Crystal structures of two alanylpiperidine analogues

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The structure of ethyl 1-[N-(4-methylphenyl)-N-(methylsulfonyl)alanyl]-piperidine-4-carboxylate, C19H28N2O5S, I, a compound of interest as activator of Ubiquitin C-terminal Hydrolase-L1 (UCH-L1), was determined by single-crystal X-ray diffraction (SCXRD) analysis. In order to find new activators, a derivative of compound I, namely, 1-[N-(4-methylphenyl)-N-(methylsulfonyl)-alanyl]piperidine-4-carboxylic acid, C17H24N2O5S, II, was studied. The synthesis and crystal structure are also reported. Despite being analogues, different crystal packings are observed. Compound II bears a carboxylic group, which favors a strong hydrogen bond. A polymorph risk assessment was carried out to study interactions in compound II.

1. Chemical context

Ubiquitin C-terminal Hydrolase-L1 is a deubiquitinase that represents 2% of the neuronal soluble proteins in the brain and is involved in the neuropathogenesis of neurodegenerative diseases. Studies have shown that several mutations have an impact on the hydrolase activity of UCH-L1 (Leroy et al., 1998; Maraganore et al., 1999) and that its down-regulation is associated with idiopathic Parkinson’s disease (Choi et al., 2004). Finding potentiators of UCH-L1 could be a therapeutic pathway for these diseases (Mitsui et al., 2010). Ethyl 1-[N-(methylsulfonyl)-N-(p-tolyl)-alanyl]piperidine-4-carboxylate was discovered through in silico drug screening as an activator of UCH-L1, with a hydrolase activity up to 111% at 63 μM (Mitsui et al., 2010). We studied the only known activator in the literature, compound I. Derivatives of compound I were then investigated as potential activators and compound II was obtained after a saponification. Compound II bears a carboxylic acid group, which opens up the possibility for co-crystallization and salification in order to modulate the physicochemical properties, such as the solubility. We report the crystal structures of these two compounds as well as a survey of the interactions observed in compound II.

2. Structural commentary

Both compounds crystallize as colorless plate-like crystals but in different space groups. Compound I crystallizes in the
triclinic $P\overline{1}$ space groups and compound II in the monoclinic $P2_1/n$ space group. The asymmetric units are shown in Fig. 1. Both compounds crystallize as a racemic mixture and have one molecule in the asymmetric unit in a similar conformation. The torsion angle $N1—C1—C2—N2$ is 156.2 (1)° and 153.5 (1)° for I and II respectively. The only slight difference between the two compounds is the geometry of $N2$. In compound I, the distance between $N2$ and the plane formed by $C2$, $C3$ and $C7$ is 0.114 (2) Å whereas in compound II this distance is 0.014 (2) Å. A more planar arrangement of $N2$ in compound II is noticed, probably caused by the crystal packing. Single crystals represent the bulk samples as the powder patterns calculated from SCXRD data are similar to the experimental ones.

3. Supramolecular features

As compound I does not have any strong hydrogen-bond acceptors, only weak hydrogen bonds are observed in the crystal structure (see Table 1). The amide oxygen atom $O1$ participates in the formation of two intramolecular hydrogen bonds [$S1(7)$ motifs; Etter et al., 1990]. The oxygen atom $O4$ is inter-connected with atom $H12C$ of the sulfonyl methyl of an adjacent molecule [$d(H···O) 2.44 Å; Table 1$], forming an $R_2^2(8)$ hydrogen bond motif along the $a$-axis direction (Fig. 2). As compound I bears a tolyl moiety, $\pi—\pi$ interactions were expected but were not observed in this crystal packing.

Compound II bearing a carboxylic moiety instead of an ester has an impact on the hydrogen bonds and thus on the

![Figure 1](image1.png)

The asymmetric units of compounds I and II, with displacement ellipsoids drawn at the 50% probability level.

![Figure 2](image2.png)

Crystal packing of I with hydrogen bonds highlighted in green (a) showing one layer of molecules, viewed down the $a$ axis and (b) showing adjacent layers of molecules.

![Figure 3](image3.png)

Crystal packing of II showing the tubular arrangement viewed down the $a$ axis. Hydrogen bonds are highlighted in green.

| Compound  | $D$—$H···A$ | $D$—$H$ | $H···A$ | $D$—$A$ | $D$—$H···A$ |
|-----------|------------|---------|---------|----------|------------|
| I         |            |         |         |          |            |
| II        |            |         |         |          |            |

Table 1
Hydrogen-bond geometry ($Å$, °) for compound I.

| Compound  | $D$—$H···A$ | $D$—$H$ | $H···A$ | $D$—$A$ | $D$—$H···A$ |
|-----------|------------|---------|---------|----------|------------|
| I         |            |         |         |          |            |
| II        |            |         |         |          |            |

Table 2
Hydrogen-bond geometry ($Å$, °) for compound II.

| Compound  | $D$—$H···A$ | $D$—$H$ | $H···A$ | $D$—$A$ | $D$—$H···A$ |
|-----------|------------|---------|---------|----------|------------|
| I         |            |         |         |          |            |
| II        |            |         |         |          |            |

Symmetry code: (i) $-x, -y, -z + 1$.
crystal packing. In compound II, a tubular arrangement (Fig. 3) can be observed, which is different from that of compound I. In compound II, a hydrogen-bonded ring with an \(R_2^2(24)\) motif is formed by a strong hydrogen bond between H3 of the carboxylic acid group and O5 from an adjacent molecule \(d(H\cdots O) 1.88(3)\;\text{Å}; \text{Table 2}\). In addition, two intra-molecular \([S_6^3(7)\) motifs] and one intermolecular \([R_2^2(10)\) motif] weak hydrogen bonds are detected. As in compound I, no \(\pi\)-\(\pi\) interactions are noticed in the crystal structure. A dimer synthon is observed in the crystal packing in both cases, but for compound I it is ensured by weak hydrogen bonds in contrast to compound II where the dimer is based on strong hydrogen bonds.

4. Database survey

Searches of the Cambridge Structural Database (CSD, version 5.42, update September 2021; Groom et al. 2016) were carried out with the exact structures of compounds I and II and with substructures containing the significant fragments (alanil-piperidine with and without the sulfonyl methyl and tolyl group). No comparable structures came out of this survey.

