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Crystal structure and Hirshfeld surface analysis of 4-methyl-*N*-[2-(5-methylfuran-2-yl)phenyl]-*N*-[(5-phenylfuran-2-yl)methyl]benzenesulfonamide

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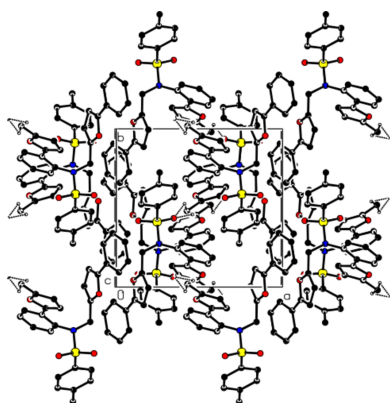
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In the title compound, C₂₉H₂₅NO₄S, the molecular conformation is stable with intramolecular C—H···O and C—H···N interactions being observed. In the crystal, molecules are linked by C—H···O interactions, forming layers parallel to the (100) plane. In addition, π – π [centroid-to-centroid distance = 3.4961 (7) Å] and C—H··· π interactions connect the molecules within the layers. The layers are also bound to each other by van der Waals interactions. A Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H···H (53.2%), C···H/H···C (28.9%) and O···H/H···O (13.8%) contacts.

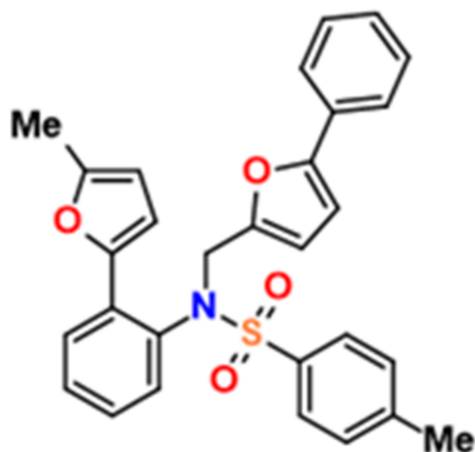
1. Chemical context

Carbon–carbon bond formation *via* Suzuki coupling of organoboronic acids and its derivatives with organic halides provides a mild method for the synthesis of various functionalized compounds (Miyaura & Suzuki, 1995; Suzuki 1999), especially biaryls (Leadbeater & Marco, 2002; Polyanskii *et al.*, 2019; Khalilov *et al.*, 2021). Recently, we have been interested in the synthesis of furan-substituted sulfonylamides because they are key intermediates in synthesis, analytical chemistry, catalysis and in the pharmaceutical industry (Demeke & Forsyth, 2002; Aliyeva *et al.*, 2005; Aliyeva *et al.*, 2024). The most widely used furan-substituted sulfonylamide is furosemide, which is a loop diuretic medication used to treat fluid build-up due to heart failure, kidney disease, or liver scarring. Continuing our research in the chemistry of furyl-substituted sulfonylamides (Guliyeva *et al.*, 2024; Mammadova *et al.*, 2023a, b; Burkin *et al.*, 2024, 2025), in this work we set out a coupling strategy using the Suzuki reaction to synthesize a sulfonylamide-substituted bifuran compound, which was hitherto unknown and is potentially important toward further transformations or the synthesis of pharmaceutical species. Moreover, the attachment of non-covalent bond-donor or acceptor centers to the sulfonylamide can be applied as a synthetic strategy in the ligand design and catalysis (Gurbanov *et al.*, 2022; Huseynov *et al.*, 2021; Mahmudov *et al.*, 2015, 2023).



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2. Structural commentary

The molecular conformation of the molecule is stable due to the intramolecular interactions $C1-H1B \cdots O4$, $C14-H14 \cdots O2$, $C19-H19 \cdots N1$ and $C24-H24 \cdots O3$, which form $S(5)$, $S(5)$, $S(6)$ and $S(5)$ ring motifs (Bernstein *et al.*, 1995), respectively (Fig. 1; Table 1). The dihedral angles between the planes of the *A* ($O1/C2-C5$), *B* ($C6-C11$), *C* ($C12-C17$), *D* ($O2/C18-C21$), and *E* ($C23-C28$) rings of the molecule are $A/B = 13.26$ (7), $A/C = 52.20$ (7), $A/D = 62.45$ (8), $A/E = 88.06$ (7), $B/C = 48.74$ (6), $B/D = 53.59$ (7), $B/E = 75.63$ (6), $C/D = 5.41$ (7), $C/E = 39.61$ (6) and $D/E = 35.00$ (7)°. The bond lengths and angles in the title compound are in good agreement with those reported for related compounds (see *Data-base survey* section).

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecules are linked by the intermolecular $C-H \cdots O$ interactions, forming layers parallel to the (100) plane (Table 1; Figs. 2 and 3). In addition, $\pi-\pi$ [$Cg1 \cdots Cg1^{ii} = 3.4961$ (7) Å, slippage = 1.016 Å; symmetry code: (ii) $-x + 2, -y + 1, -z + 1$; $Cg1$ is the centroid of the (*A*: $O1/C2-C5$) furan

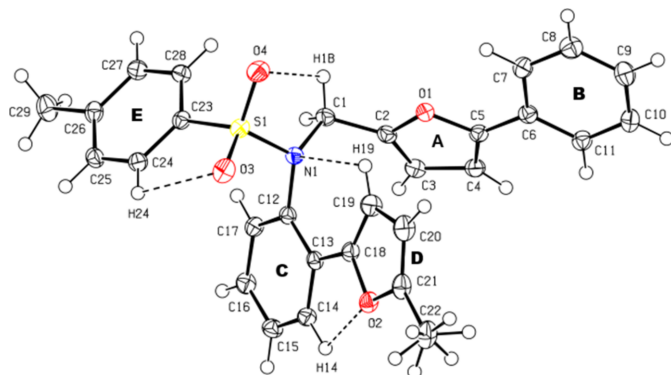


Figure 1

View of the title molecule. Displacement ellipsoids are drawn at the 50% probability level.

