Fluorenophane chlorobenzene solvate: molecular and crystal structures

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The title compound, \(1^H,7^H,3,5,9,11\)-tetraoxa-1,7(2,7)-difluorena-4,10(1,3)-dibenzenacyclododecaphane-1,7-dione (fluorenophane), exists as a solvate with chlorobenzene, \(C_8H_8O_6/C_6H_5Cl\). The fluorenophane contains two fluorenone fragments linked by two \(m\)-substituted benzene fragments. Some decrease in its macrocyclic cavity leads to a stacking interaction between the tricyclic fluorenone fragments. In the crystal, the fluorenophane and chlorobenzene molecules are linked by weak C—H\(\cdots\)C Cl hydrogen bonds. The Cl atom of chlorobenzene does not form a halogen bond. A Hirshfeld surface analysis and two-dimensional fingerprint plots were used to analyse the intermolecular contacts found in the crystal structure.

1. Chemical context

Discovered at the end of the last century, the ability of cyclophanes to form inclusion complexes makes them the central class of synthetic receptors in molecular recognition processes (Diederich, 1991). Particular attention has been paid to the possibility of cationic cyclophanes with box geometries being involved in strong donor–acceptor interactions leading to the formation of ‘guest–host’ complexes with different guests (Dale et al., 2016; Barnes et al., 2013; Gong et al., 2010). Previously we have obtained fluorenophane 1 with two fluorenone fragments linked by rigid xylyl groups (Lukyanenko et al., 2003; Simonov et al., 2006). X-ray diffraction analysis of this cyclophane revealed the box geometry with an open intramolecular cavity and the formation of inclusion complexes with DMF and nitrobenzene (Simonov et al., 2006). The other fluorenophane obtained by our group, 2, differs from the previous one in the position of the methylene groups, which are located directly at the benzene fragment in 1 or fluorenone in 2. Fluorenophane 2 forms inclusion complexes with chloroform and bromoform with a 1:2 stoichiometry. Moreover, C—Cl\(\cdots\)\(\pi\) and C—Br\(\cdots\)\(\pi\) halogen bonds (Shishkina et al., 2021) are present in the complexes. In contrast to cationic cyclophanes, there are no charged fragments in fluorenophanes. Continuing our research in this area, we have obtained fluorenophane 3 with a different position of attachment of the benzene rings compared to 2 (\(m\)- and \(p\)-isomers, respectively) and studied its complexation with chlorobenzene.
2. Structural commentary

Fluorenophane 3 was crystallized from chlorobenzene and exists in the crystal as a solvate in a 1:1 ratio rather than as an inclusion complex. Fluorenophane 3 contains two fluorenone fragments linked by two 1-substituted benzene fragments (Fig. 1). The macrocycle 3 has a boat conformation similar to structure 1 [the torsion angles C41—O6—C1—C2, C37—O5—C36—C33, C20—O3—C22—C23, and C16—O2—C15—C13 are −90.6 (4), 78.4 (4), −80.0 (4) and 91.6 (4)°, respectively]. In structure 3, the fluorenone fragments are oriented in the same directions (cis-orientation) while the orientation of these fragments is trans in structures 1 and 2. meta-Substitution of the two benzene fragments results in a smaller macrocycle cavity as compared to fluorenophanes 1 and 2 with para-substituted benzene fragments. As a result, the two fluorenones are slightly bowed inwards [the dihedral angle between C2–C7 and C8–C14 benzene rings is 12.51 (18)° in one fluorenone while the dihedral angle between the C31–C35 and C23–C28 benzene rings is 9.64 (18)° in the other fluorenone]. This can be explained by a π-stacking interaction between the C10—O1 carbonyl group and the C25/C26/C31/C30/C29 fluorenone ring [centroid Cg2, with O1...Cg2 = 3.469 (3) Å, C10...Cg2 = 3.492 (4) Å, C10...O1...Cg2 = 81.1 (2)°]. In contrast to structures 1 and 2, the macrocycle in structure 3 does not contain any molecules inside its cavity. Therefore, the structure under study is a chlorobenzene solvate of fluorenophane.

3. Supramolecular features

In the crystal, the fluorenophane and chlorobenzene molecules are linked to each other by weak C46—H46⋯O6 and C18—H18⋯C1 hydrogen bonds while the fluorenophanes are linked by weak C35—H35⋯O1 hydrogen bonds (Table 1), forming stepped ribbons. The ribbons are connected by C1—H1⋯Cl1 hydrogen bonds while the fluorenophanes are linked by weak C35—H35⋯O1 hydrogen bonds (Table 1), forming stepped ribbons. The ribbons are connected by C1—H1⋯Cl1 interactions (Table 1) to give the final three-dimensional structure. The halogen atom does not form a halogen bond in the structure of 3, in contrast to the supramolecular complexes studied earlier (Shishkina et al., 2021). The electrostatic potential for chlorobenzene was calculated using the B3LYP/6–311G(d,p) method. An area with a positive charge (σ-hole) was not found in the electrostatic potential map around the halogen atom (Fig. 2). The highest electrostatic potential at the chlorine atom is −0.08 eV. This fact can explain the absence of halogen bonds in the structure of 3.

4. Hirshfeld surface analysis

Crystal Explorer 17.5 (Turner et al., 2017) was used to analyze interactions in the crystal. Molecular Hirshfeld surfaces mapped over dnorm with a standard (high) surface resolution.

Figure 1
The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Figure 2
Electrostatic potential map of the chlorobenzene molecule in 3 calculated by the B3LYP/6-311G(d,p) method.
and a fixed colour scale of $-0.134$ (red) to $1.206$ (blue) were generated separately (Fig. 3) for the fluorenonophane and chlorobenzene molecules. The areas in red correspond to contacts that are shorter than the sum of the van der Waals radii of the closest atoms. Thus, the red spots at some hydrogen atoms and at the carbonyl oxygen atom as well as in the area of the five-membered ring indicate the existence of short C–H···O and C–H···π(ring) contacts.