A polymorph risk assessment based on the hydrogen bonds in the CSD was carried out. This statistical analysis allows us to estimate which atoms are the donors and the acceptors for hydrogen bonds in the crystal structure (Chemburkar et al., 2000; Galek et al., 2007). This quantifies the probability of hydrogen-bond formation and thus the different probable polymorphs that can arise from a specific compound. The results are summarized in Table 3. A hydrogen-bonding interaction between two carboxylic groups is predicted with the highest probability. We did not observe the carboxylic dimer but rather this group interacting with one oxygen of the sulfonyl methyl. The analysis also predicts other plausible hydrogen-bonded networks (Fig. 4), one that is statistically slightly more likely to be formed than the current one. This suggests that another potential polymorph could be obtained. Thus, we undertook a polymorph screening by several crystallization experiments of compound II. The recrystallization solvents that we tested were cyclohexane, toluene, ethyl acetate, chloroform, dichloromethane, acetone, acetonitrile, 2-propanol, ethanol and methanol. They all lead to the same polymorph.

5. Synthesis and crystallization

**Compound I:** This was purchased from Evotech (Hamburg, Germany). The product was crystallized by slow evaporation from non-anhydrous ethyl acetate, which provided colorless plate-like crystals suitable for SCXRD. M.p. 442.2 K

**Compound II:** In a round-bottom flask, compound I (405.1 mg, 1.02 mmol, 1.0 eq) dissolved in 8 mL of THF was added to a solution of LiOH (81.9 mg, 3.40 mmol, 3.4 eq) dissolved in 5 mL of water. The mixture was stirred at room temperature for 8 h. The resulting mixture was washed with ether. The aqueous phase was then acidified with HCl 37% to a pH of 2 and extracted with dichloromethane. The combined organic phases were dried over anhydrous Na\(_2\)SO\(_4\) and concentrated under vacuum to yield a white solid (351.0 mg, 93%). The product was crystallized by slow evaporation from methanol, which provided colorless plate-like crystals suitable for SCXRD. M.p. 496.2 K

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. All H atoms, except one of the -OH group in II, were refined using a riding model, with C—H = 0.93 (aromatic), 0.96 (methyl) or 0.98 Å (tertiary carbon). Coordinates of the hydrogen atom of the -OH group were refined. The isotropic atomic displacement parameters of the H atoms were set at 1.5\(U_{eq}\) of the parent atom for the methyl and alcohol groups, and at 1.2\(U_{eq}\) otherwise.

**Acknowledgements**

This work was performed on XRD equipment from the PC2 platform at UNamur and PXRD equipment has been funded...
by FRS–FNRS. The authors thank Laurie Bodard for her help on the polymorph risk assessment.

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Table 4
Experimental details.

| Crystal data | I | II |
|--------------|---|----|
| Chemical formula | C19H28N2O5S | C17H24N2O5S |
| M₀ | 396.49 | 368.44 |
| Crystal system, space group | Triclinic, P | Monoclinic, P2_1/n |
| Temperature (K) | 295 | 295 |
| a, b, c (Å) | 8.5368 (6), 9.6594 (6), 13.5173 (12) | 12.1013 (2), 12.3092 (2), 12.4348 (3) |
| α, β, γ (°) | 75.947 (6), 79.302 (6), 74.554 (5) | 90, 100.546 (2), 90 |
| V (Å³) | 1033.47 (14) | 1820.97 (6) |
| Z | 2 | 4 |
| Radiation type | Mo Ka | Mo Ka |
| µ (mm⁻¹) | 0.19 | 0.21 |
| Crystal size (mm) | 0.79 × 0.18 × 0.05 | 0.77 × 0.18 × 0.11 |

| Data collection | Oxford Diffraction Xcalibur, Gemini Ultra R | Oxford Diffraction Xcalibur, Gemini Ultra R |
|----------------|-----------------------------------------------|-----------------------------------------------|
| Diffractometer | Analytical [CrysAlis PRO (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)] | Analytical [CrysAlis PRO (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)] |
| Tmin, Tmax | 0.923, 0.991 | 0.893, 0.980 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 13200, 6870, 4304 | 29518, 6284, 4779 |
| Rint | 0.026 | 0.026 |
| (sin θ/λ)max (Å⁻¹) | 0.762 | 0.761 |

| Refinement | | |
| R[F² > 2σ(F²)], wR(F²), S | 0.054, 0.158, 1.02 | 0.043, 0.126, 1.02 |
| No. of reflections | 6870 | 6284 |
| No. of parameters | 248 | 232 |
| H-atom treatment | H-atom parameters constrained | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.34, −0.38 | 0.29, −0.29 |

Computer programs: CrysAlis PRO (Rigaku OD, 2018), SHELXT2014 (Sheldrick, 2015a), SHELXL2016 (Sheldrick, 2015b), Mercury (Macrae et al., 2020) and publCIF (Westrip, 2010).
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Computing details

For both structures, data collection: CrysAlis PRO (Rigaku OD, 2018); cell refinement: CrysAlis PRO (Rigaku OD, 2018); data reduction: CrysAlis PRO (Rigaku OD, 2018); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2016 (Sheldrick, 2015b); molecular graphics: Mercury (Macrae et al., 2020); software used to prepare material for publication: publCIF (Westrip, 2010).

Ethyl 1-[N-(4-methylphenyl)-N-(methylsulfonyl)alanyl]piperidine-4-carboxylate (I)

Crystal data

C_{19}H_{28}N_{2}O_{5}S
Mr = 396.49
Triclinic, P\bar{T}
\(a = 8.5368\) (6) Å
\(b = 9.6594\) (6) Å
\(c = 13.5173\) (12) Å
\(\alpha = 75.947\) (6)°
\(\beta = 79.302\) (6)°
\(\gamma = 74.554\) (5)°
\(V = 1033.47\) (14) Å³

Z = 2
\(F(000) = 424\)
\(D_{x} = 1.274\) Mg m⁻³
Mo Kα radiation, \(\lambda = 0.71073\) Å

Cell parameters from 3246 reflections
\(\theta_{\text{min}} = 2.2°\)
\(\theta_{\text{max}} = 32.8°\)
\(h = −12→10\)
\(k = −13→14\)
\(l = −20→19\)

Data collection

Oxford Diffraction Xcalibur, Gemini Ultra R diffractometer
Radiation source: fine-focus sealed X-ray tube
Graphite monochromator
Detector resolution: 10.3712 pixels mm⁻¹
\(\omega\) scans
Absorption correction: analytical
[CrysAlisPro (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)]

