Crystal structure and Hirshfeld surface analysis of 
2,2,2-trichloro-N,N-bis[[1(RS,4SR)-1,4-dihydro-1,4-epoxynaphthalen-1-yl]methyl]acetamide

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In the title compound, C24H18Cl3NO3, the tetrahydrofuran rings adopt envelope conformations. In the crystal, C—H⋯O hydrogen bonds connect molecules, generating layers parallel to the (001) plane. These layers are connected along the c-axis direction by C—H⋯π interactions. The packing is further stabilized by interlayer van der Waals and interhalogen interactions. The most important contributions to the surface contacts are from H⋯C (36.8%), Cl⋯H⋯C (26.6%), C⋯H⋯C (18.8%) and O⋯H⋯O (11.3%) interactions, as concluded from a Hirshfeld surface analysis.

1. Chemical context

In recent years, the IMDAF cycloaddition (the intramolecular furan Diels–Alder reaction) in combination with other known reactions in a tandem or sequential manner is pursued for the construction of several important bicyclic or polycyclic compounds, including natural ones (for some reviews on this topic, see: Zubkov et al., 2005; Takao et al., 2005; Juhl et al., 2009; Padwa et al., 2013; Parvatkar et al., 2014; Krishna et al., 2021). Cascade sequences comprising two or more successive [4 + 2] cycloaddition steps are a powerful and frequently used protocol in modern syntheses aimed at constructing cyclohexene derivatives thanks to their exceptional chemo- selectivity, regioselectivity, diastereoselectivity, and capability to create more than four chiral centers in a single synthetic step (Criado et al., 2010, 2013). It has been shown previously that the Diels–Alder reaction of bis-dienes with derivatives of maleic acid, esters of acetylene dicarboxylic acid and hexa-

![Synthesis scheme for 2,2,2-trichloro-N,N-bis[[1(RS,4SR)-1,4-dihydro-1,4-epoxynaphthalen-1(4H)-yl]methyl]acetamide (1).](https://doi.org/10.1107/S2056989021009907)
fluoro-2-butyne proceeds in all cases diastereo- and chemoselectively and leads, depending on the temperature, to annelated diepoxynaphthalenes of the ‘domino’ or ‘pincer’ type (Borisova et al., 2018a,b; Grudova et al., 2020; Kyatkovskaya et al., 2020, 2021). In order to expand the limits of the applicability of the IMDAF strategy, we tested in this study dehydrobenzene generated in situ in the role of dienophile. It was demonstrated that the products of the parallel [4 + 2] cycloaddition of two aryne moieties to both the furan fragments of the bis-diene system (Fig. 1, 1 and 2) prevails over the adduct (3) of the IMDAF reaction (Fig. 1).

On the other hand, intermolecular non-covalent interactions organize the molecular aggregates, catalytic intermediates, etc., which play crucial roles for the functional properties of heterocyclic compounds (Gurbanov et al., 2020a,b; Khalilov et al., 2018a,b; Ma et al., 2017a,b, 2020, 2021; Mahmudov et al., 2020; Mizar et al., 2012). Thus, attached –CCl3 and C=O groups can participate in intermolecular interactions and affect the properties of 1-3.

2. Structural commentary
In the title compound (1, Fig. 2), the tetrahydrofuran rings (O19/C11–C14 and O29/C21–C24) adopt envelope conformations with the O atoms as the flaps. The molecular conformation is stabilized by intramolecular C10—H10A⋯O29 and C20—H20A⋯O19 hydrogen bonds and C20—H20B⋯C11 and C20—H20B⋯C13 interactions (Table 1).

3. Supramolecular features and Hirshfeld surface analysis
In the crystal, hydrogen bonds of the C—H⋯O type link the molecules, generating layers parallel to the (001) plane (Table 1; Figs. 3, 4, 5 and 6). These layers are connected by C—H⋯π interactions (C13—H13A⋯Cg8; Table 1), where Cg8 is

| Table 1
| Hydrogen-bond geometry (Å, °).
| Cg8 is the centroid of the C24A/C25⋯C28/C28A aromatic ring.
| D—H⋯A  | D—H  | H⋯A  | D⋯A  | D—H⋯A  |
|-------|------|------|------|--------|
| C10—H10A⋯O29 | 0.97 | 2.35 | 3.074 (2) | 131 |
| C12—H12A⋯O11 | 0.93 | 2.66 | 3.494 (2) | 150 |
| C17—H17A⋯O11 | 0.93 | 2.51 | 3.427 (3) | 168 |
| C20—H20A⋯O19 | 0.97 | 2.39 | 3.068 (2) | 127 |
| C27—H27A⋯O19b | 0.93 | 2.51 | 3.438 (3) | 175 |
| C20—H20B⋯C11 | 0.97 | 2.55 | 3.174 (4) | 122 |
| C20—H20B⋯C13 | 0.97 | 2.64 | 3.292 (19) | 125 |
| C13—H13A⋯Cg8 | 0.93 | 2.90 | 3.633 (2) | 136 |

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

Figure 2
The molecule of the title compound 1 with atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.

