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Bis[5-oxo-4,5-dihydro-8*H*-2-azonia-4,8,9-trizabicyclo[4.3.0]nona-2,6,9(1)-triene] sulfate

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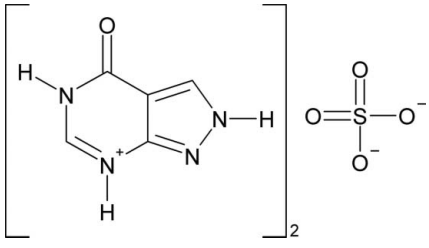
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 10.0.

In the crystal structure of the title compound, $2C_5H_5N_4O^+\cdot SO_4^{2-}$, N—H···O hydrogen bonds assemble the molecules into a two-dimensional network structure parallel to the *cb* plane. The S atom of the sulfate ion lies on a special position on a twofold axis.

Related literature

For general background, see: Elion *et al.* (1962); Rundles *et al.* (1966). For related structures, see: Prusiner & Sundaralingam (1972); Gadret *et al.* (1974); Sheldrick & Bell (1987); Singh & Pedersen (1993).



Experimental

Crystal data

$2C_5H_5N_4O^+\cdot SO_4^{2-}$
M_r = 370.32
Monoclinic, $C2/c$
a = 12.337 (3) Å
b = 10.054 (2) Å
c = 11.064 (2) Å
β = 102.42 (3)°
V = 1340.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 298 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
3561 measured reflections
1323 independent reflections
1252 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.05
1323 reflections
132 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3···O3^{i}	0.936 (10)	1.698 (14)	2.628 (2)	173 (3)
N3—H5···O2	0.819 (10)	1.957 (18)	2.753 (3)	164 (3)
N1—H1···O3^{ii}	0.906 (10)	1.838 (13)	2.716 (3)	163 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2054).

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supporting information

Acta Cryst. (2008). E**64**, o2411 [doi:10.1107/S1600536808038026]

Bis[5-oxo-4,5-dihydro-8*H*-2-azonia-4,8,9-trizabicyclo[4.3.0]nona-2,6,9(1)-triene] sulfate

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S1. Comment

Allopurinol is known to be a potent inhibitor of xanthine oxidase and is used extensively for treatment of gout (Rundles *et al.*, 1966). It is used in conjunction with anticancer drugs which impede RNA biosynthesis and as adjunct therapy in conjunction with 6-mecaptopurine in treatment of leukemia (Elion *et al.*, 1962). As part of our interest in salts and co-crystals of drugs, we have investigated the crystal structure of Allopurinol sulfate, (I) (Fig. 1). The least-squares plane of the six-membered ring makes an angle of 5.79 (11)° with the least-squares planes of the five-membered ring of the purine ring. The N3 nitrogen is protonated similar to the reported structure of chloride salt (Sheldrick & Bell, 1987). The molecules are linked *via* N—H···O hydrogen bonds,forming two-dimensional infinite chains along the *cb* plane. These sheets are linked together by the sulfate molecules which act as acceptors of H atoms, assembling the molecules in an infinite two-dimensional network (Fig. 2).

S2. Experimental

Allopurinol (0.1361 g, 1 mmol) and Sulfuric acid (0.27 ml, 5 mmol) were added to Dimethylformamide. The mixture was stirred and heated on a water bath. After solution was complete, it was filtered and allowed to evaporate at room temperature. Colorless plate like crystals, suitable for *x*-ray analysis grew over a period of two days when the solution was exposed to air.

S3. Refinement

Atoms H1, H3 and H5 on N1, N2 and N3 respectively were located in difference Fourier maps and refined isotropically. All other H atoms were found in difference map but then placed in calculated positions with C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C) for all aromatic H atoms.

