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(2*S*)-3-Carbamoyl-2-(4-methoxybenzenesulfonamido)propanoic acid

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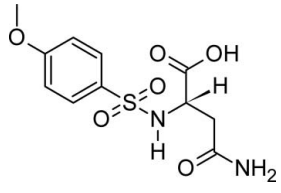
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.063; data-to-parameter ratio = 17.7.

In the title compound, $C_{11}H_{14}N_2O_6S$, an amino acid-derived sulfonamide, the acetamido group and the carboxylic group are oriented at dihedral angles of 45.84 (5)° and 47.97 (5)° respectively, with respect to the aromatic ring. In the crystal, the molecules are connected by N—H···O and O—H···O hydrogen bonds and weak C—H···O interactions, forming a three-dimensional network.

Related literature

For related structures, see: Arshad *et al.* (2009*a*,*b*), Khan *et al.* (2009). Amino acid-derived sulfonamide derivatives have been used as potent inhibitors of Procollagen C-Proteinase, see: Dankwardt *et al.* (2002).



Experimental

Crystal data

$C_{11}H_{14}N_2O_6S$
M_r = 302.30
Monoclinic, $P2_1$
a = 7.1462 (1) Å
b = 8.9874 (2) Å
c = 11.1418 (2) Å
β = 108.090 (1)°
V = 680.22 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 100 K
0.42 × 0.26 × 0.23 mm

Data collection

Siemens SMART diffractometer equipped with a Bruker APEXII detector
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.897, T_{max} = 0.942
15356 measured reflections
3434 independent reflections
3335 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.063
S = 1.04
3434 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 1581 Friedel pairs
Flack parameter: −0.01 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N2*···O3^{i}	0.812 (19)	2.147 (19)	2.9430 (15)	166.9 (16)
N2—H2*N2*···O2^{ii}	0.89 (2)	2.02 (2)	2.8808 (16)	162.9 (17)
C11—H11*B*···O3^{iii}	0.98	2.48	3.3701 (17)	150
N1—H1*N*···O5^{ii}	0.927 (18)	1.907 (18)	2.8196 (14)	167.8 (16)
O4—H4*O*···O5^{iv}	0.81 (2)	1.79 (2)	2.5875 (14)	169 (2)
C9—H9*A*···O2^{ii}	0.99	2.54	3.4055 (16)	145

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997), *PLATON* (Spek, 2009) and *X-SEED* (Barbour, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5624).

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supporting information

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(2*S*)-3-Carbamoyl-2-(4-methoxybenzenesulfonamido)propanoic acid

Hafiz Mubashar-ur-Rehman, Islam Ullah Khan, Muhammad Nadeem Arshad and K. Travis Holman

S1. Comment

Amino acid derived sulfonamide derivatives have been used as potent inhibitors of Procollagen C-Proteinase (Dankwardt *et al.*, 2002). This structure is in countinuation to already reported crystal structures of sulfonamides derived from amino acids (Arshad *et al.*, 2009*a*), (Arshad *et al.*, 2009*b*) (Khan *et al.*, 2009) by our group.

The dihedral angle between the acetamido group attached at the C7 and the carboxylic group C7/C8/O3/O4 is 38.64 (0.05)° while these two groups are oriented at dihedral angle of 45.84 (0.05)° and 47.97 (0.05)° respectively with respect to the aromatic ring. The symmetry related intermolecular N—H···O, O—H···O and weak C—H···O type interactions stabilized the structure by the formation of three dimentional network (Fig. 2, Table, 1).

S2. Experimental

To the solution of L-asparagine (0.5 g, 3.78 mmol) in distilled water (10 ml), 4-methoxybenzenesulfonyl chloride(0.78 g, 3.78 mmol) was suspended. The reaction mixture was allowed to stirr at room temperature for 2 hrs. The pH of the solution was maintained at 8–9 by 1*M* sodium carbonate solution through out the reaction. After completion of the reaction which was observed by the consumption of suspended 4-methoxybenzenesulfonyl chloride, 1 *N* HCl solution was used to adjusted the pH about 2–3, which results in the formation of a white precipitate, which was filtered off, dried and recrystallized in methanol by slow evaporation to yield colorless needles of (I).