Table 1

Hydrogen-bond geometry (Å, °).

$Cg3$ and $Cg5$ are the centroids of the $C6-C12$ and $C23-C28$ rings, respectively.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C1-H1B \cdots O4$	0.99	2.43	2.9091 (16)	109
$C14-H14 \cdots O2$	0.95	2.36	2.7265 (16)	102
$C19-H19 \cdots N1$	0.95	2.56	3.0211 (16)	110
$C22-H22C \cdots O3^i$	0.98	2.60	3.2636 (18)	125
$C22-H22D \cdots O3^i$	0.98	2.60	3.2636 (18)	126
$C24-H24 \cdots O3$	0.95	2.55	2.9196 (16)	104
$C1-H1A \cdots Cg3^{ii}$	0.99	2.94	3.6930 (14)	134
$C4-H4 \cdots Cg5^{iii}$	0.95	2.86	3.7996 (14)	172
$C29-H29C \cdots Cg3^{iv}$	0.98	2.97	3.6676 (17)	129

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

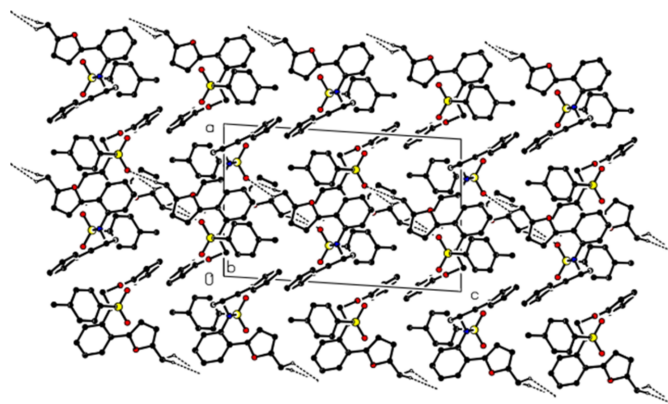


Figure 2

A view of the molecular packing along the *b* axis, showing the $C-H \cdots O$ interactions.

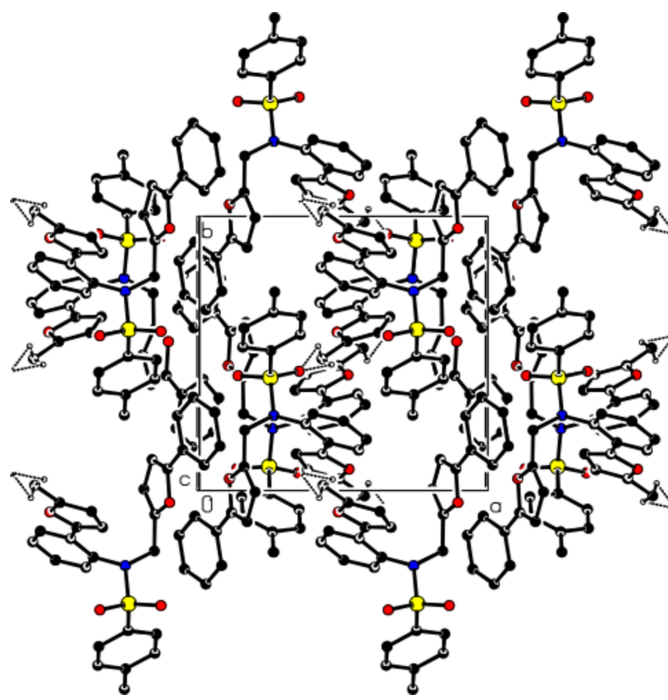


Figure 3

A view of the molecular packing along the *c* axis, showing the $C-H \cdots O$ interactions.

Table 2

Summary of short interatomic contacts (Å).

Contact	Distance	Symmetry operation
H22A...H3	2.55	$1 - x, 1 - y, 1 - z$
H29B...H22E	2.49	$1 - x, 2 - y, 1 - z$
O3...H22D	2.60	$1 - x, \frac{1}{2} + y, \frac{3}{2} - z$
O4...H10	2.60	$x, 1 + y, z$
H19...H9	2.47	$2 - x, \frac{1}{2} + y, \frac{3}{2} - z$
H28...H28	2.40	$2 - x, 2 - y, 1 - z$
H27...H10	2.55	$2 - x, 1 - y, 1 - z$
H22C...H16	2.43	$x, \frac{3}{2} - y, \frac{1}{2} + z$
H10...O4	2.60	$x, -1 + y, z$
H22D...O3	2.60	$1 - x, -\frac{1}{2} + y, \frac{3}{2} - z$
H16...H29B	2.45	$1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$

ring] and C—H... π interactions connect the molecules within layers (Fig. 4). The layers are also bound to each other by van der Waals interactions.

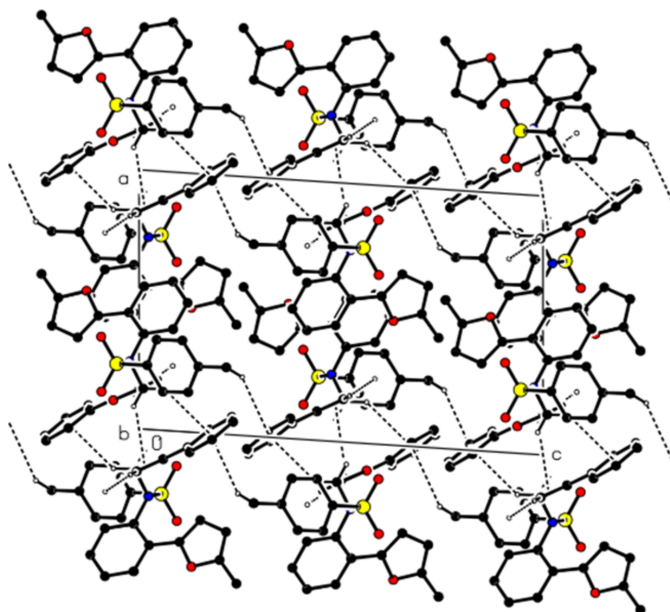


Figure 4

A view of the molecular packing along the *b* axis, showing the π - π and C—H... π interactions.

