Crystal structure and Hirshfeld surface analysis of N-[(5-(4-methylphenyl)-1,2-oxazol-3-yl)methyl]-1-phenyl-N-(prop-2-en-1-yl)methanesulfonamide

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In the title compound, C21H22N2O3S, the 1,2-oxazole ring makes the dihedral angles of 9.16 (16) and 87.91 (17)° with the toluene and phenyl rings, while they form a dihedral angle of 84.42 (15)° with each other. The C—S—N—C pr and C—S—N—C me (pr = propene, me = 3-methyl-1,2-oxazole) torsion angles are 86.8 (2) and −100.6 (3)°, respectively. In the crystal, molecules are linked by C—H···O hydrogen bonds, generating a three-dimensional network. A Hirshfeld surface analysis was performed to investigate the contributions of the different intermolecular contacts within the supramolecular structure. The major interactions are H···H (53.6%), C···H/H···C (20.8%) and O···H/H···O (17.7%).

1. Chemical context

Sulfonamide antibiotics are readily available drugs that are gradually losing their importance due to the development of bacterial resistance (Sköld, 2000). Along with the use of much less accessible antibiotics of other classes, the design of new sulfonamides to overcome this problem seems to be reasonable (Nadirova et al., 2021; Naghiyev et al., 2020). One of the possible methods for structural modification is the synthesis of drug analogues containing heterocycles. From this point of view, isothiazole (Kletskov et al., 2020; Khalilov et al., 2021) and isoxazole (Zhu et al., 2018; Abdelhamid et al., 2011) rings are of great interest. In particular, isoxazole derivatives possess a wide range of biological activity, so this heterocycle is considered to be one of the most privileged scaffolds in pharmaceutical chemistry (Altug et al., 2017; Safavora et al., 2019). Moreover, a lot of isoxazoles exhibit antibacterial properties on their own (Agrawal & Mishra, 2018; Yadigarov et al., 2009), and the widely used sulfonamide antibiotic sulfamethoxazole contains an isoxazole ring. A preliminary assessment of the biological activity of newly designed isoxazole-containing structures can be carried out in silico using molecular docking. Data on the structural parameters of promising molecules is therefore required (Gurbanov et al., 2020a,b; Ma et al., 2020,2021). All this was our motive for the synthesis and accurate structure establishment of N-allyl-N-[(5-tolylisoxazol-3-yl)methyl]benzylsulfonamide (1), which
has not previously been characterized. It was obtained from isoxazolylallylamine (2) and benzyl sulfonyl chloride using the 'green chemistry' procedure developed earlier by one of us (Kolesnik et al., 2022).

Allyl derivatives structurally similar to sulfonamide 1 are widely used as starting materials in organic synthesis for the construction of polyheterocyclic systems through intra-molecular \[4 + 2\] cycloaddition reactions (Zubkov et al., 2014; Krishna et al., 2022).

### 2. Structural commentary

In the title compound (Fig. 1), the 1,2-oxazole ring (O3/N2/C3–C5) forms dihedral angles of 9.16 (16) and 87.91 (17)/C14, respectively, with the toluene and phenyl rings (C6–C11 and C16–C21) which subtend a dihedral angle of 84.42 (15)/C14 with each other. The torsion angles C1—S1—N1—C2 and C1—S1—N1—C13 are 86.8 (2) and 100.6 (3)/C14, respectively.

### 3. Supramolecular features and Hirshfeld surface analysis

Molecules in the crystal are joined together by C—H⋯O hydrogen bonds, forming a three-dimensional network (Table 1; Figs. 2, 3 and 4).

The Hirshfeld surfaces were calculated and two-dimensional fingerprint plots generated using Crystal Explorer 17.5 (Spackman et al., 2021). Fig. 5 depicts the three-dimensional Hirshfeld surface projected over \(d_{norm}\) in the range –0.1677 to 1.4857 a.u. The bright-red patches surrounding O1, O2, and O3 and hydrogen atoms H8, H17, H19, and H21, which highlight their activities as donors and/or acceptors, can be connected with O1, O2, and O3 interactions, which play a significant role in the molecular packing (Tables 1 and 2).

