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# *tert*-Butyl *N*-[1-diazoacetyl-3-(methylsulfanyl)propyl]carbamate

**Tahir Mehmood,[a] Javid H. Zaidi[a]* and Peter G. Jones[b]**

[a]Department of Chemistry, Quaid-I-Azam University, Islamabad 45320, Pakistan, and [b]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Hagenring 30, 38106 Braunschweig, Germany
Correspondence e-mail: javid_zaidi@qau.edu.pk

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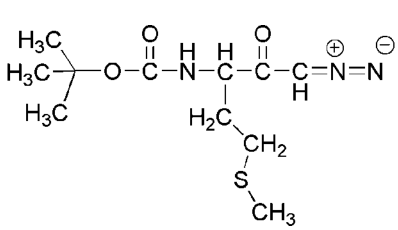
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.023; $wR$ factor = 0.060; data-to-parameter ratio = 18.0.

In the enantiomerically pure title compound, $C_{11}H_{19}N_3O_3S$, the chain C—N—C(O)—O—C—C (from the asymmetric carbon to a methyl of the *tert*-butyl group) displays an extended conformation. In the crystal, molecules are linked into chains parallel to the *c* axis by classical N—H···$O_{diazocarbonyl}$ hydrogen bonding and an unusual intermolecular three-centre interaction involving the amino acid (aa) carbonyl $O_{aa}$ and the diazocarbonyl grouping C(O)—CH—N≡N, with H···$O_{aa}$ = 2.51 Å and N···$O_{aa}$ = 2.8141 (14) Å.

## Related literature

For the applications of $\alpha$-diazocarbonyl compounds in organic and, especially, natural product synthesis, see: Padwa & Weingarten (1996). The ready availability, relative stability and facile decomposition of these compounds under various conditions make them useful intermediates, see: Doyle *et al.* (1998). $\alpha$-Diazoketones undergo a variety of transformations, see: Ye & McKervey (1994). Asymmetric versions of diazocarbonyl reactions have been reported to produce enantiomerically pure compounds, see: Doyle & McKervey (1997). The Arndt-Eistert synthesis, which consists of conversion of activated carboxylic acids to diazoketones by the action of diazomethane followed by Wolf rearrangement, has become widely used in recent years for the synthesis of $\beta$-peptides and $\beta$-amino acid derivatives from appropriately protected $\alpha$-amino acids, see: Müller *et al.* (1998).



## Experimental

### *Crystal data*

| | |
|---|---|
| $C_{11}H_{19}N_3O_3S$ | $Z$ = 3 |
| $M_r$ = 273.35 | Cu $K\alpha$ radiation |
| Trigonal, $P3_1$ | $\mu$ = 1.93 mm$^{-1}$ |
| $a$ = 9.7915 (3) Å | $T$ = 100 K |
| $c$ = 13.8581 (5) Å | 0.20 × 0.20 × 0.15 mm |
| $V$ = 1150.62 (6) Å$^3$ | |

### *Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur Nova A diffractometer | $T_{min}$ = 0.717, $T_{max}$ = 1.000 (expected range = 0.537–0.749) |
| Absorption correction: multi-scan (CrysAlis Pro; Oxford Diffraction, 2009) | 16152 measured reflections |
| | 3073 independent reflections |
| | 3051 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.032 |

### *Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.023 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.060 | $\Delta\rho_{max}$ = 0.15 e Å$^{-3}$ |
| $S$ = 1.05 | $\Delta\rho_{min}$ = −0.14 e Å$^{-3}$ |
| 3073 reflections | Absolute structure: Flack (1983), 1474 Freidel pairs |
| 171 parameters | Flack parameter: 0.023 (10) |
| 1 restraint | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H01···O3$^{i}$ | 0.842 (16) | 2.027 (16) | 2.8465 (14) | 164.1 (14) |
| C8—H8···O2$^{i}$ | 0.95 | 2.51 | 2.9686 (16) | 110 |
| C11—H11$B$···O2$^{ii}$ | 0.98 | 2.52 | 3.457 (2) | 160 |
| C3—H3$B$···O3$^{iii}$ | 0.98 | 2.67 | 3.5693 (17) | 152 |
| C1—H1$C$···S$^{iv}$ | 0.98 | 2.95 | 3.9281 (16) | 177 |

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

Data collection: *CrysAlis Pro* (Oxford Diffraction, 2009); cell refinement: *CrysAlis Pro*; data reduction: *CrysAlis Pro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2855).