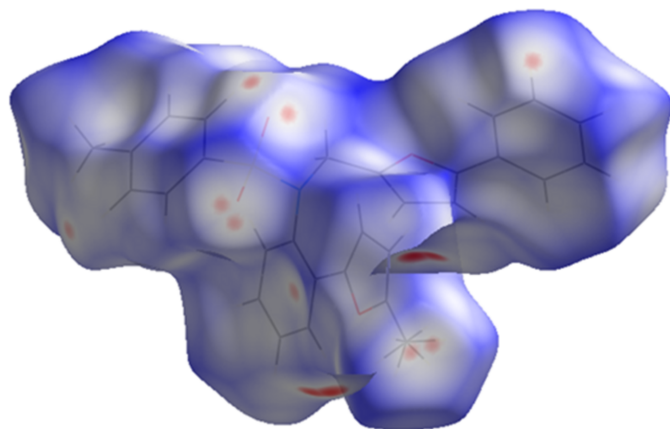


Figure 5

Hirshfeld surface of the title compound mapped with d_{norm} .

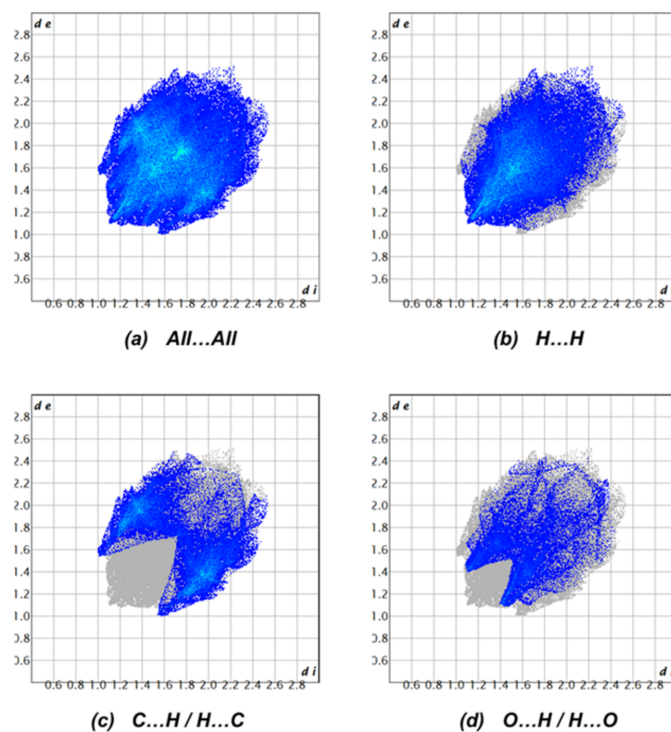


Figure 6

The two-dimensional fingerprint plots for the compound showing (a) all interactions, and delineated into (b) H...H (53.2%), (c) C...H/H...C (28.9%) and (d) O...H/H...O (13.8%) interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

A Hirshfeld surface analysis was carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021) to investigate the intermolecular interactions (Tables 1 and 2) in the crystal of the compound. The Hirshfeld surface mapped with the d_{norm} function over the range -0.1546 to 1.3220 a.u. (Fig. 5) illustrates contact distances that are equal, shorter, and longer in relation to the sum of van der Waals radii, represented by white, red, and blue colors, respectively, with bright-red spots indicating the corresponding donors and acceptors. According to the two-dimensional fingerprint plots, the intermolecular H...H, C...H/H...C and O...H/H...O contacts make the most important contributions to the Hirshfeld surface of 53.2%, 28.9% and 13.8%, respectively (Fig. 6). Smaller contributions are made by O...C/C...O (2.4%), C...C (1.6%) and O...O (0.1%) interactions.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 6.00, update April 2025; Groom *et al.*, 2016) for the 2-(furan-2-yl)-*N*-[(furan-2-yl) methyl]aniline unit gave four hits, *viz.* CSD refcodes IFUBOB (Zubkov *et al.*, 2008a), LOKWOB (Burkin *et al.*, 2024), SEBWIG (Zubkov *et al.*, 2006) and VOCWAM (Zubkov *et al.*, 2008b).

IFUBOB, LOKWOB and VOCWAM crystallize in the triclinic $P\bar{1}$ space group, and SEBWIG in the monoclinic $P2_1/c$ space group like the title compound. While in the title

compound intermolecular C—H...O hydrogen bonds, C—H... π and π – π interactions are observed, in IFUBOB, LOKWOB, SEBWIG and VOCWAM, the molecules are linked by C—H...O hydrogen bonds, forming a three-dimensional network. C—H... π interactions were also observed in all except VOCWAM.

In addition, four related compounds containing the O=S=O group are HUSFIO (Burkin *et al.*, 2025), YIKROD (Mammadova *et al.*, 2023a), KETGID (Schinke *et al.*, 2022) and LUJKUA (Vinaya *et al.*, 2024). In the crystal of HUSFIO, the molecules form sheets parallel to the (002) plane due to C—H...O and C—H...F interactions. In addition, the molecules are connected in the *a*-axis direction by S—O... π and π – π interactions, and there are van der Waals interactions between the molecular sheets. In YIKROD, molecules are connected *via* C—H...O and C—H...N hydrogen bonds, forming layers parallel to the (100) plane. These layers are interconnected by C...H interactions and weak van der Waals interactions. In KETGID, the crystal structure features three short intermolecular C—H...O contacts involving the methanesulfonyl-O atoms. In LUJKUA, the asymmetric unit contains two distinct molecules, which exhibit differences in conformation resulting from a variation in key torsion angles. These distinctions influence the molecular orientation and intermolecular interactions, with strong N—H...N and N—H...O hydrogen bonds forming a centrosymmetric tetramer stabilized by π – π stacking.

