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Crystal structure and Hirshfeld surface analysis of *N*-[2-(5-methylfuran-2-yl)phenyl]-3-nitro-*N*-[(3-nitrophenyl)sulfonyl]benzenesulfonamide

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In the title compound, $C_{23}H_{17}N_3O_9S_2$, C—H···O hydrogen bonds link adjacent molecules in a three-dimensional network, while π–π stacking interactions, with centroid–centroid distances of 3.8745 (9) Å, between the furan and an arene ring of one of the two (3-nitrophenyl)sulfonyl groups, result in chains parallel to the *a* axis. The Hirshfeld surface analysis indicates that O···H/H···O (40.1%), H···H (27.5%) and C···H/H···C (12.4%) interactions are the most significant contributors to the crystal packing.

1. Chemical context

The synthesis of sulfonamides has been given considerable attention in the literature. A large number of reports are based on various chemical and physical properties, methods of synthesis and application of sulfonamides (Safavora *et al.*, 2019). The electronic and structural properties of the sulfonamide moiety make it a bioisostere of such compounds as urea, thiourea, carbamates and sulfamides (Reitz *et al.*, 2009; Abdelhamid *et al.*, 2011; Khalilov *et al.*, 2021). Linear and cyclic compounds containing sulfonamide fragments have a wide range of biological activity – they possess antibacterial properties (Yun *et al.*, 2012; Nadirova *et al.*, 2021), show diuretic activity (Logemann *et al.*, 1959; DeStevens *et al.*, 1959), are active against seizures (Thiry *et al.*, 2008) and inhibit various enzymes like human leukocyte elastase and cathepsin G, a HIV-1 protease (Supuran *et al.*, 2003). Sulfonamides are also used as fungicidal (Chohan *et al.*, 2006, 2010) and insecticidal mixtures. The most widely used furan-substituted sulfonylamide is Furosemide, a loop diuretic medication used to treat fluid build-up due to heart failure, kidney disease or liver scarring. Typically, furan-substituted monosulfamides are obtained by treatment of the amines with the corresponding sulfonyl chlorides (Pilipenko *et al.*, 2012; Butin *et al.*, 2006; Naghiyev *et al.*, 2020). It turned out unexpectedly that the interaction of 2-(α-furyl)aniline with sulfochloride containing the electron-withdrawing 3-nitrophenyl group under the same conditions gives a double sulfarylation product (Fig. 1), which is possible only with the use of strong bases (Bartsch *et al.*, 1977; Li *et al.*, 2022). The obtained product can serve as a compound for studying furan fragment-opening (Pilipenko *et al.*, 2012; Butin *et al.*, 2006) or the Diels–Alder reactions of

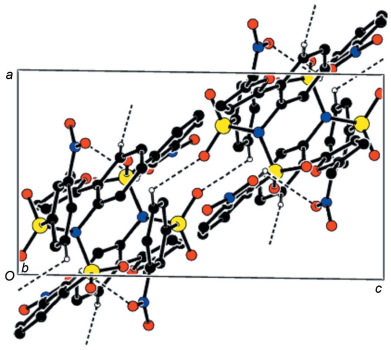
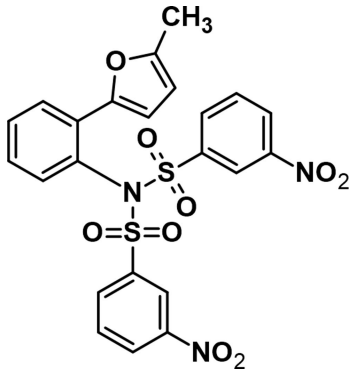


Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O4^{i}	0.95	2.31	3.226 (2)	161
C16—H16···O2^{ii}	0.95	2.53	3.0874 (18)	118
C19—H19···O4^{iii}	0.95	2.58	2.981 (2)	106

Symmetry codes: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

furans (Borisova *et al.*, 2018*a*,*b*; Krishna *et al.*, 2022; Zubkov *et al.*, 2007) and for studying biological activity. On the other hand, intermolecular noncovalent interactions organize the molecular aggregates, catalytic intermediates, *etc*., which play a critical role in the functional properties of heterocyclic compounds (Gurbanov *et al.*, 2020*a*,*b*, 2022; Ma *et al.*, 2021; Mahmoudi, *et al.*, 2017*a*,*b*; Mahmudov *et al.*, 2011, 2022).



2. Structural commentary

In the title compound (Fig. 2), the angle between the planes of the arene rings (C12–C17 and C18–C23) of the (3-nitrophenyl)sulfonyl groups are 40.87 (7)°. The furan ring (O1/C7–C10) is inclined at angles of 51.04 (8) and 12.78 (8)° with respect to the arene rings (C12–C17 and C18–C23) of the (3-nitrophenyl)sulfonyl groups, while it makes a dihedral angle of 20.77 (8)° with the plane of the arene ring (C1–C6) attached to the furan ring. The arene ring attached to the furan ring makes dihedral angles of 33.19 (7) and 17.84 (7)°, respectively, with the arene rings of the 3-nitrophenyl)sulfonyl groups. The geometric properties of the title compound are normal and consistent with those of related compounds listed in the *Database survey* (Section 4).

Table 2
Summary of short interatomic contacts (Å)..