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# supporting information

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## *tert*-Butyl *N*-[1-diazoacetyl-3-(methylsulfanyl)propyl]carbamate

**Tahir Mehmood, Javid H. Zaidi and Peter G. Jones**

### S1. Comment

*α*-Diazocarbonyl compounds find widespread applications in organic and, especially, natural product synthesis (Padwa & Weingarten, 1996). The ready availability, relative stability and facile decomposition of these compounds under various conditions (*e.g.* thermal, photochemical; acid-, base- and transition-metal-catalysis) make them useful intermediates (Doyle *et al.* 1998). Furthermore, *α*-diazoketones undergo a variety of transformations such as cyclopropanation, aziridine formation, ylide formation, C–H or C–*X* insertion reactions and cyclization reactions (Ye & McKervey, 1994). These reactions are chemoselective, and promote the formation of new carbon-carbon and carbon-heteroatom bonds under mild conditions. Asymmetric versions of diazocarbonyl reactions have been reported to produce enantiomerically pure compounds (Doyle & McKervey, 1997). One such method is the Arndt-Eistert synthesis, which consists of conversion of activated carboxylic acids to diazoketones by the action of diazomethane, followed by Wolf rearrangement. The method has become widely used in recent years for the synthesis of *β*-peptides and *β*-amino acid derivatives from appropriately protected *α*-amino acids (Müller *et al.* 1998). Here we present the structure of an *α*-diazocarbonyl compound based on methionine.

The structure of the title compound is shown in Fig. 1. Molecular dimensions may be regarded as normal. The two essentially planar groupings N1,O1,O2,C2,4,5,6 and N2,N3,O3,C6,7,8 (r.m.s. deviations 0.04, 0.02 Å) subtend an interplanar angle of 84.75 (3)°. The atom chain C2 to C6 displays an extended conformation (minimum absolute torsion angle 170°).

The main feature of the molecular packing is the classical H bond N1—H1···O3, which links the molecules *via* the $3_1$ screw operator to form chains parallel to the *z* axis (Fig. 2). Within the chains, an unusual three-centre interaction is also observed, whereby the carbonyl oxygen O2 is involved in short contacts to H8 and N2 of the diazocarbonyl group of a neighbouring molecule. The former is far from linear (angle 110°) but this is not unusual for three-centre interactions. The latter may be interpreted as a dipole-dipole interaction [dimensions: N2···O2 2.8141 (14) Å, C8—N2···O2 73.5 (1)°]. The remaining "weak" C—H···O interactions (Table 1) link neighbouring chains; H3B···O3 is implicit between the chains of Fig. 2 but is omitted for clarity.

### S2. Experimental

10 mmol of BOC-protected methionine was dissolved in 50 ml of dry distilled THF under inert conditions. To maintain basic conditions 12 mmol (1.66 ml) of triethylamine was added. Then 10 mmol (0.95 ml) of ethyl chloroformate was added, and the mixture stirred for 15 min at 248 K. 13 mmol of diazomethane were then added at 268 K and the mixture was further stirred for 30 min. After this temperature was allowed to rise to room temperature over 3 h. The reaction was then quenched with 3–4 drops of glacial acetic acid. The solvent was evaporated under vacuum. The residue was dissolved in ethyl acetate, extracted with aq. solutions of $NaHCO_3$ and $NH_4Cl$ and dried over anhydrous $MgSO_4$. The crude product was purified by column chromatography (yield 85%; m.p.326-328 K).