5. Database survey
A search of the Cambridge Structural Database (CSD, Version 5.42, update of November 2020; Groom et al., 2016) for cyclophanes containing fluorenone and benzene fragments yielded two hits: two structures with fluorenone fragments linked by rigid xylol groups (CCDC 263272 and CCDC 263273; Simonov et al., 2006). Recently, two more structures with fluorenonophanes linked by para-substituted benzene fragments were published (CCDC 647971 and CCDC 2098245; Shishkina et al., 2021). The structures found are characterized by a larger macrocyclic cavity compared to that in fluorenonophane 3.

6. Synthesis and crystallization
A solution of 1.75 g (4.78 mmol) of 2,7-bis(bromomethyl)-9H-fluoren-9-one (Haenel et al., 1985) in 200 mL of anhydrous DMF was added to a mixture of 0.526 g (4.78 mmol) of resorcinol and 3.96 g (28.7 mmol) of K$_2$CO$_3$ in 270 mL of anhydrous DMF with stirring under nitrogen for 10 h at 353–358 K. The reaction mixture was stirred at the same temperature for a further 35 h, cooled and filtered (Fig. 5). The precipitate was washed with DMF and the filtrate was evaporated under reduced pressure. The residue was dissolved

Figure 3
Hirshfeld surface mapped over $d_{norm}$, showing the conformation of the fluorenonophane and chlorobenzene molecules.

Figure 4
The two-dimensional fingerprint plots for fluorenonophane 3 (top) and chlorobenzene (bottom).
in CHCl₃ and washed with an aqueous sodium carbonate solution (50 mL), then with water (3 × 50 mL) to a neutral pH. After drying over MgSO₄, the CHCl₃ was evaporated under reduced pressure. The product was purified by chromatography on silica gel (Acros 0.060, EtOH, 500:1. The yield of cyclophane analyzed for C₄₂H₂₈O₆: C, 80.24; H, 4.49. Found: C, 80.44; H, 4.76%. Crystals were obtained by crystallization of fluorenophane 3 from chlorobenzene.

7. Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 2. Carbon-bound H atoms were added in calculated positions with C–H bond lengths of 0.95 Å for C–H, 0.92 Å for CH₂ and refined as riding atoms with Uiso(H) = 1.2Ueq(C).

Funding information

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Table 2

| Crystal data | Chemical formula | C₄₂H₂₈O₆Cl₂CH₂Cl |
|--------------|------------------|------------------|
| M₀ | 741.19 |
| Crystal system, space group | Triclinic, P1 |
| Temperature (K) | 100 |
| a, b, c (Å) | 6.2278 (6), 9.6965 (8), 14.9822 (13) |
| α, β, γ (°) | 105.288 (8), 97.126 (7), 96.919 (7) |
| V (Å³) | 854.83 (13) |
| Z | 1 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.17 |
| Crystal size (mm) | 0.6 × 0.4 × 0.2 |

Data collection

Diffractometer | Xcalibur, Sapphire3 |
Absorption correction | Multi-scan (CrysAlis PRO; Rigaku OD, 2018) |

Refinement

R[F² > 2σ(F²)], wR(F²), S | 0.064, 0.171, 1.03 |
No. of reflections | 7191 |
No. of parameters | 496 |
No. of restraints | 3 |
H-atom treatment | H-atom parameters constrained |
Δρ_{max}, Δρ_{min} (e Å⁻³) | 0.79, −0.42 |
Absolute structure | Flack x determined using 564 quotients | (Parsons et al., 2013) |
Absolute structure parameter | 0.19 (9) |

Computer programs: CrysAlis PRO (Rigaku OD, 2018), SHELXT (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b) and OLEX2 (Dolomanov et al., 2009).

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supporting information

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Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2018); cell refinement: CrysAlis PRO (Rigaku OD, 2018); data reduction: CrysAlis PRO (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

19H,79H-3,5,9,11-Tetraoxa-1,7(2,7)-difluorena-4,10(1,3)-dibenzenacyclododecaphane-19,79-dione chlorobenzene monosolvate

Crystal data

C42H28O6·C6H5Cl

Mr = 741.19

Triclinic, P1

a = 6.2278 (6) Å
b = 9.6965 (8) Å

M = 14.9822 (13) Å

α = 105.288 (8)°
β = 97.126 (7)°
γ = 96.919 (7)°

V = 854.83 (13) Å³

Z = 1

F(000) = 386

Dx = 1.440 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 1987 reflections

θ = 3.9–33.0°

μ = 0.17 mm⁻¹

T = 100 K

Block, colourless

0.6 × 0.4 × 0.2 mm

Data collection

Xcalibur, Sapphire3
diffractometer

Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.1827 pixels mm⁻¹

Absorption correction: multi-scan, CrysAlisPro; Rigaku OD, 2018

Tmin = 0.846, Tmax = 1.000

8226 measured reflections

7191 independent reflections

5307 reflections with I > 2σ(I)

Rp = 0.028
θmax = 35.0°, θmin = 3.0°

h = −9→8

k = −7→15

l = −24→20

Refinement

Refinement on F²
Least-squares matrix: full

R[F² > 2σ(F²)] = 0.064

wR(F²) = 0.171

S = 1.03

7191 reflections

496 parameters

3 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(Fc)² + (0.0825P)² + 0.017P]

where P = (Fc² + 2Fh²)/3

(Δ σ)max < 0.001

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Δρ_{max} = 0.79 \text{ e Å}^{-3}  \\
Δρ_{min} = -0.42 \text{ e Å}^{-3}

Absolute structure: Flack x determined using 564 quotients [(I)−(I)]/[(I)+(I)] (Parsons et al., 2013)

