

4-Acetamido-*N*-(4-methylpiperazinyl)benzenesulfonamide monohydrate

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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.042
 wR factor = 0.114
Data-to-parameter ratio = 17.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{13}\text{H}_{20}\text{N}_4\text{O}_3\text{S}\cdot\text{H}_2\text{O}$, is V-shaped and entraps a water molecule *via* an $\text{O}-\text{H}\cdots\text{N}$ and an $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond. In the crystal structure, the packing of the molecules is stabilized by $\text{N}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds and van der Waals forces.

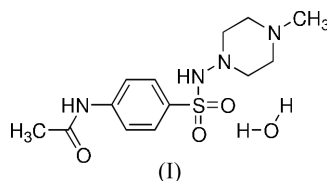
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Comment

Piperazine and its derivatives are important targets for drug discovery. The synthesis and crystal structure reported here are part of this study (Guo, 2004).



The molecular structure of the title compound, (I), is illustrated in Fig. 1. The bond distances and angles are normal, within experimental error.

In the crystal structure, symmetry-related molecules are linked by hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 4-acetamidophenylsulfonyl chloride (3.7 g) with 1-amino-4-methylpiperazine (1.8 g) under microwave irradiation for 2 min. The resulting product was dispersed in cold water (40 ml), after which 2.4 g of the colourless powder product was separated by filtration.

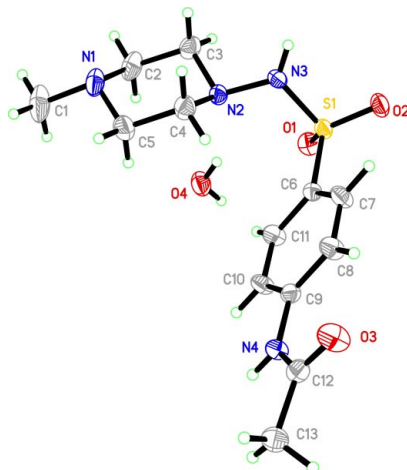


Figure 1

The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

Pure *N*-(4-methylpiperazinyl)-4-acetamidophenylsulfonamide (1.5 g) was heated and dissolved in water (20 ml). A single crystal was obtained after 8 h at room temperature.

Crystal data

$C_{13}H_{20}N_4O_3S \cdot H_2O$
 $M_r = 330.41$
 Monoclinic, $C2/c$
 $a = 28.637 (14) \text{ \AA}$
 $b = 7.140 (3) \text{ \AA}$
 $c = 19.799 (11) \text{ \AA}$
 $\beta = 124.688 (14)^\circ$
 $V = 3329 (3) \text{ \AA}^3$
 $Z = 8$

$D_x = 1.319 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 966 reflections
 $\theta = 3.4\text{--}26.5^\circ$
 $\mu = 0.22 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Block, colourless
 $0.22 \times 0.18 \times 0.14 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.965$, $T_{\max} = 0.980$
 13 322 measured reflections

3458 independent reflections
 2800 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 26.5^\circ$
 $h = -36 \rightarrow 36$
 $k = -8 \rightarrow 8$
 $l = -24 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.114$
 $S = 1.03$
 3458 reflections
 201 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 3.7269P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3 \cdots O3^i$	0.86	2.39	2.888 (2)	118
$N4-H4 \cdots O4^{ii}$	0.86	2.05	2.902 (3)	170
$O4-H4C \cdots N1^{iii}$	0.85	1.97	2.795 (3)	164
$O4-H4D \cdots O1$	0.85	2.33	2.996 (3)	135
$O4-H4D \cdots N2$	0.85	2.64	3.411 (3)	152

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

The H atoms of the water molecule were found in difference Fourier maps. However, during refinement, they were fixed at O—H distances of 0.85 \AA and their U_{iso} values were set at $1.2U_{\text{eq}}(\text{O})$. The H atoms of NH and CH groups were treated as riding, with N—H = 0.86 \AA and C—H = $0.93\text{--}0.97 \text{ \AA}$. For the H atoms attached to atom

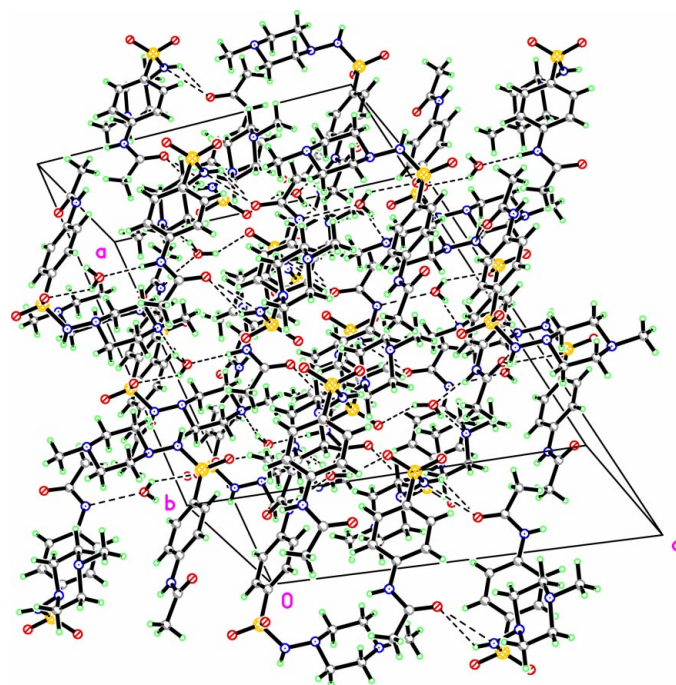


Figure 2

Packing diagram of (I), showing the hydrogen bonding (dashed lines).

$C1$, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C1})$, otherwise $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent N or C atom})$.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997); program(s) used to solve structure: SHELXTL (Sheldrick, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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