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(^{S}*S*,2*S*,3*S*)-2-(2-Methylpropan-2-sulfinamido)-3-phenylbutyronitrile

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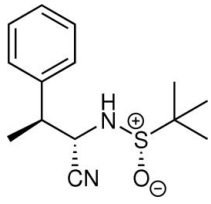
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.046; wR factor = 0.074; data-to-parameter ratio = 15.1.

The absolute configuration has been determined for the title compound, $C_{14}H_{20}N_2OS$. There are two independent molecules in the asymmetric unit. Intermolecular N—H···O hydrogen bonds are observed in the crystal packing, forming infinite chains with the base vectors [100] and [010]. Each chain contains only one of the two independent molecules.

Related literature

For uses of *tert*-butanesulfinimines, see: Ferreira *et al.* (2009). For asymmetric Strecker reactions utilizing this auxiliary, see: Davis *et al.* (1994); Li *et al.* (2003). For natural sources of (2*S*,3*S*)-β-methylphenylalanine, see: Singh *et al.* (2003); Kaneda (1992, 2002). For a related structure, see: Harms *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{20}N_2OS$
M_r = 264.38
Orthorhombic, $P2_12_12_1$
a = 9.0344 (4) Å
b = 9.0617 (5) Å
c = 35.767 (3) Å
V = 2928.1 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 100 K
0.36 × 0.08 × 0.06 mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.936, T_{max} = 1.041
15474 measured reflections
5160 independent reflections
3413 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.074
S = 0.77
5160 reflections
342 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), 2183 Friedel pairs
Flack parameter: −0.04 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.85 (2)	2.110 (17)	2.882 (3)	151 (3)
N21—H211···O21^{ii}	0.85 (2)	2.23 (2)	2.995 (4)	149 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2213).

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(^{S}*S*,2*S*,3*S*)-2-(2-Methylpropan-2-sulfinamido)-3-phenylbutyronitrile

Klaus Harms, Michael Marsch, Markus Oberthür and Peter Schüler

S1. Comment

Chiral sulfinimines have proven to be powerful and versatile precursors for the synthesis of nonproteinogenic amino acids (Ferreira *et al.*, 2008). They allow the stereoselective introduction of cyanide therefore representing an asymmetric modification of the Strecker reaction (Davis *et al.*, 1994); Li *et al.*, 2003). We have synthesized the title compound, (I), that can be hydrolyzed to give (2*S*,3*S*)-*β*-methylphenylalanine which is an amino acid found in the antibiotic families of the bottromycins and the mannopeptimycins (Singh *et al.*, 2003); Kaneda, 1992; and Kaneda, 2002). In this paper we report the crystal structure and absolute configuration of (I).

The molecular structure of (I) is presented in Fig. 1. There are two independent molecules in the asymmetric unit. The structure exhibits intermolecular N—H···O hydrogen bonds resulting in infinite one dimensional chains with the base vectors [1 0 0] and [0 1 0], respectively (details have have been provided in Table 1 and Fig. 2). Each chain contains only one of the two independent molecules.

The crystal structure and absolute configuration of a closely related compound has just been reported (Harms *et al.*, 2009).

S2. Experimental

Trimethylsilyl cyanide (TMSCN) (706 *μL*, 5.64 mmol) was added dropwise to a solution of (^{S}*S*)-(2-phenylpropyliden)-2-methyl-2-propansulfinylimin (1.12 g, 4.70 mmol) and CsF (858 mg, 5.64 mmol) in 50 ml *n*-hexane at 243 K. The mixture was stirred at this temperature for 14 h and subsequently quenched with semisaturated aqueous NH_4Cl solution. Extraction with EtOAc (2×50 mL) and drying of the combined organic phases ($MgSO_4$) yielded the crude mixture of 3*S* / 3*R* epimers. Crystallization from petrolether/EtOAc yielded 370 mg (1.41 mmol, 35%) of a 1:1 mixture of the diastereomers. Flash column chromatography of the mother liquor yielded 80 mg (303 mmol, 6%) of the pure 3*S* isomer, which had a slightly higher R_f-value (R_f=0.30 in petrol ether/EtOAc 2:1) than the 3*R* isomer of which 60 mg (227 mmol, 5%) could be isolated. The remaining fractions afforded 400 mg (1.53 mmol, 32%) of a roughly 1:1 mixture of the epimers. (^{S}*S*,2*S*,3*S*)-(2-Methylpropansulfinyl)-2-amino-3-phenylbutyronitrile was crystallized from petrol ether/THF.