Figure 3
A general view of the intermolecular C—H⋯O hydrogen bonds and C—H⋯π interactions (depicted by dashed lines) in the unit cell of the title compound 1. [Symmetry codes: (a) 1/2 – x, – 1/2 + y, 1/2 – z; (b) 1/2 – x, 1/2 + y, 1/2 – z; (c) 1/2 – x, 1/2 + y, 1/2 – z; (d) 1/2 + x, 1/2 – y, 1/2 + z].
the centroid of the C24/C25–C28/C28 aromatic ring. The intermolecular interactions in the crystal of the title compound (Table 2) were quantified using Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots (McKinnon et al., 2007) were generated. The calculations and visualization were performed using CrystalExplorer17 (Turner et al., 2017). The three-dimensional Hirshfeld surface mapped over \(d_{\text{norm}}\) in the range \(-0.1862\) to \(+1.4233\) a.u. is shown in Fig. 7. The short and long contacts are indicated as red and blue spots, respectively, on the Hirshfeld surfaces, and contacts with distances approximately equal to the sum of the van der Waals radii are represented as white spots. The Cl···H and C···H···O interactions, which play a key role in the molecular packing, can be correlated with the bright-red patches near

| Contact | Distance | Symmetry operation |
|---------|----------|--------------------|
| Cl1···H10A | 3.10 | \(x, -1 + y, z\) |
| H20A···H25A | 2.44 | \(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z\) |
| H17A···O1 | 2.51 | \(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z\) |
| H23A···Cl2 | 3.07 | \(1 - x, 1 - y, -z\) |
| C28···H16A | 2.96 | \(-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z\) |
| H14A···C25 | 2.90 | \(-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z\) |
| H15A···H14A | 2.56 | \(-1 - x, 1 - y, 1 - z\) |
Cl1, Cl2, O1 and O19 and hydrogen atoms H14A and H16A, which highlight their functions as donors and/or acceptors. Fig. 8 shows the full two-dimensional fingerprint plot (Fig. 8a) and those delineated into the major contacts: H···H (36.8%, Fig. 8b) interactions are the major factor in the crystal packing together with Cl···H/H···Cl (26.6%, Fig. 8c), C···H/H···C (18.8%, Fig. 8d) and O···H/H···O (11.3%, Fig. 8e) interactions representing the next highest contributions. The percentage contributions of other weak interactions are listed in Table 3.

4. Database survey
A search of the Cambridge Structural Database (CSD version 5.40, update of September 2019; Groom et al., 2016) for structures having the epoxyisoindole moiety gave ten hits that closely resemble the title compound, viz. 4,5-dibromo-2-[4-(trifluoromethyl)phenyl]hexahydro-3a,6-epoxyisoindol-1(4H)-one (CSD refcode IQOTOA; Mertsalov et al., 2021), 3-hydroxy-2-[(2-(4-methylbenzene-1-sulfonyl)-2,3,7a-tetrahydro-3a,6-epoxyisoindol-6(1H)-yl][methyl]-2,3-dihydro-1H-isindol-1-one (OMUTAU; Mertsalov et al., 2021b), 2-benzyl-4,5-dibromohexahydro-3a,6-epoxyisoindol-1(4H)-one (OMEMAX; Mertsalov et al., 2021c), 4,5-dibromo-6-methyl-2-phenylhexahydro-3a,6-epoxyisoindol-1(4H)-one (IMUBIE; Mertsalov et al., 2021a), (3aR,6S,7aR)-7a-chloro-2-[(4-nitropheryl)sulfonyl]-1,2,3,6,7a-hexahydro-3a,6-epoxyisoindole (AGONUH; Temel et al., 2013), (3aR,6S,7aR)-7a-chloro-6methyl-2-[(4-nitropheryl)sulfonyl]-1,2,3,6,7a-hexahydro-3a,6-epoxyisoindole (TJMIK; Demircan et al., 2013), 5-chloro-7-methyl-3-[(4-methyl-phenyl)sulfonyl]-10-oxa-3-aza-tricyclo[5.2.1.01,5]dec-8-ene (YAXCIL; Temel et al., 2012), (3aR,6S,7aR)-7a-bromo-2-[(4-methylphenyl)sulfonyl]-1,2,3,6,7a-hexahydro-3a,6-epoxyisoindole (ERIVL; Temel et al., 2011) and tert-butyl 3a-chloroper-hydro-2,6a-epoxyxiren(e)isoindole-5-carboxylate (MGTIG; Koşar et al., 2007).

