Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Diethylamino)-*N*-(2,6-dimethylphenyl)-acetamide, a low-temperature redetermination

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.150
Data-to-parameter ratio = 12.1

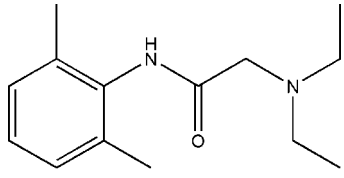
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 December 2006
Accepted 11 January 2007

The title compound, commonly known as lidocaine, $C_{14}H_{22}N_2O$, is a well known drug with local anaesthetic and anti-arrhythmic properties. Its structure has been reported previously, based on data of limited quality [Hanson & Banner (1974), *Acta Cryst.* B**30**, 2486–2488]. Results from a data collection at 173 K are now reported. The structure is characterized by the presence of two independent molecules in the asymmetric unit and by chains of hydrogen-bonded molecules in the crystal structure.

Comment

The title compound, commonly known as lidocaine, (I), is widely used as a local anaesthetic and for the management of cardiac arrhythmias (Gröningsson *et al.*, 1985; Powell, 1986). The crystal structure of lidocaine (I), previously determined at room temperature from photographic data (Hanson & Banner, 1974), has been redetermined at low temperature. Reduction of the thermal motion in (I) at 173 K reveals the presence of a twofold orientational disorder of an ethyl group for one of the two symmetry-independent molecules that was not resolved in the room-temperature study.



(I)

Selected geometric parameters for (I) are listed in Table 1. As in the earlier structure, the *trans*-amide configuration allows the formation of an intramolecular hydrogen bond (Fig. 1) and favours the formation, by the same amide H atom, of a chain of intermolecular hydrogen bonds (Table 2), parallel to the *c* axis (Fig. 2). In spite of a 4.5% decrease in the unit-cell volume, the mean values of the distances between the non-H atoms involved in the formation of the hydrogen bonds are essentially unaffected by the temperature decrease. On the other hand, the molecular conformation is affected, in particular for one of the molecules: the C2—C1—N1—C9 torsion angle (Table 1) changes by 20.3° from its value of −82.4° in the room-temperature structure.

Experimental

Samples of compound (I) were kindly provided by SIMS (SIMS srl, Reggello, Firenze, Italy). Crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone–water (1:3) solution.

Crystal data

$C_{14}H_{22}N_2O$
M_r = 234.34
Monoclinic, $P2_1/c$
a = 12.9590 (3) Å
b = 13.8003 (3) Å
c = 18.8288 (5) Å
β = 122.340 (3)°
V = 2845.00 (12) Å^3
Z = 8
D_x = 1.094 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 173 (2) K
Elongated prism, colourless
0.65 × 0.2 × 0.2 mm

Data collection

Oxford Diffraction Excalibur PX Ultra CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*ABSPACK*; Oxford Diffraction, 2006)
T_{min} = 0.763, T_{max} = 0.897
34455 measured reflections
5218 independent reflections
3547 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 68.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.150
S = 1.07
5218 reflections
433 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0974P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0015 (5)

Table 1
Selected geometric parameters (Å, °).

N1—C9	1.338 (2)	N3—C23	1.340 (2)
C9—O1	1.2314 (19)	C23—O2	1.228 (2)
C9—N1—C1	123.42 (14)	C23—N3—C15	124.13 (14)
N1—C9—C10	115.46 (14)	N3—C23—C24	115.05 (15)
C2—C1—N1—C9	−102.67 (19)	C16—C15—N3—C23	−77.9 (2)
N1—C9—C10—N2	3.1 (2)	N3—C23—C24—N4	13.0 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*N*···O1	0.89 (2)	2.14 (2)	2.8952 (18)	142.6 (17)
N1—H1*N*···O2^{i}	0.85 (2)	2.17 (2)	2.8746 (17)	140.9 (18)
N1—H1*N*···N2	0.85 (2)	2.24 (2)	2.678 (2)	112.3 (16)
N3—H3*N*···N4	0.89 (2)	2.262 (19)	2.695 (2)	109.7 (15)

