C7	0.7265 (2)	0.50523 (7)	−0.08808 (14)	0.0473 (3)
C8	0.7248 (2)	0.45456 (6)	−0.18741 (13)	0.0415 (3)
C9	0.7567 (2)	0.38670 (7)	−0.15955 (14)	0.0490 (4)
C10	0.7451 (3)	0.34165 (7)	−0.25929 (14)	0.0510 (4)
C11	0.7009 (2)	0.36315 (6)	−0.38967 (13)	0.0426 (3)
C12	0.6767 (2)	0.43024 (7)	−0.41855 (13)	0.0469 (3)
C13	0.6896 (2)	0.47504 (7)	−0.31701 (14)	0.0474 (4)
C14	0.6092 (3)	0.33325 (9)	−0.6184 (2)	0.0638 (5)
O1*W*	0.7643 (3)	0.67018 (7)	−0.04812 (14)	0.0793 (5)
O2*W*	0.9830 (3)	0.37037 (6)	0.18312 (14)	0.0723 (4)

Table 2. *Selected bond lengths (Å)*

O1—C6	1.224 (2)	C2—C3	1.363 (3)
O2—C11	1.367 (2)	C4—C5	1.382 (2)
O2—C14	1.436 (2)	C5—C6	1.496 (2)
N1—C3	1.328 (2)	C7—C8	1.461 (2)
N1—C4	1.338 (2)	C8—C13	1.385 (2)
N2—C6	1.345 (2)	C8—C9	1.396 (2)
N2—N3	1.382 (2)	C9—C10	1.375 (2)
N3—C7	1.273 (2)	C10—C11	1.393 (2)
C1—C2	1.382 (2)	C11—C12	1.378 (2)
C1—C5	1.383 (2)	C12—C13	1.384 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O2*W*^{i}	0.94 (3)	1.81 (3)	2.743 (3)	174 (2)
O1*W*—H2*W*1···O2^{ii}	0.88 (3)	2.05 (3)	2.917 (2)	169 (3)
O2*W*—H1*W*2···O1	0.89 (3)	2.31 (4)	3.002 (2)	135 (3)
O2*W*—H1*W*2···N3	0.89 (3)	2.29 (3)	3.079 (2)	148 (3)
O2*W*—H2*W*2···N1^{iii}	0.82 (3)	2.03 (3)	2.847 (2)	177 (3)
N2—H2N···O1*W*	0.84 (2)	2.18 (2)	3.003 (2)	164 (2)
C1—H1···O1*W*	0.94 (2)	2.61 (3)	3.429 (2)	146 (2)
C2—H2···O2*W*^{iv}	0.95 (2)	2.62 (2)	3.444 (2)	145 (2)
C7—H7···O1*W*	0.98 (2)	2.47 (2)	3.332 (2)	146 (2)
C9—H9···O2*W*	0.97 (2)	2.63 (2)	3.531 (2)	156 (2)

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

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) and *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*. Geometrical calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1230). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2*R*,3*R*)-3-Ammonio-1-(1-methoxycarbonyl-2-methyl-1-propenyl)-4-oxoazetidine-2-sulfonate Monohydrate: a New Chiral *β*-Lactam Synthon

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Abstract

The title compound, a novel monocyclic *β*-lactam, was prepared and chemically characterized by IR and ^1H NMR spectroscopy and X-ray structure analysis. $C_9H_{14}N_2O_6S.H_2O$ exists as a zwitterion crystallized with a water molecule. The four-membered lactam ring is slightly puckered, the ammonio and sulfonate groups are *cis*, and the aliphatic substituent is *trans* to these polar groups. Hydrogen bonds link the zwitterions and the water molecules into spirals parallel to the *b* axis, forming a two-dimensional network with base vectors along (100) and (010).

Comment

A foamy sample of 3-phenoxyacetamino-4-oxoazetidine-2-sulfonic acid methyl ester, (1), prepared as described earlier (Kovačević *et al.*, 1992) changed colour and form on standing at room temperature. The formation of three new compounds was detected by monitoring the change by TLC over approximately two weeks. The most polar product was precipitated out by acetone and isolated by filtration. IR spectroscopy showed

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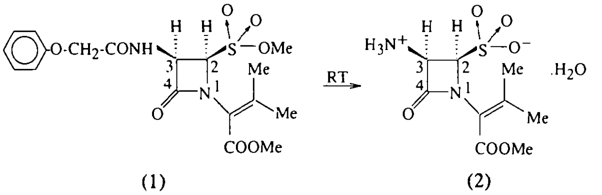
the absence of secondary amide bands at 1700 and 1520 cm^{-1} (amide I and II) and aromatic bands at 1600, 1580, 1495 and 755 cm^{-1}, and ^{1}H NMR spectroscopy proved the absence of the corresponding protons of the phenoxyacetamido group as well as the absence of the methyl sulfonate protons. These data suggested the formation of the 3-amino-4-oxoazetidine-2-sulfonic acid (2). The structure of (2), a new chiral β-lactam synthon, was confirmed by X-ray structure analysis.