Absolute structure parameter: 0.19 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

|   | x      | y      | z      | U_{iso}*/U_{eq} |
|---|--------|--------|--------|-----------------|
| O1| 0.3386 (4) | 0.2409 (3) | 0.30318 (19) | 0.0294 (6) |
| O2| 0.3907 (5) | 0.5919 (3) | 0.66155 (19) | 0.0298 (6) |
| O3| 0.3627 (5) | 0.9250 (3) | 0.47327 (18) | 0.0265 (5) |
| O4| 0.3771 (4) | 0.5007 (3) | 0.14591 (18) | 0.0265 (5) |
| O5| 0.0097 (4) | -0.0028 (3) | -0.12980 (18) | 0.0264 (5) |
| O6| 0.0323 (4) | -0.3103 (3) | 0.07559 (18) | 0.0259 (5) |
| C1| -0.1767 (6) | -0.2869 (4) | 0.0994 (3) | 0.0245 (7) |
| H1A| -0.238920 | -0.369009 | 0.120950 | 0.029* |
| H1B| -0.275117 | -0.286764 | 0.042198 | 0.029* |
| C2| -0.1747 (6) | -0.1482 (4) | 0.1741 (2) | 0.0239 (7) |
| C3| -0.3613 (6) | -0.1340 (4) | 0.2162 (3) | 0.0266 (7) |
| H3| -0.485939 | -0.207766 | 0.193390 | 0.032* |
| C4| -0.3703 (6) | -0.0156 (4) | 0.2902 (3) | 0.0267 (7) |
| H4| -0.498316 | -0.007812 | 0.318459 | 0.032* |
| C5| -0.1889 (6) | 0.0909 (4) | 0.3217 (2) | 0.0222 (6) |
| C6| -0.0046 (6) | 0.0796 (4) | 0.2766 (2) | 0.0220 (6) |
| C7| 0.0060 (6) | -0.0388 (4) | 0.2035 (2) | 0.0224 (6) |
| H7| 0.132576 | -0.045426 | 0.174069 | 0.027* |
| C8| -0.1381 (6) | 0.2194 (4) | 0.4039 (2) | 0.0225 (6) |
| C9| 0.0774 (6) | 0.2848 (4) | 0.4097 (2) | 0.0244 (7) |
| C10| 0.1658 (6) | 0.2076 (4) | 0.3267 (2) | 0.0231 (7) |
| C11| -0.2608 (6) | 0.2752 (4) | 0.4711 (3) | 0.0261 (7) |
| H11| -0.410474 | 0.235645 | 0.465987 | 0.031* |
| C12| -0.1588 (7) | 0.3914 (4) | 0.5467 (3) | 0.0278 (7) |
| H12| -0.242323 | 0.432694 | 0.592837 | 0.033* |
| C13| 0.0608 (6) | 0.4491 (4) | 0.5571 (2) | 0.0250 (7) |
| C14| 0.1794 (6) | 0.3979 (4) | 0.4864 (3) | 0.0244 (7) |
| H14| 0.327603 | 0.439670 | 0.490436 | 0.029* |
| C15| 0.1603 (7) | 0.5576 (4) | 0.6484 (3) | 0.0290 (8) |
| H15A| 0.097362 | 0.647655 | 0.652418 | 0.035* |
| H15B| 0.118770 | 0.519599 | 0.700067 | 0.035* |
| C16| 0.4764 (6) | 0.7076 (4) | 0.6331 (2) | 0.0251 (7) |
| C17| 0.6890 (7) | 0.7711 (4) | 0.6749 (3) | 0.0298 (8) |
| H17| 0.766706 | 0.736390 | 0.720920 | 0.036* |
| C18| 0.7850 (7) | 0.8861 (5) | 0.6479 (3) | 0.0316 (8) |
| Atom  | U11  | U22  | U33  | U12  | U13  | U23  |
|-------|------|------|------|------|------|------|
| H18   | 0.930083 | 0.931503 | 0.676396 | 0.038* |       |      |
| C19   | 0.6746 (6) | 0.9367 (4) | 0.5802 (3) | 0.0297 (8) |      |      |
| H19   | 0.743820 | 1.015419 | 0.561995 | 0.036* |       |      |
| C20   | 0.4627 (6) | 0.8717 (4) | 0.5394 (2) | 0.0245 (7) |      |      |
| C21   | 0.3627 (6) | 0.7559 (4) | 0.5661 (2) | 0.0256 (7) |      |      |
| H21   | 0.217055 | 0.710835 | 0.538118 | 0.031* |       |      |
| C22   | 0.1320 (6) | 0.8832 (4) | 0.4482 (3) | 0.0260 (7) |      |      |
| H22A  | 0.071510 | 0.954612 | 0.419840 | 0.031* |       |      |
| H22B  | 0.068110 | 0.886179 | 0.505817 | 0.031* |       |      |
| C23   | 0.0619 (6) | 0.7348 (4) | 0.3860 (2) | 0.0231 (7) |      |      |
| C24   | 0.1890 (6) | 0.6774 (4) | 0.3150 (2) | 0.0242 (7) |      |      |
