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A new polymorph of sulfanilic acid monohydrate

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Key indicators

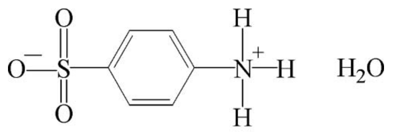
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 8.4

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

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.

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 $O_3SC_6H_4NH–CH–N(CH_3)_2{\cdot}H_2O$ (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
μ = 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.

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).

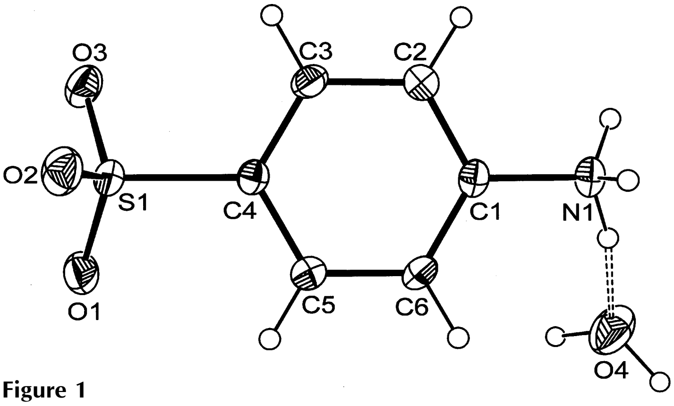


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. The hydrogen bond is shown as a dashed line.

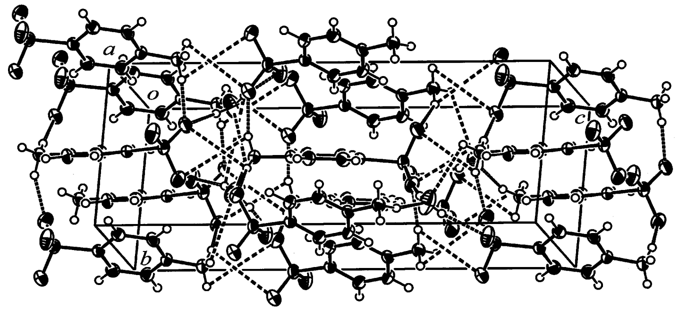


Figure 2
The molecular packing of (I), viewed approximately along the *a* axis. Dashed lines indicate the hydrogen-bonding interactions.

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A new polymorph of sulfanilic acid monohydrate