\(T_{\text{min}} = 0.923, T_{\text{max}} = 0.991\)
13200 measured reflections
6870 independent reflections
4304 reflections with \(I > 2\sigma(I)\)
\(R_{\text{int}} = 0.026\)
\(\theta_{\text{max}} = 32.8°, \theta_{\text{min}} = 2.2°\)

Refinement

Refinement on \(F^2\)
Least-squares matrix: full
\(R[F^2 > 2\sigma(F^2)] = 0.054\)
\(wR(F^2) = 0.158\)
\(S = 1.02\)
6870 reflections
248 parameters
0 restraints

Secondary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained
\(w = 1/[\sigma(F_c^2) + (0.0633P)^2 + 0.1206P]\)
where \(P = (F_c^2 + 2F_f^2)/3\)
\((\Delta/\sigma)_{\text{max}} < 0.001\)
\(\Delta\rho_{\text{max}} = 0.34\) e Å⁻³
\(\Delta\rho_{\text{min}} = −0.37\) e Å⁻³

Primary atom site location: dual

Acta Cryst. (2021). E77, 1095-1098
### 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.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

|     | x            | y            | z            | U(eq) |
|-----|--------------|--------------|--------------|-------|
| S1  | 0.21006 (5)  | 0.11226 (4)  | 0.49970 (3)  | 0.04597 (14) |
| O1  | 0.20546 (16) | 0.42592 (12) | 0.58010 (10) | 0.0542 (3)   |
| O2  | 0.2688 (3)   | −0.0438 (2)  | 1.00683 (16) | 0.1150 (8)   |
| O3  | 0.1551 (3)   | 0.16259 (18) | 1.06193 (12) | 0.0894 (5)   |
| O4  | 0.21136 (17) | 0.03480 (13) | 0.42186 (11) | 0.0618 (4)   |
| O5  | 0.25664 (18) | 0.02943 (13) | 0.59697 (11) | 0.0624 (4)   |
| N1  | 0.33461 (16) | 0.22121 (13) | 0.45549 (10) | 0.0404 (3)   |
| N2  | 0.3882 (2)   | 0.31933 (16) | 0.69243 (11) | 0.0543 (4)   |
| C1  | 0.4352 (2)   | 0.24222 (16) | 0.52607 (12) | 0.0407 (3)   |
| H1  | 0.484975     | 0.145601     | 0.564865     | 0.049*       |
| C2  | 0.3309 (2)   | 0.33501 (16) | 0.60307 (13) | 0.0435 (4)   |
| C3  | 0.2942 (3)   | 0.4081 (2)   | 0.76676 (14) | 0.0622 (5)   |
| H3A | 0.212739     | 0.487753     | 0.733487     | 0.075*       |
| H3B | 0.367076     | 0.450568     | 0.791143     | 0.075*       |
| C4  | 0.2099 (3)   | 0.3155 (2)   | 0.85723 (15) | 0.0598 (5)   |
| H4A | 0.126979     | 0.283509     | 0.834039     | 0.072*       |
| H4B | 0.155721     | 0.374364     | 0.908049     | 0.072*       |
| C5  | 0.3330 (3)   | 0.1809 (2)   | 0.90679 (14) | 0.0595 (5)   |
| H5  | 0.409068     | 0.216659     | 0.934903     | 0.071*       |
| C6  | 0.4334 (3)   | 0.0960 (2)   | 0.82547 (15) | 0.0631 (5)   |
| H6A | 0.517049     | 0.016237     | 0.856466     | 0.076*       |
| H6B | 0.362540     | 0.054116     | 0.798864     | 0.076*       |
| C7  | 0.5139 (3)   | 0.1963 (2)   | 0.73772 (15) | 0.0617 (5)   |
| H7A | 0.590425     | 0.233317     | 0.763271     | 0.074*       |
| C8  | 0.2494 (3)   | 0.0852 (2)   | 0.99538 (17) | 0.0714 (6)   |
| C9  | 0.0650 (4)   | 0.0879 (3)   | 1.1529 (2)   | 0.1069 (10)  |
| H9A | 0.004626     | 0.029494     | 1.132728     | 0.128*       |
| C10 | −0.0456 (5)  | 0.1958 (4)   | 1.2040 (2)   | 0.1357 (14)  |
| H10A| 0.012297     | 0.263307     | 1.213528     | 0.204*       |
| H10B| −0.091589    | 0.148539     | 1.269779     | 0.204*       |
| H10C| −0.131975    | 0.248210     | 1.163132     | 0.204*       |
| C11 | 0.5719 (2)   | 0.31422 (19) | 0.46630 (14) | 0.0503 (4)   |
| H11A| 0.636931     | 0.256036     | 0.418334     | 0.075*       |
| H11B| 0.639730     | 0.321567     | 0.513158     | 0.075*       |
| H11C| 0.525388     | 0.410564     | 0.429625     | 0.075*       |
| C12 | 0.0118 (2)   | 0.2217 (2)   | 0.5200 (2)   | 0.0700 (6)   |
| H12A| −0.017746    | 0.283965     | 0.455966     | 0.105*       |
### Atomic displacement parameters (Å$^2$)

