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A Triazine Derivative

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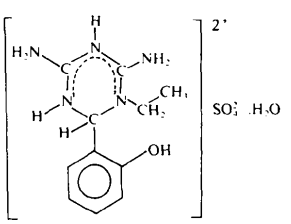
Abstract

In 4,6-diamino-1-ethyl-2-(2-hydroxyphenyl)-1,2-dihydro-3H^+,5H^+-1,3,5-triazinium sulfate monohydrate, $C_{11}H_{17}N_5O^{2+}.SO_4^{2-}.H_2O$, the sulfate group and the triazine ring are bridged *via* N—H···O hydrogen bonds. The O atom of the water molecule forms O—H···O hydrogen bonds with the sulfate moieties of two neighbouring units and O···H—N hydrogen bonds with two other neighbouring units.

Comment

Dihydrotriazines are of interest because of their antimalarial and anticancer activities (Katritzky & Rees, 1984). The Cu^{II} complex of the title compound (I) was prepared by refluxing Cu(*N*-ethylbiguanide) base with salicylaldehyde. The decomposition of the complex with H_2SO_4 (6 *N*) yielded (I) which forms interesting complexes with *d*-block metal ions (Saha, Karak & Santra, 1992). Single crystals were obtained by evaporation from an aqueous solution.



(I)

Fig. 1 shows an *ORTEP*II plot of the molecule (Johnson, 1976). The intramolecular hydrogen bond between N(7) and O(1) has the parameters N(7)—H(7.2)···O(1) = 2.743 (9), O(1)···H(7.2) = 1.859 (73) and N(7)—H(7.2) = 0.960 (68) Å. The intermolecular bonds involving the water of crystallization are

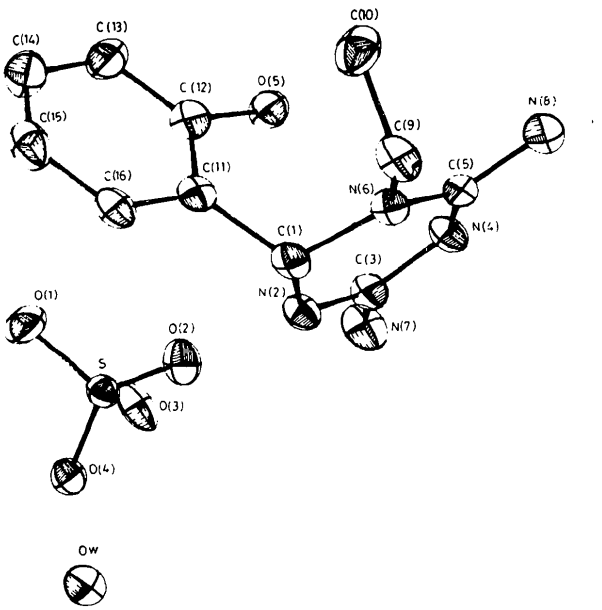


Fig. 1. Numbering scheme and displacement ellipsoids drawn at the 50% probability level.

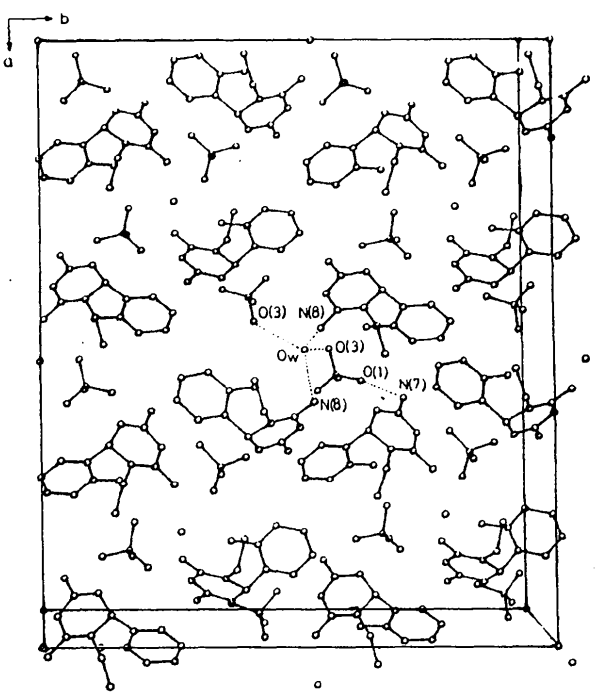


Fig. 2. Packing of molecules in the unit cell with the intermolecular hydrogen bonds marked by dotted lines.

N(8)(x, y, z)···OW($\frac{1}{4}-x$, $\frac{1}{4}+y$, $\frac{1}{4}-z$) = 2.797 (8) Å and OW(x, y, z)···O(3)(x, y, $1+z$) = 2.693 (8) Å. Fig. 2 shows the packing of molecules in the unit cell. The bond lengths and bond angles of the biguanide part of the triazine ring are comparable with those of biguanide salts (Pinkerton & Schwarzenbach, 1978).

