For both (II) and (II′), many H atoms were apparent in the difference maps but the calculated positions were used for refinement. Partial H atoms were added to resolvable C atoms with occupancies equal to those of the C atoms. For unresolvable C atoms, partial H atoms were added in the correct geometry to accord with the respective D or L framework and the occupancies set to the respective group occupancies. Isotropic displacement parameters for carboxyl and methyl H atoms were set to 1.5 times, and for methine and methylene H atoms were set to 1.2 times the equivalent isotropic displacement parameters of the atoms upon which they were riding.

Data collection: *XSCANS* (Siemens, 1996) for (I); *SMART* (Siemens, 1995) for (II). Cell refinement: *XSCANS* for (I); *SAINT* (Siemens, 1995) for (II). Data reduction: *XSCANS* for (I); *SAINT* for (II). For both compounds, program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1548). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1605–1607

(2*S*,*SR*)-*tert*-Butyl 2-(*p*-tolylsulfinylacetyl)-pyrrolidine-1-carboxylate†

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(*Received 13 January 1999; accepted 5 May 1999*)

Abstract

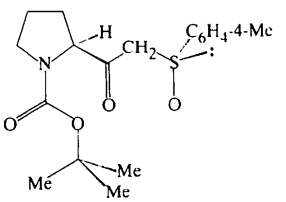
The title compound, $C_{18}H_{25}NO_4S$, is the single adduct obtained from the reaction between the *N*-(*tert*-butoxy-

† Contribution No. 1696 of the Instituto de Química, UNAM.

carbonyl)-L-proline methyl ester and the anion of (+)-(*R*)-methyl *p*-tolyl sulfoxide. The pyrrolidine ring has a twist conformation and the *Re* face of the carbonyl group is partially blocked by the *tert*-butoxycarbonyl and sulfinyl groups.

Comment

Racemic acyclic β-keto sulfoxides are readily available from the reaction of α-sulfinyl anions with esters (Corey & Chaykowsky, 1962, 1965). The method was first used by Kunieda (Kunieda *et al.*, 1974) to prepare optically pure (*R*)-α-(*p*-tolylsulfinyl)acetophenone and over the years, this synthesis has been the most widely used method for obtaining a great variety of enantiomerically pure acyclic β-keto sulfoxides (Carreño, 1995). As part of a study directed towards the synthesis of chiral β-amino alcohols using the sulfinyl group as chiral auxiliary, we prepared (2*S*,S*R*)-*tert*-butyl 2-(*p*-tolylsulfinylacetyl)pyrrolidine-1-carboxylate, (I), by the reaction of the *N*-(*tert*-butoxycarbonyl)-L-proline methyl ester and the anion of (+)-(*R*)-methyl *p*-tolyl sulfoxide. The X-ray crystallographic structure determination was undertaken in order to obtain information about its conformation and molecular geometry.



(I)

The conformation of the pyrrolidine ring can be best described as possessing a twisted conformation [q = 0.342 (7) and φ = 83.9 (10); Cremer & Pople, 1975], with an approximate $C2$ axis running through N1 and the center of the C3—C4 bond. The angles around the N atom show that it is perfectly planar. The ketosulfinyl group is in a pseudo-axial position with respect to the five-membered ring, with the carbonyl and *p*-tolylsulfinyl moieties making a dihedral angle of 69.8 (3)°. This configuration partially blocks the *Re* face of the carbonyl group towards a hypothetical reduction reaction.

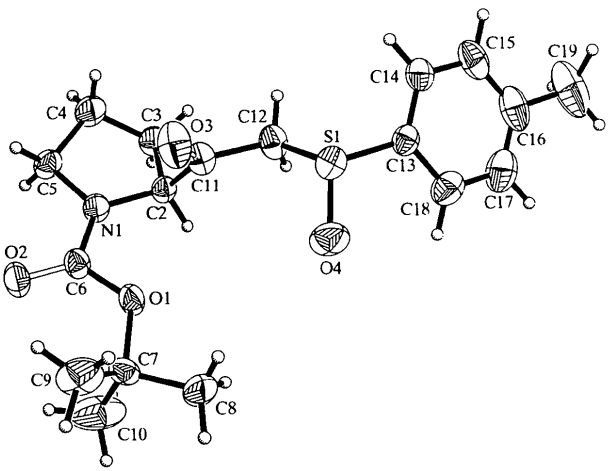


Fig. 1. A view of the title compound showing the labeling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level.

Experimental

A solution of (+)-*R*-methyl *p*-tolyl sulfoxide (8.50 mmol) in tetrahydrofuran (THF, 5 ml) was prepared and added dropwise to a solution of lithium diisopropylamide (17 mmol) in THF (50 ml). The mixture of the two solutions was then stirred for 1 h at 195 K. After this, *N*-(*tert*-butoxycarbonyl)-L-proline methyl ester (5.67 mmol) dissolved in THF (5 ml) was added dropwise and the reaction mixture was stirred for 4 h at 195 K. The unreacted sulfinyl carbanion was destroyed by addition of a saturated solution of ammonium chloride (25 ml) and the reaction product was extracted with Et_2O (2 × 20 ml). The organic phase was washed twice with a saturated solution of sodium chloride, dried and concentrated. The title compound was obtained as colorless crystals in 78% yield after flash chromatography (hexane–ethyl acetate, 3:7) and recrystallization from hexane–Et_2O solution [m.p. 365–366 K; $[\alpha]_D^{20}$ = +80.1° (c = 1.0, methanol)].