Contact	Distance	Symmetry operation
N2···O3	3.03	$-x+2, -y+1, -z+1$
H19···H15	2.58	$-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$
H16···O2	2.53	$-x+1, -y+1, -z+1$
O5···H17	2.62	$x+1, y, z$
O4···H3	2.31	$-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$
H4···H8	2.46	$x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$
H9···O8	2.67	$-x+1, -y+2, -z+1$

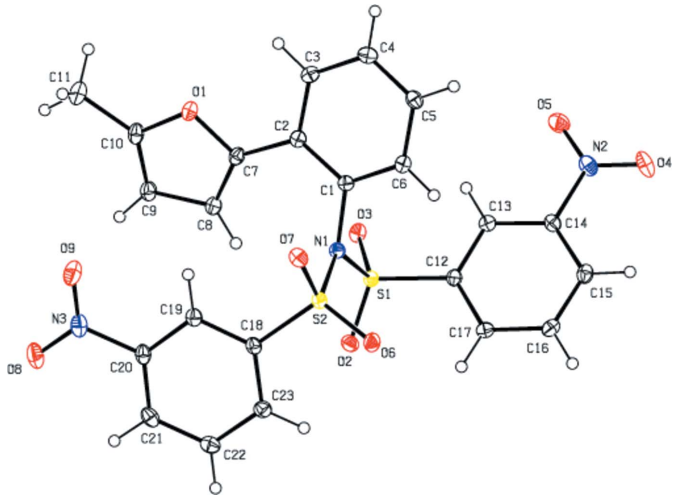


Figure 2
The molecular structure of the title compound, showing the atom labelling and with displacement ellipsoids drawn at the 50% probability level.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal of the title compound, molecules are linked by intermolecular C—H···O hydrogen bonds forming the three-dimensional network (Tables 1 and 2), while π–π stacking interactions {$Cg1\cdots Cg4^{iv}$ = 3.8745 (9) Å [symmetry code: (iv)

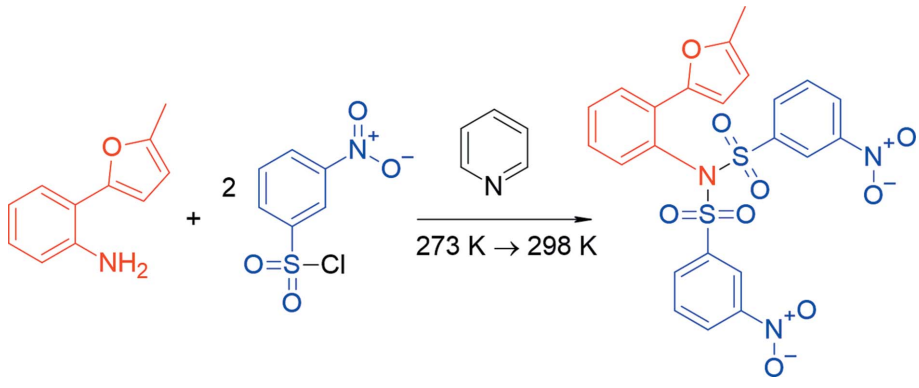


Figure 1
One-pot synthesis of *N*-[2-(5-methylfuran-2-yl)phenyl]-3-nitro-*N*-[(3-nitrophenyl)sulfonyl]benzenesulfonamide.

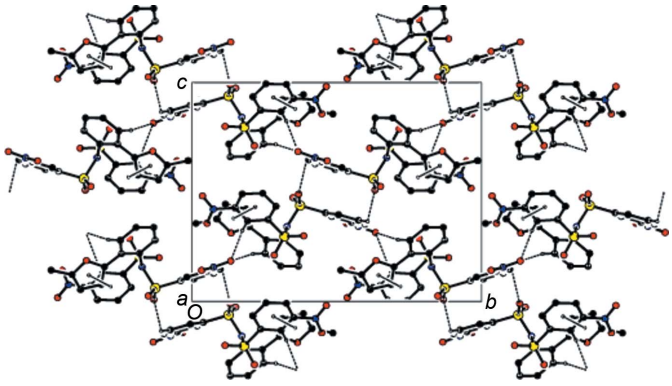


Figure 3
The crystal packing along the *a* axis, showing the C—H···O hydrogen-bond network and the π–π stacking interactions.

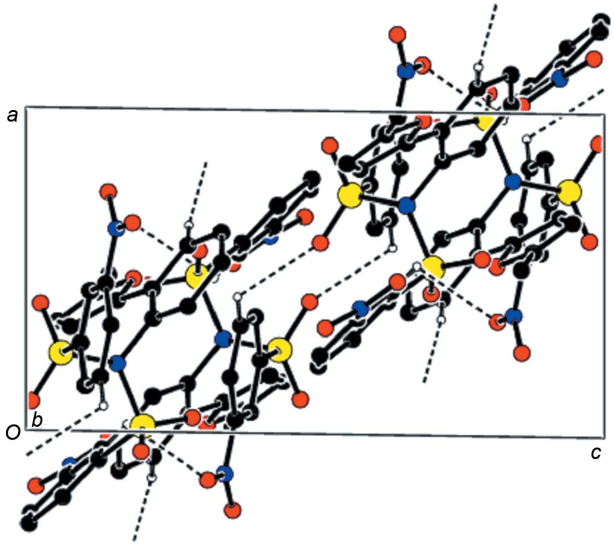


Figure 4
The crystal packing diagram along the *b* axis, showing the intermolecular C—H···O hydrogen bonds.