5. Synthesis and crystallization

A 125 mL dry Schlenk tube was charged with *N*–[(5-bromofuran-2-yl)methyl]-4-methyl-*N*–[2-(5-methylfuran-2-yl)phenyl]benzene-1-sulfonamide (500 mg, 1.03 mmol) in a mixture of ethanol/toluene (10 mL, 1:1). To the reaction mixture in the presence of a 2 *M* water solution of Na₂CO₃ (5.38 mmol, 2.71 mL), phenylboronic acid (250 mg, 2.06 mmol) was added (Fig. 7). Argon was bubbled through the solution for 10 min. Then tetrakis(triphenylphosphine)palladium (59.3 mg, 51.4 μ mol) was added in a gentle flow of argon. The reaction mixture was stirred at 383 K for 5 h. After the cooling of the reaction to room temperature, the resulting mixture was treated with water (30 mL) and extracted with EtOAc (3 $\times$ 10 mL), and treated in a usual manner to give a solid that was purified by silica gel column chromatography (eluent: heptane to heptane/ethyl acetate, 10:1). The title compound was obtained as a light-brown solid, yield 40%, 199.2 mg

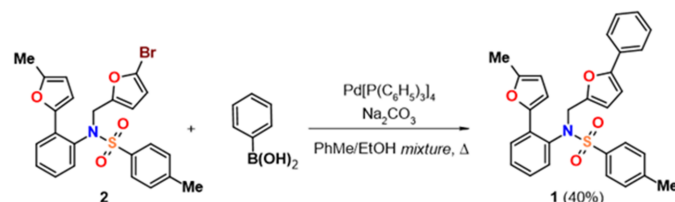


Figure 7

Synthesis of 4-methyl-*N*–[2-(5-methyl-2-furyl)phenyl]-*N*–[(5-phenyl-2-furyl)methyl]benzenesulfonamide.

Table 3

Experimental details.

Crystal data	
Chemical formula	C ₂₉ H ₂₅ NO ₄ S
<i>M_r</i>	483.56
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.71270 (9), 11.10322 (9), 18.30473 (13)
β (°)	93.6993 (7)
<i>V</i> (Å ³)	2375.55 (3)
<i>Z</i>	4
Radiation type	Cu <i>K</i> α
μ (mm ^{−1})	1.51
Crystal size (mm)	0.21 $\times$ 0.15 $\times$ 0.12
Data collection	
Diffractometer	Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector
Absorption correction	Multi-scan (CrysAlis PRO; Rigaku OD, 2021)
<i>T</i> _{min} , <i>T</i> _{max}	0.615, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	28196, 5133, 4843
<i>R</i> _{int}	0.039
(sin θ / λ) _{max} (Å ^{−1})	0.639
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.038, 0.103, 1.07
No. of reflections	5133
No. of parameters	318
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ^{−3})	0.36, −0.48

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

(0.41 mmol); m.p. 456 K. A single-crystal of the compound was grown from a heptane/ethyl acetate mixture. IR (KBr), ν (cm^{−1}): 1348 (ν_{as} SO₂), 1165 (ν_{s} SO₂). ¹H NMR (700.2 MHz, CDCl₃) (*J*, Hz): δ 7.84 (*d*, *J* = 7.9, 1H, H Ar), 7.69 (*d*, *J* = 8.1, 2H, H Ar), 7.39 (*d*, *J* = 7.6, 2H, H Ar), 7.34–7.31 (*m*, 3H, H Ar), 7.25–7.23 (*m*, 3H, H Ar), 7.08 (*d*, *J* = 3.1, 1H, H Fur), 7.01 (*t*, *J* = 7.6, 1H, H Ar), 6.65 (*d*, *J* = 7.9, 1H, H Ar), 6.39 (*d*, *J* = 3.3, 1H, H Fur), 6.08 (*d*, *J* = 3.1, 1H, H Fur), 6.03 (*d*, *J* = 3.3, 1H, H Fur), 5.01 (*d*, *J* = 15.5, 1H, NCH), 4.65 (*d*, *J* = 15.5, 1H, NCH), 2.39 (*s*, 3H, CH₃), 2.32 (*s*, 3H, CH₃). ¹³C{¹H} NMR (176.1 MHz, CDCl₃): δ 153.9, 152.0, 148.9, 148.4, 143.3, 137.4, 133.9, 131.8, 130.5, 130.3, 129.4 (2C), 128.8, 128.5 (2C), 128.0 (2C), 127.4, 127.1, 126.7, 123.7 (2C), 112.2, 111.4, 108.4, 105.5, 47.4, 21.5, 13.7. MS (ESI) *m/z*: [*M* + *H*]⁺ 484. Elemental analysis calculated (%) for C₂₉H₂₅NO₄S: C 72.03, H 5.21, N 2.90, S 6.63; found: C 71.89, H 5.48, N 3.18, S 6.33.

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 and 0.99 Å) and refined using a riding model with *U*_{iso}(H) = 1.2 or 1.5 *U*_{eq}(C). One of the methyl groups (C22) was found to be disordered; it was treated as an idealized disordered methyl group, with two positions rotated from each other by 60°, and the site-occupation factors were fixed at 0.5.

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The authors' contributions are as follows. Conceptualization, MA and GMM; synthesis and NMR analysis, SA, RZN and AEL; X-ray analysis, VNK; writing (review and editing of the manuscript) MA and GMM; funding acquisition RZN, KIH and NDS; supervision, MA and GMM.

