This work was suported by the Ministry of Science, Technology and Informatics of the Republic of Croatia.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71175 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1030]

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Benzyl 2-[(2*R*,S*S*)-2-(Benzylaminosulfinyl)-4-oxoazetidin-1-yl]-3-methylbut-2-enoate

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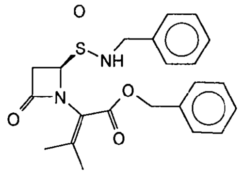
Abstract

The absolute configuration of the S atom was established as *S* using the known absolute configuration of the adjacent C atom (*R*). The 3-methylbut-2-enoate fragment is planar and it makes an angle (C3—N2—C4—C5) of −55.1 (5)° with the β-lactam ring.

Comment

The title compound (1) was synthesized and structurally characterized as part of our broader investigation of new β-lactams (Herak, Kovačević & Gašpert, 1989).



(1)

The main goal of this structure determination was to confirm the absolute configuration of the S atom (established as *S*) on the basis of the absolute configuration of C1 which was known to be *R* (Pant, Steele & Stoodley, 1982; Steele & Stoodley, 1983) (Fig. 1). The 3-methylbut-2-enoate part of the molecule is nearly planar [C5—C4—C8—O3 1.0 (6), O3—C8—O4—C9 −2.3 (5)°] while the values of the bond distances C4—C8 and C4—C5 [1.492 (4) and 1.350 (4) Å] indicate single- and double-bond character, respectively. This planar moiety lies at 58.4 (1)° to the best plane through the β-lactam ring and the torsion angle C3—N2—C4—C5 is −55.1 (5)°. The sum of the bond angles around the β-lactam nitrogen N2 is exactly 360° [C1—N2—C3 94.2 (3), C1—N2—C4 133.6 (3), C3—N2—C4 132.2 (3)° (Yang, Seiler & Dunitz, 1987; Kobal, 1991)]. The β-lactam ring is folded about a diagonal. The N2 atom is 0.07 Å out of the plane

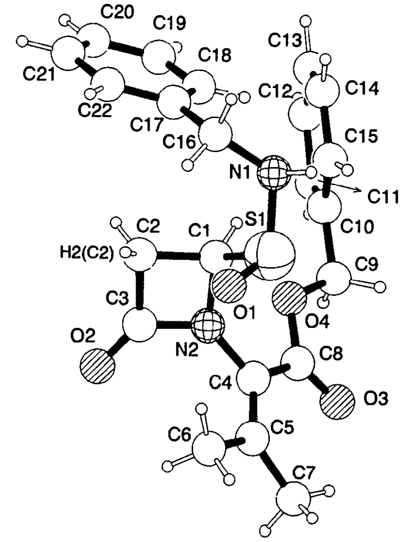


Fig. 1. *PLUTON* drawing of $C_{22}H_{24}N_2O_4S$ showing the atomic labelling scheme. H-atom labelling [except H2(C2)] omitted for clarity.

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defined by the other three atoms within the β-lactam ring (C1, C2 and C3). As a result of free rotation about C1—S1 [C2—C1—S1—O1 28.9 (3)°] the O1 and S1 atoms of the sulfinyl group are able to lie close to H2(C2) [2.598 (3) and 2.899 (1) Å, respectively; sum of the van der Waals radii of S and H is 3.03 and of O and H 2.58 Å]. Consequently, the hydrogen H2(C2) is strongly deshielded and the corresponding peak in the ^{1}H NMR ($CDCl_3$) spectrum shows a downfield shift [δ: 3.35 p.p.m.; for *R,SR* diastereomer δ: 2.94 p.p.m. (Herak, Vinković & Kojić-Prodić, 1993)]. Along the *a* axis the molecules are held together by hydrogen bonds N1—H···O2 of 2.957 (4) Å (Table 3, Fig. 2).

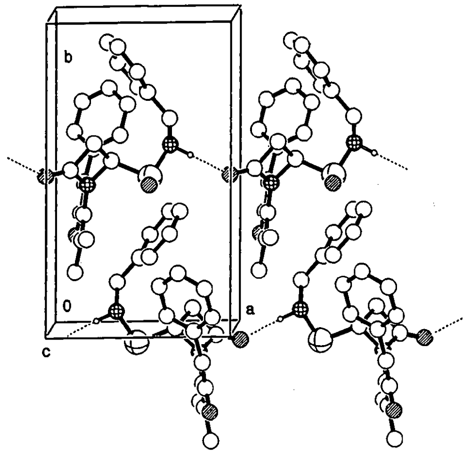

Fig. 2. *PLUTON* drawing of the unit cell of $C_{22}H_{24}N_2O_4S$. Hydrogen bonds are represented by dashed lines.

