Table 4. *Selected geometric parameters (Å, °) for (5) at 193 K*

C1—O1	1.443 (4)	C7—C8	1.548 (7)
C1—C2	1.475 (5)	C8—C9	1.475 (5)
C1—C14	1.527 (5)	C9—C10	1.189 (5)
C1—C13	1.536 (5)	C10—C11	1.486 (5)
C2—C3	1.187 (5)	C11—O11	1.450 (4)
C3—C4	1.483 (5)	C11—C15	1.522 (5)
C4—C5	1.495 (6)	C11—C12	1.531 (5)
C5—C6	1.500 (6)	C12—C13	1.533 (5)
C6—C7	1.528 (7)		
C2—C1—C14	109.2 (3)	C9—C8—C7	110.8 (4)
C2—C1—C13	110.2 (3)	C10—C9—C8	175.2 (4)
C14—C1—C13	108.4 (3)	C9—C10—C11	175.9 (4)
C3—C2—C1	173.3 (4)	C10—C11—C15	112.3 (3)
C2—C3—C4	175.3 (4)	C10—C11—C12	109.1 (3)
C3—C4—C5	111.7 (3)	C15—C11—C12	112.1 (3)
C4—C5—C6	118.0 (4)	C11—C12—C13	111.5 (3)
C5—C6—C7	113.6 (4)	C12—C13—C1	117.1 (3)
C6—C7—C8	112.0 (4)		
C1—C2—C3—C4	4 (9)	C11—C12—C13—C1	−154.6 (3)
C8—C9—C10—C11	0 (10)		

Space group *I2/a* was used, which is the third choice for space group *C2/c* (No. 15; *International Tables for Crystallography*, 1983, Vol. A), in order to avoid large correlation coefficients between the *sc* + *z* coordinates when β is much larger than 90°. At 193 K, there are fewer measurable reflections (*ca* 2%) as *N* is proportional to the volume of the unit cell, however, the number of observed reflections increased by *ca* 8%.

For both measurements, data collection: *STADI*-4 (Stoe & Cie, 1996*a*); cell refinement: *STADI*-4; data reduction: *X-RED* (Stoe & Cie, 1996*b*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1269). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 325–327

2-Amino-5-methyl-10b*H*-1,3,4-thiadiazolo[3,2-*c*]quinazolin-6-ium-10b-carboxylate Hydrate

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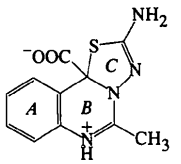
Abstract

The $C_{11}H_{10}N_4O_2S$ molecule in the title compound, $C_{11}H_{10}N_4O_2S.H_2O$, is a new heterocycle. It comprises three fused rings and exists in a zwitterionic form. The two six-membered rings are approximately coplanar, but the presence of a saturated C_{sp^3} atom at the junction of the five-membered and central six-membered rings imposes a marked non-coplanarity on these, with a dihedral angle of 23.1 (2)°. The molecule is thus chiral and the crystal structure is a racemate incorporating two molecules of water for each *R*/*S* pair of molecules. There is an extensive hydrogen-bonding network.

Comment

Many heterocyclic compounds exhibit bioactivity (Grayson, 1982), especially as antifungal, antitumour and antiviral agents (Iradyan *et al.*, 1990). Several heterocyclic compounds with a composition similar to that of the tricyclic CAM molecule in the title compound, CAM.H_2O, are essentially planar and hence achiral. We have determined the structure of CAM.H_2O by X-ray diffraction methods (Gilmore, 1983) as part



CAM

of a study of the bioactivity of CAM. The saturated sp^3-C(2) atom at the *B*/*C* ring junction (Fig. 1) induces chirality and prevents the CAM molecule from being planar.