| H24   | 0.329679 | 0.727598 | 0.314978 | 0.029* |       |      |
| C25   | 0.1081 (6) | 0.5461 (4) | 0.2498 (2) | 0.0220 (6) |      |      |
| C26   | −0.0988 (6) | 0.4699 (4) | 0.2487 (2) | 0.0222 (6) |      |      |
| C27   | −0.2240 (6) | 0.5237 (4) | 0.3154 (3) | 0.0260 (7) |      |      |
| H27   | −0.362640 | 0.471796 | 0.316539 | 0.031* |       |      |
| C28   | −0.1408 (6) | 0.6572 (4) | 0.3816 (2) | 0.0250 (7) |      |      |
| H28   | −0.224639 | 0.695843 | 0.428392 | 0.030* |       |      |
| C29   | 0.2013 (6) | 0.4666 (4) | 0.1678 (2) | 0.0230 (6) |      |      |
| C30   | 0.0310 (6) | 0.3407 (4) | 0.1173 (2) | 0.0236 (7) |      |      |
| C31   | −0.1468 (6) | 0.3419 (4) | 0.1661 (2) | 0.0222 (6) |      |      |
| C32   | 0.0253 (6) | 0.2392 (4) | 0.0333 (2) | 0.0232 (6) |      |      |
| H32   | 0.148081 | 0.238534 | 0.001502 | 0.028* |       |      |
| C33   | −0.1620 (6) | 0.1380 (4) | −0.0043 (3) | 0.0243 (7) |      |      |
| C34   | −0.3346 (6) | 0.1355 (4) | 0.0463 (3) | 0.0260 (7) |      |      |
| C35   | −0.459675 | 0.062432 | 0.021745 | 0.031* |       |      |
| C36   | −0.3285 (6) | 0.2373 (4) | 0.1318 (3) | 0.0258 (7) |      |      |
| C37   | −0.447445 | 0.234509 | 0.165682 | 0.031* |       |      |
| C38   | −0.1881 (6) | 0.0386 (4) | −0.1026 (2) | 0.0261 (7) |      |      |
| C39   | −0.292015 | −0.049858 | −0.107536 | 0.031* |       |      |
| C36B  | −0.125393 | 0.087410 | −0.147047 | 0.031* |       |      |
| C37   | 0.0872 (6) | −0.1085 (4) | −0.0968 (2) | 0.0243 (7) |      |      |
| C38   | 0.2524 (6) | −0.1682 (4) | −0.1384 (3) | 0.0294 (8) |      |      |
| H38   | 0.305188 | −0.136765 | −0.187734 | 0.035* |       |      |
| C39   | 0.3404 (6) | −0.2748 (4) | −0.1071 (3) | 0.0296 (8) |      |      |
| C39   | 0.454620 | −0.316585 | −0.135380 | 0.036* |       |      |
| C40   | 0.2643 (6) | −0.3216 (4) | −0.0352 (3) | 0.0292 (8) |      |      |
| H40   | 0.325432 | −0.394801 | −0.014078 | 0.035* |       |      |
| C41   | 0.0986 (6) | −0.2601 (4) | 0.0050 (2) | 0.0250 (7) |      |      |
| C42   | 0.0074 (6) | −0.1549 (4) | −0.0254 (3) | 0.0246 (7) |      |      |
| H42   | −0.108662 | −0.114593 | 0.002177 | 0.030* |       |      |
| C11   | 0.2053 (2) | 0.17453 (13) | 0.65929 (9) | 0.0542 (4) |      |      |
| C43   | 0.4128 (7) | 0.2615 (4) | 0.7525 (3) | 0.0306 (8) |      |      |
| C44   | 0.3680 (7) | 0.2937 (5) | 0.8418 (3) | 0.0320 (8) |      |      |
| H44   | 0.224785 | 0.265769 | 0.853168 | 0.038* |       |      |
| C45   | 0.5314 (8) | 0.3668 (5) | 0.9151 (3) | 0.0348 (9) |      |      |
| H45   | 0.502082 | 0.389116 | 0.977568 | 0.042* |       |      |
| C46   | 0.7375 (8) | 0.4077 (5) | 0.8978 (3) | 0.0406 (10) |      |      |
| H46  | 0.850033 | 0.460205 | 0.948342 | 0.049* |
|------|----------|----------|----------|--------|
| C47  | 0.7812 (9) | 0.3732 (6) | 0.8083 (4) | 0.0484 (12) |
| H47  | 0.924417 | 0.401211 | 0.796899 | 0.058* |
| C48  | 0.6189 (9) | 0.2979 (5) | 0.7341 (3) | 0.0418 (11) |
| H48  | 0.649317 | 0.271929 | 0.671735 | 0.050* |