S3. Refinement

The amino H atoms were isotropically refined with a restraint (0.85 Å) N—H distance. The other H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and allowed to ride on their parent atoms, with 1.5 $U_{eq}(C_{methyl})$ or 1.2 $U_{eq}(C)$.

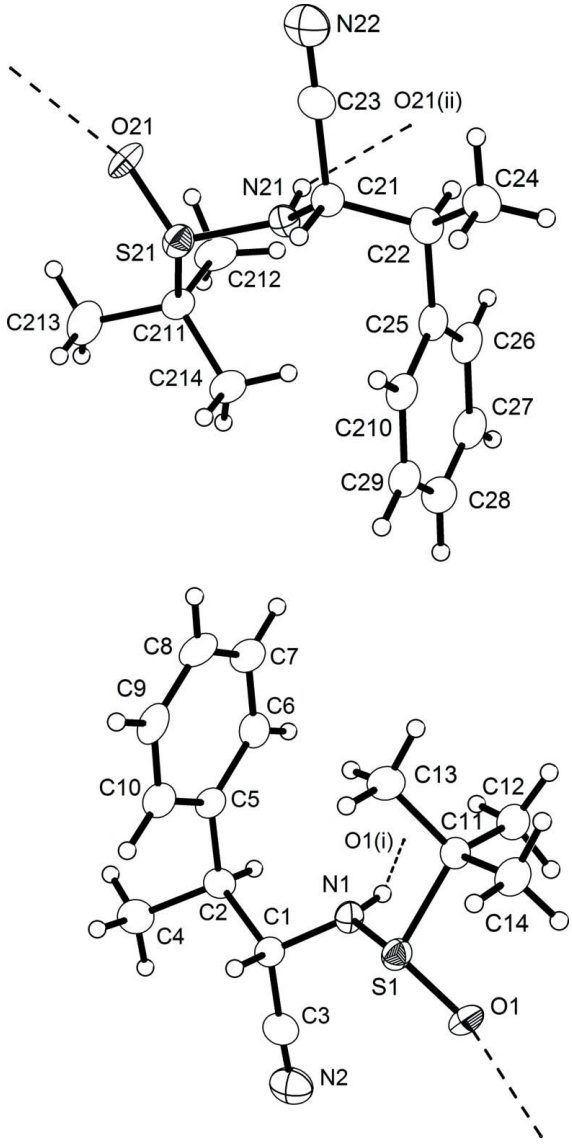

Figure 1

A view of the two molecules in the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. Symmetry operations, (i): x-1/2, -y+3/2, -z; (ii): -x, y+1/2, -z+1/2.