|          | $U^{11}$      | $U^{22}$      | $U^{33}$      | $U^{12}$      | $U^{13}$      | $U^{23}$      |
|----------|---------------|---------------|---------------|---------------|---------------|---------------|
| S1       | 0.0381 (2)    | 0.0362 (2)    | 0.0636 (3)    | −0.00995 (16) | −0.00197 (19) | −0.01207 (17) |
| O1       | 0.0444 (7)    | 0.0491 (6)    | 0.0645 (8)    | 0.0048 (5)    | −0.0079 (6)   | −0.0194 (5)   |
| O2       | 0.157 (2)     | 0.0689 (11)   | 0.1072 (15)   | −0.0226 (12)  | 0.0015 (14)   | −0.0131 (10)  |
| O3       | 0.1050 (15)   | 0.0800 (10)   | 0.0695 (10)   | −0.0174 (10)  | 0.0144 (9)    | −0.0133 (8)   |
| O4       | 0.0598 (9)    | 0.0532 (7)    | 0.0829 (9)    | −0.0212 (6)   | −0.0043 (7)   | −0.0280 (6)   |
| O5       | 0.0629 (9)    | 0.0520 (7)    | 0.0669 (8)    | −0.0211 (6)   | −0.0053 (7)   | 0.0039 (6)    |
| O6       | 0.0352 (7)    | 0.0367 (6)    | 0.0492 (7)    | −0.0082 (5)   | −0.0054 (6)   | −0.0089 (5)   |
| O7       | 0.0541 (10)   | 0.0534 (8)    | 0.0517 (8)    | 0.0017 (7)    | −0.0076 (7)   | −0.0191 (6)   |
| C1       | 0.0333 (8)    | 0.0376 (7)    | 0.0507 (9)    | −0.0047 (6)   | −0.0051 (7)   | −0.0119 (6)   |
| C2       | 0.0389 (9)    | 0.0382 (7)    | 0.0527 (9)    | −0.0075 (6)   | −0.0036 (7)   | −0.0114 (6)   |
| C3       | 0.0761 (15)   | 0.0538 (10)   | 0.0557 (11)   | −0.0053 (10)  | −0.0049 (10)  | −0.0226 (8)   |
| C4       | 0.0598 (13)   | 0.0600 (11)   | 0.0568 (11)   | −0.0007 (9)   | −0.0072 (9)   | −0.0214 (9)   |
| C5       | 0.0600 (13)   | 0.0647 (11)   | 0.0542 (11)   | −0.0060 (9)   | −0.0145 (9)   | −0.0170 (8)   |
| C6       | 0.0635 (14)   | 0.0587 (10)   | 0.0620 (12)   | 0.0094 (9)    | 0.0241 (10)   | −0.0167 (9)   |
| C7       | 0.0490 (12)   | 0.0749 (12)   | 0.0598 (11)   | 0.0038 (9)    | −0.0152 (9)   | −0.0247 (9)   |
| C8       | 0.0784 (17)   | 0.0663 (13)   | 0.0668 (13)   | −0.0071 (11)  | −0.0187 (12)  | −0.0122 (10)  |
| C9       | 0.114 (3)     | 0.0948 (18)   | 0.0859 (19)   | −0.0194 (18)  | 0.0136 (18)   | 0.0053 (15)   |
| C10      | 0.125 (3)     | 0.143 (3)     | 0.092 (2)     | −0.013 (2)    | 0.033 (2)     | 0.009 (2)     |
| C11      | 0.0363 (9)    | 0.0539 (9)    | 0.0644 (11)   | −0.0139 (7)   | −0.0001 (8)   | −0.0200 (8)   |
| C12      | 0.0332 (10)   | 0.0571 (11)   | 0.1202 (19)   | −0.0110 (8)   | 0.0035 (11)   | −0.0288 (11)  |
| C13      | 0.0396 (9)    | 0.0410 (8)    | 0.0493 (9)    | −0.0094 (7)   | −0.0059 (7)   | −0.0105 (6)   |
| C14      | 0.0528 (11)   | 0.0484 (9)    | 0.0554 (10)   | 0.0016 (8)    | −0.0105 (9)   | −0.0109 (8)   |
| C15      | 0.0567 (13)   | 0.0568 (11)   | 0.0695 (13)   | 0.0007 (9)    | −0.0188 (10)  | −0.0030 (9)   |
| C16      | 0.0607 (13)   | 0.0713 (12)   | 0.0540 (11)   | −0.0235 (10)  | −0.0112 (10)  | −0.0024 (9)   |
| C17      | 0.0791 (16)   | 0.0693 (12)   | 0.0534 (11)   | −0.0223 (11)  | 0.0062 (10)   | −0.0168 (9)   |
| C18      | 0.0627 (13)   | 0.0471 (9)    | 0.0573 (11)   | −0.0109 (8)   | 0.0035 (9)    | −0.0143 (8)   |
| C19      | 0.092 (2)     | 0.1060 (19)   | 0.0624 (14)   | −0.0224 (16)  | −0.0167 (14)  | 0.0085 (13)   |
### Geometric parameters (Å, °)