#### Table 1

| \(D—H—A\) | \(D—H\) | \(H—A\) | \(D—A\) | \(D—H—A\) |
|---|---|---|---|---|
| C8—H8···O2i | 0.95 | 2.59 | 3.404 (4) | 143 |
| C17—H17···O3ii | 0.95 | 2.57 | 3.314 (4) | 135 |
| C19—H19···O1iii | 0.95 | 2.51 | 3.434 (4) | 165 |
| C21—H21···O2iv | 0.95 | 2.50 | 3.369 (4) | 152 |

Symmetry codes: (i) \(x, 1+y, z, 1+z\); (ii) \(-x, 1+y, 1-z\); (iii) \(1-x, y, z\); (iv) \(-x+1, -y+1, z\).

Figure 1  
The title molecule with the labelling scheme and 50% probability ellipsoids.

Figure 2  
A view along the \(a\) axis of the C—H⋯O interactions in the title compound.

Figure 3  
A view along the \(b\) axis of the C—H⋯O interactions in the title compound.
Fig. 6 depicts the overall two-dimensional fingerprint plot for the title compound. The percentage contributions to the Hirshfeld surfaces from various interatomic interactions (Table 2) include $H\cdots C_1/C_1/C_1H$ (53.6%; Fig. 6a), $C\cdots C_1/C_1/C_1H/H\cdots C_1/C_1/C_1C$ (20.8%; Fig. 6c) and $O\cdots C_1/C_1/C_1H/H\cdots C_1/C_1/C_1O$ (17.7%; Fig. 6d). Other contact types, such as $N\cdots C_1/C_1/C_1H/H\cdots N$ (4.5%), $C\cdots C_1/C_1/C_1C$ (1.7%), $N\cdots C_1/C_1/C_1C/C\cdots C_1/C_1/C_1N$ (0.9%), and $O\cdots C_1/C_1/C_1C/C\cdots O$ (0.8%), account for less than 4.5% of the Hirshfeld surface and are likely to have little directional impact on the packing.

4. Database survey

Four related compounds with a methanesulfonamide unit have been reported, viz. $N$-(4-chlorophenyl)-1-(5-[[2-(phenylvinyl)sulfonyl][methyl]-1,3,4-oxadiazol-2-yl]methanesulfonamide (CEGKAC: Muralikrishna et al., 2012), $N$-(4-fluoro-phenyl)methanesulfonamide (CICPIO: Gowda et al., 2007a), $N$-(2,5-dichlorophenyl)methanesulfonamide (WIHGUQ: Gowda et al., 2007b) and $N$-(3-methylphenyl)methanesulfonamide (VIDKOJ: Gowda et al., 2007c).

In the crystal of CEGKAC, molecules are linked by $N\cdots H\cdots O$ hydrogen bonds, generating C(10) chains propagating in [001]. The packing is consolidated by $C\cdots H\cdots O$, $C\cdots H\cdots \pi$ and very weak aromatic $\pi\cdots \pi$ stacking interactions [centroid–centroid separation = 4.085 (2) Å]. In the crystal of CICPIO, the molecules are packed into a layer structure along the $a$-axis direction via $N\cdots H\cdots O$ hydrogen bonds [$H\cdots O = 2.08$ (2), $N\cdots O = 2.911$ (6) Å and $N\cdots H\cdots O = 164$ (6)°]. In the crystal of WIHGUQ, the amide H atom is available to a receptor molecule as it lies on one side of the plane of the benzene ring, while the methanesulfonyl group is on the opposite side of the plane, similar to the arrangement in other methanesulfonanilides. The molecules are packed into chains through $N\cdots H\cdots O$ and $N\cdots H\cdots Cl$ hydrogen bonding. In the crystal of VIDKOJ, the molecules are linked into chains along the $c$-axis direction through $N\cdots H\cdots O$ hydrogen bonds.
5. Synthesis and crystallization