In the crystal of IQOTOA, the asymmetric unit consists of two crystallographically independent molecules. In both molecules, the pyrrolidine and tetrahydrofuran rings adopt

Table 3
Percentage contributions of interatomic contacts to the Hirshfeld surface for the title compound (1).

| Contact | Percentage contribution |
|---------|-------------------------|
| H···H   | 36.8                    |
| Cl···H/H···Cl | 26.6                   |
| C···H/H···C | 18.8                   |
| O···H/H···O | 11.3                   |
| Cl···Cl   | 4.4                     |
| Cl···O/O···Cl | 0.8                    |
| Cl···Cl   | 0.8                     |
| O···Cl/Cl | 0.4                     |
| C···Cl   | 0.1                     |

Figure 7
Hirshfeld surface of the title molecule 1 mapped with $d_{norm}$.
envelope conformations. In the crystal, molecules are linked in pairs by C—H···O hydrogen bonds. These pairs form a tetrameric supramolecular motif, leading to molecular layers parallel to the (100) plane formed by C—H···π and C—Br···π interactions. OMUTAU also crystallizes with two independent molecules in the asymmetric unit. In the central ring systems of both molecules, the tetrahydrofuran rings adopt twisted-envelope conformations and the six-membered ring is in a boat conformation. In both molecules, the nine-membered groups attached to the central ring system are essentially planar. In the crystal, strong intermolecular O—H···O hydrogen bonds, thus generating R2(18) rings. The crystal packing is dominated by H···H, Br···H, H···π and Br···π interactions. In the crystal structures of IQOTOA, OMUTAU, OMEMAX, AGONUH, TIJMIK, YAXCIL, UPAQE1 and ERIVIL, the molecules are predominantly linked by C—H···O hydrogen bonds, giving various hydrogen-bonding pattern connectivities. In the crystal of AGONUH, the molecules are connected in zigzag chains running along the b-axis direction. In TIJMIK, two types of C—H···O hydrogen bonds are found, viz. R2(20) and R2(26) rings, with adjacent rings running parallel to the ac plane. Additionally, C—H···O hydrogen bonds form a C(6) chain, linking the molecules in the b-axis direction. In the crystal of ERIVIL, the molecules are connected into R2(8) and R2(14) rings along the b-axis direction. In MIGTIG, the molecules are linked only by weak van der Waals interactions.

5. Synthesis and crystallization

CsF (1.7 g, 0.011 mol) was added to 2,2,2-trichloro-N,N-bis(furan-2-ylmethyl)acetamide (0.0022 mol) dissolved in dry CH3CN (20 mL). Then an equivalent of 2-(trimethylsilyl)phenyl trifluoromethanesulfonate (0.54 mL, 0.022 mol) was added to the solution under an argon atmosphere. The mixture was refluxed for 4 h (TLC control). After that, one more portion of 2-(trimethylsilyl)phenyl trifluoromethanesulfonate (0.27 mL, 0.011 mol) and CsF (1.7 g, 0.011 mol) was added to the mixture, repeating all procedures. After the mixture was cooled, CsF was filtered off through a thin layer of SiO2, and the resulting solution was concentrated under reduced pressure. The residue (brown oil) was separated using column chromatography on silica gel (a mixture EtOAc/hexane = 1/25 as eluent) to give compounds 1–3 in the ratio ~30/25/45. Single crystals of compound 1 was obtained by slow crystallization from a hexane/EtOAc mixture.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. All C-bound H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93–0.98 Å, and with Uiso(H) = 1.2Ueq(C). Six reflections (I01, 011, 101, 110, 002 and 200), which were observed by the beam stop, and nine outliers (343, 253, 7, 1.15, 3.6, 11, 15, 4.4, 072, 4.6, 12, 4.3, 22 and 13.6, 2) were omitted during the final refinement cycle.