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Crystal structure and Hirshfeld surface analysis of 4-methyl-*N*-[2-(5-methylfuran-2-yl)phenyl]-*N*-[(5-phenylfuran-2-yl)methyl]benzenesulfonamide

Selbi Annadurdyeva, Anastasia E. Levina, Victor N. Khrustalev, Roya Z. Nazarova, Khudayar I. Hasanov, Nurlana D. Sadikhova, Mehmet Akkurt and Gizachew Mulugeta Manahelohe

Computing details

4-Methyl-*N*-[2-(5-methylfuran-2-yl)phenyl]-*N*-[(5-phenylfuran-2-yl)methyl]benzenesulfonamide

Crystal data

$C_{29}H_{25}NO_4S$

$M_r = 483.56$

Monoclinic, $P2_1/c$

$a = 11.71270$ (9) Å

$b = 11.10322$ (9) Å

$c = 18.30473$ (13) Å

$\beta = 93.6993$ (7)°

$V = 2375.55$ (3) Å³

$Z = 4$

$F(000) = 1016$

$D_x = 1.352$ Mg m⁻³

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

Cell parameters from 18959 reflections

$\theta = 3.8\text{--}79.3^\circ$

$\mu = 1.51$ mm⁻¹

$T = 100$ K

Prism, colourless

$0.21 \times 0.15 \times 0.12$ mm

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE

area-detector

diffractometer

Radiation source: micro-focus sealed X-ray tube

φ and ω scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2021)

$T_{\min} = 0.615$, $T_{\max} = 1.000$

28196 measured reflections

5133 independent reflections

4843 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 80.0^\circ$, $\theta_{\min} = 3.8^\circ$

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -19 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.103$

$S = 1.07$

5133 reflections

318 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.936P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.36$ e Å⁻³

$\Delta\rho_{\min} = -0.48$ e Å⁻³

Extinction correction: SHELXL-2019/2

(Sheldrick, 2015b),

$F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00047 (13)

Special details

Experimental. CrysAlisPro 1.171.43.129a (Rigaku OD, 2021). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.75420 (3)	0.91249 (3)	0.55524 (2)	0.01882 (10)	
O1	0.89563 (7)	0.54353 (8)	0.56336 (5)	0.01940 (19)	
O2	0.48043 (8)	0.57815 (8)	0.62957 (5)	0.0216 (2)	
O3	0.65373 (9)	0.93517 (9)	0.59358 (5)	0.0259 (2)	
O4	0.86552 (9)	0.91925 (9)	0.59281 (5)	0.0266 (2)	
N1	0.74228 (9)	0.77377 (9)	0.52304 (5)	0.0174 (2)	
C1	0.84333 (10)	0.72008 (12)	0.48961 (7)	0.0200 (2)	
H1A	0.840447	0.740049	0.436818	0.024*	
H1B	0.914364	0.754977	0.513030	0.024*	
C2	0.84531 (10)	0.58720 (12)	0.49885 (7)	0.0197 (3)	
C3	0.80749 (11)	0.49497 (12)	0.45546 (7)	0.0220 (3)	
H3	0.769646	0.501157	0.408125	0.026*	
C4	0.83540 (11)	0.38660 (12)	0.49453 (7)	0.0213 (3)	
H4	0.819986	0.306638	0.478271	0.026*	
C5	0.88842 (10)	0.42029 (11)	0.55961 (7)	0.0191 (2)	
C6	0.94092 (10)	0.35150 (11)	0.62083 (7)	0.0193 (2)	
C7	1.01454 (12)	0.40632 (12)	0.67457 (7)	0.0221 (3)	
H7	1.027068	0.490801	0.673127	0.027*	
C8	1.06915 (12)	0.33764 (13)	0.72983 (7)	0.0266 (3)	
H8	1.119454	0.375271	0.765674	0.032*	
C9	1.05054 (13)	0.21398 (14)	0.73295 (8)	0.0282 (3)	
H9	1.088203	0.167234	0.770716	0.034*	
C10	0.97662 (12)	0.15918 (12)	0.68057 (7)	0.0256 (3)	
H10	0.963472	0.074865	0.682719	0.031*	
C11	0.92192 (11)	0.22725 (12)	0.62513 (7)	0.0217 (3)	
H11	0.871108	0.189155	0.589761	0.026*	
C12	0.63199 (10)	0.74259 (11)	0.48753 (6)	0.0167 (2)	
C13	0.55292 (10)	0.67513 (11)	0.52494 (6)	0.0172 (2)	
C14	0.44821 (11)	0.64726 (11)	0.48680 (7)	0.0202 (2)	
H14	0.393701	0.600160	0.510350	0.024*	
C15	0.42232 (11)	0.68661 (12)	0.41580 (7)	0.0225 (3)	
H15	0.350198	0.667796	0.391789	0.027*	
C16	0.50147 (11)	0.75340 (12)	0.37972 (7)	0.0222 (3)	
H16	0.483966	0.780739	0.331103	0.027*	
C17	0.60674 (11)	0.77981 (11)	0.41561 (7)	0.0197 (2)	
H17	0.662090	0.823758	0.390805	0.024*	
C18	0.57319 (11)	0.63322 (11)	0.60056 (7)	0.0191 (2)	