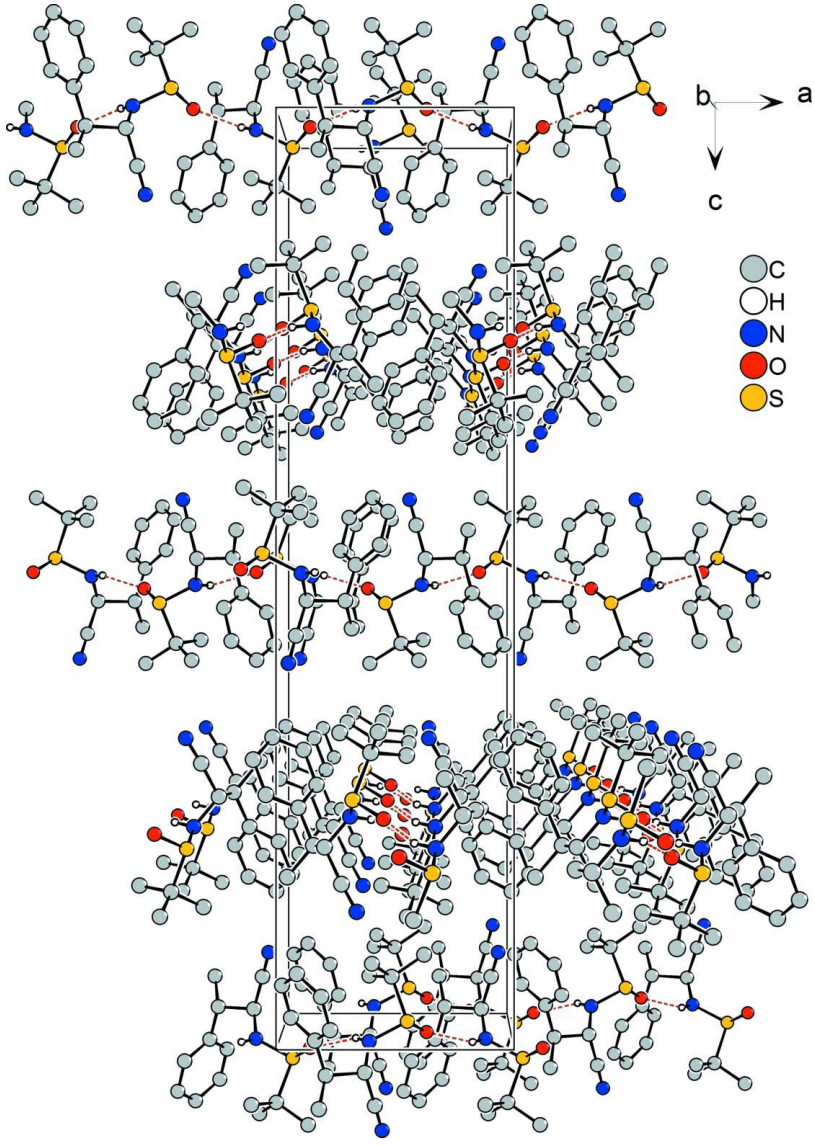

Figure 2

Unit cell packing of (I) viewed down the *b*-axis. Dotted lines indicate hydrogen bonds.

(*S*,2*S*,3*S*)-2-(2-Methylpropane-2-sulfinamido)-3- phenylbutyronitrile

Crystal data

$C_{14}H_{20}N_2OS$
$M_r = 264.38$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.0344$ (4) Å
$b = 9.0617$ (5) Å
$c = 35.767$ (3) Å
$V = 2928.1$ (3) Å^3
$Z = 8$

$F(000) = 1136$
$D_x = 1.199$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10131 reflections
$\theta = 2.3$–25°
$\mu = 0.21$ mm^{-1}
$T = 100$ K
Prism, colourless
0.36 × 0.08 × 0.06 mm

Data collection

Stoe IPDS II diffractometer
Radiation source: sealed tube
Graphite monochromator
area detetor, ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.936, T_{max} = 1.041
15474 measured reflections
5160 independent reflections
3413 reflections with $I > 2\sigma(I)$
R_{int} = 0.093
θ_{max} = 25°, θ_{min} = 2.3°
h = −10→10
k = −9→10
l = −41→42

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.074
S = 0.77
5160 reflections
342 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: CH inferred from neighbouring sites, NH located
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0105P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0011 (3)
Absolute structure: Flack (1983), 2183 Friedel pairs
Absolute structure parameter: −0.04 (9)

Special details

Experimental. ν_{max}/cm^{-1} 3232 (br), 2963 (w), 2930 (w), 2872(w), 1492 (w), 1454 (*m*), 1422 (*m*), 1364 (w), 1113 (w), 1085 (*m*), 1054 (*s*), 1016(*m*); δ_H (300 MHz; DMSO) 0.75 (s, 9H, *t*Bu), 1.37 (d, 3H, $^3J_{Me,CH}$ = 7.1 Hz, CH_3), 3.10 (dq, 1H, $^3J_{CH,CHN}$ = 10.3, $J_{CH,Me}$ = 7.1 Hz, CH), 4.51 (pt, 1H, $^3J_{CHN,CH}$ = 10.3 Hz, CHN), 6.24 (d, 1H, $^3J_{NH,CHN}$ = 10.5 Hz, NH), 7.14 – 7.32 (m, 5H, CH_{arom}); δ_C (75 MHz; DMSO-d_6)18.4 (CH_3), 21.9 ($C(CH_3)_3$), 43.2 (CH), 52.8 (CHN), 55.9 ($C(CH_3)_3$), 120.2 (CN), 126.7 (*p*-CH_{arom}), 127.7 (CH_{arom}), 128.2 (CH_{arom}), 141.9 (*i*-C_{arom}); $[\alpha]_D^{23}$ -1.0 (c 1.00 in $CHCl_3$).