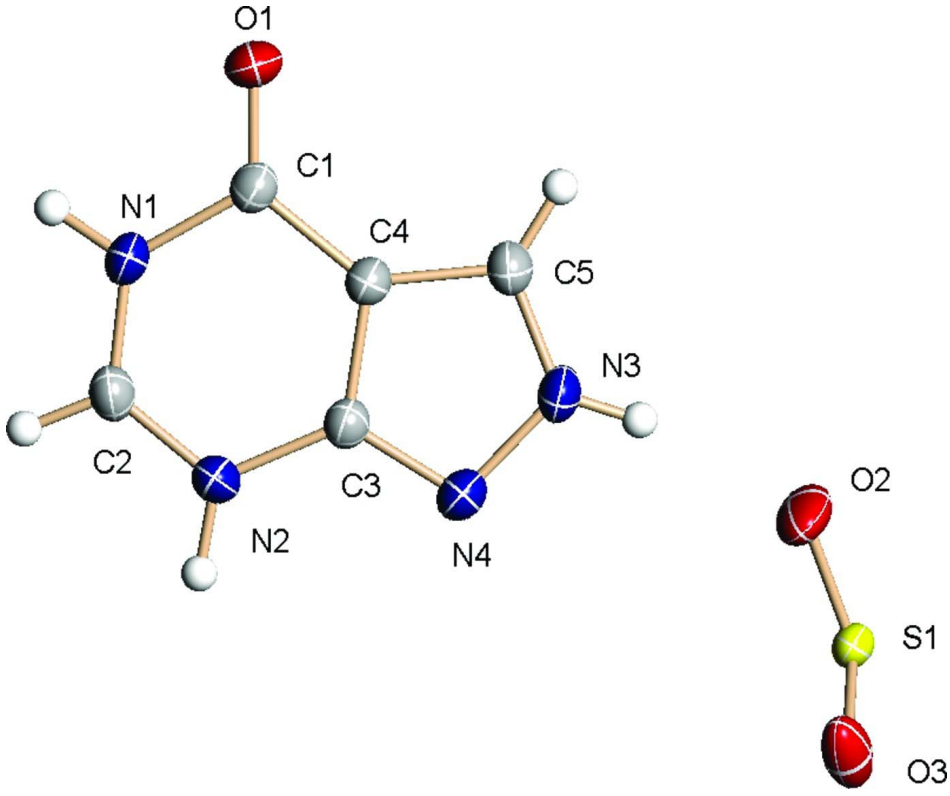

Figure 1

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

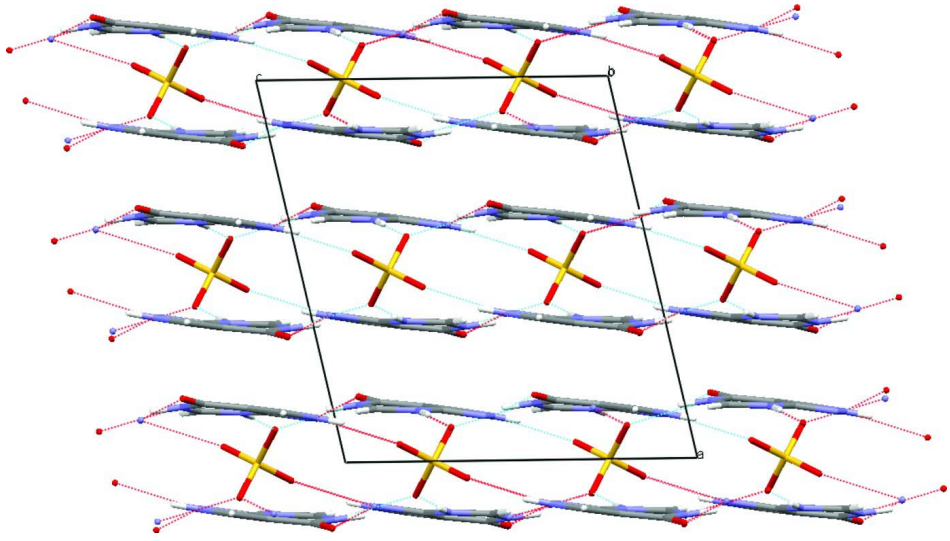

Figure 2

The packing of (I), viewed down the *b* axis, showing the layers of molecules and sulfate molecules connected by N—H···O hydrogen bonds (dashed lines).

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Crystal data

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$M_r = 370.32$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 12.337$ (3) Å
$b = 10.054$ (2) Å
$c = 11.064$ (2) Å
$\beta = 102.42$ (3)°
$V = 1340.2$ (5) Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.835$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1323 reflections
$\theta = 2.6$–$26.1°$
$\mu = 0.30$ mm^{-1}
$T = 298$ K
Plate, colourless
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
3561 measured reflections
1323 independent reflections
1252 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 26.1°$, $\theta_{min} = 2.6°$
$h = -15\rightarrow11$
$k = -10\rightarrow12$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.096$
$S = 1.05$
1323 reflections
132 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.9856P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0068 (13)

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	0.5000	0.24338 (5)	0.2500	0.0248 (2)
O3	0.41241 (12)	0.33107 (12)	0.17777 (11)	0.0458 (4)
O2	0.45510 (13)	0.16122 (14)	0.33449 (12)	0.0472 (4)

