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A low-temperature determination of butyramide

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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.

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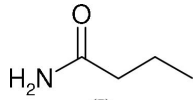
The low-temperature structure determination of butyramide, C_4H_9NO, obtained as part of a experimental polymorph screen on adenine, is reported here. Each molecule takes part in four hydrogen bonds to form a three-dimensional ribbon structure.

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

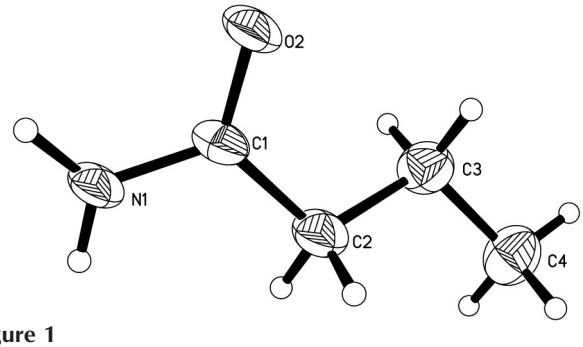


Figure 1
View of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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

Data collection

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

Refinement

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

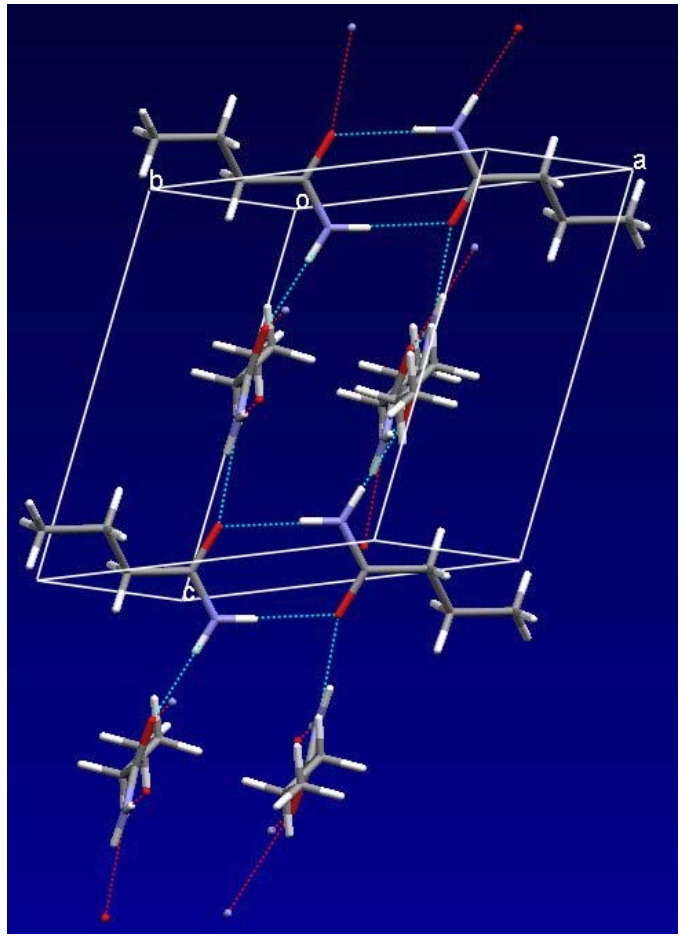


Figure 2
The packing in (I), showing the butyramide dimer unit which forms a hydrogen-bonded (dashed lines) criss-cross ribbon motif.

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