Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[3-(Dimethylamino)propyl]-10,11-dihydro-5*H*-dibenz[*a*,*d*][7]annulen-5-ol

Gustavo Portalone,[a]* Marcello Colapietro,[a] S. Bindya,[b] M. A. Ashok[b] and H.S. Yathirajan[b]

[a]Chemistry Department, University of Rome I 'La Sapienza', Piazzale Aldo Moro 5, I-00185 Rome, Italy, and [b]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India

Correspondence e-mail: g.portalone@caspur.it

Received 20 December 2006
Accepted 11 January 2007

In the crystal structure of the title compound, $C_{20}H_{25}NO$, a derivative of amitriptyline, an antidepressant drug, an intramolecular O—H···N hydrogen bond of 2.713 (2) Å between a hydroxyl group and the N atom of the dimethylaminopropyl group is observed. The crystal packing is stabilized only by van der Waals interactions.

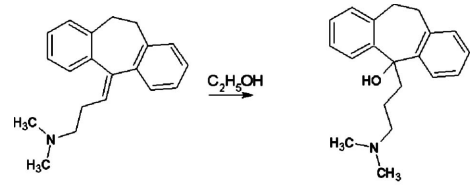
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.108
Data-to-parameter ratio = 14.7

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

Comment

Amitriptyline is the prototype of a tertiary amine tricyclic antidepressant drug. In humans, this medication is used in the treatment of anxiety, bipolar disorders and depression. In treating depression amitryptiline diplays also a sedation side-effect (Bryson & Wilde, 1996). By blocking the way cells of the nervous system transport amines, amitriptyline is able to increase the levels of circulating neurotransmitters, especially serotonin. It is metabolized to nortriptyline which inhibits the membrane pump mechanism responsible for uptake of norepinephrine and serotonin in adrenergic and serotonergic neurons. Pharmacologically this action may potentiate or prolong neuronal activity since reuptake of these biogenic amines is important physiologically in terminating transmitting activity. This interference with the reuptake of norepinephrine and/or serotonin is believed by some to underlie the antidepressant activity of amitriptyline. Sometimes it is also used to treat chronic pain, eating disorders and certain skin problems. It also can be used as a strong antihistaminic drug. Up to now, only the crystal structure of amitryptiline hydrochloride has been determined (Klein *et al.*, 1994). In view of the importance of amitriptyline, the structure of the title compound, (I), derived from the hydrolysis of the free base, is described (Fig. 1).



(I)

The cycloheptene ring is in a sofa conformation; the average endocyclic torsion angle of the ring is 34.8 (2)° (Table 1, Fig. 1). The overall molecular conformation of (I) is defined by the selected torsion angles in Table 1. The geometry of the alkylamino side chain is constrained by the intramolecular hydrogen bond between the hydroxyl group at C7 and dimethylaminopropyl N atom (Table 2, Fig. 1). The crystal packing is governed only by van der Waals interactions.

Experimental

Amitriptyline free base was obtained as a gift sample from Arvee Chem Pharma Private Limited, Mysore, India. Amitriptyline free base is susceptible to hydrolysis as it contains aliphatic double bonds (Henwood, 1967). When the free base (0.5 g) was recrystallized from ethanol (5 ml) the compound was hydrolysed owing to water present in the solvent. The hydrolysed compound (I) melts at 390 K.

Crystal data

$C_{20}H_{25}NO$
M_r = 295.41
Monoclinic, $P2_1/c$
a = 7.7278 (12) Å
b = 27.744 (3) Å
c = 8.6183 (15) Å
β = 113.841 (10)°
V = 1690.1 (5) Å^3
Z = 4
D_x = 1.161 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
Prism, colourless
0.15 × 0.15 × 0.10 mm

Data collection

Huber CS four-circle diffractometer
ω scans
Absorption correction: none
3353 measured reflections
2966 independent reflections
2115 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°
3 standard reflections
every 97 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.06
2966 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.2607P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected torsion angles (°).

C11—C5—C6—C7	0.9 (2)	C8—C9—C10—C11	63.9 (2)
C5—C6—C7—C8	56.88 (18)	C6—C5—C11—C10	−15.4 (3)
C6—C7—C8—C9	−65.58 (17)	C9—C10—C11—C5	−36.7 (2)
C7—C8—C9—C10	−4.3 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.90	2.7131 (18)	170

All H atoms were found in a difference map and refined isotropically using a riding model (C—H = 0.93–0.97 Å, O—H = 0.82 Å).

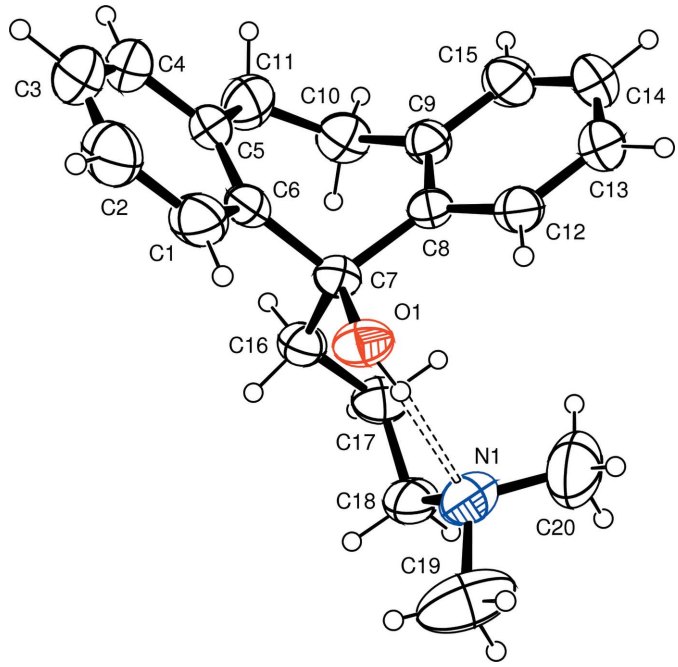


Figure 1
The molecular structure of (I) showing the atom-labelling scheme. Displacements ellipsoids are at the 50% probability level. The intramolecular hydrogen bond is indicated by a double dashed line.

The U_{iso} values of all the H atoms were set equal to 1.2 or 1.5 times U_{eq} of the parent atom.

Data collection: *XCS* (Colapietro *et al.*, 1992); cell refinement: *XCS*; data reduction: *XCS*; 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*.

SB thanks University of Mysore for research facilities and MAA thanks Arvee Chem Pharma Private Limited for the gift sample of Amitriptyline free base.

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.
Bryson, H. M. & Wilde, M. I. (1996). *Drugs Aging*, **8**, 459–476.
Colapietro, M., Cappuccio, G., Marciante, C., Pifferi, A., Spagna, R. & Helliwell, J. R. (1992). *J. Appl. Cryst.* **25**, 192–194.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Henwood, C. R. (1967). *Nature* (*London*), **216**, 1039–1040.
Klein, C. L., Lear, J., O'Rourke, S., Williams, S. & Liang, L. (1994). *J. Pharm. Sci.* **83**, 1253–1256.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.