| Bond/Distance | Length (Å) | Torsion (°) | Length (Å) | Torsion (°) |
|---------------|------------|-------------|------------|-------------|
| S1—O5         | 1.4285 (14) |             | C7—H7A     | 0.9700      |
| S1—O4         | 1.4286 (13) |             | C7—H7B     | 0.9700      |
| S1—N1         | 1.6278 (13) |             | C9—C10     | 1.428 (4)   |
| S1—C12        | 1.7526 (19) |             | C9—H9A     | 0.9700      |
| O1—C2         | 1.227 (2)   |             | C9—H9B     | 0.9700      |
| O2—C8         | 1.188 (3)   |             | C10—H10A   | 0.9600      |
| O3—C8         | 1.324 (3)   |             | C10—H10B   | 0.9600      |
| O3—C9         | 1.462 (3)   |             | C10—H10C   | 0.9600      |
| N1—C13        | 1.445 (2)   |             | C11—H11A   | 0.9600      |
| N1—C1         | 1.476 (2)   |             | C11—H11B   | 0.9600      |
| N2—C2         | 1.346 (2)   |             | C11—H11C   | 0.9600      |
| N2—C3         | 1.464 (2)   |             | C12—H12A   | 0.9600      |
| N2—C7         | 1.466 (2)   |             | C12—H12B   | 0.9600      |
| C1—C11        | 1.519 (2)   |             | C12—H12C   | 0.9600      |
| C1—C2         | 1.537 (2)   |             | C13—C14    | 1.380 (2)   |
| C1—H1         | 0.9800      |             | C13—C18    | 1.381 (2)   |
| C3—C4         | 1.516 (3)   |             | C14—C15    | 1.382 (3)   |
| C3—H3A        | 0.9700      |             | C14—H14    | 0.9300      |
| C3—H3B        | 0.9700      |             | C15—C16    | 1.379 (3)   |
| C4—C5         | 1.535 (3)   |             | C15—H15    | 0.9300      |
| C4—H4A        | 0.9700      |             | C16—C17    | 1.384 (3)   |
| C4—H4B        | 0.9700      |             | C16—C19    | 1.514 (3)   |
| C5—C8         | 1.514 (3)   |             | C17—C18    | 1.381 (3)   |
| C5—H5         | 0.9800      |             | C17—H17    | 0.9300      |
| C6—C7         | 1.521 (3)   |             | C18—H18    | 0.9300      |
| C6—H6A        | 0.9700      |             | C19—H19A   | 0.9600      |
| C6—H6B        | 0.9700      |             | C19—H19B   | 0.9600      |
| O5—S1—O4      | 118.43 (8)  |             | O2—C8—O3   | 123.4 (2)   |
| O5—S1—N1      | 106.30 (8)  |             | O2—C8—C5   | 125.1 (2)   |
| O4—S1—N1      | 108.16 (8)  |             | O3—C8—C5   | 111.50 (18) |
| O5—S1—C12     | 108.50 (11) |             | C10—C9—O3  | 108.7 (2)   |
| O4—S1—C12     | 107.33 (10) |             | C10—C9—H9A | 110.0       |
| N1—S1—C12     | 107.71 (8)  |             | O3—C9—H9A  | 110.0       |
| C8—O3—C9      | 119.1 (2)   |             | C10—C9—H9B | 110.0       |
| C13—N1—C1     | 122.65 (12) |             | O3—C9—H9B  | 110.0       |
| C13—N1—S1     | 117.57 (11) |             | H9A—C9—H9B | 108.3       |
| C1—N1—S1      | 118.92 (10) |             | C9—C10—H10A| 109.5       |
| C2—N2—C3      | 119.21 (16) |             | C9—C10—H10B| 109.5       |
| C2—N2—C7      | 126.42 (15) |             | H10A—C10—H10B| 109.5      |
| C3—N2—C7      | 112.45 (15) |             | C9—C10—H10C| 109.5       |
| N1—C1—C11     | 110.73 (13) |             | H10A—C10—H10C| 109.5      |
| N1—C1—C2      | 111.57 (13) |             | H10B—C10—H10C| 109.5      |
| C11—C1—C2     | 109.61 (12) |             | C1—C11—H11A| 109.5       |
| N1—C1—H1      | 108.3       |             | C1—C11—H11B| 109.5       |
C11—C1—H1 108.3   H11A—C11—H11B 109.5
C2—C1—H1 108.3   C1—C11—H11C 109.5
O1—C2—N2 121.94 (15)   H11A—C11—H11C 109.5
O1—C2—C1 120.33 (15)   H11B—C11—H11C 109.5
N2—C2—C1 117.58 (15)   S1—C12—H12A 109.5
N2—C3—C4 110.73 (15)   S1—C12—H12B 109.5
N2—C3—H3A 109.5   H12A—C12—H12B 109.5
C4—C3—H3A 109.5   S1—C12—H12C 109.5
N2—C3—H3B 109.5   H12B—C12—H12C 109.5
H3A—C3—H3B 108.1   C14—C13—C18 119.67 (16)
C3—C4—C5 111.24 (18)   C14—C13—N1 121.20 (15)
C3—C4—H4A 109.4   C18—C13—N1 119.13 (14)
C5—C4—H4A 109.4   C13—C14—C15 117.39 (18)
C3—C4—H4B 109.4   C13—C14—H14 120.3
C5—C4—H4B 109.4   C15—C14—H14 120.3
H4A—C4—H4B 108.0   C16—C15—C14 122.01 (18)
C8—C5—C6 112.44 (17)   C16—C15—H15 119.0
C8—C5—C4 111.67 (19)   C14—C15—H15 119.0
C6—C5—C4 110.22 (16)   C15—C16—C17 117.39 (18)
C8—C5—H5 107.4   C15—C16—C19 120.6 (2)
C6—C5—H5 107.4   C17—C16—C19 122.0 (2)
C4—C5—H5 107.4   C18—C17—C16 121.65 (19)
C7—C6—C5 110.66 (16)   C18—C17—H17 119.2
C7—C6—H6A 109.5   C16—C17—H17 119.2
C6—C5—C8 109.5   C13—C18—C17 119.76 (17)
C7—C6—H6A 109.5   C13—C18—H18 120.1
H6A—C6—H6B 108.1   C17—C18—H18 120.1
N2—C7—C6 109.7 (17)   C16—C19—C17 109.5
N2—C7—C7A 109.7   H19A—C19—H19B 109.5
C6—C7—C7A 109.7   C16—C19—H19C 109.5
N2—C7—H7B 109.7   H19A—C19—H19C 109.5
C6—C7—H7B 109.7   H19B—C19—H19C 109.5
H7A—C7—H7B 108.2

O5—S1—N1—C13 176.09 (11)   C2—N2—C7—C6 −103.4 (2)
O4—S1—N1—C13 47.92 (13)   C3—N2—C7—C6 60.5 (2)
C12—S1—N1—C13 −67.78 (14)   C5—C6—C7—C6 −57.7 (2)
O5—S1—N1—C1 −14.24 (14)   C9—O3—C8—O2 −2.2 (4)
O4—S1—N1—C1 −142.40 (12)   C9—O3—C8—C5 −179.8 (2)
C12—S1—N1—C1 −101.89 (14)   C6—C5—C8—O2 6.3 (4)
C13—N1—C1—C11 −25.36 (19)   C4—C5—C8—O2 130.8 (3)
S1—N1—C1—C11 165.51 (11)   C6—C5—C8—O3 −176.2 (2)
C13—N1—C1—C2 97.02 (16)   C4—C5—C8—O3 −51.7 (3)
S1—N1—C1—C2 −72.11 (15)   C8—O3—C9—C10 −171.3 (3)
C3—N2—C2—O1 3.0 (3)   C1—N1—C13—C14 −76.6 (2)
C7—N2—C2—O1 165.98 (18)   S1—N1—C13—C14 92.68 (17)
C3—N2—C2—C1 178.66 (15) C1—N1—C13—C18 103.66 (19)
C7—N2—C2—C1 −18.4 (3) S1—N1—C13—C18 −87.07 (18)
N1—C1—C2—O1 −28.1 (2) C18—C13—C14—C15 1.3 (3)
C11—C1—C2—O1 94.89 (19) N1—C13—C14—C15 −178.45 (17)
N1—C1—C2—N2 156.17 (14) C18—C13—C18—C17 −1.7 (3)
C11—C1—C2—N2 −80.81 (18) N1—C13—C18—C17 178.02 (17)
C2—N2—C3—C4 106.1 (2) C15—C16—C17—C18 1.7 (3)
C7—N2—C3—C4 −59.1 (2) C14—C15—C16—C17 −1.7 (3)
N2—C3—C4—C5 54.5 (2) C15—C16—C17—C18 1.7 (3)
C3—C4—C5—C8 −178.38 (17) C14—C13—C18—C17 −1.7 (3)
C3—C4—C5—C6 52.6 (2) N1—C13—C18—C17 178.02 (17)
C8—C5—C6—C7 179.54 (18) C14—C13—C14—C15 0.7 (3)
C4—C5—C6—C7 54.2 (2)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|--------|
| C12—H12B···O1 0.96 | 2.50 | 3.210 (2) | 130 |
| C12—H12C···O4i 0.96 | 2.44 | 3.376 (2) | 164 |
| C14—H14···O1 0.93 | 2.48 | 3.177 (2) | 132 |

Symmetry code: (i) −x, −y, −z+1.