(1) (2)

The bond lengths in (2) have standard values with the exception of the short N1—C4 amide bond of 1.354 (6) Å, which is close to the length of a double C=N bond [1.333 (2) Å]. Short C—N bonds have also been found in the simplest four-membered β-lactam, β-propiolactam (Yang, Seiler & Dunitz, 1987), and where C and N are *sp*^{2} hybridized (Allen *et al.*, 1987). The lactam ring is slightly puckered, with maximum deviations from the least-squares plane of the ring of 0.045 (14) Å for N1 and 0.044 (15) Å for C4. The lactam ring in β-propiolactam (Yang, Seiler & Dunitz, 1987) is planar, while in strained β-lactams (Pfaendler, Gosteli, Woodward & Rihs, 1981) the N atom is highly pyramidalized with a deviation of 0.54 Å from the plane of the three atoms to which it is bonded. In (2), N1 deviates by 0.091 (16) Å from the C2–C4–C5 plane and the bond angles at N1 sum to 359.4 (3)°, indicating a very small degree of pyramidalization. The carbonyl O atom O4 deviates by 0.138 (17) Å from the ring least-squares plane. The polar sulfonic and ammonio groups attached to the chiral C atoms C2 and C3, respectively, are *syn* with respect to the lactam ring and the aliphatic substituent attached to N1 is *anti* [C5—N1—C4—C3 −160.8 (5)°]. The 1-methoxycarbonyl-2-methyl-1-propenyl group is almost planar due to conjugation along C6—C5—C9—O5 [deviations from the least-squares plane of the group range from 0.02 (2) Å (C6) to 0.40 (2) Å (O5)].

The hydrogen bonds N2—H22···O7 [N2—H22 1.009 (6), O7···H22 2.689 (6) Å, N2—H22···O7 169.1 (4)°] and O7—H27···O1^{i} [O7—H27 0.98 (10), O1^{i}···H27 2.692 (6) Å, O7—H27···O1^{i} 109 (7)°; symmetry code: (i) *x*, 1 + *y*, *z*] form a chain along the *b* axis (Fig. 2). Two parallel chains are cross-linked by N2—H23···O2^{ii} hydrogen bonds [N2—H23 1.009 (5), O2^{ii}···H23 2.774 (5) Å, N2—H23···O2^{ii} 145.1 (4)°; symmetry code: (ii) $-x$, $\frac{1}{2}+y$, $-z$] into spirals running along the *b* axis. N2—H21 is involved in a bifurcated hydrogen bond which is intramolecular to O1 [N2—H21 1.009 (6), O1···H21 2.803 (6) Å, N2—H21···O1 126.0 (4)°] and intermolecular to a water molecule [N2—H21 1.009 (6), O7^{iii}···H21 2.927 (5) Å, N2—H21···O7^{iii} 113.0 (3)°; symmetry code: (iii) $1-x$, $y-\frac{1}{2}$, $-z$], completing the two-dimensional network.

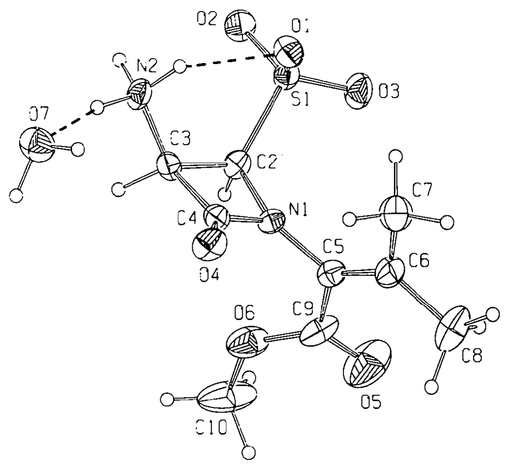


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecular structure with the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level for non-H atoms; H atoms are drawn as small circles of arbitrary radii. The intramolecular and intermolecular hydrogen bonds N2—H21···O1 and N2—H22···O7 are also shown.