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C21	0.2714 (4)	0.3582 (4)	0.27085 (9)	0.0236 (9)
H21	0.36	0.2946	0.2662	0.028*
C22	0.3222 (4)	0.5196 (4)	0.27258 (10)	0.0270 (9)
H22	0.232	0.5812	0.2769	0.032*
C23	0.2020 (4)	0.3113 (5)	0.30640 (10)	0.0288 (9)
C24	0.4285 (4)	0.5485 (5)	0.30544 (10)	0.0336 (10)
H24A	0.5189	0.4903	0.302	0.05*
H24B	0.3805	0.5198	0.3289	0.05*

H24C	0.4537	0.6536	0.3063	0.05*
C25	0.3851 (4)	0.5647 (4)	0.23521 (10)	0.0251 (9)
C26	0.3155 (4)	0.6706 (4)	0.21346 (10)	0.0310 (9)
H26	0.229	0.7179	0.2227	0.037*
C27	0.3702 (4)	0.7092 (4)	0.17816 (10)	0.0332 (10)
H27	0.3217	0.783	0.1638	0.04*
C28	0.4935 (4)	0.6406 (4)	0.16434 (11)	0.0354 (11)
H28	0.5303	0.6659	0.1403	0.042*
C29	0.5647 (4)	0.5341 (4)	0.18560 (10)	0.0308 (9)
H29	0.6501	0.4861	0.176	0.037*
C210	0.5117 (4)	0.4971 (4)	0.22081 (11)	0.0265 (9)
H210	0.5622	0.425	0.2353	0.032*
C211	0.0856 (4)	0.2374 (4)	0.17447 (11)	0.0281 (9)
C212	−0.0540 (4)	0.3289 (5)	0.17864 (10)	0.0370 (10)
H21A	−0.1007	0.3413	0.1541	0.056*
H21B	−0.0287	0.4259	0.1889	0.056*
H21C	−0.1227	0.2785	0.1956	0.056*
C213	0.0538 (5)	0.0964 (5)	0.15275 (11)	0.0448 (11)
H21D	−0.0214	0.0386	0.166	0.067*
H21E	0.1449	0.0383	0.1505	0.067*
H21F	0.0175	0.1216	0.1277	0.067*
C214	0.2103 (4)	0.3255 (5)	0.15608 (10)	0.0322 (9)
H21G	0.3006	0.2657	0.1556	0.048*
H21H	0.2283	0.4158	0.1704	0.048*
H21I	0.1817	0.3514	0.1305	0.048*
N21	0.1689 (3)	0.3395 (3)	0.23975 (7)	0.0228 (7)
N22	0.1415 (4)	0.2754 (4)	0.33308 (9)	0.0399 (9)
O21	0.0331 (3)	0.0854 (3)	0.23728 (7)	0.0316 (6)
S21	0.15503 (10)	0.17532 (11)	0.22032 (3)	0.0260 (2)
C1	0.8730 (4)	0.4752 (4)	−0.02331 (10)	0.0228 (8)
H1	0.9404	0.3911	−0.0171	0.027*
C2	0.7114 (3)	0.4153 (4)	−0.02369 (10)	0.0252 (8)
H2	0.6449	0.4999	−0.03	0.03*
C3	0.9145 (4)	0.5311 (4)	−0.06101 (11)	0.0268 (9)
C4	0.6885 (4)	0.2967 (4)	−0.05361 (10)	0.0325 (10)
H4A	0.7481	0.2097	−0.0475	0.049*
H4B	0.7189	0.3354	−0.078	0.049*
H4C	0.5836	0.2691	−0.0545	0.049*
C5	0.6698 (4)	0.3632 (4)	0.01507 (9)	0.0242 (9)
C6	0.5653 (4)	0.4409 (4)	0.03603 (10)	0.0283 (9)
H6	0.5181	0.5254	0.0258	0.034*
C7	0.5311 (4)	0.3941 (5)	0.07186 (10)	0.0320 (9)
H7	0.4586	0.4467	0.0857	0.038*
C8	0.5988 (4)	0.2738 (4)	0.08799 (11)	0.0333 (10)
H8	0.5752	0.2442	0.1128	0.04*
C9	0.7019 (4)	0.1975 (4)	0.06715 (11)	0.0317 (9)
H9	0.7496	0.1139	0.0777	0.038*
C10	0.7372 (4)	0.2407 (4)	0.03095 (11)	0.0297 (9)