### S3. Refinement

The NH hydrogen was refined freely. Methyl H atoms were identified in difference syntheses, idealized and refined as rigid groups with C—H 0.98 Å and H—C—H angles 109.5°, allowed to rotate but not tip. Other H atoms were placed in calculated positions and refined using a riding model with C—H 0.98 Å (methylene) or 0.99 Å (methine); hydrogen *U* values were fixed at 1.5 × *U*(eq) of the parent atom for methyl H and 1.2 × *U*(eq) of the parent atom for other C—H. Data are 100% complete to 2$\theta$ 145°. The absolute configuration *S* at C6 (and thus the space group $P3_1$ rather than its enantiomer $P3_2$) was determined by the Flack (1983) parameter, which refined to 0.023 (10).

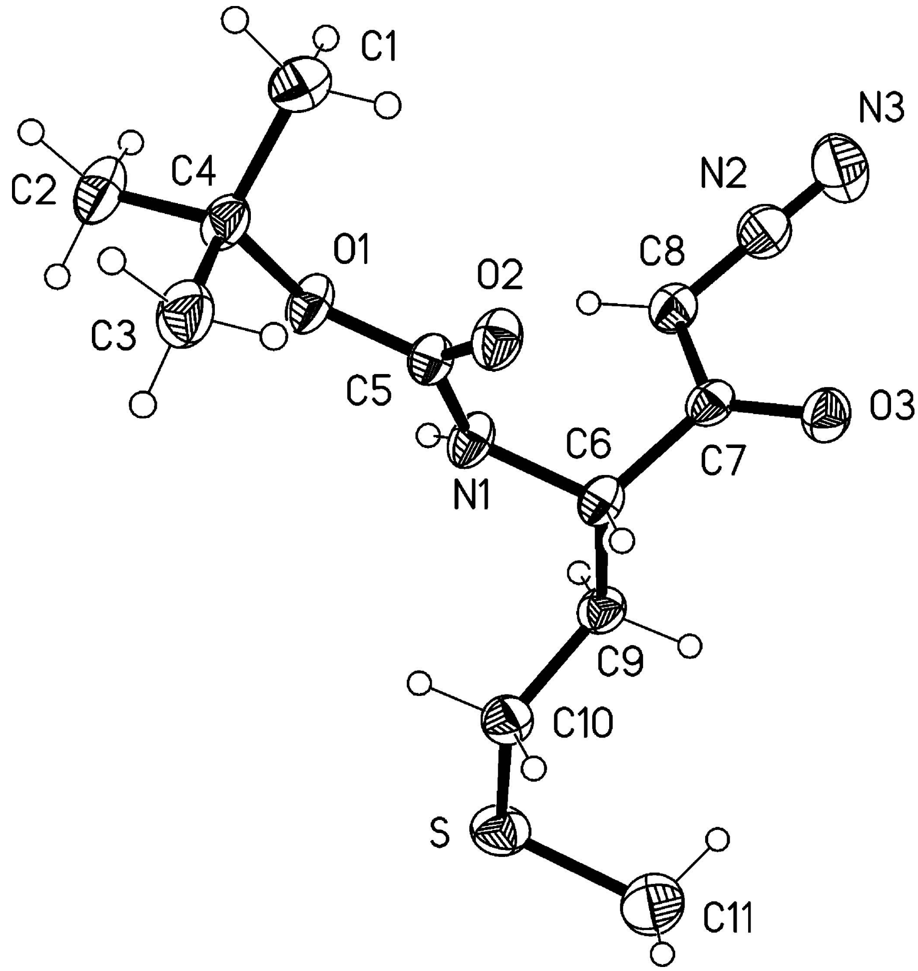


**Figure 1**

The molecule of the title compound in the crystal. Ellipsoids correspond to 50% probability levels.