C19	0.66248 (12)	0.63388 (13)	0.65182 (7)	0.0249 (3)	
H19	0.736417	0.666552	0.646406	0.030*	
C20	0.62372 (13)	0.57591 (13)	0.71549 (8)	0.0279 (3)	
H20	0.667186	0.562507	0.760376	0.033*	
C21	0.51367 (12)	0.54384 (12)	0.69972 (7)	0.0247 (3)	
C22	0.42627 (14)	0.48056 (14)	0.74114 (8)	0.0320 (3)	
H22A	0.354790	0.474037	0.710479	0.048*	0.5
H22B	0.453939	0.399773	0.754648	0.048*	0.5
H22C	0.412534	0.526204	0.785545	0.048*	0.5
H22D	0.459386	0.459306	0.789969	0.048*	0.5
H22E	0.360236	0.533570	0.745800	0.048*	0.5
H22F	0.401641	0.407138	0.714903	0.048*	0.5
C23	0.75261 (11)	1.01057 (11)	0.47944 (6)	0.0181 (2)	
C24	0.65102 (11)	1.06855 (12)	0.45602 (7)	0.0203 (2)	
H24	0.584841	1.061068	0.483077	0.024*	
C25	0.64818 (11)	1.13728 (11)	0.39262 (7)	0.0216 (3)	
H25	0.579097	1.176375	0.376150	0.026*	
C26	0.74469 (11)	1.15002 (11)	0.35268 (7)	0.0212 (3)	
C27	0.84635 (11)	1.09300 (12)	0.37789 (7)	0.0215 (3)	
H27	0.913159	1.102499	0.351658	0.026*	
C28	0.85073 (10)	1.02279 (12)	0.44071 (7)	0.0201 (2)	
H28	0.919719	0.983500	0.457156	0.024*	
C29	0.74108 (14)	1.22441 (14)	0.28389 (8)	0.0319 (3)	
H29A	0.784934	1.298663	0.292996	0.048*	
H29B	0.661506	1.244615	0.269002	0.048*	
H29C	0.774435	1.178402	0.244843	0.048*	

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02193 (17)	0.01953 (16)	0.01493 (16)	−0.00240 (10)	0.00068 (11)	−0.00034 (10)
O1	0.0181 (4)	0.0180 (4)	0.0221 (4)	0.0004 (3)	0.0010 (3)	0.0003 (3)
O2	0.0232 (4)	0.0213 (4)	0.0210 (4)	0.0005 (3)	0.0066 (3)	0.0044 (3)
O3	0.0330 (5)	0.0248 (5)	0.0210 (5)	−0.0009 (4)	0.0098 (4)	−0.0027 (4)
O4	0.0294 (5)	0.0283 (5)	0.0209 (5)	−0.0062 (4)	−0.0081 (4)	0.0019 (3)
N1	0.0158 (5)	0.0180 (5)	0.0184 (5)	−0.0002 (4)	0.0015 (4)	0.0006 (4)
C1	0.0155 (5)	0.0220 (6)	0.0230 (6)	0.0005 (4)	0.0040 (4)	0.0025 (5)
C2	0.0152 (5)	0.0231 (6)	0.0211 (6)	0.0014 (4)	0.0029 (4)	0.0026 (4)
C3	0.0183 (6)	0.0255 (7)	0.0221 (6)	0.0000 (5)	0.0006 (4)	0.0005 (5)
C4	0.0186 (6)	0.0218 (6)	0.0235 (6)	−0.0011 (5)	0.0011 (5)	−0.0022 (5)
C5	0.0161 (5)	0.0185 (6)	0.0230 (6)	−0.0004 (4)	0.0042 (4)	−0.0010 (4)
C6	0.0177 (5)	0.0206 (6)	0.0198 (6)	0.0009 (4)	0.0038 (4)	−0.0013 (5)
C7	0.0247 (6)	0.0203 (6)	0.0215 (6)	−0.0001 (5)	0.0022 (5)	−0.0023 (5)
C8	0.0281 (6)	0.0295 (7)	0.0216 (6)	−0.0010 (5)	−0.0020 (5)	−0.0020 (5)
C9	0.0310 (7)	0.0285 (7)	0.0247 (6)	0.0043 (6)	−0.0014 (5)	0.0043 (5)
C10	0.0298 (7)	0.0196 (6)	0.0277 (7)	0.0020 (5)	0.0033 (5)	0.0015 (5)
C11	0.0221 (6)	0.0206 (6)	0.0225 (6)	−0.0006 (5)	0.0024 (5)	−0.0019 (5)
C12	0.0169 (5)	0.0154 (5)	0.0180 (5)	0.0017 (4)	0.0015 (4)	−0.0015 (4)

C13	0.0175 (5)	0.0158 (5)	0.0184 (6)	0.0031 (4)	0.0024 (4)	−0.0004 (4)
C14	0.0176 (6)	0.0196 (6)	0.0235 (6)	0.0002 (4)	0.0028 (4)	−0.0005 (5)
C15	0.0199 (6)	0.0238 (6)	0.0234 (6)	0.0002 (5)	−0.0030 (5)	−0.0020 (5)
C16	0.0264 (6)	0.0224 (6)	0.0173 (6)	0.0000 (5)	−0.0028 (5)	0.0007 (5)
C17	0.0219 (6)	0.0188 (6)	0.0184 (6)	−0.0009 (5)	0.0021 (4)	0.0005 (4)
C18	0.0200 (6)	0.0175 (6)	0.0203 (6)	0.0009 (4)	0.0055 (4)	0.0017 (4)
C19	0.0257 (6)	0.0288 (7)	0.0202 (6)	−0.0007 (5)	0.0013 (5)	0.0045 (5)
C20	0.0361 (8)	0.0299 (7)	0.0178 (6)	0.0025 (6)	0.0027 (5)	0.0048 (5)
C21	0.0336 (7)	0.0217 (6)	0.0197 (6)	0.0046 (5)	0.0082 (5)	0.0035 (5)
C22	0.0402 (8)	0.0282 (7)	0.0293 (7)	0.0036 (6)	0.0157 (6)	0.0080 (6)
C23	0.0204 (6)	0.0163 (5)	0.0175 (5)	−0.0013 (4)	0.0002 (4)	−0.0018 (4)
C24	0.0191 (6)	0.0191 (6)	0.0228 (6)	0.0006 (4)	0.0028 (4)	−0.0029 (5)
C25	0.0209 (6)	0.0189 (6)	0.0248 (6)	0.0035 (5)	−0.0010 (5)	−0.0024 (5)
C26	0.0265 (6)	0.0178 (6)	0.0192 (6)	0.0012 (5)	0.0002 (5)	−0.0014 (4)
C27	0.0204 (6)	0.0226 (6)	0.0220 (6)	0.0004 (5)	0.0044 (5)	0.0001 (5)
C28	0.0177 (6)	0.0207 (6)	0.0216 (6)	0.0002 (4)	−0.0006 (4)	−0.0002 (5)
C29	0.0374 (8)	0.0320 (8)	0.0263 (7)	0.0064 (6)	0.0031 (6)	0.0082 (6)