H10	0.8079	0.1861	0.017	0.036*
C11	0.9830 (4)	0.6723 (4)	0.07127 (9)	0.0234 (8)
C12	0.8840 (4)	0.8074 (4)	0.06764 (10)	0.0320 (9)
H12A	0.7922	0.78	0.0548	0.048*
H12B	0.9355	0.8836	0.0532	0.048*
H12C	0.8606	0.8456	0.0926	0.048*
C13	0.9020 (4)	0.5441 (4)	0.08954 (11)	0.0317 (10)
H13A	0.8719	0.5721	0.1149	0.047*
H13B	0.9677	0.4582	0.0907	0.047*
H13C	0.814	0.5194	0.0748	0.047*
C14	1.1218 (4)	0.7103 (4)	0.09391 (10)	0.0326 (10)
H14A	1.0935	0.7347	0.1196	0.049*
H14B	1.1717	0.7951	0.0825	0.049*
H14C	1.189	0.6254	0.0941	0.049*
N1	0.8892 (3)	0.5844 (3)	0.00524 (8)	0.0215 (7)
N2	0.9438 (4)	0.5784 (4)	−0.08972 (9)	0.0397 (8)
O1	1.1317 (2)	0.7367 (3)	0.00919 (7)	0.0304 (6)
S1	1.05045 (10)	0.60907 (10)	0.02574 (3)	0.0241 (2)
H211	0.087 (2)	0.384 (4)	0.2437 (9)	0.031 (11)*
H1A	0.831 (3)	0.658 (2)	0.0059 (9)	0.027 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C21	0.0206 (18)	0.025 (2)	0.025 (2)	0.0023 (15)	−0.0032 (14)	0.0016 (16)
C22	0.023 (2)	0.027 (2)	0.031 (2)	0.0031 (16)	−0.0027 (16)	−0.0024 (17)
C23	0.029 (2)	0.030 (2)	0.027 (2)	0.0060 (18)	0.0026 (17)	0.001 (2)
C24	0.030 (2)	0.039 (3)	0.032 (2)	−0.0029 (19)	−0.0009 (18)	−0.0053 (18)
C25	0.027 (2)	0.021 (2)	0.0267 (19)	−0.0030 (16)	−0.0063 (16)	0.0001 (16)
C26	0.024 (2)	0.026 (2)	0.043 (2)	−0.0047 (18)	−0.0068 (16)	−0.002 (2)
C27	0.041 (2)	0.029 (2)	0.030 (2)	−0.0087 (19)	−0.0113 (19)	0.0050 (18)
C28	0.036 (2)	0.040 (3)	0.031 (2)	−0.0128 (19)	−0.0006 (17)	−0.0053 (19)
C29	0.026 (2)	0.026 (2)	0.041 (2)	−0.0049 (18)	0.0029 (19)	−0.0031 (18)
C210	0.019 (2)	0.022 (2)	0.038 (2)	−0.0009 (15)	−0.0020 (17)	0.0003 (19)
C211	0.025 (2)	0.029 (2)	0.031 (2)	−0.0043 (17)	0.0041 (16)	0.0008 (17)
C212	0.031 (2)	0.041 (2)	0.039 (2)	0.003 (2)	0.0036 (18)	0.015 (2)
C213	0.048 (2)	0.038 (3)	0.049 (3)	−0.016 (2)	−0.004 (2)	−0.002 (2)
C214	0.029 (2)	0.033 (2)	0.035 (2)	−0.0036 (19)	0.0043 (16)	0.004 (2)
N21	0.0181 (17)	0.0239 (17)	0.0263 (15)	0.0025 (14)	−0.0009 (13)	−0.0021 (14)
N22	0.039 (2)	0.038 (2)	0.043 (2)	0.0162 (17)	0.0033 (18)	0.0054 (17)
O21	0.0292 (14)	0.0269 (15)	0.0386 (15)	−0.0126 (12)	0.0023 (11)	0.0106 (13)
S21	0.0233 (5)	0.0241 (5)	0.0306 (5)	−0.0019 (4)	0.0000 (4)	0.0011 (4)
C1	0.0180 (18)	0.0209 (19)	0.030 (2)	0.0025 (14)	0.0008 (16)	0.0009 (17)
C2	0.0179 (18)	0.030 (2)	0.0282 (19)	−0.0001 (15)	−0.0006 (15)	−0.0043 (19)
C3	0.016 (2)	0.033 (2)	0.032 (2)	0.0072 (16)	−0.0033 (16)	−0.0015 (18)
C4	0.023 (2)	0.040 (3)	0.035 (2)	−0.0018 (17)	−0.0022 (16)	−0.005 (2)
C5	0.0168 (18)	0.026 (2)	0.030 (2)	−0.0042 (16)	−0.0004 (15)	−0.0012 (16)
C6	0.0199 (19)	0.027 (2)	0.038 (2)	−0.0019 (17)	−0.0033 (17)	−0.0041 (17)