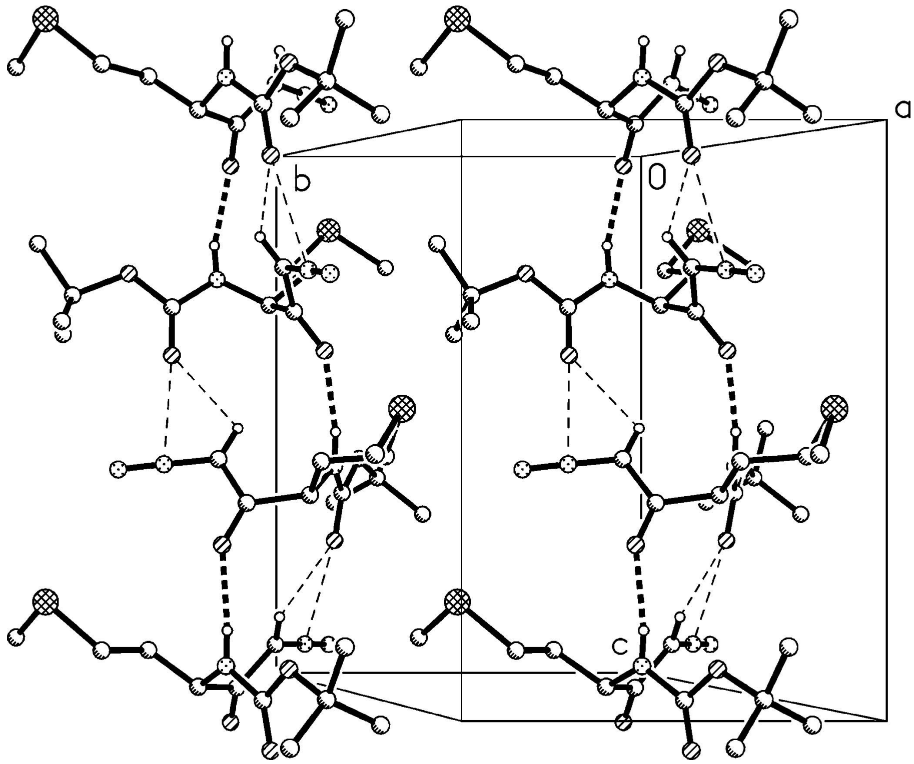


**Figure 2**

Packing diagram of the title compound viewed perpendicular to the *yz* plane. Classical H bonds are represented by thick dashed lines, and the three-centre interaction (see text) by thin dashed lines. H atoms not involved in H bonding are omitted for clarity.

### *tert*-Butyl *N*-[1-diazoacetyl-3-(methylsulfanyl)propyl]carbamate

*Crystal data*

$C_{11}H_{19}N_3O_3S$
$M_r = 273.35$
Trigonal, $P3_1$
Hall symbol: P 31
$a = 9.7915$ (3) Å
$c = 13.8581$ (5) Å
$V = 1150.62$ (6) Å$^3$
$Z = 3$
$F(000) = 438$
$D_x = 1.183$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 14422 reflections
$\theta = 3.2$–$75.7°$
$\mu = 1.93$ mm$^{-1}$
$T = 100$ K
Tablet, colourless
0.20 × 0.20 × 0.15 mm

*Data collection*

Oxford Diffraction Xcalibur Nova A diffractometer
Radiation source: Nova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3543 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.717$, $T_{max} = 1.000$
16152 measured reflections
3073 independent reflections
3051 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 75.6°$, $\theta_{min} = 5.2°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.060$
$S = 1.05$
3073 reflections
171 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.1663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$
Absolute structure: Flack (1983), 1474 Freidel pairs
Absolute structure parameter: 0.023 (10)

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Short contact: 2.8141 (14) N2 - O2_$1; 73.5 (1) C8 - N2 - O2_$1; Operator $1 - *x* + *y*+1, -*x* + 1, *z* - 1/3