S3. Refinement

The C-H H-atoms were positioned geometricaly with C—H = 0.95 Å, C—H = 0.99Å and C—H = 1.00 Å for aromatic, methylene and chiral carbon atoms respectively, and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C). Similarly the C-H H-atoms were positioned geometricaly with C—H = 0.98 Å for methyl group and were refined using a riding model with U_{iso}(H) = 1.5 U_{eq}(C). The N-H and O–H H atoms were located in difference map with N–H= 0.81 (2)—0.93 (2)Å and O—H= 0.81 (2) with U_{iso}(H) = 1.2 for N atoms and U_{iso}(H) = 1.5 for O atoms. The three reflections (001), (002) and (003) were omitted during the final refinement as these were obscured by the beam stop.

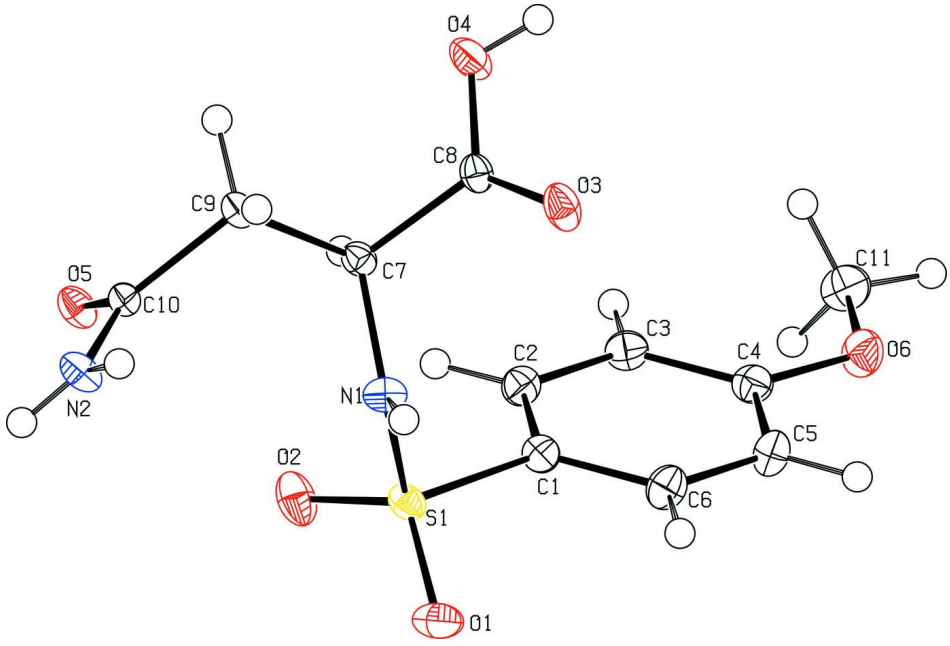

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

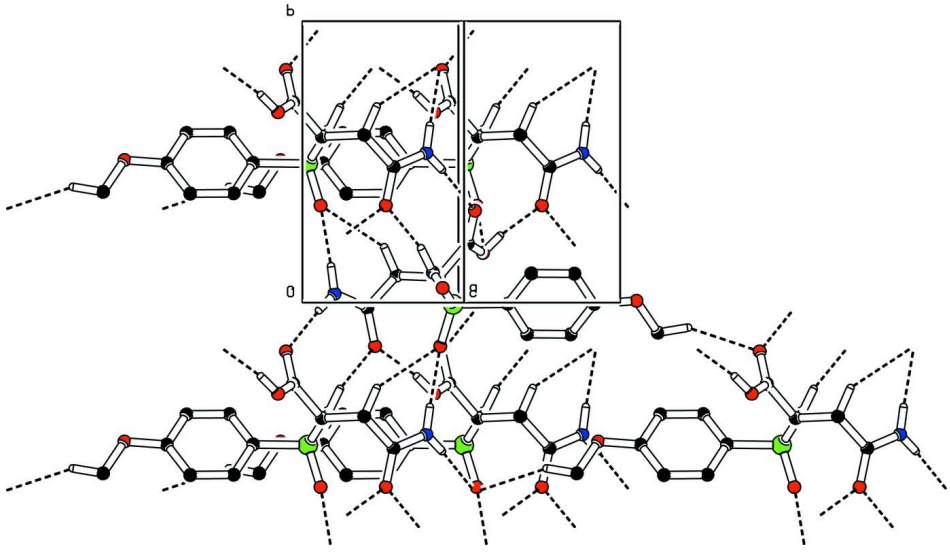

Figure 2

Unit cell packing for (I) with hydrogen bonding shown as dashed lines and the hydrogen atoms not involved in hydrogen bonding have been omitted.