C7	0.027 (2)	0.037 (2)	0.032 (2)	−0.010 (2)	0.0006 (17)	−0.002 (2)
C8	0.028 (2)	0.040 (3)	0.032 (2)	−0.0152 (18)	0.0005 (17)	0.004 (2)
C9	0.031 (2)	0.024 (2)	0.041 (2)	−0.0078 (17)	−0.0058 (17)	0.005 (2)
C10	0.0200 (19)	0.028 (2)	0.041 (2)	−0.0025 (16)	0.0004 (18)	−0.0013 (19)
C11	0.026 (2)	0.0224 (19)	0.0222 (19)	−0.0018 (16)	0.0003 (14)	−0.0002 (17)
C12	0.036 (2)	0.029 (2)	0.031 (2)	−0.0051 (18)	0.0019 (16)	−0.0009 (19)
C13	0.029 (2)	0.035 (2)	0.031 (2)	−0.0019 (17)	0.0029 (17)	0.0009 (19)
C14	0.026 (2)	0.035 (3)	0.037 (2)	−0.0069 (17)	−0.0029 (17)	−0.0021 (19)
N1	0.0145 (16)	0.0214 (18)	0.0287 (16)	0.0009 (13)	−0.0041 (12)	−0.0014 (14)
N2	0.0345 (19)	0.044 (2)	0.041 (2)	0.0121 (17)	0.0020 (17)	0.0041 (18)
O1	0.0252 (13)	0.0318 (15)	0.0342 (15)	−0.0099 (12)	0.0069 (12)	0.0021 (12)
S1	0.0194 (4)	0.0229 (5)	0.0300 (5)	−0.0018 (4)	−0.0001 (4)	−0.0006 (5)

Geometric parameters (Å, º)

C21—N21	1.457 (4)	C1—N1	1.429 (4)
C21—C23	1.480 (5)	C1—C3	1.488 (5)
C21—C22	1.535 (5)	C1—C2	1.557 (4)
C21—H21	1	C1—H1	1
C22—C25	1.509 (5)	C2—C5	1.512 (5)
C22—C24	1.540 (5)	C2—C4	1.530 (5)
C22—H22	1	C2—H2	1
C23—N22	1.146 (4)	C3—N2	1.144 (4)
C24—H24A	0.98	C4—H4A	0.98
C24—H24B	0.98	C4—H4B	0.98
C24—H24C	0.98	C4—H4C	0.98
C25—C26	1.386 (5)	C5—C10	1.387 (5)
C25—C210	1.396 (5)	C5—C6	1.396 (5)
C26—C27	1.400 (5)	C6—C7	1.385 (5)
C26—H26	0.95	C6—H6	0.95
C27—C28	1.369 (5)	C7—C8	1.377 (5)
C27—H27	0.95	C7—H7	0.95
C28—C29	1.387 (5)	C8—C9	1.379 (5)
C28—H28	0.95	C8—H8	0.95
C29—C210	1.388 (5)	C9—C10	1.390 (5)
C29—H29	0.95	C9—H9	0.95
C210—H210	0.95	C10—H10	0.95
C211—C212	1.516 (5)	C11—C13	1.520 (5)
C211—C213	1.523 (5)	C11—C12	1.522 (5)
C211—C214	1.529 (5)	C11—C14	1.532 (4)
C211—S21	1.843 (4)	C11—S1	1.831 (3)
C212—H21A	0.98	C12—H12A	0.98
C212—H21B	0.98	C12—H12B	0.98
C212—H21C	0.98	C12—H12C	0.98
C213—H21D	0.98	C13—H13A	0.98
C213—H21E	0.98	C13—H13B	0.98
C213—H21F	0.98	C13—H13C	0.98
C214—H21G	0.98	C14—H14A	0.98

