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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1293). Services for accessing these data are described at the back of the journal.

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Intermolecular C—H···N and C—H···O interactions in (2*S*,4*S*,5*R*)-(−)-3,4-dimethyl-5-phenyl-2-(1,3-thiazol-2-yl)-1,3-oxazolidine

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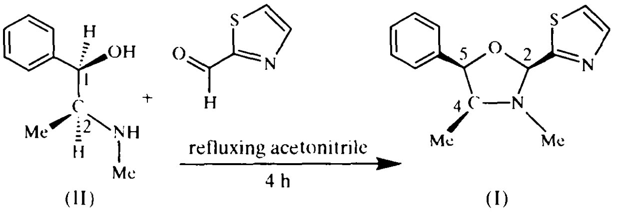
Abstract

The title compound, $C_{14}H_{16}N_2OS$, prepared from (1*R*,2*S*)-(−)-ephedrine, contains the oxazolidine ring in an envelope conformation, with the nitrogen atom 0.623 (2) Å from the plane of the other four oxazolidine-ring atoms. Intermolecular C—H···N and C—H···O interactions generate a two-dimensional hydrogen-bonded network, with shortest C···N and C···O distances of 3.403 (3) and 3.463 (2) Å, respectively.

Comment

Amino acid derivatives continue to be an important class of chiral compounds with applications in asymmetric synthesis and catalysis. The title compound, (I), a derivative of (1*R*,2*S*)-(−)-ephedrine, (II), is of current interest as a chiral auxiliary and as a new potentially bidentate ligand with an O,N or O,S donor-atom set for use in coordination chemistry.



Bond lengths and angles are unexceptional and in accord with anticipated values (Orpen *et al.*, 1994). The absolute structure can be deduced from (II) and by analysis of our X-ray data. The oxazolidine ring adopts an envelope conformation with N3 0.623 (2) Å from the (O1,C2,C4,C5) plane, which is at angles of 74.18 (6) and 86.99 (6)° to the phenyl and thiazole rings, respectively. The phenyl ring is oriented at an angle of 41.15 (8)° to the thiazolyl group. The molecular geometry in (I) compares with (2*S*,4*S*,5*R*)-(−)-2-(1*H*-imidazol-2-yl)-3,4-dimethyl-5-phenyl-1,3-oxazolidine, (III) (Gallagher *et al.*, 1998), and a related *p*-bromophenyl derivative (Just *et al.*, 1983). Examination of the structure with *PLATON* (Spek, 1997*a*) showed that there were no solvent-accessible voids in the crystal lattice.

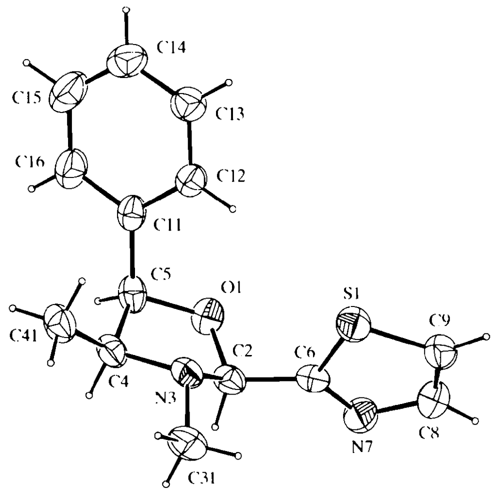


Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

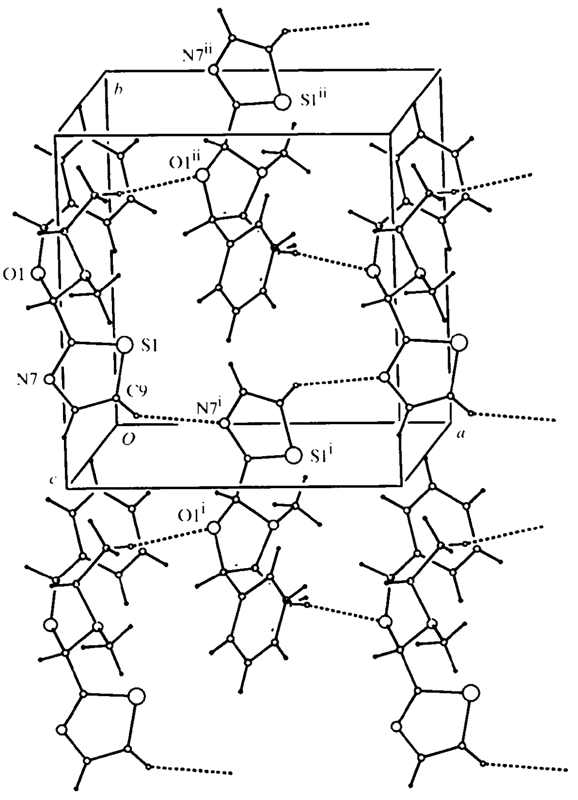


Fig. 2. A view of the intermolecular interactions in the crystal structure of (I).

