

Methyl 4,6-bis(4-fluorophenyl)-2-oxo-cyclohex-3-ene-1-carboxylate

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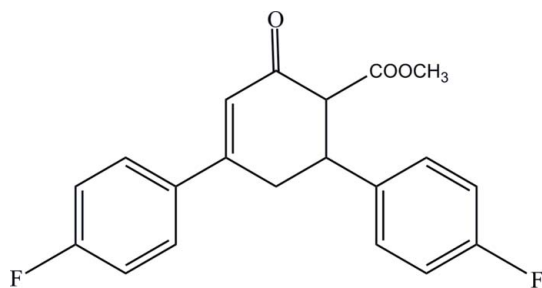
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; disorder in main residue; R factor = 0.055; wR factor = 0.169; data-to-parameter ratio = 11.2.

The 3-cyclohexene units adopt envelope conformations in each of the two independent molecules that comprise the asymmetric unit of the title compound, $\text{C}_{20}\text{H}_{16}\text{F}_2\text{O}_3$. The dihedral angles between the two fluorophenyl rings are 79.7 (2) and 73.7 (2)° in the two molecules. In one of the molecules, two C—H groups of the cyclohexene ring are disordered over two sets of sites in a 0.818 (13):0.182 (13) ratio, the major and minor components corresponding to the two enantiomeric forms of the molecule. Weak intermolecular C—H...O interactions help to stabilize the crystal structure.

Related literature

For background to the applications of cyclohexenones, see: Padmavathi *et al.* (1999; 2000; 2001*a,b*); Hiromichi *et al.* (2002); Hoye & Tennakoon (2000); Kolesnick & Golde (1994); Tanaka *et al.* (1997). For related structures, see: Fischer *et al.* (2007*a,b*, 2008*a,b*); Li *et al.* (2009); Ashalatha *et al.* (2009); Sreevidya *et al.* (2010). For ring conformations, see: Cremer & Pople (1975).



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Experimental

Crystal data

$\text{C}_{20}\text{H}_{16}\text{F}_2\text{O}_3$
 $M_r = 342.33$
Orthorhombic, $Pca2_1$
 $a = 17.3774$ (5) Å
 $b = 9.0629$ (3) Å
 $c = 22.2238$ (7) Å
 $V = 3500.02$ (19) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 296$ K
 $0.40 \times 0.32 \times 0.28$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.961$, $T_{\max} = 0.973$
38134 measured reflections
5278 independent reflections
2895 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.169$
 $S = 1.02$
5278 reflections
472 parameters
1 restraint
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C8A}-\text{H8AB}\cdots\text{O2B}^{\text{i}}$ | 0.97 | 2.38 | 3.263 (5) | 151 |
| $\text{C8B}-\text{H8BB}\cdots\text{O2A}^{\text{ii}}$ | 0.97 | 2.57 | 3.404 (5) | 144 |
| $\text{C15B}-\text{H15B}\cdots\text{O1B}^{\text{iii}}$ | 0.93 | 2.55 | 3.226 (6) | 130 |

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2744).

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supplementary materials

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Methyl 4,6-bis(4-fluorophenyl)-2-oxocyclohex-3-ene-1-carboxylate

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Comment

Cyclohexenones are efficient synthons for building spiro compounds (Padmavathi *et al.*, 2001a) or as intermediates in the synthesis of benzisoxazoles or carbazole derivatives (Padmavathi *et al.*, 1999; 2000; 2001b). Cyclohexenone derivatives are well known lead molecules for the treatment of inflammation and autoimmune diseases (Kolesnick & Golde, 1994; Tanaka *et al.*, 1997; Hoye & Tennakoon, 2000; Hiromichi *et al.*, 2002).