(2*S*)-3-Carbamoyl-2-(4-methoxybenzenesulfonamido)propanoic acid

Crystal data

$C_{11}H_{14}N_2O_6S$
$M_r = 302.30$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 7.1462\ (1)$ Å
$b = 8.9874\ (2)$ Å
$c = 11.1418\ (2)$ Å
$\beta = 108.090\ (1)°$
$V = 680.22\ (2)$ Å^3
$Z = 2$

$F(000) = 316$
$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8744 reflections
$\theta = 3.0$–$28.6°$
$\mu = 0.27$ mm^{-1}
$T = 100$ K
Needle, colorless
0.42 × 0.26 × 0.23 mm

Data collection

Siemens SMART diffractometer equipped with a Bruker APEXII detector
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.897$, $T_{max} = 0.942$
15356 measured reflections
3434 independent reflections
3335 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 28.6°$, $\theta_{min} = 3.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.063$
$S = 1.04$
3434 reflections
194 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
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 0.1181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), **1581 Friedel pairs**
Absolute structure parameter: −0.01 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.29092 (4)	−0.00600 (3)	0.24767 (3)	0.01463 (7)
O1	−0.42282 (14)	0.05232 (12)	0.31015 (9)	0.0222 (2)
N1	−0.30244 (16)	0.10725 (12)	0.13389 (10)	0.0151 (2)
C1	−0.04937 (16)	−0.00385 (17)	0.35229 (10)	0.0149 (2)
H1N	−0.375 (3)	0.193 (2)	0.1302 (16)	0.018*
H1N2	−0.739 (3)	−0.027 (2)	−0.1489 (16)	0.018*
O2	−0.32080 (14)	−0.15497 (11)	0.19714 (9)	0.0228 (2)
N2	−0.65042 (16)	0.03205 (13)	−0.13841 (11)	0.0173 (2)
C2	0.08556 (19)	−0.11149 (14)	0.34294 (12)	0.0168 (2)