C214—H21H	0.98	C14—H14B	0.98
C214—H21I	0.98	C14—H14C	0.98
N21—S21	1.647 (3)	N1—S1	1.646 (3)
N21—H211	0.85 (2)	N1—H1A	0.85 (2)
O21—S21	1.498 (2)	O1—S1	1.493 (2)
N21—C21—C23	110.7 (3)	N1—C1—C3	112.7 (3)
N21—C21—C22	109.4 (3)	N1—C1—C2	110.1 (3)
C23—C21—C22	111.5 (3)	C3—C1—C2	110.3 (3)
N21—C21—H21	108.4	N1—C1—H1	107.9
C23—C21—H21	108.4	C3—C1—H1	107.9
C22—C21—H21	108.4	C2—C1—H1	107.9
C25—C22—C21	109.6 (3)	C5—C2—C4	112.8 (3)
C25—C22—C24	113.3 (3)	C5—C2—C1	109.5 (3)
C21—C22—C24	112.3 (3)	C4—C2—C1	112.2 (3)
C25—C22—H22	107.1	C5—C2—H2	107.3
C21—C22—H22	107.1	C4—C2—H2	107.3
C24—C22—H22	107.1	C1—C2—H2	107.3
N22—C23—C21	176.6 (4)	N2—C3—C1	177.7 (4)
C22—C24—H24A	109.5	C2—C4—H4A	109.5
C22—C24—H24B	109.5	C2—C4—H4B	109.5
H24A—C24—H24B	109.5	H4A—C4—H4B	109.5
C22—C24—H24C	109.5	C2—C4—H4C	109.5
H24A—C24—H24C	109.5	H4A—C4—H4C	109.5
H24B—C24—H24C	109.5	H4B—C4—H4C	109.5
C26—C25—C210	117.9 (4)	C10—C5—C6	118.7 (3)
C26—C25—C22	120.9 (3)	C10—C5—C2	121.1 (3)
C210—C25—C22	121.1 (3)	C6—C5—C2	120.2 (3)
C25—C26—C27	121.3 (4)	C7—C6—C5	119.6 (4)
C25—C26—H26	119.4	C7—C6—H6	120.2
C27—C26—H26	119.4	C5—C6—H6	120.2
C28—C27—C26	120.0 (4)	C8—C7—C6	122.1 (4)
C28—C27—H27	120	C8—C7—H7	119
C26—C27—H27	120	C6—C7—H7	119
C27—C28—C29	119.7 (4)	C7—C8—C9	118.1 (4)
C27—C28—H28	120.1	C7—C8—H8	121
C29—C28—H28	120.1	C9—C8—H8	121
C28—C29—C210	120.4 (4)	C8—C9—C10	121.1 (4)
C28—C29—H29	119.8	C8—C9—H9	119.4
C210—C29—H29	119.8	C10—C9—H9	119.4
C29—C210—C25	120.8 (4)	C5—C10—C9	120.4 (4)
C29—C210—H210	119.6	C5—C10—H10	119.8
C25—C210—H210	119.6	C9—C10—H10	119.8
C212—C211—C213	110.6 (3)	C13—C11—C12	111.6 (3)
C212—C211—C214	111.7 (3)	C13—C11—C14	109.8 (3)
C213—C211—C214	110.9 (3)	C12—C11—C14	110.2 (3)
C212—C211—S21	111.2 (2)	C13—C11—S1	107.7 (3)
C213—C211—S21	105.2 (3)	C12—C11—S1	111.8 (2)