Experimental

Crystal data

$C_{11}H_{17}N_5O^{2+}.SO_4^{2-}.H_2O$
M_r = 349.4
Orthorhombic
*Fdd*2
a = 31.346 (1) Å
b = 26.838 (6) Å
c = 7.293 (5) Å
V = 6135.3 (3) Å^3
Z = 16
D_x = 1.51 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 16–28°
μ = 2.2 mm^{-1}
T = 300 K
Prismatic
0.60 × 0.40 × 0.32 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1675 measured reflections
1675 independent reflections
1191 observed reflections [$I > 3\sigma(I)$]
θ_{max} = 70°
h = 0 → 8
k = 0 → 30
l = 0 → 38
2 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F
R = 0.039
wR = 0.040
1191 reflections
266 parameters
$w = 1/[\sigma^2(F) + 0.005533F^2]$
$(\Delta/\sigma)_{max}$ = 0.296
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S	0.06666 (2)	0.08686 (3)	0.68920	0.0318 (5)
O(1)	0.0787 (1)	0.1371 (1)	0.6372 (6)	0.0469 (18)
O(2)	0.0645 (1)	0.0831 (1)	0.8916 (6)	0.0504 (23)
O(3)	0.0242 (1)	0.0748 (1)	0.6155 (6)	0.0461 (17)
O(4)	0.0975 (1)	0.0507 (1)	0.6223 (7)	0.0581 (21)
O(5)	0.2012 (1)	0.1457 (1)	0.9913 (6)	0.0446 (19)
OW	0	0	1.3925 (6)	0.0358 (19)
N(2)	0.1108 (1)	0.1491 (1)	1.1003 (6)	0.0347 (17)
N(4)	0.1453 (1)	0.2244 (1)	1.1245 (6)	0.0333 (16)
N(6)	0.1606 (1)	0.1648 (1)	1.3447 (6)	0.0323 (17)
N(7)	0.1000 (1)	0.2100 (2)	0.8846 (7)	0.0476 (22)
N(8)	0.1920 (1)	0.2434 (1)	1.3522 (6)	0.0387 (18)
C(1)	0.1363 (1)	0.1258 (1)	1.2424 (6)	0.0308 (18)
C(3)	0.1185 (1)	0.1935 (1)	1.0333 (6)	0.0327 (19)
C(5)	0.1667 (1)	0.2100 (1)	1.2767 (6)	0.0312 (18)
C(9)	0.1829 (1)	0.1481 (2)	1.5112 (8)	0.0408 (24)
C(10)	0.2288 (2)	0.1320 (2)	1.4743 (8)	0.0505 (27)
C(11)	0.1654 (1)	0.0860 (1)	1.1671 (6)	0.0330 (21)
C(12)	0.1977 (1)	0.0973 (1)	1.0434 (7)	0.0375 (20)
C(13)	0.2250 (1)	0.0605 (2)	0.9802 (8)	0.0474 (25)
C(14)	0.2192 (2)	0.0110 (2)	1.0402 (9)	0.0534 (28)
C(15)	0.1871 (1)	−0.0008 (2)	1.1609 (9)	0.0513 (29)
C(16)	0.1602 (1)	0.0365 (1)	1.2238 (7)	0.0413 (23)

Table 2. *Selected geometric parameters* (Å, °)

S—O(1)	1.453 (2)	C(3)—N(2)	1.311 (4)
S—O(2)	1.481 (4)	C(3)—N(7)	1.307 (6)
S—O(3)	1.471 (3)	N(2)—C(1)	1.450 (5)
S—O(4)	1.453 (4)	C(1)—C(11)	1.508 (4)
O(5)—C(12)	1.358 (4)	C(11)—C(12)	1.389 (5)
N(6)—C(5)	1.324 (4)	C(12)—C(13)	1.386 (5)
N(6)—C(1)	1.496 (4)	C(13)—C(14)	1.410 (7)
N(6)—C(9)	1.471 (6)	C(14)—C(15)	1.374 (8)
C(5)—N(4)	1.353 (5)	(15)—C(16)	1.388 (5)
C(5)—N(8)	1.318 (4)	C(16)—C(11)	1.40 (4)
N(4)—C(3)	1.355 (4)	C(9)—C(10)	1.526 (7)
O(1)—S—O(2)	109.8 (2)	C(5)—N(4)—C(3)	122.4 (3)
O(1)—S—O(3)	110.0 (2)	N(4)—C(5)—N(8)	116.6 (3)
O(1)—S—O(4)	110.9 (2)	N(4)—C(3)—N(2)	119.2 (3)
O(2)—S—O(3)	107.9 (2)	N(4)—C(3)—N(7)	118.3 (3)
O(3)—S—O(4)	109.4 (2)	C(3)—N(2)—C(1)	123.8 (3)
O(5)—C(12)—C(11)	116.7 (3)	N(2)—C(3)—C(7)	122.4 (3)
O(5)—C(12)—C(13)	122.6 (3)	N(2)—C(1)—C(11)	112.2 (3)
N(6)—C(5)—N(4)	119.9 (3)	C(1)—C(11)—C(16)	119.5 (3)
N(6)—C(5)—N(8)	123.5 (3)	C(1)—C(11)—C(12)	121.5 (3)
C(5)—N(6)—C(1)	121.8 (3)	C(11)—C(16)—C(15)	121.0 (3)
N(6)—C(1)—N(2)	109.7 (3)	C(16)—C(11)—C(12)	119.0 (3)
N(6)—C(1)—C(11)	111.6 (3)	C(11)—C(12)—C(13)	120.7 (3)
C(5)—N(6)—C(9)	121.4 (4)	C(12)—C(13)—C(14)	119.2 (4)
C(1)—N(6)—C(9)	116.1 (4)	C(13)—C(14)—C(15)	120.7 (4)
N(6)—C(9)—C(10)	112.9 (4)	C(14)—C(15)—C(16)	119.4 (4)

15 H atoms were located from difference maps and refined, two were geometrically fixed and two water H atoms could not be located. Data reduction: local programs. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Computer used: Siemens 7.580.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71609 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1054]

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