The crystal structures of some cyclohexenone derivatives viz, (8*RS*,9*SR*)-ethyl 4-(3-bromothien-2-yl)-6-(2-furyl)-2-oxocyclohex-3-ene-1-carboxylate, (7*RS*,8*SR*)-ethyl 6-(1,3-benzodioxol-5-yl)-3-(3-bromo-2-thienyl)-2-oxocyclohex-3-ene-1-carboxylate, ethyl 4-(3-bromo-2-thienyl)-2-oxo-6-phenylcyclohex-3-ene-1-carboxylate, rac-ethyl 3-(3-bromo-2-thienyl)-2-oxo-6-(4-propoxyphenyl)cyclohex-3-ene-1-carboxylate (Fischer *et al.*, 2007a,b; 2008a,b) and ethyl 6-(6-methoxy-2-naphthyl)-2-oxo-4-(2-thienyl)cyclohex-3-ene-1-carboxylate (Li *et al.*, 2009) have been reported. In a continuation of our work on cyclohexenone derivatives (Ashalatha *et al.*, 2009; Sreevidya *et al.*, 2010) and in view of the importance of these derivatives, the title compound (I) is synthesized and its crystal structure is reported here.

The asymmetric unit of the title compound consists of two crystallographically independent molecules, A and B (Fig. 1). The cyclohex-3-ene units in both molecules adopt envelope conformations with puckering parameters $Q = 0.462$ (4) Å, $\Theta = 128.3$ (5)° and $\varphi = 165.2$ (6)° for molecule A and $Q = 0.488$ (6) Å, $\Theta = 128.5$ (6)° and $\varphi = 164.2$ (7)° for molecule B (Cremer & Pople, 1975). The two fluorophenyl rings are inclined to each other forming dihedral angles of 79.7 (2)° (C1A–C6A:C13A–C18A) in molecule A and 73.7 (2)° (C1B–C6B:C13B–C18B) in molecule B.

The cyclohexene rings (C7–C12) are almost coplanar with C7A, C12A, C7B and C12B displaced by -0.322 (4) Å, 0.262 (4) Å, -0.339 (6) Å and 0.281 (5) Å. In molecule B, C7 and C12 are disordered over two sites with a 0.818 (13):0.182 (13) ratio and the minor disorder component shows displacements from the mean plane of C8B/C9B/C10B/C11B in the opposite sense to that of the major component: C7C = 0.33 (4) Å; C12C = -0.414 (18) Å. Atoms C7A, C12A, C7B and C12B are stereogenic centres. In molecule B, the major and minor disorder components correspond to the two enantiomeric forms of the molecule.

In the crystal packing (Fig. 2), the molecules are linked through weak intermolecular C8A—H8AB...O2B; C8B—H8BB...O2A and C15B—H15B...O1B hydrogen bonds (see Table 1).

Experimental

A mixture of (2*E*)-1,3-bis(4-fluorophenyl)prop-2-en-1-one (2.24g, 0.01mol) and methyl acetoacetate (0.01 mol) were refluxed for 2 hr in 10-15 ml of ethanol in presence of 0.8 ml 10% NaOH. The reaction mixture was cooled to room temperature and the solid product obtained was filtered and recrystallized from toluene (M.P.: 417K). Analytical data: found (calculated): C % : 70.08 (70.17); H% : 4.66 (4.71)

Refinement

Atoms C7B and C12B and their attached H atoms are disordered over two sets of sites with a 0.818 (13):0.182 (13) occupancy ratio. All hydrogen atoms were positioned geometrically [C–H = 0.93 or 0.96 Å] and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl groups. In the absence of large anomalous dispersion, 3128 Friedel pairs were merged for the final refinement.

Figures

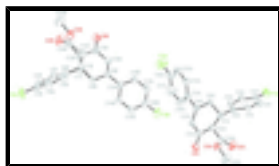


Fig. 1. The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Bonds to atoms of the minor disorder component are drawn as open lines and H atoms are omitted for clarity.

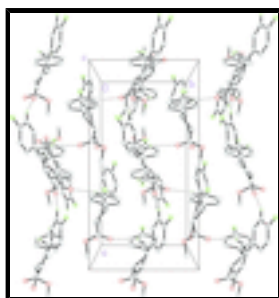


Fig. 2. The crystal packing of the title compound, showing hydrogen-bonded network (dashed lines). H atoms not involved in hydrogen bond interactions and atoms of the minor disorder component are omitted for clarity.