1-[N-(4-methylphenyl)-N-(methylsulfonyl)alanyl]piperidine-4-carboxylic acid (II)

Crystal data

C17H24N2O5S  
Mr = 368.44  
Monoclinic, P21/n  
a = 12.1013 (2) Å  
b = 12.3092 (2) Å  
c = 12.4348 (3) Å  
β = 100.546 (2)°  
V = 1820.97 (6) Å³  
Z = 4  
Dx = 1.344 Mg m⁻³  
Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 8359 reflections  
θ = 2.7–31.5°  
µ = 0.21 mm⁻¹  
T = 295 K  
Plate, colorless  
0.77 × 0.18 × 0.11 mm

Data collection

Oxford Diffraction Xcalibur, Gemini Ultra R  
Radiation source: fine-focus sealed X-ray tube  
Graphite monochromator  
Detector resolution: 10.3712 pixels mm⁻¹  
ω scans  
Absorption correction: analytical  
[CrysAlisPro (Rigaku OD, 2018), based on expressions derived by Clark & Reid (1995)]  
Tmin = 0.882, Tmax = 0.980  
θmax = 32.7°, θmin = 2.2°  
h = −18→18  
k = −18→17  
l = −17→18

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.043  
wR(F²) = 0.126  
S = 1.02  
29518 measured reflections  
6284 independent reflections  
4779 reflections with I > 2σ(I)  
Rint = 0.026  
θmax = 26.0°, θmin = 2.2°  
Primary atom site location: dual  
Secondary atom site location: dual  
232 parameters  
0 restraints
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement

\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0593P)^2 + 0.3685P} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\[ (\Delta/\sigma)_{\text{max}} = 0.001 \]
\[ \Delta \rho_{\text{max}} = 0.29 \text{ e Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.29 \text{ e Å}^{-3} \]

**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.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

| Symbol | x         | y         | z         | U_{iso} / U_{eq} |
|--------|-----------|-----------|-----------|-----------------|
| S1     | 0.58667 (2) | 0.35673 (2) | 0.60942 (2) | 0.03513 (9)     |
| O1     | 0.68078 (9)  | 0.19851 (8)  | 0.41698 (10) | 0.0531 (3)      |
| O2     | 1.04956 (12) | 0.60661 (11) | 0.37887 (13) | 0.0780 (4)      |
| O3     | 1.13713 (10) | 0.46783 (11) | 0.31848 (13) | 0.0732 (4)      |
| H3     | 1.195 (2)    | 0.509 (2)    | 0.352 (2)    | 0.110*          |
| O4     | 0.50845 (8)  | 0.37442 (9)  | 0.68112 (8)  | 0.0479 (2)      |
| O5     | 0.65804 (8)  | 0.44541 (8)  | 0.59029 (9)  | 0.0501 (2)      |
| N1     | 0.51748 (8)  | 0.32221 (9)  | 0.48974 (8)  | 0.0344 (2)      |
| N2     | 0.73982 (9)  | 0.33804 (10) | 0.32395 (10) | 0.0447 (3)      |
| C1     | 0.56084 (10) | 0.35539 (10) | 0.39054 (10) | 0.0359 (2)      |
| H1     | 0.580679     | 0.432597     | 0.397240     | 0.043*          |
| C17    | 0.67424 (14) | 0.24889 (13) | 0.66359 (13) | 0.0555 (4)      |
| H17A   | 0.726630     | 0.233560     | 0.616194     | 0.083*          |
| H17B   | 0.629402     | 0.185582     | 0.669483     | 0.083*          |
| H17C   | 0.714621     | 0.268508     | 0.734742     | 0.083*          |
| C2     | 0.66661 (10) | 0.29079 (11) | 0.37903 (10) | 0.0392 (3)      |
| C3     | 0.83897 (11) | 0.27729 (13) | 0.30503 (13) | 0.0488 (3)      |
| H3A    | 0.832698     | 0.263103     | 0.227419     | 0.059*          |
| H3B    | 0.842307     | 0.208002     | 0.342640     | 0.059*          |
| C4     | 0.94599 (11) | 0.34095 (12) | 0.34608 (11) | 0.0427 (3)      |
| H4A    | 1.009763     | 0.302094     | 0.327725     | 0.051*          |
| H4B    | 0.957054     | 0.347251     | 0.425085     | 0.051*          |
| C5     | 0.94003 (11) | 0.45455 (12) | 0.29524 (11) | 0.0438 (3)      |
| H5     | 0.933185     | 0.446018     | 0.215935     | 0.053*          |
| C6     | 0.83536 (12) | 0.51356 (12) | 0.31663 (13) | 0.0510 (3)      |
| H6A    | 0.841595     | 0.526101     | 0.394520     | 0.061*          |
| H6B    | 0.829392     | 0.583548     | 0.280182     | 0.061*          |
| C7     | 0.73079 (12) | 0.44690 (14) | 0.27497 (12) | 0.0510 (4)      |
| H7A    | 0.665534     | 0.483641     | 0.292748     | 0.061*          |
| H7B    | 0.720557     | 0.440587     | 0.196007     | 0.061*          |
| C8     | 1.04549 (13) | 0.51874 (13) | 0.33621 (12) | 0.0489 (3)      |
| C9     | 0.47176 (12) | 0.34023 (14) | 0.28791 (12) | 0.0489 (3)      |
| H9A    | 0.458064     | 0.264097     | 0.275037     | 0.073*          |
| H9B    | 0.497692     | 0.371751     | 0.226417     | 0.073*          |
### Atomic displacement parameters (Å²)