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**Table 4**

**Experimental details.**

| Crystal data | C2H16Cl3NO4 | 474.74 |
|-------------|-------------|--------|
| Chemical formula | C2H16Cl3NO4 | 474.74 |
| M | 296 |
| Temperature (K) | 15.0134 (6), 8.1336 (3), 18.2841 (6) |
| R | 504.307 (2) |
| No. of parameters | 280 |
| R1, wR2 | 0.030, 0.652 |
| No. of reflections | 4991 |
| Crystal size (mm) | 0.34 x 0.18 x 0.14 |
| Crystal system, space group | Monoclinic, P21/c |
| Detector diffractometer | Bruker Kappa APEXII area-detector diffractometer |
| Absorption correction | Multi-scan (SADABS; Bruker, 2013) |
| Tmin, Tmax | 0.743, 0.940 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 17833, 4991, 3570 |
| No. of parameters | 4991 |
| H-atom treatment | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 0.30, -0.36 |

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXT (Sheldrick, 2015a), SHELX (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2015).
Acknowledgements

The authors’ contributions are as follows. Conceptualization, MA and AB; synthesis, GZM; X-ray analysis, ZA and GZM; writing (review and editing of the manuscript), ZA, GZM and MA; supervision, MA and AB.

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**Computing details**

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

**2,2,2-Trichloro-N,N-bis\{[(1RS,4SR)-1,4-dihydro-1,4-epoxynaphthalen-1-yl]methyl\}acetamide**

**Crystal data**

\[
\begin{align*}
\text{C}_{24}\text{H}_{18}\text{Cl}_3\text{NO}_3 \\
M_r &= 474.74 \\
\text{Monoclinic, } P2_1/n \\
a &= 15.0134 (6) \ \text{Å} \\
b &= 8.1336 (3) \ \text{Å} \\
c &= 18.2841 (6) \ \text{Å} \\
\beta &= 104.307 (2) ^\circ \\
V &= 2163.48 (14) \ \text{Å}^3 \\
Z &= 4
\end{align*}
\]

\[
\begin{align*}
F(000) &= 976 \\
D_r &= 1.458 \ \text{Mg} \ \text{m}^{-3} \\
\text{Mo } K\alpha \ \text{radiation, } \lambda &= 0.71073 \ \text{Å} \\
\text{Cell parameters from } 4831 \ \text{reflections} \\
\theta &= 2.8–26.7 ^\circ \\
\mu &= 0.45 \ \text{mm}^{-1} \\
T &= 296 \ \text{K} \\
\text{Fragment, colourless} \\
\text{0.34 } \times \text{ 0.18 } \times \text{ 0.14 mm}
\end{align*}
\]

**Data collection**

Bruker Kappa APEXII area-detector diffractometer

\[
\begin{align*}
\bar{\phi} \text{ and } \omega \text{ scans} \\
\text{Absorption correction: multi-scan} \quad \text{(SADABS; Bruker, 2013)} \\
T_{\text{min}} &= 0.743, \ T_{\text{max}} = 0.940 \\
17833 \ \text{measured reflections}
\end{align*}
\]

**Refinement**

Refinement on \(F^2\)

\[
\begin{align*}
\text{Least-squares matrix: full} \\
R(F^2) &= 0.030 \\
\omega R(F^2) &= 0.118 \\
S &= 1.01 \\
4991 \ \text{reflections} \\
280 \ \text{parameters} \\
0 \ \text{restraints}
\end{align*}
\]

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\[
\begin{align*}
w &= 1/[\sigma^2(F_c^2) + (0.0576P)^2 + 0.6103P] \\
\text{where } P &= (F_c^2 + 2F_e^2)/3 \\
(\Delta \sigma)_{\text{max}} &= 0.001 \\
\Delta \rho_{\text{max}} &= 0.30 \ \text{e} \ \text{Å}^{-3} \\
\Delta \rho_{\text{min}} &= -0.36 \ \text{e} \ \text{Å}^{-3}
\end{align*}
\]
Supporting Information

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)