Methyl 4,6-bis(4-fluorophenyl)-2-oxocyclohex-3-ene-1-carboxylate

Crystal data

$\text{C}_{20}\text{H}_{16}\text{F}_2\text{O}_3$

$M_r = 342.33$

Orthorhombic, $Pca2_1$

Hall symbol: P 2c -2ac

$a = 17.3774$ (5) Å

$b = 9.0629$ (3) Å

$c = 22.2238$ (7) Å

$V = 3500.02$ (19) Å³

$Z = 8$

$F(000) = 1424$

$D_x = 1.299$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7809 reflections

$\theta = 2.5$ – 21.9°

$\mu = 0.10$ mm⁻¹

$T = 296$ K

Block, colourless

$0.40 \times 0.32 \times 0.28$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

φ and ω scans

Absorption correction: multi-scan

5278 independent reflections

2895 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\text{max}} = 30.2^\circ$, $\theta_{\text{min}} = 2.3^\circ$

$h = -24 \rightarrow 24$

(SADABS; Bruker, 2009)

$T_{\min} = 0.961$, $T_{\max} = 0.973$

38134 measured reflections

$k = -12 \rightarrow 12$

$l = -20 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.169$

$S = 1.02$

5278 reflections

472 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0821P)^2 + 0.1221P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|-------------|--------------|----------------------------------|-----------|
| F1A | 0.13044 (18) | -0.1190 (4) | 0.76691 (10) | 0.1074 (9) | |
| F2A | -0.0040 (2) | -0.1767 (4) | 0.23590 (14) | 0.1323 (12) | |
| O1A | 0.40617 (15) | 0.0074 (3) | 0.44238 (14) | 0.0808 (8) | |
| O2A | 0.39944 (18) | -0.1519 (3) | 0.57537 (17) | 0.0913 (9) | |
| O3A | 0.41115 (18) | 0.0919 (3) | 0.57886 (15) | 0.0886 (8) | |
| C1A | 0.2085 (2) | -0.2420 (4) | 0.62524 (16) | 0.0726 (9) | |
| H1AA | 0.2232 | -0.3296 | 0.6067 | 0.087* | |
| C2A | 0.1803 (3) | -0.2447 (5) | 0.68369 (19) | 0.0818 (11) | |
| H2AA | 0.1762 | -0.3329 | 0.7048 | 0.098* | |
| C3A | 0.1588 (2) | -0.1153 (5) | 0.70904 (16) | 0.0751 (11) | |
| C4A | 0.1641 (3) | 0.0180 (5) | 0.6812 (2) | 0.0809 (11) | |
| H4AA | 0.1496 | 0.1046 | 0.7006 | 0.097* | |
| C5A | 0.1921 (3) | 0.0189 (5) | 0.62256 (19) | 0.0763 (11) | |
| H5AA | 0.1957 | 0.1080 | 0.6020 | 0.092* | |
| C6A | 0.21478 (19) | -0.1097 (4) | 0.59429 (14) | 0.0601 (8) | |