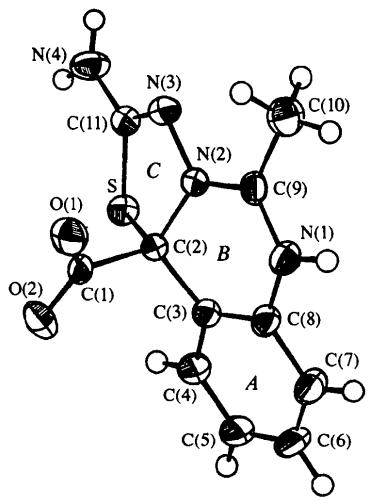


Fig. 1. The structure of the CAM molecule with the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

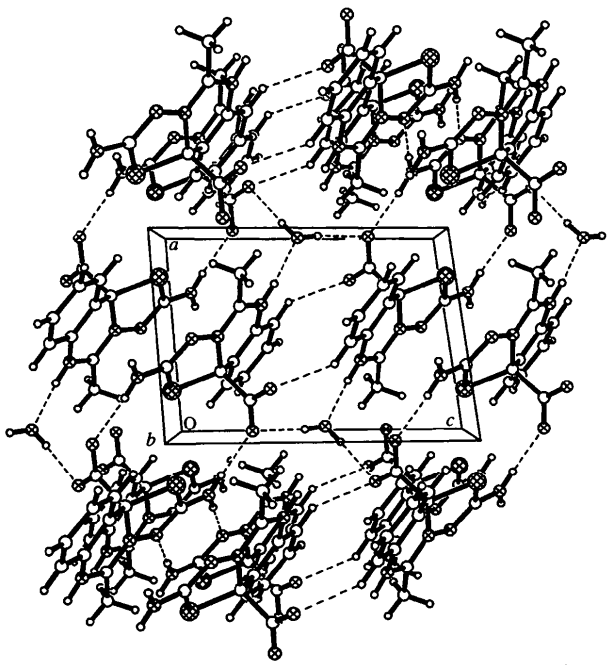


Fig. 2. Packing diagram of the unit-cell contents.

The CAM molecule is zwitterionic with a protonated N(1) atom in the central *B* ring and a deprotonated carboxylate substituent. The six-membered *A* and *B* rings are approximately coplanar [dihedral angle between mean planes 9.0 (2)°], but there is a dihedral angle of 23.1 (2)° between rings *B* and *C*.

The crystal structure is a racemate and contains two molecules of water for each *R*/*S* pair of CAM molecules. CAM molecules are linked together by a network of hydrogen bonds in which the NH_2 group interacts with carboxylate oxygen [N(4)···O(2^{i})] and imine nitrogen [N(4)···N(3^{ii})] in different molecules, and the water of crystallization interacts as an acceptor from the protonated ring nitrogen [N(1)···O(3)] and as a donor to carboxylate O atoms in different molecules [O(3)···O(1^{iii}) and O(3)···O(2^{iv})]; details and symmetry codes are given in Table 2.

Experimental

The title compound was obtained by condensation of *N*-acetylisatin with thiosemicarbazide, followed by acid-catalysed rearrangement as reported previously (Zhang, Dai, Wang & Li, 1996). It was recrystallized from 95% ethanol by slow evaporation of the solvent.

Crystal data

$C_{11}H_{10}N_4O_2S.H_2O$
M_r = 280.30
Triclinic
$P\bar{1}$
a = 8.084 (4) Å
b = 8.389 (3) Å
c = 10.216 (3) Å
α = 96.04 (3)°
β = 92.73 (3)°
γ = 115.90 (4)°
V = 616 (1) Å^3
Z = 2
D_x = 1.51 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 20 reflections
θ = 10.14–15.63°
μ = 0.260 mm^{-1}
T = 296 K
Plate
0.800 × 0.400 × 0.025 mm
Light brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Molecular Structure Corporation, 1989)
T_{min} = 0.92, T_{max} = 0.99
2586 measured reflections
2405 independent reflections