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

The positions of H atoms belonging to those parts of the molecules not affected by disorder were detected in Fourier difference maps and were refined [secondary CH_2 = 0.97 (2)–1.08 (3) Å, methyl CH_3 = 0.92 (3)–1.06 (4) Å, aromatic CH = 0.90 (3)–1.02 (2) Å and N—H = 0.85 (2)–0.89 (2) Å]. The positions of the other H atoms were calculated geometrically, with secondary CH_2 distances of 0.99 Å and methyl CH_3 distances of 0.98 Å. The constraints U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C) for methyl groups, were applied. The population parameters of the two possible orientations of the disordered ethyl group are 0.512 (7) and 0.488 (7).

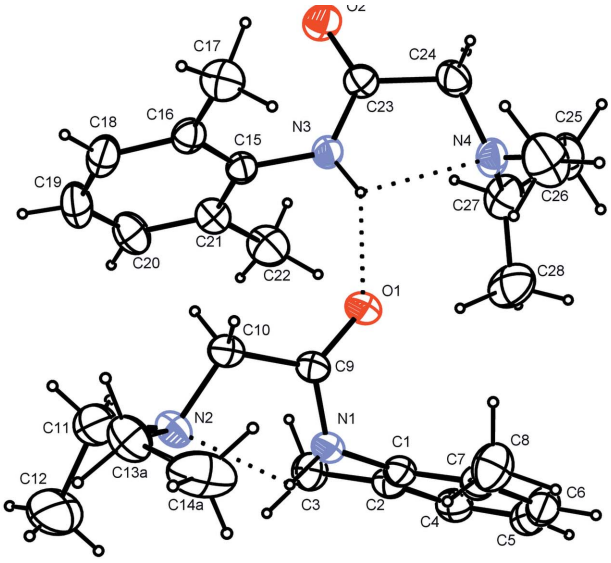


Figure 1
The asymmetric unit of (I), with 30% probability displacement ellipsoids. Only the major component of the disordered ethyl group is shown for clarity. Hydrogen bonds are indicated by dashed lines.

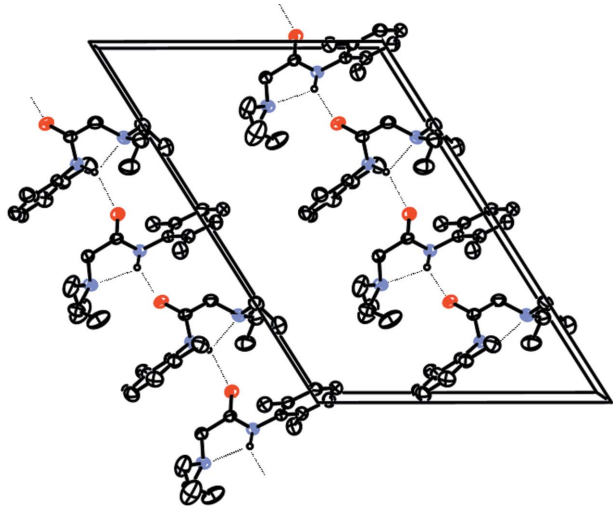


Figure 2
Part of the packing of (I), in the proximity of the *ac* face of the unit cell. H atoms have been omitted, except for those involved in the bifurcated hydrogen-bonding system. Only the major disorder component is shown.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge financial support from the Italian Ministero dell'Istruzione, dell'Universitá e della Ricerca.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gröningsson, K., Lindgren, J.-E., Lundberg, E., Sandberg, R. & Wahlén, A. (1985). *Anal. Profiles Drug. Subst.* **14**, 207–243.
Hanson, A. W. & Banner, D. W. (1974). *Acta Cryst.* B**30**, 2486–2488.
Oxford Diffraction (2006). *CrysAlis CCD* (Version 1.171.31.2), *CrysAlis RED* (Version 1.171.31.2) and *ABSPACK* in *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Powell, M. F. (1986). *Anal. Profiles Drug. Subst.* **15**, 761–779.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.