H2	0.0478	−0.1847	0.2784	0.020*
H2N2	−0.669 (2)	0.130 (2)	−0.1430 (17)	0.020*
O3	−0.03178 (13)	0.32670 (11)	0.13252 (9)	0.0204 (2)
C3	0.27541 (19)	−0.11253 (15)	0.42761 (13)	0.0181 (2)
H3	0.3675	−0.1859	0.4212	0.022*
O4	0.12194 (15)	0.17646 (12)	0.03322 (11)	0.0241 (2)
C4	0.32887 (17)	−0.00447 (18)	0.52207 (11)	0.0173 (2)
H4O	0.208 (3)	0.238 (2)	0.0560 (19)	0.026*
O5	−0.42990 (13)	−0.15430 (10)	−0.10103 (9)	0.01674 (18)
C5	0.1919 (2)	0.10294 (16)	0.53159 (12)	0.0196 (3)
H5	0.2285	0.1754	0.5968	0.024*
O6	0.51004 (13)	0.00661 (13)	0.60857 (8)	0.0223 (2)
C6	0.0043 (2)	0.10393 (16)	0.44689 (12)	0.0189 (2)
H6	−0.0877	0.1776	0.4529	0.023*
C7	−0.17978 (17)	0.09007 (14)	0.05256 (11)	0.0132 (2)
H7	−0.1132	−0.0091	0.0698	0.016*
C8	−0.02147 (17)	0.21146 (14)	0.07875 (12)	0.0139 (2)
C9	−0.30540 (17)	0.09626 (14)	−0.08778 (11)	0.0138 (2)
H9A	−0.3628	0.1968	−0.1088	0.017*
H9B	−0.2219	0.0752	−0.1420	0.017*
C10	−0.46823 (16)	−0.01830 (16)	−0.11135 (10)	0.0134 (2)
C11	0.6510 (2)	−0.10641 (19)	0.60538 (14)	0.0249 (3)
H11A	0.5988	−0.2044	0.6167	0.037*
H11B	0.7742	−0.0886	0.6734	0.037*
H11C	0.6762	−0.1032	0.5238	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01351 (12)	0.01393 (13)	0.01602 (13)	−0.00161 (11)	0.00396 (9)	0.00279 (11)
O1	0.0180 (4)	0.0296 (5)	0.0215 (4)	0.0010 (4)	0.0097 (4)	0.0059 (4)
N1	0.0158 (5)	0.0138 (5)	0.0171 (5)	0.0037 (4)	0.0074 (4)	0.0040 (4)
C1	0.0147 (5)	0.0149 (5)	0.0141 (5)	0.0006 (5)	0.0030 (4)	0.0027 (5)
O2	0.0230 (5)	0.0141 (5)	0.0264 (5)	−0.0041 (4)	0.0005 (4)	0.0023 (4)
N2	0.0125 (5)	0.0143 (6)	0.0252 (6)	−0.0011 (4)	0.0061 (4)	0.0012 (4)
C2	0.0199 (6)	0.0143 (6)	0.0167 (6)	−0.0004 (5)	0.0062 (5)	−0.0001 (5)
O3	0.0141 (4)	0.0163 (5)	0.0297 (5)	−0.0005 (3)	0.0053 (4)	−0.0082 (4)
C3	0.0180 (6)	0.0181 (6)	0.0188 (6)	0.0021 (5)	0.0066 (5)	0.0012 (5)
O4	0.0170 (5)	0.0220 (5)	0.0373 (6)	−0.0072 (4)	0.0144 (4)	−0.0125 (4)
C4	0.0161 (5)	0.0203 (6)	0.0149 (5)	0.0006 (5)	0.0040 (4)	0.0033 (6)
O5	0.0131 (4)	0.0122 (4)	0.0244 (4)	−0.0006 (3)	0.0051 (3)	−0.0009 (4)
C5	0.0214 (6)	0.0199 (6)	0.0158 (6)	0.0006 (5)	0.0033 (5)	−0.0032 (5)
O6	0.0168 (4)	0.0290 (5)	0.0180 (4)	0.0028 (4)	0.0009 (3)	−0.0013 (4)
C6	0.0211 (6)	0.0171 (6)	0.0179 (6)	0.0029 (5)	0.0054 (5)	−0.0013 (5)
C7	0.0118 (5)	0.0121 (5)	0.0159 (5)	0.0001 (4)	0.0047 (4)	−0.0003 (4)
C8	0.0108 (5)	0.0143 (6)	0.0148 (5)	0.0006 (4)	0.0012 (4)	−0.0002 (4)
C9	0.0121 (5)	0.0131 (5)	0.0157 (5)	−0.0017 (4)	0.0037 (4)	−0.0002 (5)
C10	0.0135 (5)	0.0139 (6)	0.0128 (5)	−0.0029 (4)	0.0042 (4)	−0.0009 (4)

C11	0.0170 (6)	0.0362 (8)	0.0200 (6)	0.0060 (5)	0.0034 (5)	0.0027 (6)

Geometric parameters (Å, º)