2082 reflections with $I > 3\sigma(I)$
R_{int} = 0.022
θ_{max} = 26°
h = 0 → 9
k = −10 → 10
l = −12 → 12
3 standard reflections
every 300 reflections
intensity decay: 0.7%

Refinement

Refinement on F
R = 0.038
wR = 0.050
S = 1.44

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.05
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

2082 reflections
208 parameters
H atoms refined with fixed U_{iso}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

S—C(11)	1.768 (2)	N(2)—N(3)	1.398 (2)
S—C(2)	1.830 (2)	N(2)—C(2)	1.469 (2)
O(1)—C(1)	1.233 (2)	N(3)—C(11)	1.308 (3)
O(2)—C(1)	1.255 (2)	N(4)—C(11)	1.327 (3)
N(1)—C(9)	1.325 (3)	C(1)—C(2)	1.571 (3)
N(1)—C(8)	1.404 (3)	C(2)—C(3)	1.498 (3)
N(2)—C(9)	1.318 (2)	C(3)—C(4)	1.391 (3)
C(11)—S—C(2)	87.2 (1)	N(2)—C(2)—S	102.0 (1)
C(9)—N(1)—C(8)	122.5 (2)	C(3)—C(2)—C(1)	109.4 (1)
C(9)—N(2)—N(3)	121.4 (2)	C(3)—C(2)—S	115.8 (1)
C(9)—N(2)—C(2)	123.2 (2)	C(1)—C(2)—S	108.8 (1)
N(3)—N(2)—C(2)	115.1 (1)	C(8)—C(3)—C(2)	117.5 (2)
C(11)—N(3)—N(2)	108.2 (2)	C(3)—C(8)—N(1)	119.0 (2)
O(1)—C(1)—O(2)	128.7 (2)	N(2)—C(9)—N(1)	119.3 (2)
O(1)—C(1)—C(2)	116.8 (2)	N(3)—C(11)—N(4)	123.2 (2)
O(2)—C(1)—C(2)	114.3 (2)	N(3)—C(11)—S	117.5 (1)
N(2)—C(2)—C(3)	110.2 (1)	N(4)—C(11)—S	119.3 (2)
N(2)—C(2)—C(1)	110.3 (1)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H(3)···O(3)	2.738 (3)	175
N(4)—H(1)···O(2^{i})	2.782 (3)	173
N(4)—H(2)···N(3^{ii})	3.119 (3)	178
O(3)—H(11)···O(1^{iii})	2.775 (3)	175
O(3)—H(12)···O(2^{iv})	2.832 (2)	174

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN PROCESS* and *HKL* (Molecular Structure Corporation, 1989). Program(s) used to refine structure: *TEXSAN LS* and *FOURIER*. Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: CF1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Acetyl-4-[(1*R*,2*S*,5*R*)-3-menthyloxycarbonyl]piperazine-2,5-dione†

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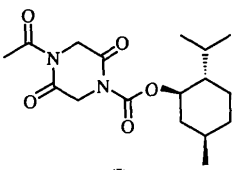
Abstract

The title compound, $C_{17}H_{26}N_2O_5$, assumes a skewed-boat conformation. The degree of folding of the piperazinedione ring given by the Hooker parameter, β, was found to be 32.06°.

Comment

X-ray crystallographic studies have shown that the presence of N and/or α-C substituents play an important role in determining the conformations of piperazine-2,5-diones (Karle, 1981). As part of our studies (Chai & King, 1995) to exploit such conformational effects to direct reactions stereoselectively, the title compound, (I), was synthesized.



(I)

The crystal structure shows that the menthyloxycarbonyl substituent extends away from the piperazinedione ring, whereas the latter adopts a skewed boat conformation. This is in contrast to the conformations adopted by glycine anhydride and its derivatives. For

† Alternative name: (1′*R*,2′*S*,5′*R*)-3-menthyl 4-acetyl-2,5-dioxopiperazine-1-carboxylate.