|   | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|---|-----------|-----------|-----------|-----------|-----------|-----------|
| S1| 0.03062 (14) | 0.03481 (15) | 0.03976 (16) | 0.00400 (11) | 0.00588 (11) | 0.00067 (11) |
| O1| 0.0514 (6) | 0.0410 (5) | 0.0722 (7) | 0.0074 (4) | 0.0248 (5) | 0.0058 (5) |
| O2| 0.0771 (9) | 0.0640 (8) | 0.0943 (10) | 0.0098 (7) | 0.0193 (7) | 0.0239 (7) |
| O3| 0.0402 (6) | 0.0697 (8) | 0.1097 (11) | 0.0137 (5) | 0.0139 (6) | 0.0030 (7) |
| O4| 0.0448 (5) | 0.0571 (6) | 0.0436 (5) | 0.0033 (4) | 0.0129 (4) | 0.0075 (4) |
| O5| 0.0436 (5) | 0.0481 (5) | 0.0563 (6) | 0.0190 (4) | 0.0033 (4) | 0.0010 (4) |
| N1| 0.0293 (4) | 0.0375 (5) | 0.0373 (5) | 0.0058 (4) | 0.0082 (4) | 0.0016 (4) |
| N2| 0.0353 (5) | 0.0490 (6) | 0.0530 (6) | 0.0044 (5) | 0.0171 (5) | 0.0037 (5) |
| C1| 0.0321 (5) | 0.0382 (6) | 0.0387 (6) | 0.0001 (5) | 0.0102 (4) | 0.0020 (5) |
| C17| 0.0548 (8) | 0.0583 (9) | 0.0515 (8) | 0.0155 (7) | 0.0051 (7) | 0.0093 (7) |
| C2| 0.0345 (6) | 0.0428 (6) | 0.0414 (6) | 0.0005 (5) | 0.0103 (5) | 0.0025 (5) |
| C3| 0.0366 (6) | 0.0525 (8) | 0.0609 (9) | 0.0005 (6) | 0.0184 (6) | 0.0099 (7) |
| C4| 0.0360 (6) | 0.0486 (7) | 0.0450 (7) | 0.0029 (5) | 0.0114 (5) | 0.0049 (5) |
| C5| 0.0388 (6) | 0.0551 (8) | 0.0387 (6) | 0.0029 (6) | 0.0107 (5) | 0.0008 (6) |
| C6| 0.0493 (8) | 0.0481 (8) | 0.0591 (8) | 0.0065 (6) | 0.0190 (7) | 0.0149 (6) |
| C7| 0.0397 (7) | 0.0658 (9) | 0.0499 (8) | 0.0079 (6) | 0.0147 (6) | 0.0195 (7) |
| C8| 0.0490 (8) | 0.0517 (8) | 0.0469 (7) | 0.0062 (6) | 0.0107 (6) | 0.0010 (6) |
| C9| 0.0407 (7) | 0.0635 (9) | 0.0411 (7) | 0.0017 (6) | 0.0039 (5) | 0.0014 (6) |
| C10| 0.0327 (5) | 0.0359 (6) | 0.0404 (6) | 0.0076 (5) | 0.0059 (5) | 0.0002 (5) |
| C11| 0.0499 (8) | 0.0397 (7) | 0.0735 (10) | 0.0066 (6) | 0.0131 (7) | 0.0083 (7) |
| C12| 0.0713 (11) | 0.0438 (8) | 0.0759 (11) | 0.0228 (8) | 0.0088 (9) | 0.0078 (7) |
| C13| 0.0513 (8) | 0.0660 (9) | 0.0443 (7) | 0.0296 (7) | 0.0026 (6) | 0.0081 (7) |
| C14| 0.0316 (6) | 0.0692 (10) | 0.0534 (8) | 0.0112 (6) | 0.0038 (5) | 0.0085 (7) |
| C15| 0.0320 (6) | 0.0447 (7) | 0.0500 (7) | 0.0045 (5) | 0.0066 (5) | 0.0028 (5) |
| C16| 0.0732 (12) | 0.1026 (16) | 0.0695 (11) | 0.0573 (12) | 0.0065 (9) | 0.0100 (11) |
Geometric parameters (Å, º)

| Bond                  | Length (Å) | Bond                  | Length (Å) | Bond                  | Length (Å) |
|-----------------------|------------|-----------------------|------------|-----------------------|------------|
| S1—O4                 | 1.4309 (10) | C5—C8                 | 1.508 (2)  | S1—O5                 | 1.4385 (10) |
| S1—O5                 | 1.4385 (10) | C5—C6                 | 1.5252 (19) | S1—N1                 | 1.6249 (10) |
| S1—N1                 | 1.6249 (10) | C5—H5                 | 0.9800     | S1—C17                | 1.7543 (15) |
| O1—C2                 | 1.2300 (16) | C6—H6A                | 0.9700     | O2—C8                 | 1.2018 (19) |
| O2—C8                 | 1.2018 (19) | C6—H6B                | 0.9700     | O3—C8                 | 1.3269 (19) |
| O3—C8                 | 1.3269 (19) | C7—H7A                | 0.9700     | O3—H3                 | 0.90 (3)   |
| N1—C10                | 1.4438 (15) | C7—H7B                | 0.9700     | N1—C1                 | 1.4834 (15) |
| N1—C1                 | 1.4834 (15) | C9—H9A                | 0.9600     | N2—C2                 | 1.3470 (17) |
| N2—C2                 | 1.3470 (17) | C9—H9B                | 0.9600     | N2—C7                 | 1.4677 (19) |
| N2—C7                 | 1.4677 (19) | C10—C15               | 1.3829 (18) | N2—C3                 | 1.4688 (17) |
| C1—C9                 | 1.5238 (18) | C11—C12               | 1.390 (2)  | C1—H1                 | 0.9800     |
| C1—C2                 | 1.5356 (17) | C11—H11               | 0.9300     | C17—H17A              | 0.9600     |
| C1—H1                 | 0.9800     | C12—C13               | 1.377 (3)  | C17—H17B              | 0.9600     |
| C17—H17A              | 0.9600     | C12—H12               | 0.9300     | C17—H17C              | 0.9600     |
| C3—C4                 | 1.5198 (19) | C13—C14               | 1.382 (2)  | C3—H3A                | 0.9700     |
| C3—H3A                | 0.9700     | C14—H14               | 0.9300     | C3—H3B                | 0.9700     |
| C3—H3B                | 0.9700     | C15—H15               | 0.9300     | C4—C5                 | 1.531 (2)  |
| C4—C5                 | 1.531 (2)  | C16—H16A              | 0.9600     | C4—H4A                | 0.9700     |
| C4—H4A                | 0.9700     | C16—H16B              | 0.9600     | C4—H4B                | 0.9700     |