supplementary materials

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|------|---------------|-------------|---------------|-------------|------------|
| C7A | 0.2441 (2) | -0.1129 (4) | 0.52993 (16) | 0.0615 (8) | |
| H7AA | 0.2645 | -0.2121 | 0.5226 | 0.074* | |
| C8A | 0.1799 (2) | -0.0870 (4) | 0.48469 (15) | 0.0610 (8) | |
| H8AA | 0.1432 | -0.1673 | 0.4876 | 0.073* | |
| H8AB | 0.1532 | 0.0035 | 0.4952 | 0.073* | |
| C9A | 0.2075 (2) | -0.0759 (4) | 0.42100 (15) | 0.0599 (8) | |
| C10A | 0.2816 (2) | -0.0426 (4) | 0.40906 (16) | 0.0650 (8) | |
| H10A | 0.2969 | -0.0383 | 0.3690 | 0.078* | |
| C11A | 0.3381 (2) | -0.0134 (4) | 0.45435 (17) | 0.0640 (9) | |
| C12A | 0.3102 (2) | -0.0035 (4) | 0.51956 (16) | 0.0611 (8) | |
| H12A | 0.2909 | 0.0966 | 0.5267 | 0.073* | |
| C13A | 0.1514 (2) | -0.1027 (4) | 0.37197 (16) | 0.0642 (8) | |
| C14A | 0.0756 (2) | -0.0588 (4) | 0.37815 (17) | 0.0698 (9) | |
| H14A | 0.0596 | -0.0133 | 0.4135 | 0.084* | |
| C15A | 0.0230 (2) | -0.0822 (5) | 0.3320 (2) | 0.0818 (11) | |
| H15A | -0.0279 | -0.0517 | 0.3358 | 0.098* | |
| C16A | 0.0481 (3) | -0.1514 (6) | 0.2809 (2) | 0.0956 (14) | |
| C17A | 0.1221 (3) | -0.1967 (7) | 0.2727 (2) | 0.1112 (18) | |
| H17A | 0.1371 | -0.2429 | 0.2372 | 0.133* | |
| C18A | 0.1741 (3) | -0.1723 (6) | 0.3184 (2) | 0.0933 (14) | |
| H18A | 0.2250 | -0.2022 | 0.3137 | 0.112* | |
| C19A | 0.3777 (2) | -0.0302 (4) | 0.56085 (17) | 0.0634 (8) | |
| C20A | 0.4811 (4) | 0.0706 (6) | 0.6153 (3) | 0.124 (2) | |
| H20A | 0.5034 | 0.1649 | 0.6246 | 0.187* | |
| H20B | 0.5176 | 0.0126 | 0.5931 | 0.187* | |
| H20C | 0.4681 | 0.0206 | 0.6520 | 0.187* | |
| F1B | 0.1337 (2) | 0.5160 (4) | -0.19775 (12) | 0.1155 (10) | |
| F2B | 0.2341 (2) | 0.2462 (4) | 0.32262 (14) | 0.1301 (11) | |
| O1B | -0.15225 (18) | 0.6114 (4) | 0.12515 (15) | 0.0933 (9) | |
| O2B | -0.1376 (3) | 0.7363 (4) | -0.0131 (2) | 0.1300 (15) | |
| O3B | -0.1543 (2) | 0.4954 (3) | -0.00956 (17) | 0.0966 (10) | |
| C1B | 0.0509 (2) | 0.6346 (5) | -0.05757 (19) | 0.0760 (10) | |
| H1BA | 0.0377 | 0.7220 | -0.0382 | 0.091* | |
| C2B | 0.0835 (2) | 0.6400 (5) | -0.11435 (18) | 0.0781 (11) | |
| H2BA | 0.0924 | 0.7299 | -0.1333 | 0.094* | |
| C3B | 0.1021 (3) | 0.5116 (5) | -0.14166 (17) | 0.0788 (11) | |
| C4B | 0.0910 (3) | 0.3776 (5) | -0.1145 (2) | 0.0861 (12) | |
| H4BA | 0.1053 | 0.2906 | -0.1337 | 0.103* | |
| C5B | 0.0582 (3) | 0.3747 (5) | -0.0583 (2) | 0.0793 (11) | |
| H5BA | 0.0498 | 0.2842 | -0.0397 | 0.095* | |
| C6B | 0.0378 (2) | 0.5006 (4) | -0.02920 (16) | 0.0640 (9) | |
| C7B | 0.0036 (3) | 0.4823 (7) | 0.0331 (3) | 0.0566 (14) | 0.818 (13) |
| H7BA | -0.0238 | 0.3878 | 0.0343 | 0.068* | 0.818 (13) |
| C7C | 0.0136 (17) | 0.552 (4) | 0.0413 (11) | 0.063 (7) | 0.182 (13) |
| H7CA | 0.0175 | 0.6596 | 0.0457 | 0.076* | 0.182 (13) |
| C8B | 0.06828 (19) | 0.4779 (4) | 0.08113 (15) | 0.0580 (8) | |
| H8BA | 0.1001 | 0.3917 | 0.0741 | 0.070* | |
| H8BB | 0.1006 | 0.5645 | 0.0763 | 0.070* | |
| C9B | 0.0387 (2) | 0.4732 (4) | 0.14515 (16) | 0.0600 (8) | |

