

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

Tahir Mehmood,^a Javid H. Zaidi^{a*} and Peter G. Jones^b

^aDepartment of Chemistry, Quaid-I-Azam University, Islamabad 45320, Pakistan, and ^bInstitut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Hagenring 30, 38106 Braunschweig, Germany

Correspondence e-mail: javid_zaidi@qau.edu.pk

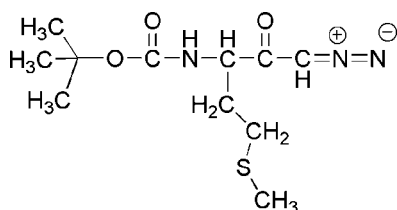
Received 2 August 2009; accepted 3 August 2009

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

In the enantiomerically pure title compound, $\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$, the chain $\text{C}-\text{N}-\text{C}(\text{O})-\text{O}-\text{C}-\text{C}$ (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 $\text{N}-\text{H}\cdots\text{O}$ diazocarbonyl hydrogen bonding and an unusual intermolecular three-centre interaction involving the amino acid (aa) carbonyl O_{aa} and the diazocarbonyl grouping $\text{C}(\text{O})-\text{CH}-\text{N}\equiv\text{N}$, with $\text{H}\cdots\text{O}_{\text{aa}} = 2.51$ Å and $\text{N}\cdots\text{O}_{\text{aa}} = 2.8141$ (14) Å.

Related literature

For the applications of α -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). α -Diazoketones undergo a variety of transformations, see: Ye & McKerverey (1994). Asymmetric versions of diazocarbonyl reactions have been reported to produce enantiomerically pure compounds, see: Doyle & McKerverey (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 β -peptides and β -amino acid derivatives from appropriately protected α -amino acids, see: Müller *et al.* (1998).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$
 $M_r = 273.35$
 Trigonal, $P3_1$
 $a = 9.7915$ (3) Å
 $c = 13.8581$ (5) Å
 $V = 1150.62$ (6) Å³
 $Z = 3$
 Cu $K\alpha$ radiation
 $\mu = 1.93$ mm⁻¹
 $T = 100$ K
 $0.20 \times 0.20 \times 0.15$ mm

Data collection

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

Refinement

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

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H01}\cdots\text{O3}^{\text{i}}$	0.842 (16)	2.027 (16)	2.8465 (14)	164.1 (14)
$\text{C8}-\text{H8}\cdots\text{O2}^{\text{i}}$	0.95	2.51	2.9686 (16)	110
$\text{C11}-\text{H11B}\cdots\text{O2}^{\text{ii}}$	0.98	2.52	3.457 (2)	160
$\text{C3}-\text{H3B}\cdots\text{O3}^{\text{iii}}$	0.98	2.67	3.5693 (17)	152
$\text{C1}-\text{H1C}\cdots\text{S}^{\text{iv}}$	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*.

The authors are grateful to the Department of Chemistry, Quaid-I-Azam University, Islamabad, Pakistan, and the Institute for Inorganic Chemistry, University of Frankfurt, Germany, for providing laboratory and analytical facilities.

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

References

- Doyle, M. P. & McKerverey, M. A. (1997). *J. Chem. Soc. Chem. Commun.* pp. 983–989.
 Doyle, M. P., McKerverey, M. & Ye, T. (1998). In *Modern Catalytic Methods for Organic Synthesis with Diazo compounds from Cyclopropanone to Ylides*. New York: Wiley-Interscience.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Müller, A., Vogt, C. & Sewald, N. (1998). *Synthesis*, pp. 837–841.
 Oxford Diffraction (2009). *CrysAlis Pro*. Oxford Diffraction Ltd, Yarnton, England.

Padwa, A. & Weingarten, M. (1996). *Chem. Rev.* **96**, 223–269.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Siemens (1994). *XP*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Ye, T. & McKervey, M. A. (1994). *Chem. Rev.* **94**, 1091–1160.

supplementary materials

Acta Cryst. (2009). E65, o2120-o2121 [doi:10.1107/S1600536809030815]

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

T. Mehmood, J. H. Zaidi and P. G. Jones

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 \cdots 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 \cdots O2 2.8141 (14) Å, C8—N2 \cdots O2 73.5 (1)°]. The remaining "weak" C—H \cdots O interactions (Table 1) link neighbouring chains; H3B \cdots O3 is implicit between the chains of Fig. 2 but is omitted for clarity.

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 NaHCO₃ and NH₄Cl and dried over anhydrous MgSO₄. The crude product was purified by column chromatography (yield 85%; m.p.326-328 K).

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 $1.5 \times U(\text{eq})$ of the parent atom for methyl H and $1.2 \times U(\text{eq})$ of the parent atom for other C—H. Data are 100% complete to 2θ 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).

Figures

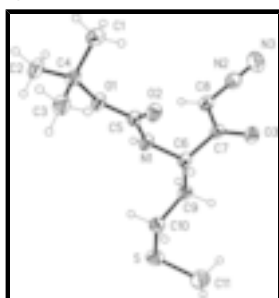


Fig. 1. The molecule of the title compound in the crystal. Ellipsoids correspond to 50% probability levels.

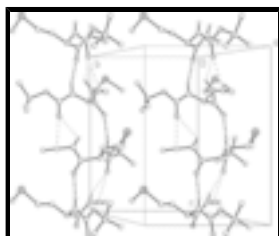


Fig. 2. Packing diagram of the title compound viewed perpendicular to the *yz* plane. Classical H bonds are represented by thick dashed lines, and the three-centre interaction (see text) by thin dashed lines. H atoms not involved in H bonding are omitted for clarity.