$x + 1, y, z$], where *Cg*1 and *Cg*4 are the centroids of the furan ring (atoms O1/C7–C10) and the arene ring (atoms C18–C23) of one of the two (3-nitrophenyl)sulfonyl groups; slippage = 1.389 Å} form chains along the *a* axis (Figs. 3 and 4).

Hirshfeld surfaces were generated for the title molecule using *CrystalExplorer17* (Spackman *et al.*, 2021). The d_{norm} mappings was performed in the range from −0.3170 to +1.1777 a.u. The C—H···O interactions are indicated by red areas on the Hirshfeld surfaces [Figs. 5(*a*) and 5(*b*)]. Fingerprint plots (Fig. 6) reveal that, while O···H/H···O interactions (40.1%) make the largest contributions to the surface contacts (Tables 1 and 2), H···H (27.5%) and C···H/H···C (12.4%) contacts are also important. Other less notable linkages are O···C/C···O (6.0%), O···O (5.7%), C···C (4.9%), O···N/N···O (2.0%), N···H/H···N (1.2%), S···C/C···S (0.1%) and S···O/O···S (0.1%).

4. Database survey

The nine related compounds found as a result of the search for '*N*-(methanesulfonyl)-*N*-methyl methanesulfonamide' in the Cambridge Structural Database (CSD, Version 5.42, update of September 2021; Groom *et al.*, 2016) are for *N*-(2-formylphenyl)-4-methyl-*N*-[(4-methylphenyl)sulfonyl]benzenesulfonamide, *i.e.* CSD refcodes JOBTIF (Kim, 2014), CEGMIM (Mughal *et al.*, 2012*a*), YAXKAL (Taher & Smith, 2012*a*), OCABUR (Abbassi *et al.*, 2011), CEGSUE (Mughal *et al.*, 2012*b*), EFASUB (Taher & Smith, 2012*b*), PONZIC (Rizzoli *et al.*, 2009), AYUPUG (Arshad *et al.*, 2011) and ROGJON (Li & Song, 2008).

In JOBTIF (space group $P2_1/n$), molecules are linked by pairs of C—H···O hydrogen bonds, forming inversion dimers. In CEGMIM (space group *Pbca*), molecules are connected by C—H···O interactions into sheets in the *ab* plane. In YAXKAL (space group $P\overline{1}$), molecules associate *via* pairs of N—H···N hydrogen bonds, forming a centrosymmetric eight-

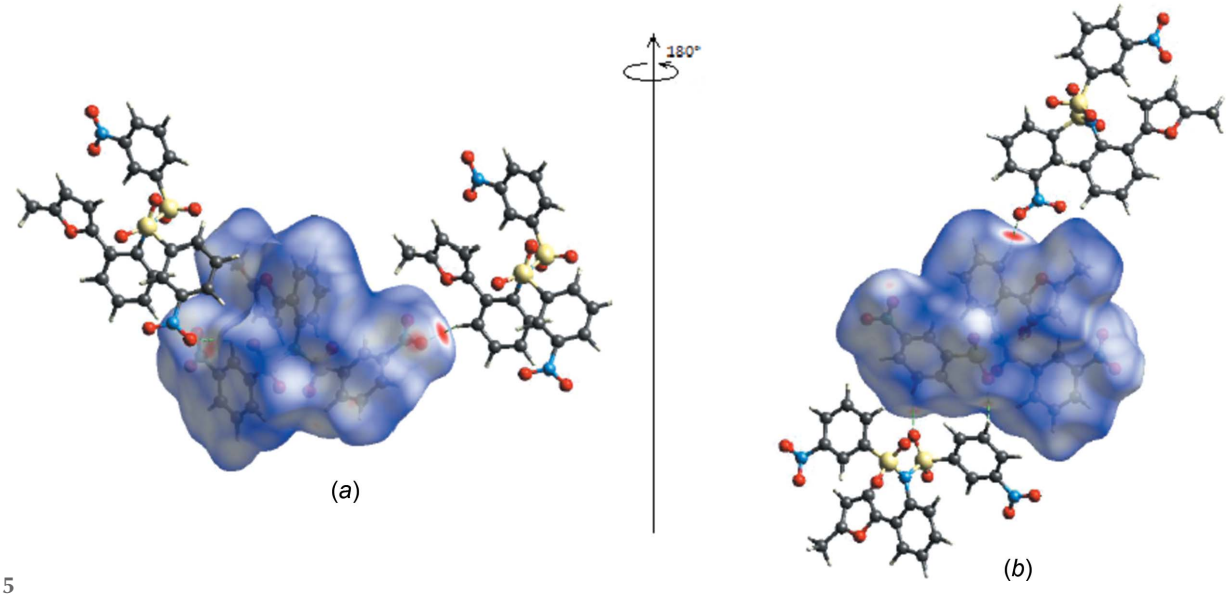


Figure 5
(*a*) Front and (*b*) back views of the three-dimensional Hirshfeld surface, with some intermolecular C—H···O interactions shown.