**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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S | 0.08692 (4) | −0.10432 (4) | 0.14682 (2) | 0.03648 (9) |
| O1 | 0.57182 (11) | 0.62115 (10) | 0.24352 (6) | 0.02868 (19) |
| O2 | 0.57776 (11) | 0.51506 (10) | 0.38885 (6) | 0.02855 (19) |
| O3 | 0.66256 (11) | 0.16891 (11) | 0.38136 (6) | 0.02857 (19) |
| N1 | 0.49938 (13) | 0.36907 (12) | 0.25188 (8) | 0.0259 (2) |
| H01 | 0.4999 (17) | 0.3757 (17) | 0.1913 (12) | 0.021 (3)* |
| N2 | 0.88980 (14) | 0.31145 (15) | 0.24685 (9) | 0.0340 (3) |
| N3 | 1.00277 (17) | 0.3112 (2) | 0.25596 (10) | 0.0492 (4) |
| C1 | 0.76526 (17) | 0.85794 (16) | 0.33133 (11) | 0.0350 (3) |
| H1A | 0.7632 | 0.7991 | 0.3890 | 0.052* |
| H1B | 0.7907 | 0.9647 | 0.3499 | 0.052* |
| H1C | 0.8455 | 0.8640 | 0.2865 | 0.052* |
| C2 | 0.60734 (19) | 0.86229 (16) | 0.19163 (11) | 0.0369 (3) |
| H2A | 0.6887 | 0.8685 | 0.1477 | 0.055* |
| H2B | 0.6307 | 0.9690 | 0.2085 | 0.055* |
| H2C | 0.5042 | 0.8059 | 0.1600 | 0.055* |
| C3 | 0.47294 (18) | 0.75163 (17) | 0.35029 (12) | 0.0364 (3) |
| H3A | 0.3714 | 0.6914 | 0.3172 | 0.055* |
| H3B | 0.4883 | 0.8548 | 0.3693 | 0.055* |
| H3C | 0.4739 | 0.6940 | 0.4079 | 0.055* |
| C4 | 0.60502 (15) | 0.77449 (14) | 0.28283 (10) | 0.0278 (3) |
| C5 | 0.55262 (14) | 0.50412 (13) | 0.30286 (9) | 0.0244 (2) |

| | | | | |
|---|---|---|---|---|
| C6 | 0.48307 (14) | 0.22893 (14) | 0.29774 (9) | 0.0240 (2) |
| H6 | 0.4329 | 0.2173 | 0.3624 | 0.029* |
| C7 | 0.64131 (14) | 0.23513 (13) | 0.31215 (9) | 0.0234 (2) |
| C8 | 0.75513 (15) | 0.31186 (15) | 0.23793 (10) | 0.0288 (3) |
| H8 | 0.7356 | 0.3609 | 0.1847 | 0.035* |
| C9 | 0.37572 (14) | 0.08310 (13) | 0.23693 (9) | 0.0257 (2) |
| H9A | 0.4220 | 0.0957 | 0.1718 | 0.031* |
| H9B | 0.3707 | −0.0110 | 0.2670 | 0.031* |
| C10 | 0.20871 (15) | 0.05652 (15) | 0.22743 (10) | 0.0300 (3) |
| H10A | 0.2144 | 0.1544 | 0.2032 | 0.036* |
| H10B | 0.1588 | 0.0339 | 0.2920 | 0.036* |
| C11 | 0.0623 (2) | −0.26955 (18) | 0.21754 (14) | 0.0502 (4) |
| H11A | 0.1660 | −0.2559 | 0.2331 | 0.075* |