C214—C211—S21	107.0 (2)	C14—C11—S1	105.5 (2)
C211—C212—H21A	109.5	C11—C12—H12A	109.5
C211—C212—H21B	109.5	C11—C12—H12B	109.5
H21A—C212—H21B	109.5	H12A—C12—H12B	109.5
C211—C212—H21C	109.5	C11—C12—H12C	109.5
H21A—C212—H21C	109.5	H12A—C12—H12C	109.5
H21B—C212—H21C	109.5	H12B—C12—H12C	109.5
C211—C213—H21D	109.5	C11—C13—H13A	109.5
C211—C213—H21E	109.5	C11—C13—H13B	109.5
H21D—C213—H21E	109.5	H13A—C13—H13B	109.5
C211—C213—H21F	109.5	C11—C13—H13C	109.5
H21D—C213—H21F	109.5	H13A—C13—H13C	109.5
H21E—C213—H21F	109.5	H13B—C13—H13C	109.5
C211—C214—H21G	109.5	C11—C14—H14A	109.5
C211—C214—H21H	109.5	C11—C14—H14B	109.5
H21G—C214—H21H	109.5	H14A—C14—H14B	109.5
C211—C214—H21I	109.5	C11—C14—H14C	109.5
H21G—C214—H21I	109.5	H14A—C14—H14C	109.5
H21H—C214—H21I	109.5	H14B—C14—H14C	109.5
C21—N21—S21	118.4 (2)	C1—N1—S1	120.2 (2)
C21—N21—H211	112 (2)	C1—N1—H1A	120 (2)
S21—N21—H211	115 (3)	S1—N1—H1A	115 (2)
O21—S21—N21	112.12 (15)	O1—S1—N1	111.33 (15)
O21—S21—C211	106.03 (15)	O1—S1—C11	105.92 (16)
N21—S21—C211	97.20 (16)	N1—S1—C11	98.29 (15)
N21—C21—C22—C25	−54.4 (4)	N1—C1—C2—C5	−53.4 (4)
C23—C21—C22—C25	−177.1 (3)	C3—C1—C2—C5	−178.3 (3)
N21—C21—C22—C24	178.7 (3)	N1—C1—C2—C4	−179.5 (3)
C23—C21—C22—C24	56.0 (4)	C3—C1—C2—C4	55.6 (4)
N21—C21—C23—N22	−17 (8)	N1—C1—C3—N2	−44 (10)
C22—C21—C23—N22	105 (7)	C2—C1—C3—N2	80 (10)
C21—C22—C25—C26	114.1 (4)	C4—C2—C5—C10	58.2 (4)
C24—C22—C25—C26	−119.7 (4)	C1—C2—C5—C10	−67.5 (4)
C21—C22—C25—C210	−63.4 (4)	C4—C2—C5—C6	−123.8 (3)
C24—C22—C25—C210	62.8 (4)	C1—C2—C5—C6	110.4 (3)
C210—C25—C26—C27	−0.1 (5)	C10—C5—C6—C7	−0.3 (5)
C22—C25—C26—C27	−177.7 (3)	C2—C5—C6—C7	−178.3 (3)
C25—C26—C27—C28	0.8 (5)	C5—C6—C7—C8	1.1 (5)
C26—C27—C28—C29	−0.6 (5)	C6—C7—C8—C9	−1.1 (5)
C27—C28—C29—C210	−0.2 (5)	C7—C8—C9—C10	0.2 (5)
C28—C29—C210—C25	0.9 (5)	C6—C5—C10—C9	−0.5 (5)
C26—C25—C210—C29	−0.8 (5)	C2—C5—C10—C9	177.5 (3)
C22—C25—C210—C29	176.8 (3)	C8—C9—C10—C5	0.5 (5)
C23—C21—N21—S21	−83.2 (3)	C3—C1—N1—S1	−85.7 (3)
C22—C21—N21—S21	153.6 (2)	C2—C1—N1—S1	150.7 (2)
C21—N21—S21—O21	93.0 (3)	C1—N1—S1—O1	98.6 (3)
C21—N21—S21—C211	−156.4 (2)	C1—N1—S1—C11	−150.6 (3)

C212—C211—S21—O21	59.6 (3)	C13—C11—S1—O1	−178.8 (2)
C213—C211—S21—O21	−60.2 (3)	C12—C11—S1—O1	58.2 (3)
C214—C211—S21—O21	−178.2 (2)	C14—C11—S1—O1	−61.6 (3)
C212—C211—S21—N21	−56.0 (3)	C13—C11—S1—N1	66.1 (3)
C213—C211—S21—N21	−175.7 (2)	C12—C11—S1—N1	−56.9 (3)
C214—C211—S21—N21	66.2 (3)	C14—C11—S1—N1	−176.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.85 (2)	2.11 (2)	2.882 (3)	151 (3)
N21—H211···O21^{ii}	0.85 (2)	2.23 (2)	2.995 (4)	149 (3)

Symmetry codes: (i) $x-1/2, -y+3/2, -z$; (ii) $-x, y+1/2, -z+1/2$.