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sulfanilic acid monohydrate

Crystal data

$C_6H_7NO_3S{\cdot}H_2O$
$M_r = 191.20$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.1630$ (6) Å
$b = 6.9607$ (5) Å
$c = 18.3251$ (10) Å
$V = 786.12$ (10) Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.616$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 957 reflections
$\theta = 2.9–26.3°$
$\mu = 0.39$ mm^{-1}
$T = 150$ K
Block, pale yellow
0.25 × 0.22 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω/θ scans
Absorption correction: part of the refinement model (ΔF) (Walker & Stuart, 1983)
$T_{\min} = 0.910$, $T_{\max} = 0.927$
3 measured reflections
957 independent reflections
793 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 26.3°$, $\theta_{\min} = 2.2°$
$h = -7 \rightarrow 0$
$k = -8 \rightarrow 2$
$l = -22 \rightarrow 22$
1534 standard reflections every 134 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.092$
$S = 1.04$
957 reflections
114 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}
Absolute structure: Flack (1983)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	−0.01053 (14)	0.48483 (12)	0.63841 (4)	0.0291 (2)
O1	0.2228 (4)	0.5066 (5)	0.63447 (13)	0.0463 (7)
O2	−0.1130 (5)	0.6505 (4)	0.67294 (13)	0.0386 (7)
O3	−0.0795 (5)	0.3055 (4)	0.67078 (13)	0.0428 (7)
O4	0.0515 (4)	0.6429 (5)	0.25576 (15)	0.0663 (11)
N1	−0.3138 (4)	0.5290 (4)	0.32896 (13)	0.0307 (7)
C1	−0.1055 (5)	0.4873 (4)	0.54710 (15)	0.0252 (7)
C2	−0.3124 (6)	0.5482 (5)	0.53154 (17)	0.0303 (8)
H2	−0.4084	0.5827	0.5699	0.036*
C3	−0.3807 (6)	0.5591 (5)	0.45936 (17)	0.0307 (8)
H3	−0.5234	0.6011	0.4480	0.037*
C4	−0.2392 (5)	0.5081 (5)	0.40465 (16)	0.0273 (7)
C5	−0.0359 (6)	0.4401 (5)	0.41925 (16)	0.0304 (8)
H5	0.0554	0.4006	0.3803	0.036*
C6	0.0381 (4)	0.4282 (4)	0.49055 (13)	0.0299 (8)
H6	0.1797	0.3823	0.5013	0.036*
H1A	−0.3781	0.4113	0.3158	0.053 (13)*
H1B	−0.1976	0.5436	0.2970	0.041 (11)*
H1C	−0.4142	0.6317	0.3197	0.059 (14)*
H4A	0.1434	0.6163	0.2156	0.12 (2)*
H4B	0.1615	0.7184	0.2777	0.15 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0329 (4)	0.0347 (4)	0.0195 (3)	0.0022 (5)	−0.0003 (3)	0.0014 (4)
O1	0.0293 (13)	0.079 (2)	0.0311 (12)	−0.0029 (16)	−0.0032 (10)	0.0047 (17)
O2	0.0500 (18)	0.0387 (13)	0.0270 (12)	0.0017 (14)	0.0012 (14)	−0.0070 (11)
O3	0.0549 (19)	0.0419 (14)	0.0315 (13)	0.0020 (14)	0.0023 (15)	0.0098 (11)
O4	0.052 (2)	0.096 (3)	0.0513 (16)	−0.025 (2)	0.0144 (17)	−0.0228 (18)
N1	0.0376 (16)	0.0327 (16)	0.0217 (12)	−0.0033 (15)	−0.0041 (13)	0.0000 (12)
C1	0.0299 (16)	0.0241 (15)	0.0216 (13)	−0.0018 (18)	0.0004 (12)	0.0007 (13)
C2	0.0306 (18)	0.0334 (18)	0.0268 (16)	0.0028 (16)	0.0057 (14)	−0.0008 (14)
C3	0.0296 (19)	0.0331 (17)	0.0294 (16)	0.0016 (16)	−0.0019 (14)	−0.0002 (14)
C4	0.0353 (17)	0.0254 (16)	0.0212 (15)	0.0001 (17)	−0.0030 (12)	−0.0001 (15)
C5	0.0321 (19)	0.0353 (17)	0.0237 (14)	0.0036 (15)	0.0054 (15)	−0.0067 (13)

C6	0.031 (2)	0.0305 (16)	0.0284 (15)	0.0041 (15)	0.0018 (15)	−0.0026 (13)

Geometric parameters (Å, º)

S1—O1	1.448 (3)	C1—C2	1.374 (5)
S1—O2	1.459 (3)	C1—C6	1.424 (4)
S1—O3	1.446 (3)	C2—C3	1.390 (4)
S1—C1	1.773 (3)	C2—H2	0.9500
O4—H4A	0.9476	C3—C4	1.376 (5)
O4—H4B	0.9474	C3—H3	0.9500
N1—C4	1.468 (4)	C4—C5	1.366 (5)
N1—H1A	0.9415	C5—C6	1.386 (4)
N1—H1B	0.9315	C5—H5	0.9500
N1—H1C	0.9606	C6—H6	0.9500
O1—S1—O2	111.64 (19)	C1—C2—C3	119.7 (3)
O1—S1—O3	113.77 (19)	C1—C2—H2	120.2
O1—S1—C1	106.26 (15)	C3—C2—H2	120.2
O2—S1—O3	112.15 (15)	C4—C3—C2	119.2 (3)
O2—S1—C1	105.00 (15)	C4—C3—H3	120.4
O3—S1—C1	107.36 (15)	C2—C3—H3	120.4
H4A—O4—H4B	90.5	C5—C4—C3	121.9 (3)
C4—N1—H1A	106.8	C5—C4—N1	120.4 (3)
C4—N1—H1B	111.4	C3—C4—N1	117.7 (3)
H1A—N1—H1B	104.9	C4—C5—C6	120.5 (3)
C4—N1—H1C	116.3	C4—C5—H5	119.8
H1A—N1—H1C	109.3	C6—C5—H5	119.8
H1B—N1—H1C	107.6	C5—C6—C1	117.7 (3)
C2—C1—C6	121.0 (3)	C5—C6—H6	121.2
C2—C1—S1	120.4 (2)	C1—C6—H6	121.2
C6—C1—S1	118.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4a···O1^{i}	0.95	1.90	2.821 (3)	163
O4—H4b···O2^{ii}	0.95	1.89	2.838 (4)	175
N1—H1a···O3^{iii}	0.94	1.97	2.846 (4)	154
N1—H1b···O4	0.93	1.84	2.738 (3)	160
N1—H1c···O2^{iv}	0.96	1.95	2.895 (4)	166

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