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3-Hydroxy-*N*,*N*-dimethyl-3-(2-thienyl)propanamine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.099
Data-to-parameter ratio = 15.0

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

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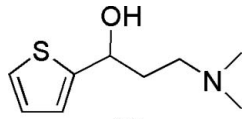
The title compound, $C_9H_{15}NOS$, was prepared by the reduction of 2-thienyl 2-(dimethylamino)ethyl ketone hydrochloride with sodium borohydride. In the crystal structure, O—H···N hydrogen bonds link pairs of molecules into centrosymmetric dimers.

Comment

Duloxetine is a pharmaceutical now under development as an antidepressant. It inhibits the uptake of both norepinephrine and serotonin and is presently in clinical evaluation (Wong *et al.*, 1988).



(I)

The title compound, (I), is the most important intermediate product in the preparation of duloxetine and is synthesized by the reduction of 2-thienyl 2-(dimethylamino)ethyl ketone hydrochloride with sodium borohydride. Its structure is reported here (Fig. 1). In the crystal structure, O—H···N hydrogen bonds link pairs of molecules into centrosymmetric dimers (Table 1).

Experimental

Colourless crystals of the title compound were prepared according to the methods of Berglund (1994) and Deeter *et al.* (1990). Crystals suitable for X-ray analysis were grown by slow evaporation of an absolute methanol solution at room temperature over a period of 20 d.

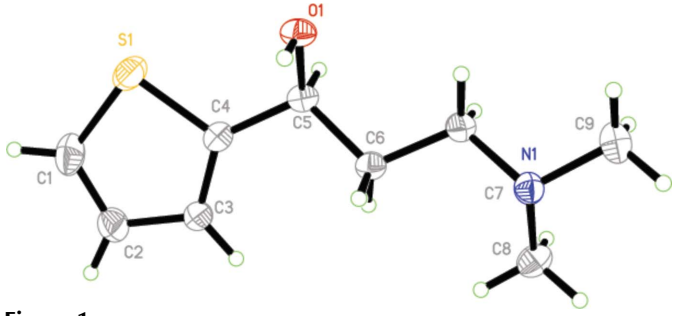


Figure 1
The molecular structure of (I), drawn with 30% probability displacement ellipsoids.

Crystal data

$C_9H_{15}NOS$
M_r = 185.28
Triclinic, $P\overline{1}$
a = 6.0670 (13) Å
b = 7.6467 (17) Å
c = 11.678 (3) Å
α = 96.004 (4)°
β = 104.036 (3)°
γ = 105.835 (3)°
V = 497.11 (19) Å^3
Z = 2
D_x = 1.238 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1735 reflections
θ = 2.8–26.3°
μ = 0.28 mm^{-1}
T = 294 (2) K
Block, colorless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.936, T_{max} = 0.951
2505 measured reflections
1726 independent reflections
1520 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°
$h = -7 \rightarrow 6$
$k = -9 \rightarrow 8$
$l = -7 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.06
1726 reflections
115 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.2073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.841 (10)	2.021 (10)	2.855 (2)	172 (2)

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

All H atoms attached to C atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). The OH hydrogen was located in a difference Fourier map and refined freely with an isotropic displacement parameter.

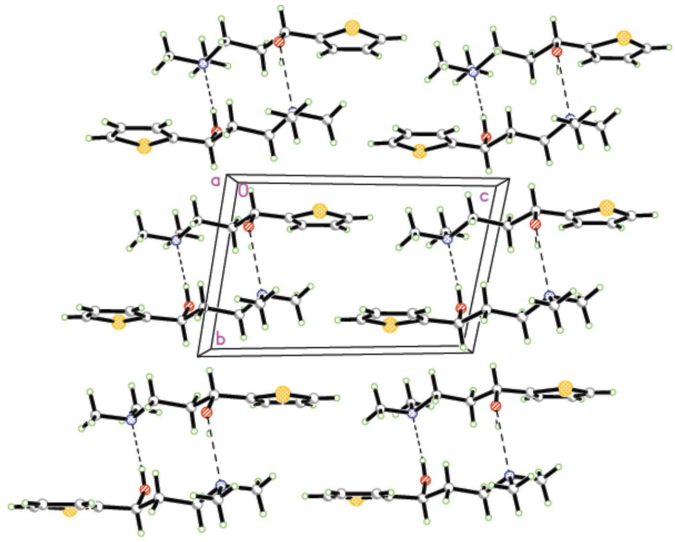


Figure 2
The crystal structure of (I), viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

Data collection: *SMART* (Bruker 1997); cell refinement: *SAINT* (Bruker 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 1997); software used to prepare material for publication: *SHELXTL*.

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