A mixture of 1,2-oxazolylallylamine 2 (1 mmol), benzyl sulfonyl chloride (1.2 mmol) and Na₂CO₃ (1.2 mmol) in water (15 mL) was refluxed for 4 h. After cooling, the reaction mixture was extracted with CH₂Cl₂ (3 × 10 mL). The combined organic fractions were washed with water (2 × 10 mL) and dried over Na₂SO₄. The solvent was evaporated under reduced pressure. The resulting oil was purified by flash chromatography (eluent CH₂Cl₂) and crystallized from MeOH as colourless crystals, yield 0.16 g (41%), m.p. 371–373 K. IR (KBr, v (cm⁻¹)): 1642, 1618, 1599, 1568 (1,2-oxazole), 1343 (S=O), 1151, 1128 (SO₂), 698 (N—SO₂), 541 (Aryl). ¹H NMR (500 MHz, CDCl₃, 293 K): δ = 2.40 (s, 3H, H12A, H12B, H12C), 3.71–3.73 (d, 2H, H13A, H13B, J = 6.7, 4.21 (s, 2H, H2A, H2B), 4.33 (s, 2H, H1A, H1B), 5.22–5.29 (m, 2H, H15A, H15B), 5.63–5.71 (m, 1H, H14), 6.47 (s, 1H, H4), 7.25–7.27 (m, 2H, H8, H10), 7.36–7.41 (m, 5H, H17, H18, H19, H20, H21), 7.64–7.65 (d, 2H, H7, H11, J = 8.2). ¹³C NMR (126 MHz, CDCl₃, 293 K): δ = 21.66, 42.55, 50.58, 59.53, 98.99, 120.50, 124.64, 125.95 (2C), 129.01 (2C), 129.06, 129.85 (2C), 130.94 (2C), 132.24, 140.86, 160.95, 170.91. MS (APCI): m/z = 383 [M + H]⁺.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. The C-bound H atoms were positioned with idealized geometry and refined using a riding model with C—H = 0.95 Å (CH aromatic), 0.99 Å (CH₂) and 0.98 Å (CH₃). Isotropic displacement parameters for all H atoms were set equal to 1.2 or 1.5Ueq (parent atom). The crystal studied was refined as an inversion twin.

Acknowledgements

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Table 3

| Crystal data                                                                 | Chemical formula       | C₂₁H₂₂N₂O₃S             | M₅     |
|------------------------------------------------------------------------------|------------------------|--------------------------|--------|
| Crystal system, space group                                                  |                        | Monoclinic, Ia            |        |
| Temperature (K)                                                               |                        | 100                      |        |
| a, b, c (Å)                                                                  |                        | 17.7316 (2)               |        |
| β (°)                                                                        |                        | 100.526 (1)               |        |
| V (Å³)                                                                       |                        | 1924.55 (19)              |        |
| Radiation type                                                               |                        | Cu Kα                     |        |
| μ (mm⁻¹)                                                                     |                        | 1.69                      |        |
| Crystal size (mm)                                                            |                        | 0.24 × 0.22 × 0.14        |        |

| Data collection                                                              |                        | XtaLa Synergy, DualFlex, HyPix |        |
|------------------------------------------------------------------------------|------------------------|--------------------------|--------|
| Absorption correction                                                        |                        | Multi-scan (CrysAlis PRO) | Rigaku od, 2021 |
| Refinement                                                                   |                        |                          |        |
| R[F² > 2σ(F²)] , wR(F²), S                                                   |                        | 0.045, 0.125, 1.09       |        |
| No. of reflections                                                           |                        | 3572                     |        |
| No. of restraints                                                            |                        | ≥277                     |        |
| No. of parameters                                                             |                        | ≤2                        |        |
| H-atom treatment                                                             |                        | H-atom parameters constrained |     |
| Δρmax, Δρmin (e Å⁻³)                                                         |                        | 0.47, –0.58               |        |
| Absolute structure                                                           |                        | Refined as an inversion twin |     |
| Absolute structure parameter                                                 |                        | 0.00 (2)                  |        |

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT (Sheldrick, 2015a), SHELXL2018 (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

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Crystal structure and Hirshfeld surface analysis of N-[[5-(4-methylphenyl)-1,2-oxazol-3-yl]methyl]-1-phenyl-N-(prop-2-en-1-yl)methanesulfonamide

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Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2021); cell refinement: CrysAlis PRO (Rigaku OD, 2021); data reduction: CrysAlis PRO (Rigaku OD, 2021); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018 (Sheldrick, 2015b); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2020).