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

Crystal data

$\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$

$M_r = 273.35$

Trigonal, $P3_1$

Hall symbol: P 31

$a = 9.7915$ (3) Å

$b = 9.7915$ (3) Å

$c = 13.8581$ (5) Å

$\alpha = 90^\circ$

$\beta = 90^\circ$

$\gamma = 120^\circ$

$V = 1150.62$ (6) Å³

$Z = 3$

$F_{000} = 438$

$D_x = 1.183$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 14422 reflections

$\theta = 3.2\text{--}75.7^\circ$

$\mu = 1.93$ mm⁻¹

$T = 100$ K

Tablet, colourless

$0.20 \times 0.20 \times 0.15$ mm

Data collection

Oxford Diffraction Xcalibur Nova A diffractometer	3073 independent reflections
Radiation source: Nova (Cu) X-ray Source	3051 reflections with $I > 2\sigma(I)$
Monochromator: mirror	$R_{\text{int}} = 0.032$
Detector resolution: 10.3543 pixels mm^{-1}	$\theta_{\text{max}} = 75.6^\circ$
$T = 100$ K	$\theta_{\text{min}} = 5.2^\circ$
ω scans	$h = -12 \rightarrow 12$
Absorption correction: multi-scan (CrysAlis Pro; Oxford Diffraction, 2009)	$k = -12 \rightarrow 12$
$T_{\text{min}} = 0.717$, $T_{\text{max}} = 1.000$	$l = -17 \rightarrow 15$
16152 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.023$	$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.1663P]$
$wR(F^2) = 0.060$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3073 reflections	$\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{\AA}^{-3}$
171 parameters	$\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1474 Freidel pairs
Secondary atom site location: difference Fourier map	Flack 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 (\AA^2)

x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
-----	-----	-----	----------------------------------

supplementary materials

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 (\AA^2)

	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, °)

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

supplementary materials

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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H01 \cdots O3 ⁱ	0.842 (16)	2.027 (16)	2.8465 (14)	164.1 (14)
C8—H8 \cdots O2 ⁱ	0.95	2.51	2.9686 (16)	110
C11—H11B \cdots O2 ⁱⁱ	0.98	2.52	3.457 (2)	160
C3—H3B \cdots O3 ⁱⁱⁱ	0.98	2.67	3.5693 (17)	152
C1—H1C \cdots 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) $x+1, y+1, z$.

Fig. 1

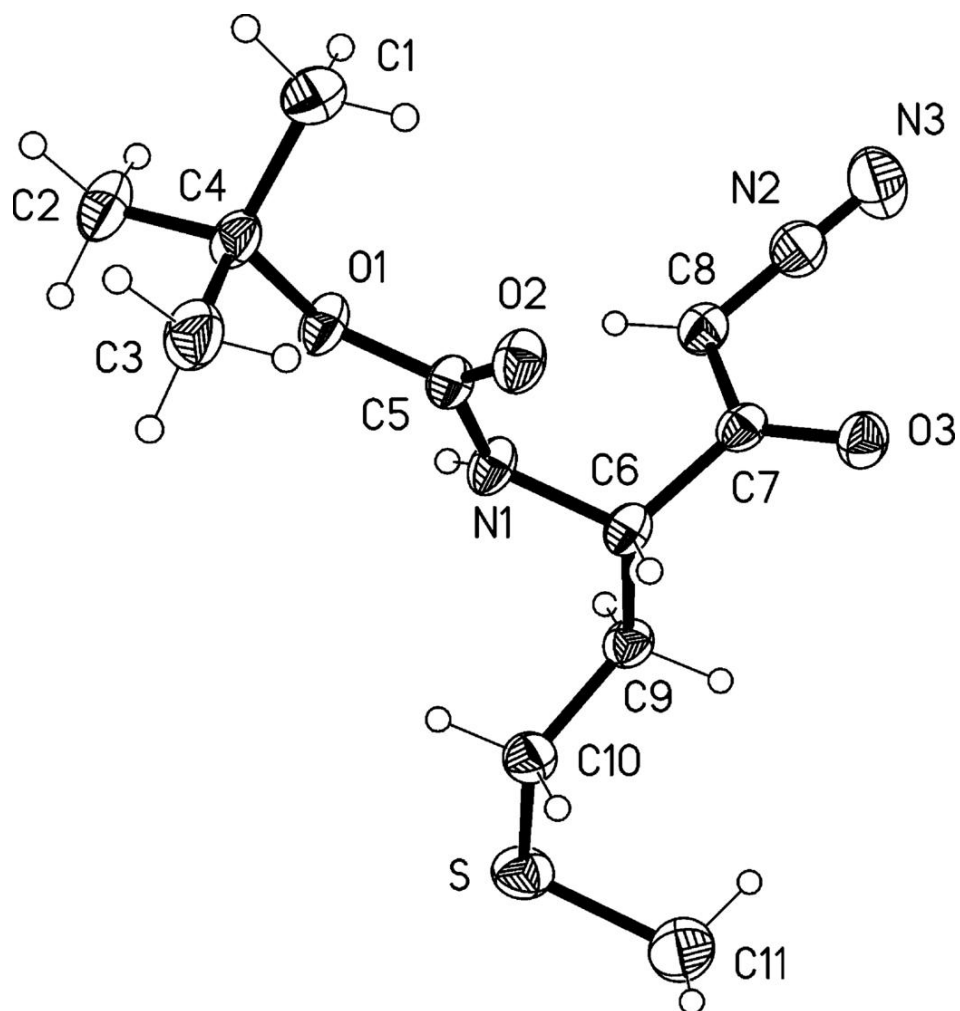


Fig. 2

