C4—H8	0.93 (2)
C2—H3	0.950 (16)	C4—H9	0.990 (16)
C1—N1—H1	118.8 (8)	C2—C3—C4	112.36 (11)
C1—N1—H2	120.7 (9)	C2—C3—H5	106.1 (9)
H1—N1—H2	120.5 (13)	C4—C3—H5	109.0 (9)
O2—C1—N1	121.64 (11)	C2—C3—H6	108.5 (9)

O2—C1—C2	121.28 (10)	C4—C3—H6	110.7 (9)
N1—C1—C2	117.05 (10)	H5—C3—H6	110.0 (13)
C3—C2—C1	114.40 (10)	C3—C4—H7	110.9 (11)
C3—C2—H3	112.4 (10)	C3—C4—H8	111.5 (11)
C1—C2—H3	109.9 (10)	H7—C4—H8	107.2 (16)
C3—C2—H4	108.6 (10)	C3—C4—H9	111.3 (9)
C1—C2—H4	105.5 (10)	H7—C4—H9	106.4 (14)
H3—C2—H4	105.5 (13)	H8—C4—H9	109.4 (14)
O2—C1—C2—C3	-30.35 (18)	C1—C2—C3—C4	177.41 (12)
N1—C1—C2—C3	151.62 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O2 ⁱ	0.919 (16)	2.030 (16)	2.9470 (15)	175.9 (13)
N1—H2 \cdots O2 ⁱⁱ	0.887 (15)	1.976 (15)	2.8496 (14)	167.9 (13)

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