| Atom | x    | y    | z    | U(eq) |
|------|------|------|------|-------|
| C1   | 0.57432 (13) | 0.4170 (2) | 0.17193 (10) | 0.0396 (4) |
| C2   | 0.53382 (14) | 0.2461 (3) | 0.14061 (12) | 0.0450 (5) |
| C10  | 0.57234 (13) | 0.6612 (2) | 0.24521 (11) | 0.0386 (4) |
| H10A | 0.533570    | 0.757230   | 0.231289     | 0.046*   |
| H10B | 0.628106    | 0.679444   | 0.228600     | 0.046*   |
| C11  | 0.59728 (12) | 0.6449 (2) | 0.32991 (11) | 0.0353 (4) |
| C12  | 0.64935 (13) | 0.7922 (2) | 0.37350 (12) | 0.0445 (5) |
| H12A | 0.686507    | 0.866559   | 0.356181     | 0.053*   |
| C13  | 0.63075 (14) | 0.7916 (2) | 0.44014 (12) | 0.0473 (5) |
| H13A | 0.651717    | 0.865687   | 0.479366     | 0.057*   |
| C14  | 0.56784 (13) | 0.6447 (2) | 0.43983 (11) | 0.0417 (4) |
| H14A | 0.530748    | 0.647835   | 0.477024     | 0.050*   |
| C14A | 0.62536 (12) | 0.4899 (2) | 0.44023 (11) | 0.0390 (4) |
| C15  | 0.65706 (14) | 0.3678 (3) | 0.49181 (12) | 0.0466 (5) |
| H15A | 0.643575    | 0.368000   | 0.538795     | 0.056*   |
| C16  | 0.71008 (15) | 0.2436 (3) | 0.47154 (13) | 0.0531 (5) |
| H16A | 0.731376    | 0.158196   | 0.505157     | 0.064*   |
| C17  | 0.73137 (15) | 0.2450 (3) | 0.40299 (13) | 0.0521 (5) |
| H17A | 0.767642    | 0.161342   | 0.391117     | 0.062*   |
| C18  | 0.69959 (13) | 0.3700 (2) | 0.35035 (12) | 0.0436 (4) |
| H18A | 0.714646    | 0.371239   | 0.303977     | 0.052*   |
| C18A | 0.64551 (11) | 0.4905 (2) | 0.36943 (10) | 0.0356 (4) |
| C20  | 0.42684 (12) | 0.5001 (2) | 0.20562 (10) | 0.0359 (4) |
| H20A | 0.422213    | 0.498360   | 0.257597     | 0.043*   |
| H20B | 0.404568    | 0.395232   | 0.183106     | 0.043*   |
| C21  | 0.36523 (12) | 0.6346 (2) | 0.16411 (10) | 0.0348 (4) |
| C22  | 0.37783 (13) | 0.7010 (3) | 0.08860 (11) | 0.0440 (4) |
| H22A | 0.405680    | 0.647375   | 0.055171     | 0.053*   |
| C23  | 0.34135 (14) | 0.8485 (3) | 0.08083 (13) | 0.0503 (5) |
| H23A | 0.338084    | 0.920840   | 0.040843     | 0.060*   |
| H24A | 0.30557 (14) | 0.8769 (2) | 0.15056 (12) | 0.0467 (5) |
| H24A | 0.296146    | 0.991967   | 0.162742     | 0.056*   |
| C24  | 0.22319 (13) | 0.7630 (2) | 0.14421 (10) | 0.0396 (4) |
| C25  | 0.12998 (14) | 0.7862 (3) | 0.13138 (11) | 0.0485 (5) |
| H25A | 0.104624    | 0.891139   | 0.125152     | 0.058*   |
| C26  | 0.07468 (15) | 0.6477 (3) | 0.12801 (12) | 0.0556 (6) |
| H26A | 0.011307    | 0.660262   | 0.118843     | 0.067*   |
| C27  | 0.11209 (14) | 0.4931 (3) | 0.13797 (12) | 0.0558 (6) |
| H27A | 0.073843    | 0.402457   | 0.136186     | 0.067*   |
|    |                  | U\(^{11}\)  | U\(^{22}\)  | U\(^{33}\)  | U\(^{12}\)  | U\(^{13}\)  | U\(^{23}\)  |
|----|------------------|------------|------------|------------|------------|------------|------------|
| C1 | 0.0400 (10)      | 0.0380 (10)| 0.0425 (10)| −0.0010 (8)| 0.0136 (8) | 0.0011 (8) |
| C2 | 0.0436 (11)      | 0.0415 (10)| 0.0510 (11)| 0.0025 (8) | 0.0138 (9) | −0.0063 (9)|
| C10| 0.0366 (10)      | 0.0304 (9) | 0.0486 (11)| −0.0056 (7)| 0.0101 (8) | 0.0000 (7) |
| C11| 0.0276 (8)       | 0.0296 (9) | 0.0489 (10)| −0.0020 (7)| 0.0096 (7) | −0.0022 (7)|
| C12| 0.0393 (11)      | 0.0305 (9) | 0.0603 (13)| −0.0053 (8)| 0.0060 (9) | −0.0042 (8)|
| C13| 0.0465 (12)      | 0.0361 (10)| 0.0552 (12)| −0.0006 (9)| 0.0051 (9) | −0.0105 (9)|
| C14| 0.0358 (10)      | 0.0437 (11)| 0.0449 (10)| −0.0001 (8)| 0.0088 (8) | −0.0059 (8)|
| C14A|0.0309 (9)        | 0.0356 (10)| 0.0485 (10)| −0.0048 (7)| 0.0062 (7) | −0.0043 (8)|
| C15| 0.0417 (12)      | 0.0463 (11)| 0.0489 (11)| −0.0066 (9)| 0.0056 (9) | 0.0023 (9) |
| C16| 0.0491 (12)      | 0.0401 (11)| 0.0632 (14)| 0.0007 (9) | 0.0006 (10)| 0.0081 (10)|
| C17| 0.0450 (12)      | 0.0366 (10)| 0.0707 (14)| 0.0091 (9) | 0.0067 (10)| −0.0039 (10)|
| C18| 0.0382 (10)      | 0.0389 (10)| 0.0536 (12)| 0.0009 (8) | 0.0109 (8) | −0.0047 (8)|
| C18A|0.0266 (8)        | 0.0312 (9) | 0.0473 (10)| −0.0040 (7)| 0.0060 (7) | −0.0008 (7)|
| C20| 0.0323 (9)       | 0.0329 (9) | 0.0431 (10)| −0.0024 (7)| 0.0102 (7) | 0.0010 (7) |
| C21| 0.0346 (9)       | 0.0309 (9) | 0.0383 (9) | −0.0023 (7)| 0.0082 (7) | −0.0031 (7)|
| C22| 0.0381 (10)      | 0.0508 (12)| 0.0455 (11)| −0.0010 (9)| 0.0150 (8) | 0.0056 (9)|
| C23| 0.0445 (11)      | 0.0458 (12)| 0.0601 (13)| −0.0024 (9)| 0.0118 (9) | 0.0166 (10)|
| C24| 0.0445 (11)      | 0.0323 (10)| 0.0595 (13)| 0.0040 (8) | 0.0058 (9) | −0.0005 (9)|
| C24A|0.0406 (10)       | 0.0413 (10)| 0.0370 (9) | 0.0020 (8) | 0.0098 (7) | −0.0010 (8)|
| C25| 0.0463 (12)      | 0.0594 (13)| 0.0416 (10)| 0.0136 (10)| 0.0142 (9) | 0.0030 (9)|
| C26| 0.0345 (10)      | 0.0849 (18)| 0.0496 (12)| 0.0021 (11)| 0.0149 (9) | 0.0111 (11)|