O4—S1—O5  118.24 (6)  C7—C6—C5  110.50 (13)
O4—S1—N1  108.76 (6)  C7—C6—H6A  109.5
O5—S1—N1  105.74 (6)  C5—C6—H6A  109.5
O4—S1—C17 107.33 (7)  C7—C6—H6B  109.5
O5—S1—C17 107.40 (7)  C5—C6—H6B  109.5
N1—S1—C17 109.13 (7)  H6A—C6—H6B  108.1
C8—O3—H3  105.0 (16)  N2—C7—C6  110.97 (12)
C10—N1—C1 122.13 (10) N2—C7—H7A  109.4
C10—N1—S1 118.32 (8)  C6—C7—H7A  109.4
C1—N1—S1  119.22 (8)  N2—C7—H7B  109.4
C2—N2—C7  126.82 (11) C6—C7—H7B  109.4
C2—N2—C3  119.62 (12) C7—H7A—C7—H7B  108.0
C7—N2—C3  113.53 (11) O2—C8—O3  122.04 (15)
N1—C1—C9  111.04 (10) O2—C8—C5  125.76 (15)
N1—C1—C2  111.26 (10) O3—C8—C5  112.20 (13)
C9—C1—C2  109.42 (11) C1—C9—H9A  109.5
N1—C1—H1  108.3 C1—C9—H9B  109.5
C9—C1—H1  108.3 H9A—C9—H9B  109.5
C2—C1—H1  108.3 C1—C9—H9C  109.5
S1—C17—H17A 109.5 H9A—C9—H9C  109.5
| Bond                  | Value 1 | Bond                  | Value 2 | Bond                  | Value 3 |
|----------------------|---------|----------------------|---------|----------------------|---------|
| S1—C17—H17B         | 109.5   | H9B—C9—H9C          | 109.5   |                      |         |
| H17A—C17—H17B       | 109.5   | C15—C10—C11         | 119.68  | (12)                 |
| S1—C17—H17C         | 109.5   | C15—C10—N1          | 119.04  | (11)                 |
| H17A—C17—H17C       | 109.5   | C11—C10—N1          | 121.26  | (12)                 |
| H17B—C17—H17C       | 109.5   | C10—C11—C12         | 119.43  | (15)                 |
| O1—C2—N2            | 122.37  | (12)                 |         |                      |         |
| O1—C2—C1            | 120.17  | (11)                 |         |                      |         |
| N2—C2—C1            | 117.44  | (11)                 |         |                      |         |
| N2—C3—C4            | 110.77  | (12)                 |         |                      |         |
| N2—C3—H3A           | 109.5   | C11—C12—H12         | 119.1   |                     |
| C4—C3—H3A           | 109.5   | C12—C13—C14         | 118.04  | (13)                 |
| N2—C3—H3B           | 109.5   | C12—C13—C16         | 120.88  | (18)                 |
| C4—C3—H3B           | 109.5   | C12—C13—C16         | 120.88  | (18)                 |
| H3A—C3—H3B          | 108.1   | C13—C14—C15         | 121.38  | (15)                 |
| C3—C4—C5            | 111.06  | (12)                 |         |                      |         |
| C3—C4—H4A           | 109.4   | C13—C14—H14         | 119.3   |                     |
| C5—C4—H4A           | 109.4   | C15—C14—H14         | 119.3   |                     |
| C3—C4—H4B           | 109.4   | C10—C15—C14         | 119.76  | (14)                 |
| C5—C4—H4B           | 109.4   | C10—C15—H15         | 120.1   |                     |
| H4A—C4—H4B          | 108.0   | C14—C15—H15         | 120.1   |                     |
| C8—C5—C6            | 111.72  | (13)                 |         |                      |         |
| C8—C5—C4            | 111.47  | (12)                 |         |                      |         |
| C6—C5—C4            | 109.92  | (11)                 |         |                      |         |
| C8—C5—H5            | 107.9   | H16A—C16—H16B       | 109.5   |                     |
| C6—C5—H5            | 107.9   | H16B—C16—H16C       | 109.5   |                     |
| C4—C5—H5            | 107.9   | H16B—C16—H16C       | 109.5   |                     |

| Bond                  | Value 1 | Bond                  | Value 2 | Bond                  | Value 3 |
|----------------------|---------|----------------------|---------|----------------------|---------|
| O4—S1—N1—C10        | −39.35  | (11)                 |         |                      |         |
| O5—S1—N1—C10        | −167.31 | (9)                  |         |                      |         |
| C17—S1—N1—C10       | 77.45   | (11)                 |         |                      |         |
| O4—S1—N1—C1         | 147.15  | (9)                  |         |                      |         |
| O5—S1—N1—C1         | 19.20   | (11)                 |         |                      |         |
| C17—S1—N1—C1        | −96.05  | (11)                 |         |                      |         |
| C10—N1—C1—C9        | 20.64   | (16)                 |         |                      |         |
| S1—N1—C1—C9         | −166.13 | (9)                  |         |                      |         |
| C10—N1—C1—C2        | −101.50 | (13)                 |         |                      |         |
| S1—N1—C1—C2         | 71.73   | (12)                 |         |                      |         |
| C7—N2—C2—O1         | 179.61  | (14)                 |         |                      |         |
| C3—N2—C2—O1         | 1.8 (2) |                      |         |                      |         |
| C7—N2—C2—C1         | 1.4 (2) |                      |         |                      |         |
| C3—N2—C2—C1         | −176.43 | (12)                 |         |                      |         |
| N1—C1—C2—O1         | 28.24   | (17)                 |         |                      |         |
| C9—C1—C2—O1         | −94.83  | (15)                 |         |                      |         |
| N1—C1—C2—N2         | −153.51 | (11)                 |         |                      |         |
| C9—C1—C2—N2         | 83.42   | (15)                 |         |                      |         |
| C2—N2—C3—C4         | −125.84 | (14)                 |         |                      |         |
| C7—N2—C3—C4         | 56.05   | (17)                 |         |                      |         |
| N2—C3—C4—C5         | −54.68  | (15)                 |         |                      |         |
**Hydrogen-bond geometry (Å, ‚)\(^{\circ}\)**

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|---------|
| O3—H3···O5\(^{i}\) | 0.90 (3) | 1.88 (3) | 2.7463 (15) | 161 (2) |
| C17—H17A···O1 | 0.96 | 2.48 | 3.144 (2) | 127 |
| C4—H4B···O2\(^{i}\) | 0.97 | 2.52 | 3.471 (2) | 167 |
| C11—H11···O1 | 0.93 | 2.56 | 3.2558 (19) | 132 |

Symmetry code: (i) \(-x+2, -y+1, -z+1\).