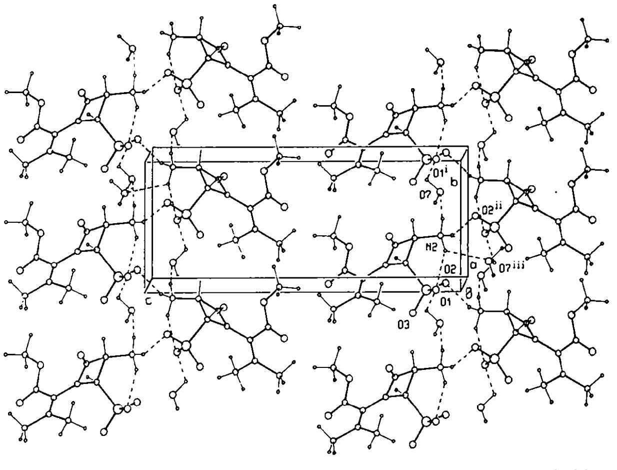


Fig. 2. A view of the molecular packing showing hydrogen-bonded helices along the *b* axis separated by hydrophobic regions containing methyl groups. The intermolecular hydrogen bonds N2—H22···O7, O7—H27···O1^{i}, N2—H23···O2^{ii} and N2—H21···O7^{iii}, and the intramolecular hydrogen bond N2—H21···O1 are shown [symmetry codes: (i) *x*, 1 + *y*, *z*; (ii) $-x$, $\frac{1}{2}+y$, $-z$; (iii) $1-x$, $y-\frac{1}{2}$, $-z$]. The atoms O5 and O6 protrude into the hydrophobic region; a C10—H102···O5 contact with a C···O distance of 3.231 (7) Å is found.

Experimental

2.1 g (4.9 mmol) of (2*R*,3*R*)-3-phenoxyacetamido-1-(1-methyloxycarbonyl-2-methylprop-1-enyl)-4-oxoazetidine-2-sulfonic acid methyl ester (Kovačević *et al.*, 1992), (1), was kept at room temperature for two weeks. 0.66 g of (2) precipitated as crystals on adding acetone (48.5% yield); an additional 0.12 g (8.8%) of (2) was obtained from the mother liquor residue upon separation by silica-gel column chromatography using methylene chloride–methanol (4:1) as eluant: m.p. (uncorrected, Fisher–Johns apparatus) 508–509 K (decomposed); R_f = 0.25 in CH_2Cl_2–MeOH (4:1). The compound was characterized by chemical and spectroscopic methods. Thermogravimetric analysis showed a 6% loss in mass in the region 367–420 K. A single crystal of (2) suitable for X-ray structure analysis was obtained from a mixture of acetone and ether.

Crystal data

$C_9H_{14}N_2O_6S.H_2O$
$M_r = 296.28$
Monoclinic
$P2_1$
$a = 6.097\,(7)$ Å
$b = 6.804\,(5)$ Å
$c = 16.92\,(2)$ Å
$\beta = 97.66\,(6)°$
$V = 696\,(1)$ Å^3
$Z = 2$
$D_x = 1.414$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7–18°$
$\mu = 0.250$ mm^{-1}
$T = 295\,(3)$ K
Plate
0.40 × 0.29 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
2023 measured reflections
1520 independent reflections
1382 observed reflections [$F > 2\sigma(F)$]
$R_{\rm int} = 0.0112$

$\theta_{\rm max} = 20°$
$h = -1 \rightarrow 5$
$k = -1 \rightarrow 6$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 87 reflections frequency: 180 min intensity decay: 0.1%

Refinement

Refinement on F
$R = 0.0408$
$wR = 0.0431$
$S = 0.46$
1379 reflections
188 parameters
H-atom parameters not refined for H21, H22 and H23, all H-atom parameters refined for H17, H27, H2 and H3, and only U's refined for all other H atoms
$w = 1.7539/[\sigma^2(F_o) + 0.0004(F_o^2)]$

$(\Delta/\sigma)_{\rm max} = 0.21$
$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)
Absolute configuration: assigned to agree with the known chiralities at C2 and C3 of its precursor, natural phenoxymethylpenicillin