| C27| 0.0412 (11)      | 0.0735 (16)| 0.0533 (12)| −0.0185 (11)| 0.0125 (9) | 0.0043 (11)|
| C28| 0.0438 (11)      | 0.0458 (11)| 0.0446 (10)| −0.0064 (9)| 0.0106 (8) | 0.0017 (9)|
| C28A|0.0346 (9)        | 0.0410 (10)| 0.0302 (8) | −0.0022 (7)| 0.0093 (7) | −0.0020 (7)|
| N1 | 0.0325 (8)       | 0.0314 (8) | 0.0418 (8) | −0.0030 (6)| 0.0086 (6) | −0.0015 (6)|
| O1 | 0.0475 (9)       | 0.0566 (9) | 0.0848 (11)| −0.0086 (7)| 0.0368 (8) | −0.0112 (8)|
| O19| 0.0279 (6)       | 0.0422 (7) | 0.0460 (7) | 0.0007 (5) | 0.0081 (5) | −0.0044 (6)|
| O29| 0.0429 (8)       | 0.0311 (7) | 0.0497 (8) | 0.0011 (6) | 0.0011 (6) | −0.0067 (6)|
| C11| 0.0690 (4)       | 0.0354 (3) | 0.0743 (4) | −0.0035 (2)| 0.0234 (3) | 0.0056 (2)|
| C12| 0.0716 (4)       | 0.0686 (4) | 0.1080 (6) | 0.0077 (3) | 0.0409 (4) | −0.0319 (4)|
| C13| 0.0757 (4)       | 0.0686 (4) | 0.0586 (4) | −0.0006 (3)| −0.0076 (3)| −0.0129 (3)|
| Bond                  | Length (Å) | Torsion (°) |
|----------------------|------------|-------------|
| C1—O1                | 1.209 (2)  |             |
| C1—N1                | 1.351 (2)  |             |
| C1—C2                | 1.568 (3)  |             |
| C2—Cl2               | 1.755 (2)  |             |
| C2—Cl1               | 1.767 (2)  |             |
| C2—Cl3               | 1.770 (2)  |             |
| C10—N1               | 1.472 (2)  |             |
| C10—C11              | 1.506 (3)  |             |
| C10—H10A             | 0.9700     |             |
| C10—H10B             | 0.9700     |             |
| C11—O19              | 1.459 (2)  |             |
| C11—C18A             | 1.538 (2)  |             |
| C11—C12              | 1.540 (2)  |             |
| C12—C13              | 1.316 (3)  |             |
| C12—H12A             | 0.9300     |             |
| C13—C14              | 1.522 (3)  |             |
| C13—H13A             | 0.9300     |             |
| C14—O19              | 1.449 (2)  |             |
| C14—C14A             | 1.526 (3)  |             |
| C14—H14A             | 0.9800     |             |
| C14A—C15             | 1.371 (3)  |             |
| C14A—C18A            | 1.400 (3)  |             |
| C15—C16              | 1.392 (3)  |             |
| C15—H15A             | 0.9300     |             |
| C16—C17              | 1.368 (3)  |             |
| C16—H16A             | 0.9300     |             |
| C17—C18              | 1.400 (3)  |             |
| O1—C1—N1             | 123.52 (18)|             |
| O1—C1—C2             | 116.95 (17)|             |
| N1—C1—C2             | 119.42 (16)|             |
| C1—C2—Cl2            | 109.31 (13)|             |
| C1—C2—C11            | 110.74 (13)|             |
| C12—C2—C11           | 107.36 (11)|             |
| C1—C2—Cl3            | 110.99 (14)|             |
| C12—C2—C13           | 107.91 (11)|             |
| C11—C2—C13           | 110.41 (11)|             |
| N1—C10—C11           | 114.17 (14)|             |
| N1—C10—H10A          | 108.7      |             |
| C11—C10—H10A         | 108.7      |             |
| N1—C10—H10B          | 108.7      |             |
| C11—C10—H10B         | 108.7      |             |
| H10A—C10—H10B        | 107.6      |             |
| O19—C11—C10          | 112.73 (14)|             |
| O19—C11—C18A         | 98.64 (13) |             |
| C10—C11—C18A         | 121.53 (15)|             |
O19—C11—C12 99.35 (14)  C22—C23—H23A 127.1
C10—C11—C12 115.42 (15)  C24—C23—H23A 127.1
C18A—C11—C12 105.78 (15)  O29—C24—C23 100.59 (15)
C13—C12—C11 106.25 (17)  O29—C24—C24A 99.27 (15)
C13—C12—H12A 126.9  C23—C24—C24A 106.97 (16)
C11—C12—H12A 126.9  O29—C24—H24A 115.9
C12—C13—C14 105.83 (17)  C23—C24—H24A 115.9
C12—C13—H13A 127.1  C24A—C24—H24A 115.9
C14—C13—H13A 127.1  C25—C24A—C28A 121.17 (18)
O19—C14—C13 100.44 (15)  C25—C24A—C24 134.57 (19)
O19—C14—C14A 99.30 (14)  C28A—C24A—C24 104.24 (16)
C13—C14—C14A 107.32 (15)  C24A—C25—C26 117.9 (2)
O19—C14—H14A 115.8  C24A—C25—H25A 121.0
C13—C14—H14A 115.8  C26—C25—H25A 121.0
C14A—C14—H14A 115.8  C27—C26—C25 121.08 (19)
C15—C14A—C18A 121.38 (18)  C27—C26—H26A 119.5
C15—C14A—C14 134.49 (18)  C25—C26—H26A 119.5
C18A—C14A—C14 104.12 (16)  C26—C27—C28 120.9 (2)
C14A—C15—C16 117.8 (2)  C26—C27—H27A 119.6
C14A—C15—H15A 121.1  C28—C27—H27A 119.6
C16—C15—H15A 121.1  C28A—C28—C27 117.6 (2)
C17—C16—C15 121.1 (2)  C28A—C28—H28A 121.2
C17—C16—H16A 119.5  C27—C28—H28A 121.2
C15—C16—H16A 119.5  C28—C28A—C24A 121.36 (17)
C16—C17—C18 121.3 (2)  C28—C28A—C21 134.20 (17)
C16—C17—H17A 119.4  C24A—C28A—C21 104.43 (15)
C18—C17—H17A 119.4  C1—N1—C20 127.57 (15)
C18A—C18—C17 117.81 (19)  C1—N1—C10 116.44 (15)
C18A—C18—H18A 121.1  C20—N1—C10 115.99 (14)
C17—C18—H18A 121.1  C14—O19—C11 96.05 (13)
C18—C18A—C14A 120.62 (17)  C24—O29—C21 96.01 (13)