N-[[5-(4-Methylphenyl)-1,2-oxazol-3-yl]methyl]-1-phenyl-N-(prop-2-en-1-yl)methanesulfonamide

Crystal data

C21H22N2O3S
Mr = 382.46
Monoclinic, Ia
a = 10.7979 (1) Å
b = 10.2238 (10) Å
b = 10.2238 (10) Å
c = 17.7316 (2) Å
β = 100.526 (1)°
V = 1924.55 (19) Å³
Z = 4

F(000) = 808
Dx = 1.320 Mg m⁻³
Cu Kα radiation, λ = 1.54178 Å
Cell parameters from 18074 reflections
θ = 5.0–79.2°
μ = 1.69 mm⁻¹
T = 100 K
Prism, colourless
0.24 × 0.22 × 0.14 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan
(CrysAlisPro; Rigaku OD, 2021)
Tmin = 0.668, Tmax = 0.779
21251 measured reflections
3572 independent reflections
3542 reflections with I > 2σ(I)
Rint = 0.051
θmax = 79.6°, θmin = 5.0°
h = −13→13
k = −12→13
l = −22→22

Refinement

Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.045
wR(F²) = 0.125
S = 1.09
3572 reflections
247 parameters
2 restraints
Primary atom site location: SHELXT
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
w = 1/[σ²(Fo²) + (0.1004P)² + 0.3109P]
where P = (Fo² + 2Fc²)/3
(D/σ)max < 0.001
Δρmax = 0.47 e Å⁻³
Δρmin = −0.58 e Å⁻³
Extinction correction: SHELXL-2018/3
(Sheldrick, 2015b),
$F^c = kF^c [1 + 0.001xF^c^2 sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (4)
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.00 (2)

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.** Refined as a two-component inversion twin.

| Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\AA^2$) |
|---|---|---|---|
|  | $x$ | $y$ | $z$ | $U_{eq}$ |
| S1 | 0.20850 (6) | 0.59215 (6) | 0.37939 (4) | 0.0181 (2) |
| O1 | 0.1034 (2) | 0.6795 (2) | 0.36066 (12) | 0.0253 (5) |
| O2 | 0.1867 (2) | 0.4534 (2) | 0.37509 (14) | 0.0271 (5) |
| O3 | 0.1016 (2) | 0.7878 (2) | 0.64904 (11) | 0.0248 (5) |
| N1 | 0.2802 (2) | 0.6254 (2) | 0.46571 (13) | 0.0183 (5) |
| N2 | 0.2125 (3) | 0.7725 (3) | 0.61968 (15) | 0.0259 (6) |
| C1 | 0.3167 (3) | 0.6294 (3) | 0.31677 (16) | 0.0206 (6) |
| H1A | 0.390384 | 0.570343 | 0.328837 | 0.025* |
| H1B | 0.275212 | 0.612336 | 0.263131 | 0.025* |
| C2 | 0.2760 (3) | 0.7586 (3) | 0.49593 (15) | 0.0180 (5) |
| H2A | 0.258277 | 0.820906 | 0.452560 | 0.022* |
| H2B | 0.359354 | 0.780818 | 0.526667 | 0.022* |
| C3 | 0.1778 (3) | 0.7734 (3) | 0.54474 (16) | 0.0178 (5) |
| C4 | 0.0450 (3) | 0.7881 (3) | 0.52263 (15) | 0.0179 (5) |
| H4 | −0.003053 | 0.791270 | 0.472112 | 0.022* |
| C5 | 0.0023 (3) | 0.7966 (3) | 0.58978 (15) | 0.0180 (5) |
| C6 | −0.1207 (3) | 0.8145 (3) | 0.61079 (15) | 0.0173 (5) |
| C7 | −0.1342 (3) | 0.8064 (3) | 0.68787 (15) | 0.0199 (6) |
| H7 | −0.063522 | 0.785401 | 0.726289 | 0.024* |
| C8 | −0.2503 (3) | 0.8290 (3) | 0.70805 (15) | 0.0193 (5) |
| H8 | −0.258482 | 0.822084 | 0.760347 | 0.023* |
| C9 | −0.3554 (3) | 0.8615 (3) | 0.65319 (16) | 0.0189 (6) |
| C10 | −0.3414 (3) | 0.8690 (3) | 0.57646 (16) | 0.0214 (6) |
| H10 | −0.412186 | 0.890631 | 0.538263 | 0.026* |
| C11 | −0.2261 (3) | 0.8453 (3) | 0.55508 (16) | 0.0209 (6) |
| H11 | −0.218664 | 0.850017 | 0.502582 | 0.025* |
| C12 | −0.4808 (3) | 0.8853 (3) | 0.67664 (17) | 0.0235 (6) |
| H12A | −0.470660 | 0.949937 | 0.718118 | 0.035* |
| H12B | −0.512271 | 0.803142 | 0.694488 | 0.035* |
| H12C | −0.540911 | 0.918217 | 0.632572 | 0.035* |
| C13 | 0.3384 (3) | 0.5231 (3) | 0.51956 (18) | 0.0233 (6) |
| H13A | 0.325804 | 0.436904 | 0.493807 | 0.028* |
| H13B | 0.295250 | 0.521026 | 0.564246 | 0.028* |
| C14 | 0.4759 (3) | 0.5443 (3) | 0.5472 (2) | 0.0264 (6) |
| H14 | 0.528386 | 0.555444 | 0.510125 | 0.032* |
| Atomic displacement parameters (Å²) |
|-------------------------------------|
|                                | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| S1                                | 0.0203 (3) | 0.0186 (3) | 0.0162 (3) | −0.0028 (2) | 0.0055 (2) | −0.0023 (2) |
| O1                                | 0.0217 (11) | 0.0310 (11) | 0.0225 (10) | 0.0011 (8) | 0.0021 (8) | 0.0003 (8) |
| O2                                | 0.0370 (14) | 0.0212 (10) | 0.0251 (10) | −0.0095 (9) | 0.0107 (10) | −0.0038 (9) |
| O3                                | 0.0178 (10) | 0.0414 (12) | 0.0150 (10) | 0.0038 (8) | 0.0028 (8) | −0.0014 (8) |
| N1                                | 0.0245 (12) | 0.0164 (10) | 0.0141 (10) | 0.0019 (9) | 0.0032 (9) | −0.0022 (9) |
| N2                                | 0.0209 (12) | 0.0395 (15) | 0.0182 (12) | 0.0047 (11) | 0.0061 (10) | −0.0024 (10) |
| C1                                | 0.0271 (15) | 0.0194 (12) | 0.0176 (11) | −0.0009 (11) | 0.0099 (11) | −0.0021 (10) |
| C2                                | 0.0204 (13) | 0.0169 (11) | 0.0175 (12) | −0.0005 (10) | 0.0055 (10) | −0.0014 (9) |
| C3                                | 0.0192 (13) | 0.0180 (12) | 0.0165 (12) | 0.0013 (9) | 0.0042 (10) | −0.0010 (9) |
| C4                                | 0.0193 (13) | 0.0199 (11) | 0.0143 (11) | 0.0007 (9) | 0.0022 (10) | 0.0008 (9) |
| C5                                | 0.0201 (14) | 0.0178 (11) | 0.0160 (12) | 0.0011 (10) | 0.0031 (10) | −0.0007 (10) |
| C6                                | 0.0216 (14) | 0.0150 (11) | 0.0160 (12) | 0.0004 (9) | 0.0049 (10) | −0.0005 (9) |
| C7                                | 0.0241 (14) | 0.0197 (13) | 0.0162 (12) | 0.0014 (10) | 0.0044 (10) | 0.0012 (10) |
| C8                                | 0.0245 (14) | 0.0188 (11) | 0.0158 (12) | −0.0011 (10) | 0.0065 (10) | 0.0002 (10) |
| C9                                | 0.0213 (13) | 0.0146 (12) | 0.0220 (13) | 0.0002 (9) | 0.0073 (11) | −0.0013 (9) |
| C10                               | 0.0222 (14) | 0.0235 (14) | 0.0181 (13) | 0.0012 (11) | 0.0022 (10) | 0.0015 (10) |
| C11                               | 0.0206 (14) | 0.0259 (13) | 0.0164 (12) | 0.0007 (10) | 0.0042 (10) | 0.0000 (11) |
| C12                               | 0.0223 (15) | 0.0256 (13) | 0.0247 (14) | 0.0003 (11) | 0.0105 (12) | −0.0012 (12) |
| C13                               | 0.0274 (15) | 0.0190 (13) | 0.0229 (14) | 0.0014 (10) | 0.0030 (11) | 0.0044 (10) |
| C14                               | 0.0257 (16) | 0.0237 (14) | 0.0297 (15) | 0.0047 (11) | 0.0046 (13) | 0.0013 (12) |
| C15                               | 0.0347 (18) | 0.0248 (15) | 0.0369 (18) | 0.0049 (13) | −0.0035 (14) | −0.0022 (13) |
| C16                               | 0.0235 (14) | 0.0192 (13) | 0.0156 (12) | 0.0001 (10) | 0.0079 (10) | −0.0015 (9) |
| C17                               | 0.0242 (14) | 0.0244 (14) | 0.0175 (11) | −0.0017 (12) | 0.0036 (11) | 0.0006 (11) |
| C18                               | 0.0360 (18) | 0.0213 (13) | 0.0212 (14) | 0.0011 (12) | 0.0061 (12) | 0.0029 (11) |
| C19                               | 0.0388 (19) | 0.0216 (13) | 0.0199 (13) | −0.0089 (12) | 0.0076 (13) | 0.0001 (10) |
| C20                               | 0.0276 (16) | 0.0323 (16) | 0.0218 (14) | −0.0082 (13) | 0.0055 (12) | 0.0018 (12) |
| C21                               | 0.0220 (14) | 0.0255 (13) | 0.0204 (13) | 0.0013 (11) | 0.0068 (11) | 0.0033 (11) |
### Geometric parameters (Å, °)