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

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

	x	y	z	$U_{\rm eq}$
S1	0.1124 (2)	0.0	0.11020 (10)	0.0425 (3)
O1	0.3295 (5)	−0.0226 (6)	0.0855 (2)	0.0525 (10)
O2	−0.0493 (6)	0.0695 (6)	0.0458 (2)	0.0618 (11)
O3	0.0441 (6)	−0.1667 (6)	0.1525 (2)	0.0596 (11)
O4	0.6511 (5)	0.3488 (6)	0.2408 (2)	0.0569 (13)
O5	0.1044 (8)	0.0509 (10)	0.4205 (2)	0.119 (3)
O6	0.1298 (7)	0.3309 (8)	0.3556 (2)	0.0775 (18)
N1	0.3233 (5)	0.1690 (6)	0.2441 (2)	0.0375 (10)
N2	0.3407 (6)	0.3876 (6)	0.0716 (2)	0.0451 (11)
C2	0.1423 (7)	0.2019 (8)	0.1780 (2)	0.0395 (14)
C3	0.2819 (7)	0.3749 (7)	0.1530 (2)	0.0405 (14)
C4	0.4612 (7)	0.3039 (7)	0.2186 (2)	0.0398 (14)
C5	0.3306 (7)	0.0654 (8)	0.3178 (2)	0.0430 (14)
C6	0.4775 (9)	−0.0783 (9)	0.3361 (3)	0.0583 (16)
C7	0.6319 (10)	−0.1426 (10)	0.2791 (3)	0.070 (2)
C8	0.5068 (13)	−0.1848 (13)	0.4144 (3)	0.096 (3)
C9	0.1773 (9)	0.1437 (11)	0.3704 (3)	0.064 (2)
C10	−0.0175 (11)	0.4276 (16)	0.4019 (3)	0.120 (4)
O7	0.6287 (6)	0.6885 (7)	0.0799 (2)	0.0655 (14)

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

S1—O1	1.448 (3)	N1—C4	1.354 (6)
S1—O2	1.448 (4)	N1—C5	1.428 (5)
S1—O3	1.432 (4)	N2—C3	1.471 (5)
S1—C2	1.783 (5)	C2—C3	1.544 (7)
O4—C4	1.208 (5)	C3—C4	1.529 (5)
O5—C9	1.190 (8)	C5—C6	1.334 (8)
O6—C9	1.323 (9)	C5—C9	1.474 (7)
O6—C10	1.429 (9)	C6—C7	1.500 (8)
N1—C2	1.480 (5)	C6—C8	1.500 (8)
O1—S1—O2	112.0 (2)	N2—C3—C4	117.3 (3)
O1—S1—O3	113.4 (2)	C2—C3—C4	85.8 (3)
O1—S1—C2	104.4 (2)	O4—C4—N1	133.1 (4)
O2—S1—O3	114.9 (2)	O4—C4—C3	135.1 (4)
O2—S1—C2	103.5 (2)	N1—C4—C3	91.8 (3)
O3—S1—C2	107.5 (2)	N1—C5—C6	120.4 (4)
C9—O6—C10	118.5 (5)	N1—C5—C9	113.6 (4)
C2—N1—C4	95.1 (3)	C6—C5—C9	125.8 (4)
C2—N1—C5	131.9 (3)	C5—C6—C7	121.6 (5)
C4—N1—C5	131.7 (3)	C5—C6—C8	123.8 (5)
S1—C2—N1	111.8 (3)	C7—C6—C8	114.6 (5)
S1—C2—C3	115.1 (3)	O5—C9—O6	123.6 (6)
N1—C2—C3	86.5 (3)	O5—C9—C5	124.5 (7)
N2—C3—C2	120.9 (3)	O6—C9—C5	112.0 (5)
O1—S1—C2—N1	−55.6 (3)	N1—C2—C3—C4	6.0 (3)
C10—O6—C9—O5	−0.2 (8)	C2—C3—C4—O4	174.1 (5)
C4—N1—C2—S1	108.9 (3)	C2—C3—C4—N1	−6.6 (3)
C4—N1—C2—C3	−6.8 (3)	N2—C3—C4—N1	−129.3 (4)
C2—N1—C4—C3	6.9 (3)	C9—C5—C6—C7	−177.8 (5)
C5—N1—C4—C3	−160.8 (5)	C9—C5—C6—C8	0.7 (10)
C2—N1—C5—C6	123.9 (5)	C6—C5—C9—O5	−29.5 (9)
C2—N1—C5—C9	−60.6 (7)		