Geometric parameters (Å, °)

S1—O3	1.4309 (10)	C14—C15	1.3860 (18)
S1—O4	1.4363 (10)	C14—H14	0.9500
S1—N1	1.6518 (11)	C15—C16	1.3873 (19)
S1—C23	1.7630 (13)	C15—H15	0.9500
O1—C5	1.3724 (15)	C16—C17	1.3905 (18)
O1—C2	1.3737 (15)	C16—H16	0.9500
O2—C21	1.3715 (16)	C17—H17	0.9500
O2—C18	1.3823 (15)	C18—C19	1.3593 (18)
N1—C12	1.4500 (15)	C19—C20	1.4307 (19)
N1—C1	1.4913 (15)	C19—H19	0.9500
C1—C2	1.4850 (18)	C20—C21	1.350 (2)
C1—H1A	0.9900	C20—H20	0.9500
C1—H1B	0.9900	C21—C22	1.4888 (19)
C2—C3	1.3530 (19)	C22—H22A	0.9800
C3—C4	1.4268 (18)	C22—H22B	0.9800
C3—H3	0.9500	C22—H22C	0.9800
C4—C5	1.3599 (18)	C22—H22D	0.9800
C4—H4	0.9500	C22—H22E	0.9800
C5—C6	1.4589 (18)	C22—H22F	0.9800
C6—C11	1.4004 (18)	C23—C28	1.3953 (17)
C6—C7	1.4044 (18)	C23—C24	1.3958 (18)
C7—C8	1.3894 (19)	C24—C25	1.3876 (19)
C7—H7	0.9500	C24—H24	0.9500
C8—C9	1.392 (2)	C25—C26	1.3924 (18)
C8—H8	0.9500	C25—H25	0.9500
C9—C10	1.390 (2)	C26—C27	1.4003 (18)
C9—H9	0.9500	C26—C29	1.5043 (18)
C10—C11	1.3884 (19)	C27—C28	1.3873 (18)

C10—H10	0.9500	C27—H27	0.9500
C11—H11	0.9500	C28—H28	0.9500
C12—C17	1.3933 (17)	C29—H29A	0.9800
C12—C13	1.4034 (17)	C29—H29B	0.9800
C13—C14	1.4060 (17)	C29—H29C	0.9800
C13—C18	1.4650 (17)		
O3—S1—O4	120.39 (6)	C13—C14—H14	119.1
O3—S1—N1	106.61 (6)	C14—C15—C16	120.16 (12)
O4—S1—N1	105.83 (6)	C14—C15—H15	119.9
O3—S1—C23	108.07 (6)	C16—C15—H15	119.9
O4—S1—C23	107.92 (6)	C15—C16—C17	119.16 (11)
N1—S1—C23	107.39 (5)	C15—C16—H16	120.4
C5—O1—C2	106.70 (10)	C17—C16—H16	120.4
C21—O2—C18	107.46 (10)	C16—C17—C12	120.77 (11)
C12—N1—C1	115.36 (10)	C16—C17—H17	119.6
C12—N1—S1	115.78 (8)	C12—C17—H17	119.6
C1—N1—S1	117.91 (8)	C19—C18—O2	109.02 (11)
C2—C1—N1	110.94 (10)	C19—C18—C13	136.14 (12)
C2—C1—H1A	109.5	O2—C18—C13	114.84 (11)
N1—C1—H1A	109.5	C18—C19—C20	106.82 (12)
C2—C1—H1B	109.5	C18—C19—H19	126.6
N1—C1—H1B	109.5	C20—C19—H19	126.6
H1A—C1—H1B	108.0	C21—C20—C19	107.05 (12)
C3—C2—O1	110.09 (11)	C21—C20—H20	126.5
C3—C2—C1	133.08 (12)	C19—C20—H20	126.5
O1—C2—C1	116.83 (11)	C20—C21—O2	109.65 (12)
C2—C3—C4	106.76 (11)	C20—C21—C22	133.93 (13)
C2—C3—H3	126.6	O2—C21—C22	116.42 (13)
C4—C3—H3	126.6	C21—C22—H22A	109.5
C5—C4—C3	106.50 (12)	C21—C22—H22B	109.5
C5—C4—H4	126.7	H22A—C22—H22B	109.5
C3—C4—H4	126.7	C21—C22—H22C	109.5
C4—C5—O1	109.95 (11)	H22A—C22—H22C	109.5
C4—C5—C6	132.45 (12)	H22B—C22—H22C	109.5
O1—C5—C6	117.52 (11)	H22D—C22—H22E	109.5
C11—C6—C7	118.70 (12)	H22D—C22—H22F	109.5
C11—C6—C5	119.89 (11)	H22E—C22—H22F	109.5
C7—C6—C5	121.36 (12)	C28—C23—C24	120.86 (12)
C8—C7—C6	120.33 (12)	C28—C23—S1	119.52 (10)
C8—C7—H7	119.8	C24—C23—S1	119.47 (10)
C6—C7—H7	119.8	C25—C24—C23	118.97 (12)
C7—C8—C9	120.36 (13)	C25—C24—H24	120.5
C7—C8—H8	119.8	C23—C24—H24	120.5
C9—C8—H8	119.8	C24—C25—C26	121.23 (12)
C10—C9—C8	119.71 (13)	C24—C25—H25	119.4
C10—C9—H9	120.1	C26—C25—H25	119.4
C8—C9—H9	120.1	C25—C26—C27	118.88 (12)

