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## Key indicators

Single-crystal X-ray study  
T = 298 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
R factor = 0.040  
wR factor = 0.108  
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

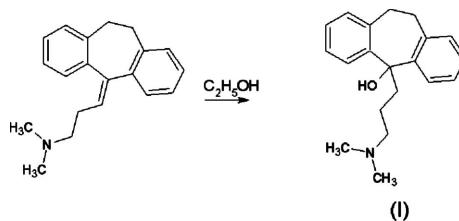
## 5-[3-(Dimethylamino)propyl]-10,11-dihydro-5H-dibenz[a,d][7]annulen-5-ol

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In the crystal structure of the title compound,  $\text{C}_{20}\text{H}_{25}\text{NO}$ , a derivative of amitriptyline, an antidepressant drug, an intramolecular  $\text{O}-\text{H} \cdots \text{N}$  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.

## 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 amitriptyline displays 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 anti-histaminic drug. Up to now, only the crystal structure of amitriptyline 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).



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

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

### Data collection

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

### Refinement

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

**Table 1**

Selected torsion angles ( $^\circ$ ).

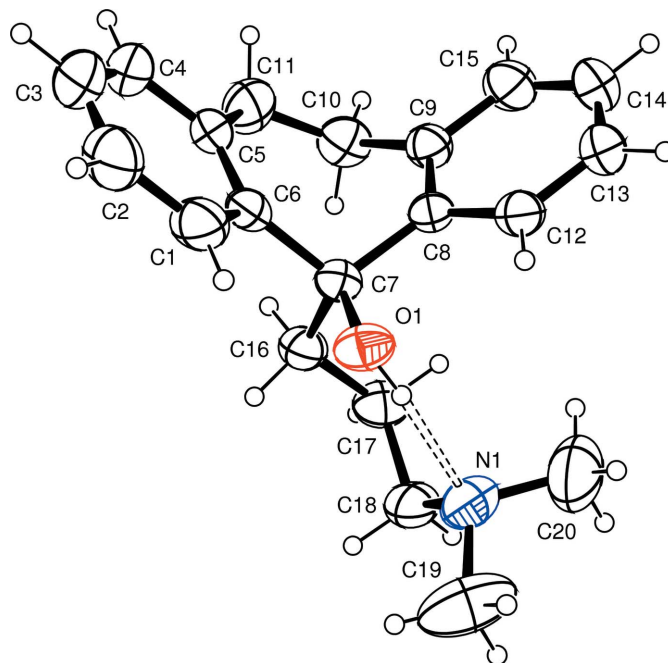
C11–C5–C6–C7	0.9 (2)	C8–C9–C10–C11	63.9 (2)
C5–C6–C7–C8	56.88 (18)	C6–C5–C11–C10	–15.4 (3)
C6–C7–C8–C9	–65.58 (17)	C9–C10–C11–C5	–36.7 (2)
C7–C8–C9–C10	–4.3 (2)		

**Table 2**

Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1 \cdots N1$	0.82	1.90	2.7131 (18)	170

All H atoms were found in a difference map and refined isotropically using a riding model ( $C-H = 0.93\text{--}0.97 \text{ \AA}$ ,  $O-H = 0.82 \text{ \AA}$ ).



**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  $U_{\text{iso}}$  values of all the H atoms were set equal to 1.2 or 1.5 times  $U_{\text{eq}}$  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.

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## 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., Marcianite, 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.