Data collection and cell refinement were carried out using *CAD*-4 *Software* (Enraf–Nonius, 1988) and the data were corrected for Lorentz and polarization effects using the Enraf–Nonius *SDP*/*VAX* package (B. A. Frenz & Associates Inc., 1982). The structure was solved by the Patterson method using *SHELXS*86 (Sheldrick, 1990). Refinement was carried out by full-matrix least squares on F minimizing $\Sigma w(F_o - F_c)^2$ (*SHELX*76; Sheldrick, 1976). All methyl H atoms were placed in geometrically calculated positions, while water and ammonio H atoms and those on C2 and C3 were located from successive difference Fourier syntheses. All H atoms except those in the water molecule and on C2 and C3 were refined riding on their respective atoms. The displacement parameters of the ammonio H atoms were kept fixed at 0.1 Å^2, while those of the methyl H atoms were refined as a common free variable. The y coordinate of S1 was held fixed in order to fix the origin in the polar space group $P2_1$. All non-H atoms were refined anisotropically. Three reflections for which the difference in the observed and calculated structure factors was large (131, 102 and 303) were omitted. The molecular geometry was calculated using the program *PLATON* (Spek, 1990). Fig. 1

was prepared using *ORTEP* (Johnson, 1965) incorporated in *PLATON* and Fig. 2 was prepared using *PLUTON* (Spek, 1991). Material was prepared for publication using *PLATON*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1146). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,10-Diiodo-5,10-dihydroboranthrene

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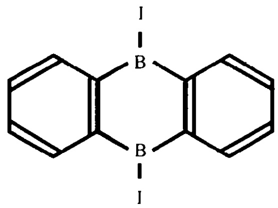
Abstract

The structure of the title compound, 9,10-diiodo-9,10-dihydro-9,10-diboranthracene, $C_{12}H_8B_2I_2$, has been determined. The nearly planar skeleton of the molecule suggests that the central six-membered boron-containing ring is conjugated with the outer two benzene rings. The molecules stack along the *c* axis and the B atoms of one molecule lie just above the I atoms of the next molecule.

Comment

As part of our study of organic donor–acceptor molecular complexes, the title compound was interesting because it was possible that it possessed an electron-accepting property. Only four kinds of 5,10-disubstituted-5,10-dihydroboranthrene compound have been synthesized, namely, the diiodo derivative (Siebert, Schaper & Asgarouladi, 1974), the dibromo derivative (Asgarouladi, Full, Schaper & Siebert, 1974), the dichloro derivative (Clement, 1965) and the dimethyl derivative (Schacht & Kaufmann, 1987). However, none of these compounds has so far been structurally analyzed. This report is concerned with the diiodo derivative, (I).



(I)

Since there are two molecules of (I) in the unit cell of space group *C*2/*m*, a quarter of the molecule corresponds to an asymmetric unit. The symmetry of the molecule is C_{2h} and is very close to D_{2h}. The mirror plane passes through the I—B···B—I atoms perpendicular to the molecular plane and a twofold axis, perpendicular to the mirror, is present on the molecular plane. All the atoms of the molecule lie on a plane, within experimental error, except for the I atoms. The angle between the least-squares molecular plane and the I—B bond is 175.6°. Though the concentrated electron density at the I atoms (about 54% in the whole molecule) results in an average bond-length e.s.d. as large as 0.01 Å, the C(1)—C(2), C(2)—C(3) and C(3)—C(3^{i}) (symmetry codes are in Table 2) bond lengths are in the conjugated C_{sp^2}—C_{sp^2} range. A slightly large value for the C(1)—C(1^{i}) bond length, however, suggests that the π-bond character of this bond is weakened. In addition, the B—C(1) bond length of 1.52 (1) Å is significantly shorter than the distance of 1.578 (1) Å found in $B(CH_3)_3$ (Bartell & Carroll, 1965) and the distances 1.571 (3)–1.589 (5) Å found in $B(Ph)_3$ (Zettler, Hausen & Hess, 1974). These facts imply that the π electrons of the outer benzene rings are partly donated to the empty 2*p* orbitals of the B atoms and that the conjugated system is enlarged to include the whole molecule. The I—B bond length of 2.18 (1) Å is in the range of ordinary covalent bonds (Cotton & Wilkinson, 1980), suggesting that electron donation does not occur from the I to the B atom.

All the molecules stack along the *c* axis, with their molecular planes parallel to each other, as shown in Fig. 2, and at an angle of 58.5° to the *c* axis. The interplanar