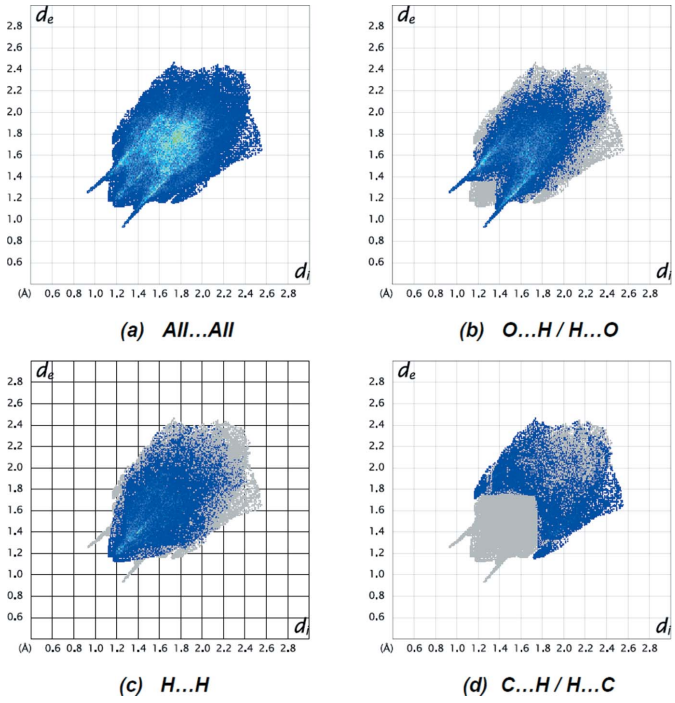


Figure 6
The 2D fingerprint plots for the title molecule, showing (*a*) all interactions, and delineated into (*b*) O···H/H···O, (*c*) H···H and (*d*) C···H/H···C interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

membered {···HNCN}$_2$ synthon. The crystal structure of OCABUR (space group $P2_1/c$) is stabilized by intermolecular C—H···O hydrogen bonds. In the crystal of CEGSUE (space group $P\overline{1}$), the only possible directional interactions are very weak C—H···π interactions and very weak π–π stacking between parallel methylphenyl rings. In EFASUB (space group $C2/c$), molecules associate *via* N—H···N and N—H···O hydrogen bonds, forming extended hydrogen-bonded sheets that lie parallel to the *bc* plane. The N—H···N hydrogen bonds propagate along the *b*-axis direction, while the N— H···O hydrogen bonds propagate along the *c*-axis direction. In the crystal packing of PONZIC (space group $P\overline{1}$), molecules are linked into chains parallel to the *a* axis by intermolecular C—H···O hydrogen bonds and π–π stacking interactions. In the crystal structure of AYUPUG (space group $P2_1/c$), weak C—H···O interactions connect the molecules in a zigzag manner along the *a* axis. In ROGJON (space group *Pbca*), the crystal sructure exhibits weak intermolecular N—H···O, C—H···O and C—H···N hydrogen bonds and π–π interactions.

5. Synthesis and crystallization

To a solution of 2-(5-methylfuran-2-yl)aniline (1.09 g, 0.0058 mol) in 7 ml of pyridine under stirring and cooling in an ice-water bath, *m*-nitrobenzenesulfonyl chloride (2.59 g, 0.0117 mol) was added gradually. The mixture was stirred for 7 h and after completion of the reaction [thin-layer chromatography (TLC) monitoring], the mixture was poured into 90 ml of 6 *M* hydrochloric acid. The oil which separated was washed with water until it crystallized. The crystals were filtered off, dried and crystallized from an ethanol/dimethylformamide (DMF) mixture to give the target disulfonamide as a yellow solid. A single crystal of *N*-[2-(5-methylfuran-2-yl)-phenyl]-3-nitro-*N*-[(3-nitrophenyl)sulfonyl]benzenesulfonamide was obtained by slow crystallization from an ethanol/DMF mixture (yield 62%, 1.94 g; m.p. 467–469 K). IR (KBr), ν (cm^{-1}): 1176 (ν_s SO$_2$), 1352 (*br*, ν_{as} SO$_2$, ν_s NO$_2$), 1530 (ν_{as} NO$_2$). ^{1}H NMR (600.2 MHz, DMSO-d_6) (J, Hz): δ 8.60 (*dd*, J = 8.1, 1.5 Hz, 2H), 8.36 (*t*, J = 1.5 Hz, 2H), 8.25 (*d*, J = 8.1 Hz, 2H), 7.94 (*t*, J = 8.1 Hz, 2H), 7.75 (*dd*, J = 8.1, 1.5 Hz, 1H), 7.59 (*dt*, J = 8.1, 1.0 Hz, 1H), 7.38 (*dt*, J = 8.1, 1.0 Hz, 1H), 7.12 (*d*, J = 8.1 Hz, 1H), 6.60 (*d*, J = 3.5 Hz, 1H), 5.81 (*d*, J = 3.5 Hz, 1H), 1.86 (*s*, 3H). ^{13}C{^{1}H} NMR (150.9 MHz, DMSO-d_6): δ 153.2 (2C), 148.2, 147.9, 140.4, 134.9, 133.8, 132.2, 132.1, 132.0, 129.7, 129.0, 128.8, 123.4, 112.0, 108.5, 13.3; MS (ESI) *m*/*z*: [M + H]$^+$ 544.37. Analysis calculated (%) for C$_{23}$H$_{17}$N$_3$O$_9$S$_2$: C 50.82, H 3.15, N 7.73, S 11.80; found: C 51.07, H 3.17, N 7.56, S 12.03.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{17}N_3O_9S_2$
M_r	543.52
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	8.10683 (6), 19.20010 (15), 14.49754 (10)
β (°)	90.8104 (7)
V (Å^3)	2256.35 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.71
Crystal size (mm)	0.33 × 0.12 × 0.11
Data collection	
Diffractometer	Rigaku XtaLAB Synergy Dualflex HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.411, 0.725
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30531, 4812, 4547
R_{int}	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.100, 1.07
No. of reflections	4812
No. of parameters	335
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.54, −0.50

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXL2016* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All C-bound H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and included as

riding contributions with isotropic displacement parameters fixed at $1.2U_{eq}$(C) (1.5 for the methyl groups).