Atomic displacement parameters (Å²)

|         | \(U_{11}^{2}\) | \(U_{22}^{2}\) | \(U_{33}^{2}\) | \(U_{12}\) | \(U_{13}\) | \(U_{23}\) |
|---------|----------------|----------------|----------------|----------|----------|----------|
| O1      | 0.0216 (13)    | 0.0295 (14)    | 0.0336 (14)    | −0.0023 (10) | 0.0080 (10) | 0.0040 (11)|
| O2      | 0.0278 (14)    | 0.0216 (12)    | 0.0279 (13)    | −0.0000 (10) | 0.0036 (10) | 0.0015 (9) |
| O3      | 0.0219 (13)    | 0.0244 (13)    | 0.0315 (13)    | 0.0000 (10)  | 0.0054 (10) | 0.0058 (10)|
| O4      | 0.0267 (14)    | 0.0236 (12)    | 0.0270 (12)    | −0.0000 (10) | 0.0049 (10) | 0.0057 (10)|
| O5      | 0.0216 (17)    | 0.0219 (16)    | 0.0274 (15)    | 0.0000 (12)  | 0.0033 (13) | 0.0072 (12)|
| O6      | 0.0211 (17)    | 0.0246 (17)    | 0.0348 (18)    | −0.0000 (13) | 0.0064 (13) | 0.0077 (14)|
| C1      | 0.0204 (16)    | 0.0256 (17)    | 0.0326 (18)    | −0.0005 (13) | 0.0067 (13) | 0.0064 (14)|
| C2      | 0.0227 (17)    | 0.0215 (16)    | 0.0219 (15)    | 0.0037 (12)  | 0.0035 (12) | 0.0052 (12)|
| C3      | 0.0212 (16)    | 0.0236 (12)    | 0.0227 (15)    | 0.0016 (12)  | 0.0042 (12) | 0.0074 (12)|
| C4      | 0.0210 (16)    | 0.0221 (16)    | 0.0232 (15)    | 0.0006 (12)  | 0.0052 (12) | 0.0053 (12)|
| C5      | 0.0222 (16)    | 0.0214 (16)    | 0.0233 (15)    | 0.0009 (12)  | 0.0072 (12) | 0.0048 (12)|
| C6      | 0.0242 (17)    | 0.0220 (16)    | 0.0262 (17)    | 0.0018 (13)  | 0.0055 (13) | 0.0056 (13)|
| C7      | 0.0206 (16)    | 0.0224 (16)    | 0.0245 (16)    | 0.0021 (12)  | 0.0032 (12) | 0.0044 (12)|
| C8      | 0.0225 (17)    | 0.0262 (18)    | 0.0282 (17)    | 0.0003 (13)  | 0.0065 (13) | 0.0058 (13)|
| C9      | 0.0289 (19)    | 0.0269 (18)    | 0.0275 (17)    | 0.0025 (14)  | 0.0083 (14) | 0.0067 (14)|
| C10     | 0.0271 (18)    | 0.0231 (17)    | 0.0237 (16)    | −0.0005 (13) | 0.0044 (13) | 0.0064 (13)|
| C11     | 0.0258 (18)    | 0.0199 (16)    | 0.0263 (16)    | 0.0007 (13)  | 0.0040 (13) | 0.0061 (12)|
| C12     | 0.032 (2)      | 0.0248 (17)    | 0.0268 (18)    | −0.0030 (14) | 0.0045 (14) | 0.0051 (14)|
| C13     | 0.0253 (17)    | 0.0234 (16)    | 0.0229 (16)    | 0.0005 (13)  | 0.0049 (13) | 0.0009 (12)|
| C14     | 0.0252 (18)    | 0.032 (2)      | 0.0271 (17)    | 0.0011 (14)  | 0.0012 (13) | 0.0030 (14)|
| C15     | 0.0242 (18)    | 0.035 (2)      | 0.0291 (18)    | −0.0017 (14) | 0.0027 (14) | 0.0006 (15)|
| C16     | 0.0227 (18)    | 0.0254 (18)    | 0.0348 (19)    | −0.0057 (13) | 0.0051 (14) | 0.0019 (14)|
| C17     | 0.0242 (17)    | 0.0224 (16)    | 0.0252 (16)    | 0.0018 (12)  | 0.0055 (12) | 0.0041 (12)|
| C18     | 0.0254 (17)    | 0.0243 (16)    | 0.0243 (16)    | −0.0028 (13) | 0.0027 (12) | 0.0022 (12)|
| C19     | 0.0239 (17)    | 0.0257 (17)    | 0.0255 (16)    | 0.0027 (13)  | 0.0027 (13) | 0.0031 (13)|
| C20     | 0.0229 (17)    | 0.0220 (16)    | 0.0240 (16)    | 0.0032 (13)  | 0.0021 (12) | 0.0067 (12)|
| C21     | 0.0222 (17)    | 0.0242 (16)    | 0.0235 (16)    | −0.0005 (13) | 0.0045 (12) | 0.0036 (12)|
| C22     | 0.0189 (16)    | 0.0238 (16)    | 0.0227 (15)    | 0.0011 (12)  | 0.0039 (12) | 0.0062 (12)|
| C23     | 0.0189 (15)    | 0.0236 (16)    | 0.0207 (15)    | 0.0000 (12)  | 0.0018 (11) | 0.0024 (12)|
| C24     | 0.0214 (16)    | 0.0218 (15)    | 0.0238 (15)    | 0.0027 (12)  | 0.0039 (12) | 0.0073 (12)|
| C25     | 0.0212 (16)    | 0.0238 (16)    | 0.0245 (16)    | 0.0021 (12)  | 0.0043 (12) | 0.0069 (13)|
| C26     | 0.0228 (17)    | 0.0223 (16)    | 0.0273 (16)    | 0.0005 (12)  | 0.0025 (13) | 0.0083 (13)|
Geometric parameters (Å, °)

| Bond                  | Distance  | Angle         |
|-----------------------|-----------|---------------|
| O1—C10                | 1.207 (4) |               |
| O2—C15                | 1.410 (5) |               |
| O2—C16                | 1.374 (5) |               |
| O3—C20                | 1.353 (5) |               |
| O3—C22                | 1.420 (5) |               |
| O4—C29                | 1.214 (4) |               |
| O5—C36                | 1.415 (5) |               |
| O5—C37                | 1.362 (4) |               |
| O6—C1                 | 1.420 (4) |               |
| O6—C41                | 1.363 (4) |               |
| C1—H1A                | 0.9900    |               |
| C1—H1B                | 0.9900    |               |
| C2—H22B               |           | 0.9900        |
| C2—C23                | 1.502 (5) |               |
| C2—C24                | 1.383 (5) |               |
| C3—C25                | 1.383 (5) |               |
| C4—C26                | 1.492 (5) |               |
| C5—C27                | 1.377 (5) |               |
| C6—C28                | 1.492 (5) |               |
| C7—C29                | 1.400 (5) |               |
| C8—C30                | 1.400 (5) |               |
| C9—C31                | 1.370 (5) |               |
| C10—C32               | 0.9500    |               |
| C11—C33               | 1.400 (5) |               |
| C12—C34               | 1.382 (5) |               |
| C13—C35               | 1.389 (5) |               |
| C14—C36               | 1.400 (5) |               |
| C15—C37               | 1.382 (5) |               |
| C16—C38               | 1.383 (5) |               |
| C17—C39               | 1.377 (5) |               |
| C18—C40               | 1.400 (5) |               |
| C19—C41               | 1.382 (5) |               |
| C20—C42               | 0.9500    |               |
| C21—C43               | 1.382 (5) |               |
| C22—C44               | 1.382 (5) |               |
| C23—C45               | 0.9500    |               |
| C24—C46               | 1.400 (5) |               |
| C25—C47               | 1.382 (5) |               |
| C26—C48               | 0.9500    |               |