| H11B | 0.0012 | −0.3671 | 0.1806 | 0.075* |
| H11C | 0.0062 | −0.2755 | 0.2774 | 0.075* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S | 0.03136 (16) | 0.03576 (17) | 0.03360 (17) | 0.01025 (14) | −0.00604 (13) | −0.00369 (14) |
| O1 | 0.0426 (5) | 0.0199 (4) | 0.0260 (4) | 0.0176 (4) | −0.0038 (4) | −0.0017 (3) |
| O2 | 0.0390 (5) | 0.0245 (4) | 0.0246 (5) | 0.0177 (4) | −0.0050 (3) | −0.0027 (3) |
| O3 | 0.0319 (4) | 0.0295 (4) | 0.0243 (4) | 0.0153 (4) | 0.0004 (3) | 0.0041 (3) |
| N1 | 0.0384 (6) | 0.0196 (5) | 0.0208 (5) | 0.0154 (4) | −0.0023 (4) | −0.0016 (4) |
| N2 | 0.0324 (6) | 0.0379 (6) | 0.0292 (6) | 0.0157 (5) | 0.0058 (4) | 0.0081 (4) |
| N3 | 0.0392 (7) | 0.0717 (10) | 0.0426 (8) | 0.0320 (7) | 0.0095 (6) | 0.0198 (7) |
| C1 | 0.0336 (7) | 0.0248 (6) | 0.0424 (8) | 0.0115 (5) | −0.0030 (6) | −0.0028 (5) |
| C2 | 0.0521 (8) | 0.0249 (6) | 0.0381 (8) | 0.0224 (6) | −0.0028 (6) | 0.0013 (5) |
| C3 | 0.0400 (8) | 0.0309 (7) | 0.0456 (8) | 0.0230 (6) | 0.0044 (6) | 0.0018 (6) |
| C4 | 0.0334 (6) | 0.0184 (5) | 0.0335 (7) | 0.0145 (5) | −0.0017 (5) | −0.0019 (5) |
| C5 | 0.0271 (6) | 0.0203 (5) | 0.0282 (6) | 0.0137 (5) | −0.0001 (4) | −0.0001 (4) |
| C6 | 0.0293 (6) | 0.0198 (5) | 0.0239 (6) | 0.0131 (5) | 0.0010 (4) | 0.0005 (4) |
| C7 | 0.0275 (6) | 0.0170 (5) | 0.0228 (6) | 0.0091 (4) | −0.0009 (4) | −0.0021 (4) |
| C8 | 0.0289 (6) | 0.0277 (6) | 0.0289 (6) | 0.0135 (5) | 0.0024 (5) | 0.0051 (5) |
| C9 | 0.0273 (6) | 0.0199 (5) | 0.0289 (6) | 0.0110 (5) | −0.0003 (5) | −0.0015 (4) |
| C10 | 0.0274 (6) | 0.0261 (6) | 0.0356 (7) | 0.0126 (5) | −0.0017 (5) | −0.0014 (5) |
| C11 | 0.0479 (9) | 0.0284 (7) | 0.0653 (11) | 0.0124 (7) | −0.0165 (8) | −0.0071 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S—C11 | 1.8015 (17) | C1—H1A | 0.9800 |
| S—C10 | 1.8089 (13) | C1—H1B | 0.9800 |
| O1—C5 | 1.3450 (14) | C1—H1C | 0.9800 |
| O1—C4 | 1.4727 (14) | C2—H2A | 0.9800 |
| O2—C5 | 1.2107 (16) | C2—H2B | 0.9800 |
| O3—C7 | 1.2323 (15) | C2—H2C | 0.9800 |
| N1—C5 | 1.3528 (15) | C3—H3A | 0.9800 |
| N1—C6 | 1.4468 (15) | C3—H3B | 0.9800 |

