(2*R*)-*N*-{3-[Bis(methylsulfanyl)methyleneamino]-propionoyl}bornane-10,2-sultam

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Key indicators

Single-crystal X-ray study
$T = 100$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.062
Data-to-parameter ratio = 24.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{16}H_{26}N_2O_3S_3$, adjacent molecules are arranged in a head-to-tail manner and linked *via* weak C—H···S interactions, forming helical chains parallel to the *a* axis. These chains are connected *via* C—H···O interactions.

We dedicate this paper to the memory of Professor Leszek B. Łankiewicz (1960–2003), an outstanding scientist and magnificent person, who was our inspiration in so many ways.

Comment

β-Amino acids are a key part of many natural products displaying antibiotic, antifungal and cytotoxic properties (Cardillo & Tomasini, 1996), precursors of β-lactam antibiotics (Georg, 1993), and peptidomimetics showing remarkable resistance for enzymatic degradation (Steer *et al.*, 2000). β-Amino acids are also very interesting because of their tendency to form highly stable helical structures: 14-helix, 12-helix, 10/12-helix for penta-, hexa- and heptamers, composed of appropriately substituted β-amino acids (Seebach *et al.*, 1998; Cheng *et al.*, 2001). The stability of these structures is higher than those of similar ones formed by α-amino acids.

(I)
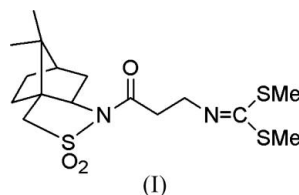
Even though β-amino acids can be obtained from natural sources and *via* chemical synthesis (Liu & Sibi, 2002), taking into account their significance and their further applications as peptide mimetics, there is a need for elaboration of new, effective and stereoselective methods for their preparation. One of the possible synthetic routes leading to the above compounds involves synthesis of a simple precursor (*e.g.* β-alanine) attached to a chiral auxiliary group. Among many chiral auxiliaries, bornane-10,2-sultam is one of the most popular and effective groups to create new chiral centres. This group was introduced by Oppolzer *et al.* (1984), and henceforth has been widely used for the synthesis of many compounds (Oppolzer, 1990; Szymańska *et al.*, 2000). We applied the Schiff base obtained from β-alanine and Oppolzer's sultam for the synthesis of some β^2-substituted β-amino acids (Woźniak *et al.*, 2006). For this purpose we modified the methodology described by Ponsinet *et al.* (2000). The precursor was chosen on the basis of the simplicity of its synthesis, the very high asymmetric induction characteristic for the sultam group and the high tendency of *N*-acylsultam derivatives to be crystalline.

In the title compound, (I), bond lengths and angles are typical for substituted bornane-10,2-sultam (Hughes *et al.*,
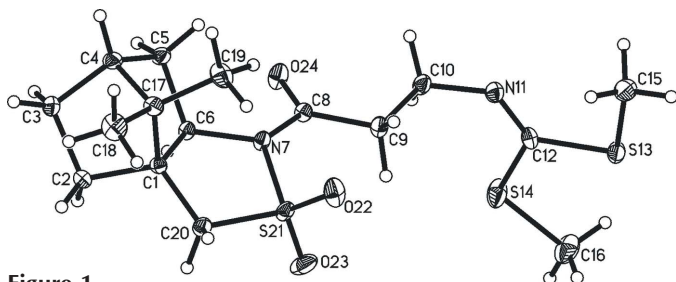
Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids.
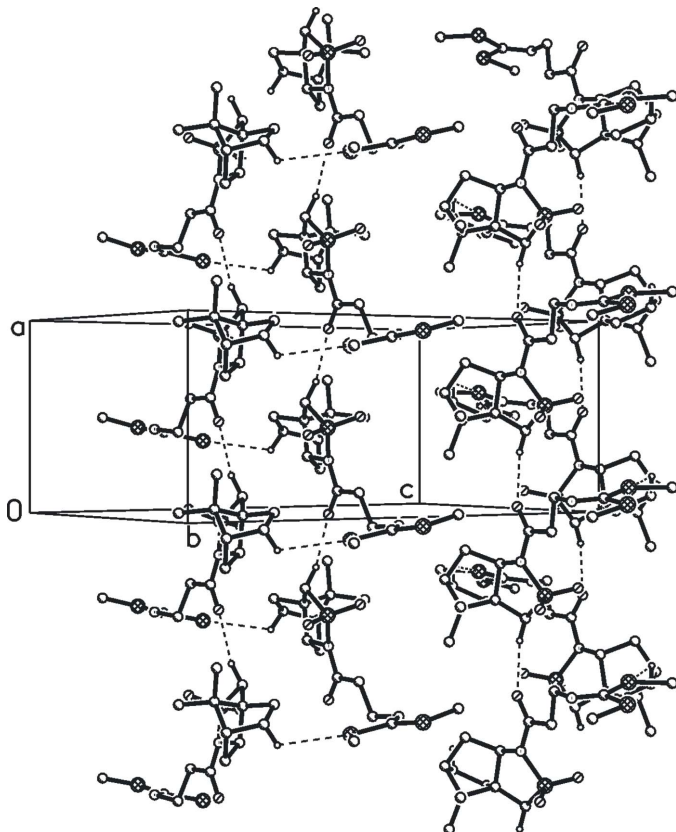
Figure 2
The packing of (I). The C—H···O and C—H···S interactions are represented by dashed lines. H atoms not involved in these interactions have been omitted.