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| Bond       | Length (Å) | Bond       | Length (Å) | Bond       | Length (Å) |
|------------|------------|------------|------------|------------|------------|
| C11—C12   | 1.390 (5)  | C38—C39   | 1.384 (6)  |
| C12—H12   | 0.9500     | C39—H39   | 0.9500     |
| C12—C13   | 1.387 (5)  | C39—C40   | 1.387 (6)  |
| C13—C14   | 1.386 (5)  | C40—H40   | 0.9500     |
| C13—C15   | 1.495 (5)  | C40—C41   | 1.377 (5)  |
| C14—H14   | 0.9500     | C41—C42   | 1.375 (5)  |
| C15—H15A  | 0.9900     | C42—H42   | 0.9500     |
| C15—H15B  | 0.9900     | C42—C43   | 1.732 (4)  |
| C16—C17   | 1.385 (5)  | C43—C44   | 1.362 (5)  |
| C16—C21   | 1.372 (5)  | C43—C48   | 1.371 (6)  |
| C17—H17   | 0.9500     | C44—H44   | 0.9500     |
| C17—C18   | 1.378 (6)  | C44—C45   | 1.372 (6)  |
| C18—H18   | 0.9500     | C45—H45   | 0.9500     |
| C18—C19   | 1.383 (6)  | C45—C46   | 1.373 (7)  |
| C19—H19   | 0.9500     | C46—H46   | 0.9500     |
| C19—C20   | 1.382 (5)  | C46—C47   | 1.364 (7)  |
| C20—C21   | 1.392 (5)  | C47—H47   | 0.9500     |
| C21—H21   | 0.9500     | C47—C48   | 1.381 (7)  |
| C22—H22A  | 0.9900     | C48—H48   | 0.9500     |
| C16—O2—C15| 117.1 (3)  | C24—C23—C28| 119.8 (3) |
| C20—O3—C22| 116.5 (3)  | C28—C23—C22| 118.7 (3) |
| C37—O5—C36| 116.8 (3)  | C23—C24—H24| 120.6    |
| C41—O6—C1 | 118.1 (3)  | C25—C24—C23| 118.8 (3) |
| O6—C1—H1A| 108.6      | C25—C24—H24| 120.6    |
| O6—C1—H1B| 108.6      | C24—C25—C26| 121.5 (3) |
| O6—C1—C2 | 114.5 (3)  | C24—C25—C29| 129.7 (3) |
| H1A—C1—H1B| 107.6     | C26—C25—C29| 108.5 (3) |
| C2—C1—H1A| 108.6      | C25—C26—C31| 108.6 (3) |
| C2—C1—H1B| 108.6      | C27—C26—C25| 120.2 (3) |
| C3—C2—C1 | 117.3 (3)  | C27—C26—C31| 131.1 (3) |
| C7—C2—C1 | 122.5 (3)  | C26—C27—H27| 121.0    |
| C7—C2—C3 | 120.2 (3)  | C26—C27—C28| 118.0 (3) |
| C2—C3—H3 | 119.1      | C28—C27—H27| 121.0    |
| C4—C3—C2 | 121.9 (3)  | C23—C28—C27| 121.6 (3) |
| C4—C3—H3 | 119.1      | C23—C28—H28| 119.2    |
| C3—C4—H4 | 120.9      | C27—C28—H28| 119.2    |
| C5—C4—C3 | 118.3 (3)  | O4—C29—C25| 127.1 (3) |
| C5—C4—H4 | 120.9      | O4—C29—C30| 127.5 (3) |
| C4—C5—C6 | 119.9 (3)  | C30—C29—C25| 105.3 (3) |
| C4—C5—C8 | 131.2 (3)  | C31—C30—C29| 109.1 (3) |
| C6—C5—C8 | 108.7 (3)  | C32—C30—C29| 129.5 (3) |
| C5—C6—C10| 107.9 (3)  | C32—C30—C31| 121.3 (3) |
| C7—C6—C5 | 122.1 (3)  | C30—C31—C26| 108.4 (3) |
| C7—C6—C10| 129.9 (3)  | C35—C31—C26| 131.4 (3) |
| C2—C7—H7 | 121.2      | C35—C31—C30| 120.1 (3) |
| C6—C7—C2 | 117.7 (3)  | C30—C32—H32| 120.5    |
| C6—C7—H7 | 121.2      | C30—C32—C33| 119.0 (3) |
| Bond                  | Distance (Å) | Angle (°)     |
|----------------------|--------------|--------------|
| C9—C8—C5            | 108.5 (3)    | C33—C32—H32 | 120.5 |
| C11—C8—C5           | 131.3 (3)    | C32—C33—C34 | 119.5 (3) |
| C11—C8—C9           | 120.1 (3)    | C32—C33—C36 | 120.7 (3) |
| C8—C9—C10           | 108.7 (3)    | C34—C33—C36 | 119.7 (3) |
| C14—C9—C8           | 121.4 (3)    | C33—C34—H34 | 119.2 |
| C14—C9—C10          | 129.8 (3)    | C35—C34—C33 | 121.6 (3) |
| O1—C10—C6           | 126.6 (3)    | C35—C34—H34 | 119.2 |
| O1—C10—C9           | 127.7 (3)    | C31—C35—C34 | 118.4 (3) |
| C9—C10—C6           | 105.7 (3)    | C31—C35—H35 | 120.8 |
| C8—C11—H11          | 121.1        | C34—C35—H35 | 120.8 |
| C8—C11—C12          | 117.9 (3)    | O5—C36—C33  | 114.3 (3) |
| C12—C11—H11         | 121.1        | O5—C36—H36A | 108.7 |
| C11—C12—H12         | 118.9        | O5—C36—H36B | 108.7 |
| C13—C12—C11         | 122.3 (4)    | C33—C36—H36A| 108.7 |
| C13—C12—H12         | 118.9        | C33—C36—H36B| 108.7 |
| C12—C13—C15         | 117.1 (3)    | H36A—C36—H36B| 107.6 |
| C14—C13—C12         | 119.3 (3)    | O5—C37—C38  | 116.1 (3) |
| C14—C13—C15         | 123.4 (3)    | O5—C37—C42  | 123.0 (3) |
| C9—C14—C13          | 118.6 (3)    | C38—C37—C42 | 120.9 (4) |
| C9—C14—H14          | 120.7        | C37—C38—H38 | 120.5 |
| C13—C14—H14         | 120.7        | C37—C38—C39 | 118.9 (3) |
| O2—C15—C13          | 114.4 (3)    | C39—C38—H38 | 120.5 |
| O2—C15—H15A         | 108.7        | C39—C38—H39 | 119.5 |
| O2—C15—H15B         | 108.7        | C38—C39—C40 | 121.0 (4) |
| C13—C15—H15A        | 108.7        | C40—C39—H39 | 119.5 |
| C13—C15—H15B        | 108.7        | C39—C40—H40 | 120.6 |
| H15A—C15—H15B       | 107.6        | C41—C40—C39 | 118.8 (4) |
| O2—C16—C17          | 115.7 (3)    | C41—C40—H40 | 120.6 |
| C21—C16—O2          | 122.8 (3)    | O6—C41—C40  | 115.9 (3) |
| C21—C16—C17         | 121.5 (3)    | O6—C41—C42  | 122.9 (3) |