O1—C1—C2—C12 4.6 (2)  C21—C22—C23—C24 0.0 (2)
N1—C1—C2—C12 −171.75 (15)  C22—C23—C24—O29 33.4 (2)
O1—C1—C2—C11 122.72 (17)  C22—C23—C24—C24A −69.8 (2)
N1—C1—C2—C11 −53.7 (2)  O29—C24—C24A—C25 146.2 (2)
O1—C1—C2—C13 −114.28 (18)  C23—C24—C24A—C25 −109.7 (2)
N1—C1—C2—C13 69.3 (2)  O29—C24—C24A—C28A −35.51 (18)
N1—C10—C11—O19 −67.92 (19)  C23—C24—C24A—C28A 68.64 (19)
N1—C10—C11—C18A 48.7 (2)  C28A—C24A—C25—C26 0.7 (3)
N1—C10—C11—C12 178.87 (15)  C24—C24A—C25—C26 178.8 (2)
O19—C11—C12—C13 32.80 (19)  C24A—C25—C26—C27 0.7 (3)
C10—C11—C12—C13 153.59 (17)  C25—C26—C27—C28 −0.9 (3)
C18A—C11—C12—C13 −69.00 (19)  C26—C27—C28—C28A −0.3 (3)
C11—C12—C13—C14 0.4 (2)  C27—C28—C28A—C24A 1.7 (3)
C12—C13—C14—O19 −33.89 (19)  C27—C28—C28A—C21 −179.31 (18)
C12—C13—C14—C14A 69.4 (2)  C25—C24A—C28A—C28 −1.9 (3)
O19—C14—C14A—C15 −144.6 (2)  C24—C24A—C28A—C28 179.46 (17)