1999; Raczko *et al.*, 2000; Szymański *et al.*, 2001) (Fig. 1 and Table 1). The cyclohexane ring adopts a fairly regular boat conformation, with ring puckering parameters $Q = 0.993$ (2) Å, $\theta = 88.65$ (7)° and $\varphi = 355.21$ (7)° (Cremer & Pople, 1975) and asymmetry parameter $\Delta C_s(C1) = 9.4$ (1)° (Duax *et al.*, 1976). Adjacent molecules of (I) are arranged in a head-to-tail manner and linked *via* weak C—H···S interactions (Table 2), forming helical chains parallel to the *a* axis (Fig. 2). These chains are connected *via* C—H···O interactions (Table 2).

Experimental

Compound (I) was synthesized from bornane-10,2-sultam and methyl 3-{[bis(methylthio)methylene]amino}propionate, according to the

procedure given by Oppolzer *et al.* (1989). Such a compound can, after enolization by treatment with either *n*-butyllithium or lithium diisopropylamide at 195 K, be alkylated by selected alkyl halides yielding precursors of β^2-amino acids (superscript 2 denotes the position of the introduced group α with reference to the carboxyl group). Crystals of (I) suitable for X-ray study were grown from an ethanol solution by slow evaporation at room temperature (m.p. 363–364 K).

Crystal data

$C_{16}H_{26}N_2O_3S_3$	Mo $K\alpha$ radiation
$M_r = 390.57$	Cell parameters from 16987
Orthorhombic, $P2_12_12_1$	reflections
$a = 7.742$ (2) Å	$\theta = 3$–$30°$
$b = 12.348$ (3) Å	$\mu = 0.42$ mm^{-1}
$c = 19.405$ (3) Å	$T = 100$ (2) K
$V = 1855.1$ (7) Å^3	Prism, colourless
$Z = 4$	$0.7 \times 0.5 \times 0.3$ mm
$D_x = 1.398$ Mg m^{-3}	

Data collection

Kuma KM-4 CCD diffractometer	$R_{int} = 0.035$
ω scans	$\theta_{max} = 30.0°$
Absorption correction: none	$h = -10 \rightarrow 10$
23438 measured reflections	$k = -17 \rightarrow 17$
5320 independent reflections	$l = -27 \rightarrow 25$
5197 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$+ 0.344P]$
$wR(F^2) = 0.062$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.007$
5320 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
221 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	Absolute structure: Flack (1983),
	2253 Friedel pairs
	Flack parameter: 0.02 (4)

Table 1
Selected geometric parameters (Å, °).

N7—C8	1.3921 (14)	C12—S14	1.7780 (13)
N7—S21	1.7019 (10)	C20—S21	1.7880 (12)
C10—N11	1.4737 (16)	S21—O22	1.4353 (11)
N11—C12	1.2700 (16)	S21—O23	1.4395 (10)
C12—S13	1.7738 (13)		
C1—C2—C3—C4	−3.28 (12)	C1—C6—N7—S21	26.16 (11)
C20—C1—C6—N7	−33.23 (12)	C5—C6—N7—S21	140.87 (8)
C4—C5—C6—C1	−7.82 (11)	S21—N7—C8—C9	−24.08 (15)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···S14[i]	0.99	2.95	3.484 (2)	115
C20—H20B···O24[ii]	0.99	2.28	3.242 (2)	164

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

All H atoms were positioned geometrically and refined as riding atoms, with C—H distances of 0.98–1.00 Å, and with U_{iso}(H) values of $1.5U_{eq}$(C) (for methyl H atoms) or $1.2U_{eq}$(C) (for the remaining H atoms). The absolute configuration based on the Flack (1983) parameter is consistent with that of bornane-10,2-sultam (Oppolzer, 1990; Kiegiel & Jurczak, 1999).

Data collection: *KM-4 CCD Software* (Oxford Diffraction, 2003); cell refinement: *KM-4 CCD Software*; data reduction: *KM-4 CCD Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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