| C16—C17—H17         | 120.9        | C42—C41—C40 | 121.2 (3) |
| C18—C17—C16         | 118.3 (4)    | C37—C42—H42 | 120.5 |
| C18—C17—H17         | 120.9        | C41—C42—C37 | 119.1 (3) |
| C17—C18—H18         | 119.3        | C41—C42—H42 | 120.5 |
| C17—C18—C19         | 121.5 (4)    | C44—C43—C11 | 119.8 (3) |
| C19—C18—H18         | 119.3        | C44—C43—C48 | 121.6 (4) |
| C18—C19—H19         | 120.3        | C48—C43—C11 | 118.6 (3) |
| C20—C19—C18         | 119.3 (3)    | C43—C44—H44 | 120.3 |
| C20—C19—H19         | 120.3        | C43—C44—C45 | 119.5 (4) |
| O3—C20—C19          | 116.9 (3)    | C45—C44—H44 | 120.3 |
| O3—C20—C21          | 123.1 (3)    | C44—C45—H45 | 120.1 |
| C19—C20—C21         | 120.0 (4)    | C44—C45—C46 | 119.8 (4) |
| C16—C21—C20         | 119.4 (3)    | C46—C45—H45 | 120.1 |
| C16—C21—H21         | 120.3        | C45—C46—H46 | 119.9 |
| C20—C21—H21         | 120.3        | C47—C46—C45 | 120.2 (4) |
| O3—C22—H22A         | 108.7        | C47—C46—H46 | 119.9 |
| O3—C22—H22B         | 108.7        | C46—C47—H47 | 119.7 |
| O3—C22—C23          | 114.1 (3)    | C46—C47—C48 | 120.6 (4) |
| Bond                  | Angle (°) | Bond                  | Angle (°) |
|----------------------|-----------|----------------------|-----------|
| H22A—C22—H22B       | 107.6     | C48—C47—H47          | 119.7     |
| C23—C22—H22A        | 108.7     | C43—C48—C47          | 118.3 (4) |
| C23—C22—H22B        | 108.7     | C43—C48—H48          | 120.8     |
| C24—C23—C22         | 121.4 (3) | C47—C48—H48          | 120.8     |
| O2—C16—C17—C18      | 178.9 (3) | C19—C20—C21—C16     | 0.1 (5)   |
| O2—C16—C21—C20      | −178.5 (3)| C20—O3—C22—C23      | −80.0 (4) |
| O3—C20—C21—C16      | 179.4 (3) | C21—C16—C17—C18     | 0.5 (6)   |
| O3—C22—C23—C24      | −30.7 (5) | C22—O3—C20—C19      | −165.2 (3)|
| O3—C22—C23—C28      | 152.4 (3) | C22—O3—C20—C21      | 15.4 (5)  |
| O4—C29—C30—C31      | −179.6 (4)| C22—C23—C24—C25     | −174.6 (3)|
| O4—C29—C30—C32      | −3.9 (7)  | C22—C23—C28—C27     | 174.6 (3) |
| O5—C37—C38—C39      | 179.1 (3) | C23—C24—C25—C26     | −0.1 (5)  |
| O5—C37—C42—C41      | −178.6 (3)| C23—C24—C25—C29     | 174.4 (3) |
| O6—C1—C2—C3         | −165.4 (3)| C24—C23—C28—C27     | −2.3 (5)  |
| O6—C1—C2—C7         | 12.6 (5)  | C24—C25—C26—C27     | −1.9 (5)  |
| O6—C41—C42—C37      | 179.0 (3) | C24—C25—C26—C31     | 174.7 (3) |
| C1—O6—C41—C40       | −160.6 (3)| C24—C25—C29—O4      | 4.5 (6)   |
| C1—O6—C41—C42       | 19.3 (5)  | C24—C25—C29—C30     | −173.6 (4)|
| C1—C2—C3—C4         | 175.2 (4) | C25—C26—C27—C28     | 1.8 (5)   |
| C1—C2—C7—C6         | −175.9 (3)| C25—C26—C31—C30     | 0.0 (4)   |
| C2—C3—C4—C5         | 0.5 (6)   | C25—C26—C31—C35     | −177.1 (4)|
| C3—C2—C7—C6         | 2.1 (5)   | C25—C29—C30—C31     | −1.5 (4)  |
| C3—C4—C5—C6         | 2.3 (5)   | C25—C29—C30—C32     | 174.2 (4) |
| C3—C4—C5—C8         | −172.2 (4)| C26—C25—C29—O4      | 179.6 (4) |
| C4—C5—C6—C7         | −3.1 (5)  | C26—C25—C29—C30     | 1.5 (4)   |
| C4—C5—C6—C10        | −179.0 (3)| C26—C27—C28—C23     | 0.3 (6)   |
| C4—C5—C8—C9         | 174.1 (4) | C26—C31—C35—C34     | 174.0 (4) |
| C4—C5—C8—C11        | −3.6 (7)  | C27—C26—C31—C30     | 176.1 (4) |
| C5—C6—C7—C2         | 0.8 (5)   | C27—C26—C31—C35     | −1.0 (7)  |
| C5—C6—C10—O1        | −173.4 (4)| C28—C23—C24—C25     | 2.2 (5)   |
| C5—C6—C10—C9        | 6.1 (4)   | C29—C25—C26—C27     | −177.5 (3)|
| C5—C8—C9—C10        | 4.8 (4)   | C29—C25—C26—C31     | −0.9 (4)  |
| C5—C8—C9—C14        | −172.1 (3)| C29—C30—C31—C26     | 1.0 (4)   |
| C5—C8—C11—C12       | 173.4 (4) | C29—C30—C31—C35     | 178.4 (3) |
| C6—C5—C8—C9         | −0.9 (4)  | C29—C30—C32—C33     | −174.1 (4)|
| C6—C5—C8—C11        | −178.6 (4)| C30—C31—C35—C34     | −2.8 (5)  |
| C7—C2—C3—C4         | −2.9 (5)  | C30—C32—C33—C34     | −4.0 (5)  |
| C7—C6—C10—O1        | 11.1 (6)  | C30—C32—C33—C36     | 171.1 (3) |
| C7—C6—C10—C9        | −169.4 (4)| C31—C26—C27—C28     | −173.9 (4)|
| C8—C5—C6—C7         | 172.6 (3) | C31—C30—C32—C33     | 1.2 (5)   |
| C8—C5—C6—C10        | −3.3 (4)  | C32—C30—C31—C35     | −175.2 (3)|
| C8—C9—C10—O1        | 172.7 (4) | C32—C30—C34—C35     | 2.3 (5)   |
| C8—C9—C10—C6        | −6.7 (4)  | C32—C33—C34—C35     | 3.5 (5)   |
| C8—C9—C14—C13       | −2.0 (5)  | C32—C33—C36—O5      | 33.9 (5)  |
| C8—C11—C12—C13      | −1.6 (6)  | C33—C34—C35—C31     | 0.0 (6)   |
| C9—C8—C11—C12       | −4.0 (5)  | C34—C33—C36—O5      | −151.0 (3)|
| C10—C6—C7—C2        | 175.7 (3) | C36—O5—C37—C38      | 167.2 (3) |
C10—C9—C14—C13  
\(-178.2 \, (4)\)  
C36—O5—C37—C42  
\(-13.0 \, (5)\)