S1—O1	1.4336 (10)	O4—H4O	0.81 (2)
S1—O2	1.4424 (11)	C4—O6	1.3565 (14)
S1—N1	1.6078 (11)	C4—C5	1.402 (2)
S1—C1	1.7576 (11)	O5—C10	1.2500 (17)
N1—C7	1.4514 (15)	C5—C6	1.3790 (18)
N1—H1N	0.927 (18)	C5—H5	0.9500
C1—C2	1.3924 (18)	O6—C11	1.4389 (18)
C1—C6	1.3948 (18)	C6—H6	0.9500
N2—C10	1.3217 (16)	C7—C8	1.5330 (17)
N2—H1N2	0.812 (19)	C7—C9	1.5435 (16)
N2—H2N2	0.89 (2)	C7—H7	1.0000
C2—C3	1.3914 (18)	C9—C10	1.5144 (17)
C2—H2	0.9500	C9—H9A	0.9900
O3—C8	1.2101 (16)	C9—H9B	0.9900
C3—C4	1.3954 (19)	C11—H11A	0.9800
C3—H3	0.9500	C11—H11B	0.9800
O4—C8	1.3151 (16)	C11—H11C	0.9800
O1—S1—O2	119.27 (6)	C4—O6—C11	116.86 (11)
O1—S1—N1	105.72 (6)	C5—C6—C1	119.57 (12)
O2—S1—N1	108.34 (6)	C5—C6—H6	120.2
O1—S1—C1	109.41 (6)	C1—C6—H6	120.2
O2—S1—C1	105.36 (6)	N1—C7—C8	111.01 (10)
N1—S1—C1	108.38 (6)	N1—C7—C9	110.77 (10)
C7—N1—S1	122.14 (9)	C8—C7—C9	109.26 (10)
C7—N1—H1N	120.1 (11)	N1—C7—H7	108.6
S1—N1—H1N	117.0 (11)	C8—C7—H7	108.6
C2—C1—C6	120.31 (11)	C9—C7—H7	108.6
C2—C1—S1	120.20 (10)	O3—C8—O4	124.77 (12)
C6—C1—S1	119.46 (10)	O3—C8—C7	123.27 (11)
C10—N2—H1N2	118.7 (12)	O4—C8—C7	111.95 (10)
C10—N2—H2N2	118.0 (11)	C10—C9—C7	108.97 (10)
H1N2—N2—H2N2	123.3 (16)	C10—C9—H9A	109.9
C3—C2—C1	120.40 (12)	C7—C9—H9A	109.9
C3—C2—H2	119.8	C10—C9—H9B	109.9
C1—C2—H2	119.8	C7—C9—H9B	109.9
C2—C3—C4	119.19 (12)	H9A—C9—H9B	108.3
C2—C3—H3	120.4	O5—C10—N2	121.87 (12)
C4—C3—H3	120.4	O5—C10—C9	120.94 (10)
C8—O4—H4O	109.0 (14)	N2—C10—C9	117.14 (12)
O6—C4—C3	124.36 (12)	O6—C11—H11A	109.5
O6—C4—C5	115.50 (12)	O6—C11—H11B	109.5
C3—C4—C5	120.14 (11)	H11A—C11—H11B	109.5
C6—C5—C4	120.38 (12)	O6—C11—H11C	109.5

C6—C5—H5	119.8	H11A—C11—H11C	109.5
C4—C5—H5	119.8	H11B—C11—H11C	109.5
O1—S1—N1—C7	176.40 (10)	C3—C4—O6—C11	−3.51 (19)
O2—S1—N1—C7	−54.66 (11)	C5—C4—O6—C11	176.95 (12)
C1—S1—N1—C7	59.18 (11)	C4—C5—C6—C1	0.8 (2)
O1—S1—C1—C2	148.92 (10)	C2—C1—C6—C5	−0.26 (19)
O2—S1—C1—C2	19.54 (12)	S1—C1—C6—C5	177.93 (11)
N1—S1—C1—C2	−96.25 (11)	S1—N1—C7—C8	−108.87 (11)
O1—S1—C1—C6	−29.28 (12)	S1—N1—C7—C9	129.56 (10)
O2—S1—C1—C6	−158.65 (10)	N1—C7—C8—O3	−18.96 (16)
N1—S1—C1—C6	85.55 (11)	C9—C7—C8—O3	103.49 (14)
C6—C1—C2—C3	−0.15 (19)	N1—C7—C8—O4	162.62 (11)
S1—C1—C2—C3	−178.33 (10)	C9—C7—C8—O4	−74.93 (13)
C1—C2—C3—C4	0.06 (19)	N1—C7—C9—C10	−54.36 (13)
C2—C3—C4—O6	−179.08 (12)	C8—C7—C9—C10	−176.96 (10)
C2—C3—C4—C5	0.4 (2)	C7—C9—C10—O5	−65.42 (14)
O6—C4—C5—C6	178.71 (12)	C7—C9—C10—N2	112.13 (12)
C3—C4—C5—C6	−0.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O3^{i}	0.812 (19)	2.147 (19)	2.9430 (15)	166.9 (16)
N2—H2*N*2···O2^{ii}	0.89 (2)	2.02 (2)	2.8808 (16)	162.9 (17)
C11—H11*B*···O3^{iii}	0.98	2.48	3.3701 (17)	150
N1—H1*N*···O5^{ii}	0.927 (18)	1.907 (18)	2.8196 (14)	167.8 (16)
O4—H4*O*···O5^{iv}	0.81 (2)	1.79 (2)	2.5875 (14)	169 (2)
C9—H9*A*···O2^{ii}	0.99	2.54	3.4055 (16)	145

Symmetry codes: (i) −*x*−1, *y*−1/2, −*z*; (ii) −*x*−1, *y*+1/2, −*z*; (iii) −*x*+1, *y*−1/2, −*z*+1; (iv) −*x*, *y*+1/2, −*z*.