| | | | |
|---|---|---|---|
| N2—N3 | 1.1145 (18) | C3—H3C | 0.9800 |
| N2—C8 | 1.3264 (18) | C6—H6 | 1.0000 |
| C1—C4 | 1.5163 (19) | C8—H8 | 0.9500 |
| C2—C4 | 1.5223 (19) | C9—H9A | 0.9900 |
| C3—C4 | 1.5189 (19) | C9—H9B | 0.9900 |
| C6—C7 | 1.5330 (17) | C10—H10A | 0.9900 |
| C6—C9 | 1.5340 (16) | C10—H10B | 0.9900 |
| C7—C8 | 1.4237 (17) | C11—H11A | 0.9800 |
| C9—C10 | 1.5276 (17) | C11—H11B | 0.9800 |
| N1—H01 | 0.842 (16) | C11—H11C | 0.9800 |

| | | | |
|---|---|---|---|
| C11—S—C10 | 100.36 (7) | H2A—C2—H2B | 109.5 |
| C5—O1—C4 | 120.53 (10) | C4—C2—H2C | 109.5 |
| C5—N1—C6 | 120.23 (11) | H2A—C2—H2C | 109.5 |
| N3—N2—C8 | 178.84 (15) | H2B—C2—H2C | 109.5 |
| O1—C4—C1 | 110.78 (10) | C4—C3—H3A | 109.5 |
| O1—C4—C3 | 109.87 (10) | C4—C3—H3B | 109.5 |
| C1—C4—C3 | 112.46 (12) | H3A—C3—H3B | 109.5 |
| O1—C4—C2 | 101.65 (10) | C4—C3—H3C | 109.5 |
| C1—C4—C2 | 110.15 (11) | H3A—C3—H3C | 109.5 |
| C3—C4—C2 | 111.43 (11) | H3B—C3—H3C | 109.5 |
| O2—C5—O1 | 126.21 (10) | N1—C6—H6 | 108.5 |
| O2—C5—N1 | 124.23 (11) | C7—C6—H6 | 108.5 |
| O1—C5—N1 | 109.57 (11) | C9—C6—H6 | 108.5 |
| N1—C6—C7 | 112.93 (10) | N2—C8—H8 | 121.7 |
| N1—C6—C9 | 109.93 (10) | C7—C8—H8 | 121.7 |
| C7—C6—C9 | 108.48 (9) | C10—C9—H9A | 109.1 |
| O3—C7—C8 | 123.12 (12) | C6—C9—H9A | 109.1 |
| O3—C7—C6 | 120.91 (11) | C10—C9—H9B | 109.1 |
| C8—C7—C6 | 115.84 (11) | C6—C9—H9B | 109.1 |
| N2—C8—C7 | 116.62 (12) | H9A—C9—H9B | 107.8 |
| C10—C9—C6 | 112.51 (10) | C9—C10—H10A | 109.1 |
| C9—C10—S | 112.65 (9) | S—C10—H10A | 109.1 |
| C5—N1—H01 | 117.4 (10) | C9—C10—H10B | 109.1 |
| C6—N1—H01 | 120.3 (10) | S—C10—H10B | 109.1 |
| C4—C1—H1A | 109.5 | H10A—C10—H10B | 107.8 |
| C4—C1—H1B | 109.5 | S—C11—H11A | 109.5 |
| H1A—C1—H1B | 109.5 | S—C11—H11B | 109.5 |
| C4—C1—H1C | 109.5 | H11A—C11—H11B | 109.5 |
| H1A—C1—H1C | 109.5 | S—C11—H11C | 109.5 |
| H1B—C1—H1C | 109.5 | H11A—C11—H11C | 109.5 |
| C4—C2—H2A | 109.5 | H11B—C11—H11C | 109.5 |
| C4—C2—H2B | 109.5 | | |

| | | | |
|---|---|---|---|
| C5—O1—C4—C1 | 66.13 (15) | C9—C6—C7—O3 | −89.46 (13) |
| C5—O1—C4—C3 | −58.74 (14) | N1—C6—C7—C8 | −35.64 (15) |
| C5—O1—C4—C2 | −176.85 (11) | C9—C6—C7—C8 | 86.47 (12) |
| C4—O1—C5—O2 | −9.28 (19) | O3—C7—C8—N2 | −0.93 (19) |

| | | | |
|---|---|---|---|
| C4—O1—C5—N1 | 170.45 (10) | C6—C7—C8—N2 | −176.76 (11) |
| C6—N1—C5—O2 | −5.96 (19) | N1—C6—C9—C10 | −62.16 (13) |
| C6—N1—C5—O1 | 174.30 (10) | C7—C6—C9—C10 | 173.92 (10) |
| C5—N1—C6—C7 | −76.13 (14) | C6—C9—C10—S | 174.67 (9) |
| C5—N1—C6—C9 | 162.57 (10) | C11—S—C10—C9 | 69.91 (11) |
| N1—C6—C7—O3 | 148.43 (11) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H01···O3$^{i}$ | 0.842 (16) | 2.027 (16) | 2.8465 (14) | 164.1 (14) |
| C8—H8···O2$^{i}$ | 0.95 | 2.51 | 2.9686 (16) | 110 |
| C11—H11*B*···O2$^{ii}$ | 0.98 | 2.52 | 3.457 (2) | 160 |
| C3—H3*B*···O3$^{iii}$ | 0.98 | 2.67 | 3.5693 (17) | 152 |
| C1—H1*C*···S$^{iv}$ | 0.98 | 2.95 | 3.9281 (16) | 177 |

Symmetry codes: (i) −*x*+*y*+1, −*x*+1, *z*−1/3; (ii) −*x*+*y*, −*x*, *z*−1/3; (iii) *x*, *y*+1, *z*; (iv) *x*+1, *y*+1, *z*.