C11—C8—C9—C10  
\(-177.2 \, (3)\)  
C36—C33—C34—C35  
\(-171.7 \, (3)\)

C11—C8—C9—C14  
\(5.9 \, (6)\)  
C36—O5—C37—C42  
\(78.4 \, (4)\)

C11—C12—C13—C14  
\(5.5 \, (6)\)  
C37—C38—C42—C41  
\(0.1 \, (6)\)

C11—C12—C13—C15  
\(-170.5 \, (4)\)  
C38—C37—C42—C41  
\(1.3 \, (5)\)

C12—C13—C14—C9  
\(-3.6 \, (5)\)  
C38—C39—C40—C41  
\(0.0 \, (6)\)

C12—C13—C15—O2  
\(168.8 \, (3)\)  
C39—C40—C41—O6  
\(-179.6 \, (3)\)

C14—C9—C10—O1  
\(-10.7 \, (7)\)  
C39—C40—C41—C42  
\(0.5 \, (5)\)

C14—C9—C10—C6  
\(169.9 \, (4)\)  
C40—C41—C42—C37  
\(-1.2 \, (5)\)

C14—C13—C15—O2  
\(-7.0 \, (5)\)  
C41—O6—C1—C2  
\(-90.6 \, (4)\)

C15—O2—C16—C21  
\(158.0 \, (3)\)  
C41—O6—C1—C2  
\(-90.6 \, (4)\)

C14—C13—C15—O2  
\(-7.0 \, (5)\)  
C11—C43—C44—C45  
\(-177.9 \, (3)\)

C15—O2—C16—C21  
\(-23.6 \, (5)\)  
C11—C43—C44—C45  
\(-177.9 \, (3)\)

C15—O2—C16—C17  
\(91.6 \, (4)\)  
C43—C44—C45—C46  
\(0.5 \, (6)\)

C16—O2—C16—C17  
\(-158.0 \, (3)\)  
C44—C45—C46—C47  
\(-2.2 \, (7)\)

C17—O2—C16—C17  
\(0.7 \, (6)\)  
C45—C46—C47—C48  
\(-1.4 \, (7)\)

C17—O2—C16—C21  
\(-0.2 \, (6)\)  
C45—C46—C47—C48  
\(-1.4 \, (7)\)

C18—C19—C20—O3  
\(-179.7 \, (3)\)  
C46—C47—C48—C43  
\(-1.3 \, (7)\)

C18—C19—C20—C21  
\(-0.3 \, (6)\)  
C46—C47—C48—C43  
\(-1.3 \, (7)\)

Hydrogen-bond geometry (Å, °)

\(D—H···A\)  
\(D—H\)  
\(H···A\)  
\(D···A\)  
\(D—H···A\)

C18—H18···Cl1i  
0.95  
2.83  
3.547 (4)  
133

C35—H35···O1ii  
0.95  
2.58  
3.491 (5)  
161

C46—H46···O6iii  
0.95  
2.55  
3.418 (5)  
152

C1—H1A···Cg2iv  
0.99  
2.95  
3.610 (4)  
125

C22—H22A···Cg1v  
0.99  
2.73  
3.711 (4)  
170

C36—H36B···Cg15vi  
0.99  
2.84  
3.713 (4)  
148

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