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### C13—C14—C14A—C15 111.3 (2) C25—C24A—C28A—C21 178.80 (17)
O19—C14—C14A—C18A 36.13 (17) C24—C24A—C28A—C21 0.19 (18)
C13—C14—C14A—C18A −67.96 (18) O29—C21—C28A—C28A −144.1 (2)
C18A—C14A—C15—C16 −0.1 (3) C20—C21—C28A—C28A −23.4 (3)
C14—C14A—C15—C16 −179.27 (19) C22—C21—C28A—C28A 113.2 (2)
C14A—C15—C16—C17 1.3 (3) O29—C21—C28A—C24A 35.00 (16)
C15—C16—C17—C18 −0.9 (3) C20—C21—C28A—C24A 155.77 (15)
C16—C17—C18—C18A −0.6 (3) C22—C21—C28A—C24A −67.64 (17)
C17—C18—C18A—C14A 1.8 (3) O1—C1—N1—C20 172.92 (18)
C17—C18—C18A—C11 −179.78 (19) C2—C1—N1—C20 −10.9 (3)
C15—C14A—C18A—C11 1.5 (3) O1—C1—N1—C10 −6.9 (3)
C14—C14A—C18A—C11 −179.68 (16) C2—C1—N1—C10 169.28 (16)
C15—C14A—C18A—C11 179.68 (16) C21—C20—N1—C1 −114.5 (2)
C14—C14A—C18A—C11 −0.92 (17) C21—C20—N1—C10 65.2 (2)
O19—C11—C18A—C18 147.2 (2) C11—C10—N1—C1 −104.29 (19)
C10—C11—C18A—C18 23.7 (3) C11—C10—N1—C20 75.91 (19)
C12—C11—C18A—C18 −110.5 (2) C13—C14—O19—C11 52.63 (15)
O19—C11—C18A—C14A −34.20 (16) C14A—C14—O19—C11 −57.06 (15)
C10—C11—C18A—C14A −157.68 (16) C10—C11—O19—C14 174.45 (14)
C12—C11—C18A—C14A 68.14 (18) C18A—C11—O19—C14 55.97 (14)
N1—C20—C21—O29 −76.82 (19) C12—C11—O19—C14 −51.72 (15)
N1—C20—C21—C22 40.7 (2) C23—C24—O29—C21 −52.34 (16)
N1—C20—C21—C28A −170.63 (14) C24A—C24—O29—C21 57.01 (16)
O29—C21—C22—C23 −33.12 (19) C20—C21—O29—C24 178.99 (15)
C20—C21—C22—C23 −157.33 (17) C22—C21—O29—C24 51.68 (16)
C28A—C21—C22—C23 68.77 (19) C28A—C21—O29—C24 −56.40 (15)

**Hydrogen-bond geometry (Å, °)**

Cg8 is the centroid of the C24A/C25–C28/C28A aromatic ring.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| C10—H10.4···O29 | 0.97 | 2.35 | 3.074 (2) | 131 |
| C12—H12.4···O1i | 0.93 | 2.66 | 3.494 (2) | 150 |
| C17—H17.4···O1ii | 0.93 | 2.51 | 3.427 (3) | 168 |
| C20—H20.4···O19 | 0.97 | 2.39 | 3.068 (2) | 127 |
| C27—H27.4···O19v | 0.93 | 2.51 | 3.438 (3) | 175 |
| C20—H20B···Cl1 | 0.97 | 2.55 | 3.1744 (18) | 122 |
| C20—H20B···Cl3 | 0.97 | 2.64 | 3.2921 (19) | 125 |
| C13—H13A···Cg8vi | 0.93 | 2.90 | 3.633 (2) | 136 |

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