C11—C10—C9	120.23 (13)	C25—C26—C29	121.02 (12)
C11—C10—H10	119.9	C27—C26—C29	120.10 (12)
C9—C10—H10	119.9	C28—C27—C26	120.82 (12)
C10—C11—C6	120.65 (12)	C28—C27—H27	119.6
C10—C11—H11	119.7	C26—C27—H27	119.6
C6—C11—H11	119.7	C27—C28—C23	119.22 (12)
C17—C12—C13	120.85 (11)	C27—C28—H28	120.4
C17—C12—N1	118.77 (11)	C23—C28—H28	120.4
C13—C12—N1	120.37 (10)	C26—C29—H29A	109.5
C12—C13—C14	117.20 (11)	C26—C29—H29B	109.5
C12—C13—C18	123.97 (11)	H29A—C29—H29B	109.5
C14—C13—C18	118.83 (11)	C26—C29—H29C	109.5
C15—C14—C13	121.82 (12)	H29A—C29—H29C	109.5
C15—C14—H14	119.1	H29B—C29—H29C	109.5
O3—S1—N1—C12	45.99 (10)	N1—C12—C13—C18	0.86 (18)
O4—S1—N1—C12	175.28 (8)	C12—C13—C14—C15	−1.40 (18)
C23—S1—N1—C12	−69.63 (9)	C18—C13—C14—C15	178.37 (12)
O3—S1—N1—C1	−171.44 (9)	C13—C14—C15—C16	1.3 (2)
O4—S1—N1—C1	−42.15 (10)	C14—C15—C16—C17	0.2 (2)
C23—S1—N1—C1	72.93 (10)	C15—C16—C17—C12	−1.59 (19)
C12—N1—C1—C2	−66.81 (13)	C13—C12—C17—C16	1.51 (19)
S1—N1—C1—C2	150.47 (9)	N1—C12—C17—C16	−179.11 (11)
C5—O1—C2—C3	−0.08 (13)	C21—O2—C18—C19	−0.35 (14)
C5—O1—C2—C1	−179.58 (10)	C21—O2—C18—C13	179.55 (10)
N1—C1—C2—C3	95.11 (16)	C12—C13—C18—C19	−6.0 (2)
N1—C1—C2—O1	−85.53 (13)	C14—C13—C18—C19	174.20 (15)
O1—C2—C3—C4	−0.06 (14)	C12—C13—C18—O2	174.10 (11)
C1—C2—C3—C4	179.34 (13)	C14—C13—C18—O2	−5.66 (16)
C2—C3—C4—C5	0.17 (14)	O2—C18—C19—C20	0.37 (15)
C3—C4—C5—O1	−0.23 (14)	C13—C18—C19—C20	−179.50 (14)
C3—C4—C5—C6	−176.77 (13)	C18—C19—C20—C21	−0.25 (16)
C2—O1—C5—C4	0.19 (13)	C19—C20—C21—O2	0.04 (16)
C2—O1—C5—C6	177.31 (10)	C19—C20—C21—C22	179.25 (15)
C4—C5—C6—C11	−13.0 (2)	C18—O2—C21—C20	0.19 (15)
O1—C5—C6—C11	170.63 (11)	C18—O2—C21—C22	−179.18 (11)
C4—C5—C6—C7	164.31 (14)	O3—S1—C23—C28	168.18 (10)
O1—C5—C6—C7	−12.02 (17)	O4—S1—C23—C28	36.52 (12)
C11—C6—C7—C8	1.41 (19)	N1—S1—C23—C28	−77.17 (11)
C5—C6—C7—C8	−175.97 (12)	O3—S1—C23—C24	−16.16 (12)
C6—C7—C8—C9	−0.7 (2)	O4—S1—C23—C24	−147.82 (10)
C7—C8—C9—C10	−0.2 (2)	N1—S1—C23—C24	98.49 (11)
C8—C9—C10—C11	0.3 (2)	C28—C23—C24—C25	1.11 (19)
C9—C10—C11—C6	0.5 (2)	S1—C23—C24—C25	−174.49 (10)
C7—C6—C11—C10	−1.30 (19)	C23—C24—C25—C26	−0.57 (19)
C5—C6—C11—C10	176.12 (12)	C24—C25—C26—C27	−0.65 (19)
C1—N1—C12—C17	−63.82 (14)	C24—C25—C26—C29	179.86 (13)
S1—N1—C12—C17	79.71 (12)	C25—C26—C27—C28	1.35 (19)

C1—N1—C12—C13	115.57 (12)	C29—C26—C27—C28	−179.15 (13)
S1—N1—C12—C13	−100.90 (12)	C26—C27—C28—C23	−0.82 (19)
C17—C12—C13—C14	−0.01 (17)	C24—C23—C28—C27	−0.43 (19)
N1—C12—C13—C14	−179.38 (11)	S1—C23—C28—C27	175.17 (10)
C17—C12—C13—C18	−179.77 (11)		

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

*Cg*3 and *Cg*5 are the centroids of the C6–C12 and C23–C28 rings, respectively.

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C1—H1 <i>B</i> $\cdots$ O4	0.99	2.43	2.9091 (16)	109
C14—H14 $\cdots$ O2	0.95	2.36	2.7265 (16)	102
C19—H19 $\cdots$ N1	0.95	2.56	3.0211 (16)	110
C22—H22 <i>C</i> $\cdots$ O3 ⁱ	0.98	2.60	3.2636 (18)	125
C22—H22 <i>D</i> $\cdots$ O3 ⁱ	0.98	2.60	3.2636 (18)	126
C24—H24 $\cdots$ O3	0.95	2.55	2.9196 (16)	104
C1—H1 <i>A</i> $\cdots$ <i>Cg</i> 3 ⁱⁱ	0.99	2.94	3.6930 (14)	134
C4—H4 $\cdots$ <i>Cg</i> 5 ⁱⁱⁱ	0.95	2.86	3.7996 (14)	172
C29—H29 <i>C</i> $\cdots$ <i>Cg</i> 3 ^{iv}	0.98	2.97	3.6676 (17)	129

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