There is an intermolecular Csp^3—H···O interaction to O1 and a C(thiazole)—H···N(thiazole) interaction to N7, which generates a two-dimensional hydrogen-bonded network, as depicted in Fig. 2, with details in Table 2. The closest H···S distance is the intramolecular H12···S1 of 3.00 Å, which is equal to the sum of the van der Waals radii (Bondi, 1964). The crystal structure of (I) contrasts with that of (III) where N—H···N hydrogen bonds, Csp^3—H···π(C=C, imidazole), C(arene)—H···π(arene) and C(arene)—H···O interactions generate a three-dimensional network (Gallagher *et al.*, 1998). The difference in hydrogen-bonding abilities when comparing S(thiazolyl) in (I) and N—H(imidazolyl) in (III) accounts for the lack of strong hydrogen-bond formation and the presence of weak C—H···N and C—H···O hydrogen bonds in (I). Further comparative studies are in progress on related 1,3-oxazolidine systems.

Experimental

The compound was prepared by refluxing thiazole-2-carboxaldehyde (500 mg, 4.42 mmol) and (1*R*,2*S*)-(−)-ephedrine (730 mg, 4.42 mmol) in 20 cm^3 of acetonitrile for 4 h. On cooling, the product was filtered and recrystallized from ethanol, yield 0.98 g (85%), m.p. 354–355 K. IR ν_{max}(KBr) cm^{-1}; 3097 (*m*), 2800 (*m*), 2377 (*m*), 1455 (*s*), 1340 (*s*), 1059 (*s*). $[\alpha]_D^{20} = -75°$ (c = 1.0, ethanol). ^{1}H NMR (400 MHz) (δ, CDCl$_3$) p.p.m.: 0.78 (*d*, 3H, CCH$_3$), 2.39 (*s*, 3H, NCH$_3$), 3.06 (*m*, 1H, MeCH), 5.12 (*s*, 1H, OCH), 5.20 (*d*, 1H, PhCH), 7.30–7.46 (*m*, 5H, C$_6$H$_5$), 7.46 (*d*, 1H, thiazole SCH), 7.83 (*d*, 1H, thiazole NCH).

Crystal data

$C_{14}H_{16}N_2OS$
$M_r = 260.35$
Orthorhombic
$P2_12_12_1$
$a = 10.2981\ (8)$ Å
$b = 10.8896\ (9)$ Å
$c = 12.1733\ (7)$ Å
$V = 1365.14\ (17)$ Å^3
$Z = 4$
$D_x = 1.267$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.73$–$19.75°$
$\mu = 0.227$ mm^{-1}
$T = 294\ (1)$ K
Block
0.30 × 0.30 × 0.29 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3406 measured reflections
3118 independent reflections
2613 reflections with $I > 2\sigma(I)$

$R_{int} = 0.012$
$\theta_{max} = 27.4°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity variation: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.091$
$S = 1.024$
3118 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.1202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0120 (18)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.01 (8)

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

S1—C6	1.7250 (18)	C2—C6	1.483 (2)
S1—C9	1.703 (2)	N7—C6	1.289 (2)
O1—C2	1.414 (2)	N7—C8	1.373 (3)
O1—C5	1.445 (2)	C8—C9	1.337 (3)
C6—S1—C9	89.54 (10)	O1—C5—C11	110.17 (15)
C2—O1—C5	108.36 (14)	S1—C6—N7	114.31 (14)
O1—C2—N3	104.45 (14)	S1—C6—C2	121.20 (12)
O1—C2—C6	109.74 (14)	S1—C9—C8	109.48 (16)
O1—C5—C4	104.16 (15)		
O1—C2—C6—S1	81.84 (16)	O1—C5—C11—C12	−38.7 (2)
O1—C2—C6—N7	−95.7 (2)	O1—C5—C11—C16	141.50 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N7^{i}	0.93	2.73	3.403 (3)	130
C41—H41C···O1^{ii}	0.96	2.62	3.463 (2)	147

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

Molecule (I) is chiral and a complete 'Friedel' data set ($\pm h,+k,+l$) was collected and the absolute structure determined [Flack parameter −0.01 (8)] by our X-ray analysis. The known absolute configuration of the (1*R*,2*S*)-(−)-ephedrine starting material, (II), is retained in the three chiral centres (2*S*,4*S*,5*R*) of the oxazolidine ring in (I). H atoms were allowed for as riding atoms with C—H in the range of 0.93 to 0.98 Å.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD*-4-*PC Software*. Data reduction: *DATRD*2 in *NRCVAX*96 (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *NRCVAX*96 and *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *NRCVAX*96, *ORTEP* (Johnson, 1976), *PLATON* (Spek, 1997*a*) and *PLUTON* (Spek 1997*b*). Software used to prepare material for publication: *NRCVAX*96, *SHELXL*97 and *WordPerfect* macro *PRPCIF*97 (Ferguson, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1296). Services for accessing these data are described at the back of the journal.

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Bis(*m*-anisidinium) dihydrogénodiphosphate

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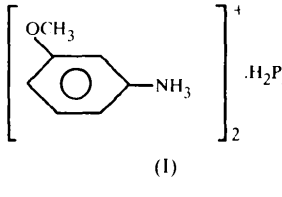
(*Reçu le 14 mars 1997, accepté le 7 septembre 1998*)

Abstract

The structure consists of an alternate stacking of $(H_2P_2O_7)^{2-}$ anions and $(1,3\text{-}CH_3OC_6H_4NH_3)^+$ cations held together by N—H···O intermolecular hydrogen bonds.

Commentaire

L'arrangement moléculaire de cette structure, (I), est constitué de couches d'anions phosphates $(H_2P_2O_7)^{2-}$



(I)

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