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supporting information

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Crystal structure and Hirshfeld surface analysis of *N*-[2-(5-methylfuran-2-yl)phenyl]-3-nitro-*N*-[(3-nitrophenyl)sulfonyl]benzenesulfonamide

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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: *SHELXL2016* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015*b*); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

N-[2-(5-Methylfuran-2-yl)phenyl]-3-nitro-*N*-[(3-nitrophenyl)sulfonyl]benzenesulfonamide

Crystal data

$C_{23}H_{17}N_3O_9S_2$
M_r = 543.52
Monoclinic, $P2_1/n$
a = 8.10683 (6) Å
b = 19.20010 (15) Å
c = 14.49754 (10) Å
β = 90.8104 (7)°
V = 2256.35 (3) Å^3
Z = 4
$F(000)$ = 1120
D_x = 1.600 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 20996 reflections
θ = 3.8–77.7°
μ = 2.71 mm^{-1}
T = 100 K
Prismatic needle, yellow
0.33 × 0.12 × 0.11 mm

Data collection

Rigaku XtaLAB Synergy Dualflex HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)
T_{min} = 0.411, T_{max} = 0.725
30531 measured reflections
4812 independent reflections
4547 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 77.8°, θ_{min} = 3.8°
h = −8→10
k = −24→24
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.07
4812 reflections
335 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.9267P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.74119 (4)	0.62643 (2)	0.56167 (2)	0.01729 (11)
S2	0.51739 (4)	0.68042 (2)	0.70267 (2)	0.01806 (11)
O1	0.97431 (14)	0.86445 (5)	0.68665 (7)	0.0223 (2)
O2	0.59124 (13)	0.63170 (6)	0.50939 (7)	0.0225 (2)
O3	0.89353 (13)	0.65098 (5)	0.52573 (7)	0.0220 (2)
O4	1.14629 (16)	0.36623 (7)	0.68499 (9)	0.0341 (3)
O5	1.23988 (15)	0.46683 (7)	0.64260 (9)	0.0340 (3)
O6	0.43825 (13)	0.61417 (6)	0.70008 (8)	0.0241 (2)
O7	0.54611 (13)	0.71571 (6)	0.78814 (7)	0.0239 (2)
O8	0.32788 (17)	0.96375 (7)	0.51794 (10)	0.0377 (3)
O9	0.47463 (18)	0.94819 (6)	0.64251 (9)	0.0357 (3)
N1	0.71051 (15)	0.67238 (6)	0.65814 (8)	0.0179 (2)
N2	1.12743 (17)	0.42581 (7)	0.65689 (9)	0.0247 (3)
N3	0.38951 (18)	0.92648 (7)	0.57790 (10)	0.0278 (3)
C1	0.85072 (17)	0.68131 (7)	0.72031 (10)	0.0175 (3)
C2	0.94303 (17)	0.74318 (7)	0.71871 (10)	0.0186 (3)
C3	1.07789 (18)	0.74767 (8)	0.78022 (10)	0.0220 (3)
H3	1.1415	0.7892	0.7821	0.026*
C4	1.12052 (19)	0.69311 (9)	0.83828 (11)	0.0239 (3)
H4	1.2142	0.6972	0.8780	0.029*
C5	1.02746 (19)	0.63238 (8)	0.83891 (10)	0.0224 (3)
H5	1.0569	0.5949	0.8787	0.027*
C6	0.89046 (18)	0.62723 (7)	0.78039 (10)	0.0200 (3)
H6	0.8238	0.5866	0.7815	0.024*
C7	0.90565 (18)	0.80227 (8)	0.65835 (10)	0.0199 (3)
C8	0.82198 (19)	0.81274 (8)	0.57740 (11)	0.0224 (3)
H8	0.7638	0.7786	0.5424	0.027*
C9	0.8383 (2)	0.88476 (8)	0.55513 (11)	0.0242 (3)
H9	0.7929	0.9077	0.5025	0.029*
C10	0.9303 (2)	0.91413 (8)	0.62300 (11)	0.0238 (3)
C11	0.9849 (2)	0.98641 (8)	0.64350 (12)	0.0303 (4)
H11A	0.9245	1.0043	0.6966	0.045*
H11B	0.9628	1.0161	0.5897	0.045*
H11C	1.1034	0.9866	0.6577	0.045*
C12	0.77285 (17)	0.53925 (7)	0.59476 (10)	0.0183 (3)
C13	0.93448 (18)	0.51767 (8)	0.61163 (10)	0.0193 (3)