| Bond/Angle | Distance/Angle |
|------------|----------------|
| S1—O1      | 1.434 (2)      |
| S1—O2      | 1.438 (2)      |
| S1—N1      | 1.620 (2)      |
| S1—C1      | 1.794 (3)      |
| O3—C5      | 1.360 (3)      |
| O3—N2      | 1.399 (3)      |
| N1—C2      | 1.468 (3)      |
| N1—C13     | 1.477 (4)      |
| N2—C3      | 1.313 (4)      |
| C1—C16     | 1.506 (4)      |
| C1—H1A     | 0.9900         |
| C2—C3      | 1.494 (4)      |
| C2—H2A     | 0.9900         |
| C3—C4      | 1.424 (4)      |
| C4—C5      | 1.355 (4)      |
| C5—C6      | 1.455 (4)      |
| C6—C7      | 1.403 (3)      |
| C7—C8      | 1.385 (4)      |
| C7—H7      | 0.9500         |
| C8—C9      | 1.393 (4)      |
| C9—C10     | 1.398 (4)      |
| O1—S1—O2   | 119.14 (15)    |
| O1—S1—N1   | 108.03 (13)    |
| O2—S1—N1   | 107.60 (13)    |
| O1—S1—C1   | 107.57 (14)    |
| O2—S1—C1   | 107.18 (14)    |
| N1—S1—C1   | 106.70 (14)    |
| C5—O3—N2   | 109.1 (2)      |
| C2—N1—C13  | 117.3 (2)      |
| C2—N1—S1   | 119.87 (19)    |
| C13—N1—S1  | 122.40 (19)    |
| C3—N2—O3   | 105.7 (2)      |
| C16—C1—S1  | 112.94 (19)    |
| C16—C1—H1A | 109.0          |
| S1—C1—H1A  | 109.0          |
| S1—C1—H1B  | 109.0          |
| H1A—C1—H1B | 107.8          |
| N1—C2—C3   | 112.2 (2)      |
| N1—C2—H2A  | 109.2          |