Experimental

Crystal data

$C_{22}H_{24}N_2O_4S$
M_r = 412.50
Monoclinic
$P2_1$
a = 8.242 (4) Å
b = 13.423 (8) Å
c = 9.935 (7) Å
β = 107.99 (5)°
V = 1045 (1) Å^3
Z = 2
D_x = 1.31 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8–18°
μ = 0.176 mm^{-1}
T = 293 K
Prism
0.65 × 0.31 × 0.22 mm
Colourless

Data collection

Stoe-modified Philips PW1100 diffractometer
ω scans
Absorption correction: none
3368 measured reflections
3286 independent reflections
2370 observed reflections [$F > 3.0\sigma(F)$]

θ_{max} = 30.01°
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity variation: 4.5%

Refinement

Refinement on F
Final R = 0.034
wR = 0.057
S = 0.9569
2370 reflections
286 parameters
Only H-atom U's refined except for H16, H26, H36, H17, H27, H37: parameters not refined

Calculated weights $w = 1/[\sigma^2(F)+0.00284*F^2]$
$(\Delta/\sigma)_{max}$ = 0.047
$\Delta\rho_{max}$ = 0.203 e Å^{-3}
$\Delta\rho_{min}$ = −0.211 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Stoe & Cie (1992) software. Cell refinement: Stoe & Cie (1992) software. Data reduction: *REDU4S* (Stoe & Cie, 1992). Program(s) used to solve structure: *SIR88* (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *PLUTON* (Spek, 1982).

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

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

	x	y	z	U_{eq}
S1	0.4798 (1)	−0.0280	0.7623 (1)	0.0397 (2)
O1	0.4764 (3)	−0.0458 (2)	0.6140 (2)	0.0551 (8)
O2	1.0443 (2)	−0.0227 (2)	0.7707 (3)	0.0547 (7)
O3	0.8985 (3)	−0.2327 (2)	1.1559 (3)	0.0577 (9)
O4	0.8513 (3)	−0.0684 (1)	1.1359 (2)	0.0423 (7)
N1	0.3705 (3)	0.0727 (2)	0.7788 (3)	0.0467 (9)
N2	0.8236 (2)	−0.0563 (2)	0.8678 (2)	0.0336 (6)
C1	0.6935 (3)	0.0211 (2)	0.8514 (3)	0.0332 (7)
C2	0.7824 (3)	0.0797 (2)	0.7604 (3)	0.0408 (8)
C3	0.9117 (3)	−0.0047 (2)	0.7944 (3)	0.0389 (8)
C4	0.8460 (3)	−0.1536 (2)	0.9292 (3)	0.0337 (6)
C5	0.8511 (4)	−0.2328 (2)	0.8469 (3)	0.0458 (9)
C6	0.8184 (5)	−0.2222 (3)	0.6910 (4)	0.0605 (13)
C7	0.8907 (6)	−0.3374 (3)	0.9018 (4)	0.0728 (16)
C8	0.8698 (3)	−0.1587 (2)	1.0841 (3)	0.0377 (8)
C9	0.8621 (5)	−0.0671 (2)	1.2859 (3)	0.0510 (10)
C10	0.8185 (4)	0.0351 (2)	1.3227 (3)	0.0413 (9)
C11	0.9241 (4)	0.0838 (2)	1.4388 (3)	0.0484 (10)
C12	0.8828 (5)	0.1770 (3)	1.4777 (4)	0.0562 (11)
C13	0.7359 (5)	0.2229 (3)	1.4003 (4)	0.0605 (13)
C14	0.6283 (5)	0.1762 (3)	1.2845 (4)	0.0667 (14)
C15	0.6671 (4)	0.0809 (3)	1.2455 (4)	0.0590 (12)
C16	0.3697 (4)	0.1609 (2)	0.6887 (3)	0.0499 (9)
C17	0.4936 (3)	0.2412 (2)	0.7618 (3)	0.0413 (8)
C18	0.5535 (4)	0.2514 (2)	0.9084 (3)	0.0454 (8)
C19	0.6664 (4)	0.3271 (2)	0.9699 (4)	0.0536 (12)
C20	0.7174 (4)	0.3953 (3)	0.8861 (5)	0.0598 (12)
C21	0.6538 (5)	0.3879 (3)	0.7406 (5)	0.0637 (13)
C22	0.5444 (4)	0.3110 (3)	0.6785 (3)	0.0523 (11)

Table 2. *Geometric parameters* (Å, °)