H13	1.0250	0.5487	0.6053	0.023*
C14	0.95731 (18)	0.44913 (8)	0.63798 (10)	0.0201 (3)
C15	0.8286 (2)	0.40242 (8)	0.64729 (10)	0.0227 (3)
H15	0.8491	0.3557	0.6658	0.027*
C16	0.66862 (19)	0.42513 (8)	0.62896 (10)	0.0228 (3)
H16	0.5788	0.3937	0.6342	0.027*
C17	0.63988 (18)	0.49387 (8)	0.60302 (10)	0.0203 (3)
H17	0.5306	0.5097	0.5911	0.024*
C18	0.41201 (17)	0.73702 (7)	0.62594 (10)	0.0190 (3)
C19	0.44463 (18)	0.80786 (8)	0.63208 (10)	0.0203 (3)
H19	0.5225	0.8258	0.6755	0.024*
C20	0.35845 (18)	0.85112 (8)	0.57207 (11)	0.0219 (3)
C21	0.24370 (19)	0.82640 (9)	0.50838 (11)	0.0248 (3)
H21	0.1887	0.8575	0.4672	0.030*
C22	0.21043 (19)	0.75567 (9)	0.50568 (11)	0.0254 (3)
H22	0.1298	0.7381	0.4635	0.030*
C23	0.29462 (18)	0.71020 (8)	0.56445 (11)	0.0221 (3)
H23	0.2724	0.6616	0.5627	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01830 (18)	0.01684 (18)	0.01676 (18)	0.00090 (11)	0.00114 (13)	−0.00100 (11)
S2	0.01788 (18)	0.01795 (18)	0.01841 (18)	0.00142 (12)	0.00264 (12)	0.00124 (12)
O1	0.0280 (5)	0.0168 (5)	0.0222 (5)	−0.0019 (4)	0.0030 (4)	0.0000 (4)
O2	0.0224 (5)	0.0235 (5)	0.0213 (5)	0.0036 (4)	−0.0038 (4)	−0.0022 (4)
O3	0.0243 (5)	0.0196 (5)	0.0222 (5)	−0.0006 (4)	0.0055 (4)	0.0000 (4)
O4	0.0361 (7)	0.0335 (6)	0.0329 (6)	0.0170 (5)	0.0079 (5)	0.0117 (5)
O5	0.0209 (6)	0.0362 (7)	0.0449 (7)	0.0040 (5)	0.0013 (5)	0.0015 (5)
O6	0.0231 (5)	0.0197 (5)	0.0296 (6)	−0.0012 (4)	0.0038 (4)	0.0043 (4)
O7	0.0242 (5)	0.0280 (6)	0.0195 (5)	0.0049 (4)	0.0022 (4)	−0.0011 (4)
O8	0.0431 (7)	0.0264 (6)	0.0436 (7)	0.0081 (5)	0.0027 (6)	0.0128 (5)
O9	0.0519 (8)	0.0216 (6)	0.0337 (7)	−0.0010 (5)	0.0018 (6)	−0.0020 (5)
N1	0.0170 (6)	0.0182 (6)	0.0184 (6)	0.0010 (4)	0.0010 (4)	−0.0020 (4)
N2	0.0251 (7)	0.0284 (7)	0.0207 (6)	0.0082 (5)	0.0039 (5)	−0.0002 (5)
N3	0.0324 (7)	0.0216 (7)	0.0297 (7)	0.0050 (5)	0.0085 (6)	0.0032 (5)
C1	0.0161 (6)	0.0195 (7)	0.0169 (6)	0.0005 (5)	0.0016 (5)	−0.0019 (5)
C2	0.0188 (6)	0.0190 (7)	0.0181 (6)	0.0002 (5)	0.0042 (5)	−0.0008 (5)
C3	0.0206 (7)	0.0234 (7)	0.0220 (7)	−0.0033 (6)	0.0016 (5)	−0.0023 (6)
C4	0.0205 (7)	0.0304 (8)	0.0207 (7)	−0.0003 (6)	−0.0009 (5)	−0.0005 (6)
C5	0.0243 (7)	0.0245 (7)	0.0184 (7)	0.0035 (6)	−0.0002 (6)	0.0022 (5)
C6	0.0217 (7)	0.0189 (7)	0.0194 (7)	−0.0006 (5)	0.0036 (5)	−0.0003 (5)
C7	0.0205 (7)	0.0172 (6)	0.0221 (7)	−0.0010 (5)	0.0049 (5)	−0.0022 (5)
C8	0.0243 (7)	0.0195 (7)	0.0235 (7)	−0.0012 (5)	0.0011 (6)	0.0016 (5)
C9	0.0265 (7)	0.0206 (7)	0.0257 (8)	0.0007 (6)	0.0033 (6)	0.0041 (6)
C10	0.0276 (7)	0.0185 (7)	0.0257 (7)	0.0014 (6)	0.0083 (6)	0.0022 (6)
C11	0.0398 (9)	0.0199 (7)	0.0315 (8)	−0.0020 (6)	0.0061 (7)	−0.0004 (6)
C12	0.0197 (7)	0.0173 (6)	0.0179 (6)	0.0013 (5)	0.0017 (5)	−0.0019 (5)

C13	0.0186 (7)	0.0210 (7)	0.0182 (6)	−0.0007 (5)	0.0023 (5)	−0.0019 (5)
C14	0.0209 (7)	0.0221 (7)	0.0172 (6)	0.0044 (5)	0.0014 (5)	−0.0021 (5)
C15	0.0312 (8)	0.0187 (7)	0.0181 (7)	0.0004 (6)	0.0009 (6)	−0.0011 (5)
C16	0.0266 (7)	0.0218 (7)	0.0199 (7)	−0.0060 (6)	0.0001 (6)	−0.0011 (5)
C17	0.0193 (7)	0.0225 (7)	0.0190 (7)	−0.0018 (5)	0.0001 (5)	−0.0020 (5)
C18	0.0167 (6)	0.0197 (7)	0.0207 (7)	0.0030 (5)	0.0040 (5)	0.0005 (5)
C19	0.0205 (7)	0.0202 (7)	0.0202 (7)	0.0021 (5)	0.0042 (5)	−0.0009 (5)
C20	0.0229 (7)	0.0187 (7)	0.0243 (7)	0.0039 (5)	0.0072 (6)	0.0019 (6)
C21	0.0217 (7)	0.0293 (8)	0.0235 (7)	0.0071 (6)	0.0040 (6)	0.0048 (6)
C22	0.0199 (7)	0.0312 (8)	0.0250 (7)	0.0024 (6)	−0.0006 (6)	−0.0011 (6)
C23	0.0182 (7)	0.0237 (7)	0.0245 (7)	0.0005 (5)	0.0021 (5)	−0.0014 (6)