O1	0.32972 (10)	0.07703 (11)	0.91973 (10)	0.0354 (3)
N1	0.35100 (11)	0.30200 (13)	0.92837 (12)	0.0279 (3)
C4	0.35629 (12)	0.19500 (15)	0.73986 (13)	0.0244 (3)
N3	0.38403 (11)	0.18821 (14)	0.55266 (13)	0.0289 (3)
N4	0.38369 (11)	0.32129 (13)	0.57759 (12)	0.0290 (3)
N2	0.36693 (11)	0.43601 (13)	0.76273 (12)	0.0281 (3)
C1	0.34391 (12)	0.17773 (15)	0.86560 (14)	0.0254 (3)
C3	0.36700 (12)	0.32242 (14)	0.69190 (14)	0.0245 (3)
C5	0.36919 (13)	0.11072 (16)	0.64455 (14)	0.0275 (3)
C2	0.36240 (13)	0.41996 (16)	0.87928 (14)	0.0287 (4)
H2	0.3674 (16)	0.4935 (19)	0.9297 (18)	0.033 (5)*
H4	0.3710 (16)	0.018 (2)	0.6442 (18)	0.036 (5)*
H3	0.3781 (18)	0.522 (2)	0.735 (2)	0.049 (6)*
H5	0.3967 (18)	0.168 (2)	0.485 (2)	0.045 (6)*
H1	0.3606 (18)	0.299 (2)	1.012 (2)	0.044 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0361 (3)	0.0221 (3)	0.0175 (3)	0.000	0.0085 (2)	0.000
O3	0.0595 (8)	0.0395 (7)	0.0306 (6)	0.0168 (6)	−0.0078 (6)	−0.0102 (5)
O2	0.0712 (9)	0.0445 (8)	0.0339 (7)	−0.0155 (7)	0.0293 (7)	−0.0026 (6)
O1	0.0502 (7)	0.0289 (6)	0.0290 (6)	−0.0007 (5)	0.0131 (5)	0.0064 (5)
N1	0.0360 (7)	0.0293 (7)	0.0196 (7)	0.0007 (5)	0.0085 (5)	−0.0018 (5)
C4	0.0280 (7)	0.0244 (8)	0.0212 (7)	−0.0003 (5)	0.0063 (6)	−0.0002 (5)
N3	0.0348 (7)	0.0323 (8)	0.0208 (6)	0.0000 (5)	0.0088 (5)	−0.0051 (5)
N4	0.0354 (7)	0.0306 (7)	0.0220 (7)	0.0002 (5)	0.0081 (5)	0.0016 (5)
N2	0.0372 (7)	0.0218 (7)	0.0256 (7)	0.0006 (5)	0.0075 (5)	0.0001 (5)
C1	0.0268 (7)	0.0279 (8)	0.0220 (7)	0.0008 (6)	0.0065 (6)	−0.0001 (5)
C3	0.0267 (7)	0.0259 (8)	0.0209 (7)	0.0008 (5)	0.0051 (6)	−0.0011 (5)
C5	0.0320 (8)	0.0264 (8)	0.0246 (7)	−0.0005 (6)	0.0071 (6)	−0.0044 (6)
C2	0.0333 (8)	0.0273 (8)	0.0251 (7)	0.0017 (6)	0.0056 (6)	−0.0052 (6)

Geometric parameters (Å, º)

S1—O2^{i}	1.4442 (12)	C4—C1	1.442 (2)
S1—O2	1.4443 (12)	N3—C5	1.324 (2)
S1—O3	1.4873 (13)	N3—N4	1.366 (2)
S1—O3^{i}	1.4873 (13)	N3—H5	0.82 (2)
O1—C1	1.2081 (18)	N4—C3	1.324 (2)
N1—C2	1.324 (2)	N2—C2	1.313 (2)
N1—C1	1.423 (2)	N2—C3	1.3851 (19)
N1—H1	0.91 (2)	N2—H3	0.94 (2)
C4—C5	1.388 (2)	C5—H4	0.94 (2)
C4—C3	1.404 (2)	C2—H2	0.92 (2)
O2^{i}—S1—O2	110.23 (11)	C2—N2—C3	117.38 (13)
O2^{i}—S1—O3	109.07 (8)	C2—N2—H3	118.7 (13)

O2—S1—O3	110.57 (9)	C3—N2—H3	123.5 (13)
O2^{i}—S1—O3^{i}	110.57 (9)	O1—C1—N1	119.61 (14)
O2—S1—O3^{i}	109.07 (8)	O1—C1—C4	129.47 (14)
O3—S1—O3^{i}	107.30 (11)	N1—C1—C4	110.91 (13)
C2—N1—C1	125.94 (13)	N4—C3—N2	124.51 (14)
C2—N1—H1	116.4 (14)	N4—C3—C4	113.56 (13)
C1—N1—H1	116.7 (14)	N2—C3—C4	121.80 (13)
C5—C4—C3	103.56 (13)	N3—C5—C4	106.34 (14)
C5—C4—C1	135.43 (14)	N3—C5—H4	125.2 (12)
C3—C4—C1	120.84 (13)	C4—C5—H4	128.4 (12)
C5—N3—N4	114.47 (13)	N2—C2—N1	122.93 (14)
C5—N3—H5	129.3 (15)	N2—C2—H2	119.1 (12)
N4—N3—H5	116.1 (15)	N1—C2—H2	118.0 (12)
C3—N4—N3	102.07 (12)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H3···O3^{ii}	0.936 (10)	1.698 (14)	2.628 (2)	173 (3)
N3—H5···O2	0.819 (10)	1.957 (18)	2.753 (3)	164 (3)
N1—H1···O3^{iii}	0.906 (10)	1.838 (13)	2.716 (3)	163 (3)

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