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C3—C2—H2A 109.2  C15—C14—C13 123.4 (3)
N1—C2—H2B 109.2  C15—C14—H14 118.3
C3—C2—H2B 109.2  C13—C14—H14 118.3
H2A—C2—H2B 107.9  C14—C15—H15A 120.0
N2—C3—C4 111.5 (3)  C14—C15—H15B 120.0
N2—C3—C2 118.9 (3)  H15A—C15—H15B 120.0
C4—C3—C2 129.6 (3)  C21—C16—C17 119.3 (3)
C5—C4—C3 104.6 (2)  C21—C16—C1 120.5 (3)
C5—C4—H4 127.7  C17—C16—C1 120.2 (3)
C3—C4—H4 127.7  C17—C16—C1 120.1 (3)
C4—C5—O3 109.2 (3)  C18—C17—C16 120.0
C4—C5—C6 134.8 (3)  C18—C17—H17 120.0
O3—C5—C6 116.0 (2)  C16—C17—H17 120.0
C11—C6—C7 119.0 (3)  C14—C15—H15A 122.2 (3)
C11—C6—C5 120.7 (2)  C14—C15—H15B 122.2 (3)
C7—C6—C5 120.3 (3)  H15A—C15—H15B 122.2 (3)
C8—C7—C6 120.1 (3)  C14—C15—H15A 122.2 (3)
C8—C7—H7 119.9  C14—C15—H15B 122.2 (3)
C6—C7—H7 119.9  C14—C15—H15A 122.2 (3)
C7—C8—C9 121.3 (2)  C14—C15—H15B 122.2 (3)
C7—C8—H8 119.3  C14—C15—H15A 122.2 (3)
C9—C8—H8 119.3  C14—C15—H15B 122.2 (3)
C8—C9—C10 118.3 (3)  C14—C15—H15A 122.2 (3)
C8—C9—C12 120.3 (3)  C14—C15—H15B 122.2 (3)

O1—S1—N1—C2  28.6 (3)  O3—C5—C6—C7  7.5 (4)
O2—S1—N1—C2  158.5 (2)  C11—C6—C7—C8  0.2 (4)
C1—S1—N1—C2  86.8 (2)  C5—C6—C7—C8  177.3 (3)
O1—S1—N1—C13 144.0 (2)  C6—C7—C8—C9  0.8 (4)
O2—S1—N1—C13 14.2 (3)  C7—C8—C9—C10  1.0 (4)
C1—S1—N1—C13 −100.6 (3)  C7—C8—C9—C12 179.9 (3)
C5—O3—N2—C3  0.4 (3)  C8—C9—C10—C11  0.3 (4)
O1—S1—C1—C16  58.5 (2)  C12—C9—C10—C11 −179.1 (3)
O2—S1—C1—C16 −172.3 (2)  C9—C10—C11—C6  0.6 (4)
N1—S1—C1—C16 −57.3 (2)  C7—C6—C11—C10  0.8 (4)
C13—N1—C2—C3  75.0 (3)  C5—C6—C11—C10 −176.6 (3)
S1—N1—C2—C3  98.1 (3)  C2—N1—C13—C14  65.6 (3)
O3—N2—C3—C4 −0.4 (3)  S1—N1—C13—C14  121.6 (3)
O3—N2—C3—C2 −179.9 (2)  N1—C13—C14—C15  126.2 (3)
N1—C2—C3—N2 101.3 (3)  S1—C1—C16—C21  105.6 (3)
N1—C2—C3—C4 −78.2 (4)  S1—C1—C16—C17 −74.6 (3)
N2—C3—C4—C5  0.2 (3)  C21—C16—C17—C18  0.1 (4)
C2—C3—C4—C5  179.7 (3)  C1—C16—C17—C18 −179.7 (3)
C3—C4—C5—O3  0.0 (3)  C16—C17—C18—C19  0.5 (5)
C3—C4—C5—C6  178.8 (3)  C17—C18—C19—C20 −0.6 (5)
N2—O3—C5—C4 −0.2 (3)  C18—C19—C20—C21  0.1 (5)
N2—O3—C5—C6 −179.3 (2)  C17—C16—C21—C20  0.6 (4)
C4—C5—C6—C11 −8.8 (5)  C1—C16—C21—C20  179.2 (3)
Hydrogen-bond geometry (Å, °)

| D—H···A  | D—H | H···A | D···A    | D—H···A |
|----------|------|-------|----------|---------|
| C7—H7···O3 | 0.95 | 2.44  | 2.763 (4) | 100     |
| C8—H8···O2i | 0.95 | 2.59  | 3.404 (4) | 143     |
| C13—H13A···O2 | 0.99 | 2.36  | 2.867 (4) | 111     |
| C17—H17···O3ii | 0.95 | 2.57  | 3.314 (4) | 135     |
| C19—H19···O1iii | 0.95 | 2.51  | 3.434 (4) | 165     |
| C21—H21···O2iv | 0.95 | 2.50  | 3.369 (4) | 152     |

Symmetry codes: (i) x−1/2, y+1/2, z+1/2; (ii) x, −y+3/2, z−1/2; (iii) x+1/2, −y+2, z; (iv) x+1/2, −y+1, z.