Geometric parameters (Å, º)

S1—O2	1.4269 (11)	C8—C9	1.427 (2)
S1—O3	1.4274 (11)	C8—H8	0.9500
S1—N1	1.6752 (12)	C9—C10	1.350 (2)
S1—C12	1.7591 (15)	C9—H9	0.9500
S2—O6	1.4249 (11)	C10—C11	1.485 (2)
S2—O7	1.4283 (11)	C11—H11A	0.9800
S2—N1	1.7089 (12)	C11—H11B	0.9800
S2—C18	1.7667 (15)	C11—H11C	0.9800
O1—C10	1.3709 (19)	C12—C17	1.393 (2)
O1—C7	1.3773 (18)	C12—C13	1.393 (2)
O4—N2	1.2231 (18)	C13—C14	1.382 (2)
O5—N2	1.2247 (19)	C13—H13	0.9500
O8—N3	1.227 (2)	C14—C15	1.384 (2)
O9—N3	1.228 (2)	C15—C16	1.390 (2)
N1—C1	1.4507 (18)	C15—H15	0.9500
N2—C14	1.4720 (19)	C16—C17	1.391 (2)
N3—C20	1.471 (2)	C16—H16	0.9500
C1—C6	1.390 (2)	C17—H17	0.9500
C1—C2	1.404 (2)	C18—C19	1.388 (2)
C2—C3	1.404 (2)	C18—C23	1.393 (2)
C2—C7	1.462 (2)	C19—C20	1.385 (2)
C3—C4	1.385 (2)	C19—H19	0.9500
C3—H3	0.9500	C20—C21	1.385 (2)
C4—C5	1.389 (2)	C21—C22	1.385 (2)
C4—H4	0.9500	C21—H21	0.9500
C5—C6	1.392 (2)	C22—C23	1.392 (2)
C5—H5	0.9500	C22—H22	0.9500
C6—H6	0.9500	C23—H23	0.9500
C7—C8	1.362 (2)		
O2—S1—O3	121.18 (7)	C10—C9—H9	126.5
O2—S1—N1	105.71 (6)	C8—C9—H9	126.5
O3—S1—N1	105.68 (6)	C9—C10—O1	109.63 (13)
O2—S1—C12	109.40 (7)	C9—C10—C11	134.12 (15)

O3—S1—C12	106.87 (6)	O1—C10—C11	116.20 (14)
N1—S1—C12	107.24 (6)	C10—C11—H11A	109.5
O6—S2—O7	120.94 (7)	C10—C11—H11B	109.5
O6—S2—N1	108.91 (6)	H11A—C11—H11B	109.5
O7—S2—N1	103.39 (6)	C10—C11—H11C	109.5
O6—S2—C18	108.60 (7)	H11A—C11—H11C	109.5
O7—S2—C18	109.02 (7)	H11B—C11—H11C	109.5
N1—S2—C18	104.78 (6)	C17—C12—C13	121.75 (13)
C10—O1—C7	107.60 (12)	C17—C12—S1	120.58 (11)
C1—N1—S1	117.17 (9)	C13—C12—S1	117.67 (11)
C1—N1—S2	117.94 (9)	C14—C13—C12	116.99 (13)
S1—N1—S2	120.70 (7)	C14—C13—H13	121.5
O4—N2—O5	124.61 (14)	C12—C13—H13	121.5
O4—N2—C14	117.32 (13)	C13—C14—C15	123.07 (14)
O5—N2—C14	118.07 (13)	C13—C14—N2	117.55 (13)
O8—N3—O9	124.14 (15)	C15—C14—N2	119.37 (13)
O8—N3—C20	117.73 (15)	C14—C15—C16	118.73 (14)
O9—N3—C20	118.13 (13)	C14—C15—H15	120.6
C6—C1—C2	121.55 (13)	C16—C15—H15	120.6
C6—C1—N1	118.27 (12)	C15—C16—C17	120.10 (14)
C2—C1—N1	120.18 (12)	C15—C16—H16	119.9
C3—C2—C1	116.87 (13)	C17—C16—H16	119.9
C3—C2—C7	119.05 (13)	C16—C17—C12	119.34 (14)
C1—C2—C7	124.08 (13)	C16—C17—H17	120.3
C4—C3—C2	121.68 (14)	C12—C17—H17	120.3
C4—C3—H3	119.2	C19—C18—C23	122.06 (14)
C2—C3—H3	119.2	C19—C18—S2	118.18 (12)
C3—C4—C5	120.55 (14)	C23—C18—S2	119.69 (11)
C3—C4—H4	119.7	C20—C19—C18	116.97 (14)
C5—C4—H4	119.7	C20—C19—H19	121.5
C4—C5—C6	118.98 (14)	C18—C19—H19	121.5
C4—C5—H5	120.5	C19—C20—C21	122.77 (14)
C6—C5—H5	120.5	C19—C20—N3	118.00 (14)
C1—C6—C5	120.32 (13)	C21—C20—N3	119.22 (14)
C1—C6—H6	119.8	C20—C21—C22	118.91 (15)
C5—C6—H6	119.8	C20—C21—H21	120.5
C8—C7—O1	108.84 (13)	C22—C21—H21	120.5
C8—C7—C2	136.62 (14)	C21—C22—C23	120.27 (15)
O1—C7—C2	114.51 (13)	C21—C22—H22	119.9
C7—C8—C9	106.95 (14)	C23—C22—H22	119.9
C7—C8—H8	126.5	C22—C23—C18	118.97 (14)
C9—C8—H8	126.5	C22—C23—H23	120.5
C10—C9—C8	106.96 (14)	C18—C23—H23	120.5