S1—O1	1.484 (3)	C5—C7	1.505 (5)
S1—N1	1.660 (3)	C9—C10	1.492 (4)
S1—C1	1.832 (3)	C10—C11	1.377 (4)
O2—C3	1.211 (4)	C10—C15	1.390 (4)
O3—C8	1.203 (4)	C11—C12	1.383 (5)
O4—C8	1.344 (3)	C12—C13	1.365 (5)
O4—C9	1.465 (4)	C13—C14	1.369 (5)
N1—C16	1.483 (4)	C14—C15	1.402 (6)
N2—C1	1.466 (4)	C16—C17	1.508 (4)
N2—C3	1.366 (4)	C17—C18	1.393 (4)
N2—C4	1.429 (4)	C17—C22	1.397 (5)
C1—C2	1.543 (4)	C18—C19	1.386 (4)
C2—C3	1.520 (4)	C19—C20	1.387 (6)

C4—C5	1.350 (4)	C20—C21	1.381 (7)
C4—C8	1.492 (4)	C21—C22	1.384 (5)
C5—C6	1.495 (5)		
N1—S1—C1	97.4 (2)	O4—C8—C4	111.1 (3)
O1—S1—C1	105.0 (2)	O3—C8—C4	126.1 (3)
O1—S1—N1	112.6 (2)	O3—C8—O4	122.9 (3)
C8—O4—C9	115.0 (2)	O4—C9—C10	108.5 (3)
S1—N1—C16	119.5 (3)	C9—C10—C15	121.0 (3)
C3—N2—C4	132.2 (3)	C9—C10—C11	120.3 (3)
C1—N2—C4	133.6 (3)	C11—C10—C15	118.7 (3)
C1—N2—C3	94.2 (3)	C10—C11—C12	121.2 (3)
S1—C1—N2	111.1 (2)	C11—C12—C13	120.2 (4)
N2—C1—C2	87.9 (2)	C12—C13—C14	119.9 (4)
S1—C1—C2	117.4 (2)	C13—C14—C15	120.4 (4)
C1—C2—C3	85.2 (2)	C10—C15—C14	119.6 (4)
N2—C3—C2	92.6 (3)	N1—C16—C17	113.8 (3)
O2—C3—C2	135.9 (3)	C16—C17—C22	118.4 (3)
O2—C3—N2	131.5 (3)	C16—C17—C18	122.9 (3)
N2—C4—C8	116.2 (3)	C18—C17—C22	118.6 (3)
N2—C4—C5	119.0 (3)	C17—C18—C19	120.4 (3)
C5—C4—C8	124.8 (3)	C18—C19—C20	120.4 (4)
C4—C5—C7	124.0 (3)	C19—C20—C21	119.6 (4)
C4—C5—C6	121.6 (3)	C20—C21—C22	120.3 (4)
C6—C5—C7	114.5 (3)	C17—C22—C21	120.6 (4)

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

D	H	*A*	*D*—H	*D*···*A*	*D*—H···*A*
N1	H1N1	O2^{i}	0.959 (2)	2.957 (4)	163.3 (3)

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

This work was supported by the Ministry of Science, Technology and Informatics of the Republic of Croatia.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71201 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1032]

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Structure of an Antimalarial Alkaloid, Jatrorrhizine

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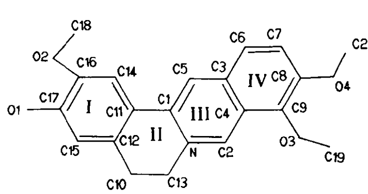
Abstract

Jatrorrhizine is a naturally occurring antimalarial alkaloid. The structure of jatrorrhizine, determined from its chloride (5,6-dihydro-3-hydroxy-2,9,10-trimethoxydibenzo[*a*,*g*]quinolizinium chloride dihydrate), consists of four fused rings. The conformation of ring I (benzo) is a boat and ring II (dihydropyrido moiety) is deviated from its twist-boat conformation. The planar rings III (pyrido moiety) and IV (benzo) are very similar to the quinoline part of several quinine derivatives. Hydrogen-bonded water molecules stabilize the crystal.

Comment

The title compound, jatrorrhizine (1) was isolated as its chloride from the bark of *Enantia chlorantha*. The protoberberine alkaloid, jatrorrhizine was tested for antimalarial activity *in vitro* against *Plasmodium jalciparum* and *in vivo* against *Plasmodium berghei*, and exhibited a potency comparable to that of quinine *in vitro* but not *in vivo*.



(1)

The antimalarial compound jatrorrhizine contains a moiety closely resembling the quinoline moiety of several quinine derivatives (Dupont, Konour, Lewinski & Oleksyn, 1985). The bond lengths and bond angles of rings III and IV are very close to those found in the quinoline part of cinchona alkaloids. However, the main differences are as follows: the methoxy group normally attached to the

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