Crystal data

$C_{18}H_{25}NO_4S$	Mo $K\alpha$ radiation
M_r = 351.45	λ = 0.71073 Å
Orthorhombic	Cell parameters from 26 reflections
$P2_12_12_1$	θ = 5.2–12.5°
a = 9.450 (2) Å	μ = 0.188 mm^{-1}
b = 11.581 (1) Å	T = 293 (2) K
c = 17.544 (1) Å	Block
V = 1920.0 (5) Å^3	0.60 × 0.50 × 0.40 mm
Z = 4	Colorless
D_x = 1.216 Mg m^{-3}	
D_m not measured	

Data collection

Siemens *P*4/PC diffractometer	θ_{max} = 25°
$\theta/2\theta$ scans	h = 0 → 11
Absorption correction: none	k = 0 → 13
1946 measured reflections	l = 0 → 20
1946 independent reflections	3 standard reflections every 97 reflections
1298 reflections with $I > 2\sigma(I)$	intensity decay: <3%

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
$wR(F^2)$ = 0.135	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
S = 1.013	
1946 reflections	

218 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.1227P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
Extinction coefficient: 0.011 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.0 (2)

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

S1—O4	1.479 (5)	N1—C5	1.462 (7)
S1—C13	1.789 (6)	C2—C11	1.512 (8)
S1—C12	1.802 (6)	C2—C3	1.544 (7)
O1—C6	1.342 (6)	C3—C4	1.489 (8)
O1—C7	1.463 (6)	C4—C5	1.517 (8)
O2—C6	1.212 (6)	C7—C10	1.457 (9)
O3—C11	1.196 (7)	C7—C9	1.477 (9)
N1—C6	1.342 (7)	C7—C8	1.517 (8)
N1—C2	1.449 (7)	C11—C12	1.521 (8)
O4—S1—C13	107.5 (3)	O1—C6—N1	111.6 (4)
O4—S1—C12	106.5 (3)	C10—C7—O1	110.7 (5)
C13—S1—C12	95.5 (3)	C10—C7—C9	112.9 (7)
C6—O1—C7	121.3 (4)	O1—C7—C9	110.3 (5)
C6—N1—C2	124.8 (4)	C10—C7—C8	111.0 (7)
C6—N1—C5	121.6 (4)	O1—C7—C8	102.1 (4)
C2—N1—C5	113.6 (4)	C9—C7—C8	109.3 (5)
N1—C2—C11	112.5 (5)	O3—C11—C2	123.3 (5)
N1—C2—C3	102.2 (4)	O3—C11—C12	121.9 (6)
C11—C2—C3	111.1 (5)	C2—C11—C12	114.7 (5)
C4—C3—C2	103.7 (5)	C11—C12—S1	110.5 (4)
C3—C4—C5	105.5 (5)	C18—C13—C14	119.2 (6)
N1—C5—C4	102.7 (4)	C18—C13—S1	119.9 (5)
O2—C6—O1	125.7 (5)	C14—C13—S1	120.9 (5)
O2—C6—N1	122.7 (5)		

The proper enantiomer was chosen on the basis of the known configuration of the substrates and the method described by Flack (1983) was used to confirm the absolute configuration.

Data collection: *XSCANS* (Siemens, 1993). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*97.

We thank the Consejo Nacional de Ciencia e Tecnología (CONACyT–Mexico) for partial financial support (project 26375-E).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1464). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1607–1610

Intermolecular N—H···N and C—H···O interactions form one-dimensional chains comprising the two independent molecules of *N*,*N*′-dicyclohexyl-*N*-nicotinoylurea

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(*Received 12 May 1999; accepted 3 June 1999*)

Abstract

The title compound, $C_{19}H_{27}N_3O_2$, crystallizes in space group $P\bar{1}$ with two molecules in the asymmetric unit which differ slightly in conformation. Intermolecular N—H···N and C—H···O interactions generate a hydrogen-bonded ring system between the alternating molecules, graph set $R_2^2(16)$, with N···N distances of 3.021 (3) and 3.041 (3) Å, and C···O distances of 3.219 (3) and 3.296 (3) Å along the hydrogen-bonded chains.

Comment

The general principles underlying molecular recognition processes are reasonably well understood and hydrogen bonding in crystal structures can usually be rationalized in preferred combinations of hydrogen-bond donors and acceptors (Etter *et al.*, 1990). This allows comparison studies to be undertaken between classes of compounds containing analogous functional groups with a view to crystal engineering. However, compounds which are geometrically similar at the molecular level may differ at the supramolecular level, *e.g.* 2,2′-dipyridyl ketone and 2,2′-dipyridyl thioketone (Norsten *et al.*, 1999). Thus, in molecules where several different potential hydrogen-bond donors and acceptors are present (with cooperativity and/or competition among these interactions), the ability to deduce in advance the molecular packing