O2—S1—N1—C1	−174.19 (10)	O2—S1—C12—C17	−25.39 (14)
O3—S1—N1—C1	−44.58 (11)	O3—S1—C12—C17	−158.26 (11)
C12—S1—N1—C1	69.16 (11)	N1—S1—C12—C17	88.80 (13)
O2—S1—N1—S2	29.26 (10)	O2—S1—C12—C13	154.26 (11)

O3—S1—N1—S2	158.87 (8)	O3—S1—C12—C13	21.39 (13)
C12—S1—N1—S2	−87.39 (9)	N1—S1—C12—C13	−91.55 (12)
O6—S2—N1—C1	−112.41 (11)	C17—C12—C13—C14	−0.6 (2)
O7—S2—N1—C1	17.40 (12)	S1—C12—C13—C14	179.70 (10)
C18—S2—N1—C1	131.55 (11)	C12—C13—C14—C15	0.4 (2)
O6—S2—N1—S1	43.96 (10)	C12—C13—C14—N2	−179.44 (12)
O7—S2—N1—S1	173.77 (8)	O4—N2—C14—C13	176.14 (13)
C18—S2—N1—S1	−72.08 (9)	O5—N2—C14—C13	−4.6 (2)
S1—N1—C1—C6	−81.02 (15)	O4—N2—C14—C15	−3.7 (2)
S2—N1—C1—C6	76.19 (15)	O5—N2—C14—C15	175.51 (14)
S1—N1—C1—C2	98.73 (14)	C13—C14—C15—C16	0.3 (2)
S2—N1—C1—C2	−104.06 (13)	N2—C14—C15—C16	−179.83 (13)
C6—C1—C2—C3	0.4 (2)	C14—C15—C16—C17	−0.9 (2)
N1—C1—C2—C3	−179.36 (12)	C15—C16—C17—C12	0.7 (2)
C6—C1—C2—C7	−178.88 (13)	C13—C12—C17—C16	0.1 (2)
N1—C1—C2—C7	1.4 (2)	S1—C12—C17—C16	179.75 (11)
C1—C2—C3—C4	1.5 (2)	O6—S2—C18—C19	166.10 (11)
C7—C2—C3—C4	−179.21 (14)	O7—S2—C18—C19	32.49 (13)
C2—C3—C4—C5	−1.6 (2)	N1—S2—C18—C19	−77.64 (12)
C3—C4—C5—C6	−0.1 (2)	O6—S2—C18—C23	−11.09 (14)
C2—C1—C6—C5	−2.1 (2)	O7—S2—C18—C23	−144.70 (12)
N1—C1—C6—C5	177.64 (13)	N1—S2—C18—C23	105.16 (12)
C4—C5—C6—C1	1.9 (2)	C23—C18—C19—C20	−1.9 (2)
C10—O1—C7—C8	0.89 (16)	S2—C18—C19—C20	−179.03 (10)
C10—O1—C7—C2	179.24 (12)	C18—C19—C20—C21	0.3 (2)
C3—C2—C7—C8	158.66 (17)	C18—C19—C20—N3	179.36 (12)
C1—C2—C7—C8	−22.1 (3)	O8—N3—C20—C19	171.70 (14)
C3—C2—C7—O1	−19.06 (19)	O9—N3—C20—C19	−8.6 (2)
C1—C2—C7—O1	160.19 (13)	O8—N3—C20—C21	−9.2 (2)
O1—C7—C8—C9	−0.59 (17)	O9—N3—C20—C21	170.48 (14)
C2—C7—C8—C9	−178.40 (16)	C19—C20—C21—C22	1.5 (2)
C7—C8—C9—C10	0.06 (18)	N3—C20—C21—C22	−177.54 (13)
C8—C9—C10—O1	0.49 (17)	C20—C21—C22—C23	−1.8 (2)
C8—C9—C10—C11	−176.60 (17)	C21—C22—C23—C18	0.2 (2)
C7—O1—C10—C9	−0.86 (16)	C19—C18—C23—C22	1.7 (2)
C7—O1—C10—C11	176.82 (13)	S2—C18—C23—C22	178.77 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1	0.95	2.41	2.7460 (18)	101
C3—H3···O4^{i}	0.95	2.31	3.226 (2)	161
C13—H13···O3	0.95	2.51	2.8637 (18)	102
C16—H16···O2^{ii}	0.95	2.53	3.0874 (18)	118
C19—H19···O4^{iii}	0.95	2.58	2.981 (2)	106
C23—H23···O6	0.95	2.56	2.9252 (19)	103

Symmetry codes: (i) −*x*+5/2, *y*+1/2, −*z*+3/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+3/2.