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A new polymorph of sulfanilic acid monohydrate

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An orthorhombic polymorph of sulfanilic acid monohydrate, $C_6H_7NO_3S{\cdot}H_2O$, is described in which there are significant hydrogen-bonding interactions between the components of the structure.

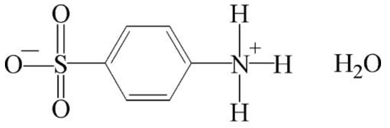
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 8.4

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

Comment

The crystal structure of a monoclinic form ($P2_1/n$) of sulfanilic acid monohydrate, (II), has been described (Rae & Maslen, 1962). Here, the structure of an orthorhombic form, (I) ($P2_12_12_1$), obtained by recrystallization from a methanol solution of the compound, is described (Fig. 1 and Table 1).



(I)

The C—S and C—N bond lengths in (I) (Table 1) are close to the corresponding distances in (II) and $O_3SC_6H_4NH\text{–}CH\text{–}N(CH_3)_2{\cdot}H_2O$ (Hempel *et al.*, 1999). The S—O bond distances in (I) are similar to those found in (II) (Rae & Maslen, 1962), in metanilic acid (Hall & Maslen, 1965), and in 2,5-dichlorobenzenesulfonic acid and 2,5-dibromobenzenesulfonic acid (Lundgren & Lundin, 1972). The C—S—O and O—S—O angles deviate from 109.5° in the expected manner.

The crystal structure of (I) is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds (Table 2), which result in the formation of a hydrogen-bonded network (Fig. 2). The water molecule is hydrogen bonded to the amine group (N1/H1*B*). The distance between the two parallel structures, with symmetry (1 + *x*, *y*, *z*), in the packing diagram (Fig. 2) is 6.163 (3) Å.

Experimental

Sulfanilic acid (1.732 g, 1 mmol) was dissolved in methanol (20 ml) and stirred for 1 h. After filtration, the clear solution was left for crystallization, and after two weeks, pale-yellow crystals were obtained.

Crystal data

$C_6H_7NO_3S{\cdot}H_2O$
$M_r = 191.20$
Orthorhombic, $P2_12_12_1$
$a = 6.1630\ (6)$ Å
$b = 6.9607\ (5)$ Å
$c = 18.3251\ (10)$ Å
$V = 786.12\ (10)$ Å^3
$Z = 4$
$D_x = 1.616$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.39$ mm^{-1}
$T = 150\ (2)$ K
Block, pale yellow
0.25 × 0.22 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: part of the refinement model (ΔF) (Walker & Stuart, 1983)
$T_{\min} = 0.910$, $T_{\max} = 0.927$
1822 measured reflections
957 independent reflections
793 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 26.3°$
3 standard reflections every 134 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.092$
$S = 1.04$
957 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.1313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.448 (3)	S1—C1	1.773 (3)
S1—O2	1.459 (3)	N1—C4	1.468 (4)
S1—O3	1.446 (3)		
O1—S1—O2	111.64 (19)	O2—S1—O3	112.15 (15)
O1—S1—O3	113.77 (19)	O2—S1—C1	105.00 (15)
O1—S1—C1	106.26 (15)	O3—S1—C1	107.36 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O1$^{\rm i}$	0.95	1.90	2.821 (3)	163
O4—H4B···O2$^{\rm ii}$	0.95	1.89	2.838 (4)	175
N1—H1A···O3$^{\rm iii}$	0.94	1.97	2.846 (4)	154
N1—H1B···O4	0.93	1.84	2.738 (3)	160
N1—H1C···O2$^{\rm iv}$	0.96	1.95	2.895 (4)	166

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

In the absence of significant anomalous scattering, Friedel pairs were merged before the final refinement. C-bound H atoms were included in the riding model approximation with C—H = 0.95 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. H atoms attached to N and O(water) were located from an electron density map, fixed in these positions and assigned individual isotropic displacement parameters; see Table 2 for bond distances.

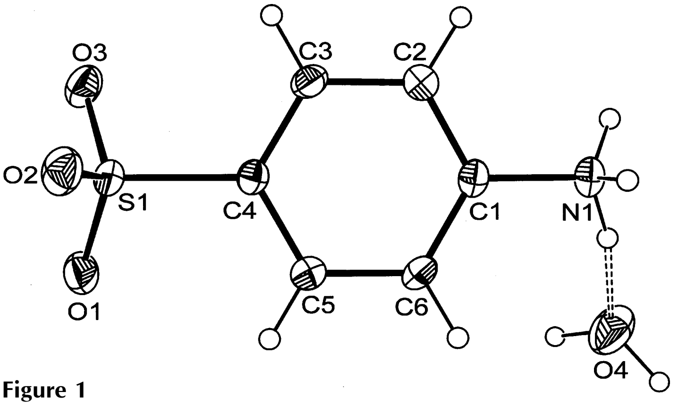


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. The hydrogen bond is shown as a dashed line.

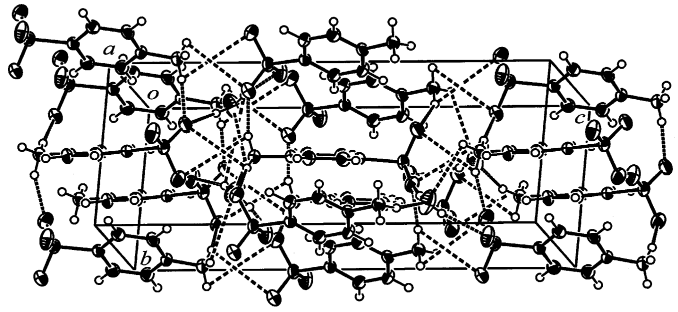


Figure 2
The molecular packing of (I), viewed approximately along the *a* axis. Dashed lines indicate the hydrogen-bonding interactions.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *CAD-4 Processing Program* (Hursthouse, 1976); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Enraf–Nonius (1992). *CAD-4 EXPRESS*. Enraf-Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hall, S. R. & Maslen, E. N. (1965). *Acta Cryst.* **18**, 301–306.
Hempel, A., Camerman, N., Mastropaolo, D. & Camerman, A. (1999). *Acta Cryst.* C**55**, 697–698.
Hursthouse, M. B. (1976). *CAD-4 Processing Program*. Queen Mary College, London.
Lundgren, J.-O. & Lundin, P. (1972). *Acta Cryst.* B**28**, 486–491.
Rae, A. I. M. & Maslen, E. N. (1962). *Acta Cryst.* **15**, 1285–1291.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Gottingen, Germany.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.