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Methyclothiazide: a hydrogen-bonded layer structure

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Key indicators

Single-crystal X-ray study
T = 187 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 15.4

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

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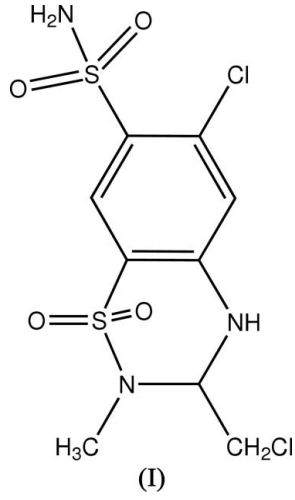
In the title compound, $C_9H_{11}Cl_2N_3O_4S_2$, systematic name 6-chloro-3-chloromethyl-3,4-dihydro-2-methyl-7-sulfamoyl-1,2,4-benzothiadiazine 1,1-dioxide, three kinds of N—H···O hydrogen bond generate a sheet structure. The combination of four N—H···O hydrogen bonds yields an $R_4^4(26)$ ring, with adjacent rings linked into two-dimensional layers parallel to the (001) plane. Pairs of adjacent planes are further connected by N—H···O hydrogen bonds.

Comment

Methyclothiazide (Fig. 1) is a well known diuretic and blood-pressure-lowering agent (Seller *et al.*, 1962). Although this drug is widely used in clinical therapy, its crystal structure has not yet been reported. The crystal structure of methyclothiazide, (I), is presented here.



(I)

In the crystal structure (Fig. 1), hydrogen bonds are observed as listed in Table 1. The combination of two N1—H1*B*···O4 hydrogen bonds and two N3—H3*A*···O2 hydrogen bonds generates an $R_4^4(26)$ ring. These $R_4^4(26)$ rings are linked to each other by sharing N—H···O hydrogen bonds, forming a two-dimensional layer parallel to the (001) plane. N1—H1*A*···O3 hydrogen bonds further connect two adjacent layers, forming pairs of sheets (Fig. 2).

Experimental

Methanol (3 ml, distilled from magnesium) was added to methyclothiazide (111 mg, 0.3 mmol, obtained from Jilin MaYinglong Pharmaceutical Co. Ltd). This mixture was heated under reflux until the methyclothiazide had dissolved. The solution was left to stand at room temperature for crystallization. After a few days colourless, prismatic crystals of methyclothiazide were obtained.

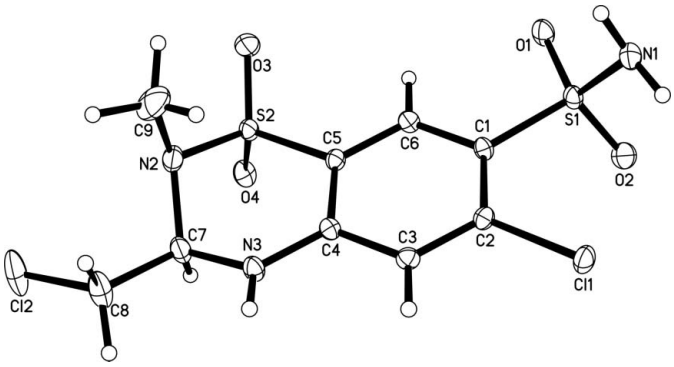


Figure 1
A view of methyclothiazide, showing the atom-labelling scheme and 30% probability displacement ellipsoids.

Crystal data

$C_9H_{11}Cl_2N_3O_4S_2$
M_r = 360.23
Orthorhombic, *Pbca*
a = 9.5251 (5) Å
b = 15.4476 (8) Å
c = 19.3226 (11) Å
V = 2843.1 (3) Å^3
Z = 8
D_x = 1.683 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 187 (2) K
Prism, colourless
0.41 × 0.24 × 0.22 mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SAINT*; Bruker, 2003)
T_{min} = 0.746, T_{max} = 0.849
14885 measured reflections
2794 independent reflections
2624 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.09
2794 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 2.8362P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.92	2.07	2.917 (2)	152
N1—H1B···O4^{ii}	0.90	2.25	3.114 (2)	162
N3—H3A···O2^{iii}	0.89	2.25	3.095 (2)	158

Symmetry codes: (i) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (ii) $x-1, y, z$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

H atoms bonded to N1 and N3 were found in a difference map and fixed in these positions, with N—H distances of 0.89–0.92 Å. Other H atoms were positioned geometrically and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C,N)] using a riding model, with C—H distances of 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (CH).

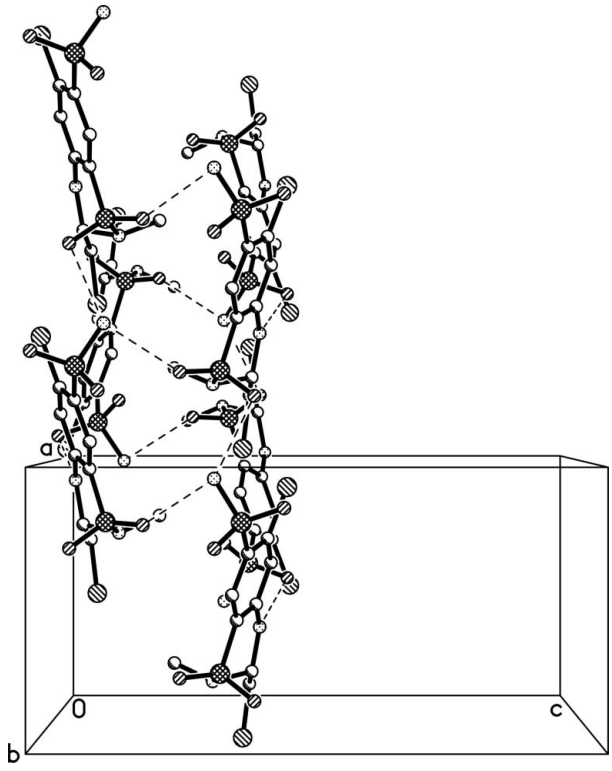


Figure 2
A view showing the hydrogen bonds (dashed lines) linking a pair of layers parallel to the (001) plane. For the sake of clarity, H atoms have been omitted.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2003); 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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