| | | | | | |
|------|--------------|------------|--------------|-------------|------------|
| C10B | -0.0333 (2) | 0.5192 (4) | 0.15751 (17) | 0.0681 (9) | |
| H10B | -0.0503 | 0.5133 | 0.1971 | 0.082* | |
| C11B | -0.0858 (2) | 0.5770 (5) | 0.11294 (19) | 0.0733 (10) | |
| C12B | -0.0537 (3) | 0.6048 (6) | 0.0490 (2) | 0.0618 (16) | 0.818 (13) |
| H12B | -0.0253 | 0.6982 | 0.0499 | 0.074* | 0.818 (13) |
| C12C | -0.0661 (11) | 0.504 (2) | 0.0472 (9) | 0.056 (6) | 0.182 (13) |
| H12C | -0.0764 | 0.3988 | 0.0421 | 0.067* | 0.182 (13) |
| C13B | 0.0905 (2) | 0.4144 (4) | 0.19204 (16) | 0.0652 (8) | |
| C14B | 0.1692 (2) | 0.4082 (4) | 0.18360 (18) | 0.0715 (9) | |
| H14B | 0.1900 | 0.4412 | 0.1475 | 0.086* | |
| C15B | 0.2173 (3) | 0.3544 (5) | 0.2275 (2) | 0.0835 (11) | |
| H15B | 0.2702 | 0.3523 | 0.2213 | 0.100* | |
| C16B | 0.1867 (3) | 0.3042 (6) | 0.2801 (2) | 0.0904 (13) | |
| C17B | 0.1109 (3) | 0.3063 (8) | 0.2898 (2) | 0.118 (2) | |
| H17B | 0.0911 | 0.2706 | 0.3258 | 0.142* | |
| C18B | 0.0621 (3) | 0.3607 (7) | 0.2468 (2) | 0.1047 (17) | |
| H18B | 0.0094 | 0.3620 | 0.2541 | 0.126* | |
| C19B | -0.1188 (3) | 0.6222 (6) | 0.0055 (2) | 0.0869 (13) | |
| C20B | -0.2189 (4) | 0.5075 (7) | -0.0513 (4) | 0.135 (2) | |
| H20D | -0.2467 | 0.4160 | -0.0520 | 0.202* | |
| H20E | -0.1998 | 0.5289 | -0.0909 | 0.202* | |
| H20F | -0.2525 | 0.5854 | -0.0385 | 0.202* | |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| F1A | 0.116 (2) | 0.162 (3) | 0.0439 (12) | -0.0081 (18) | 0.0163 (13) | 0.0076 (14) |
| F2A | 0.108 (2) | 0.193 (3) | 0.0960 (19) | -0.007 (2) | -0.0281 (18) | -0.033 (2) |
| O1A | 0.0687 (16) | 0.0973 (19) | 0.0764 (18) | -0.0123 (13) | 0.0137 (14) | 0.0075 (14) |
| O2A | 0.104 (2) | 0.0625 (15) | 0.108 (2) | 0.0139 (14) | -0.0235 (19) | 0.0038 (16) |
| O3A | 0.105 (2) | 0.0661 (15) | 0.095 (2) | 0.0003 (14) | -0.0244 (18) | -0.0034 (15) |
| C1A | 0.090 (2) | 0.070 (2) | 0.058 (2) | -0.0041 (19) | 0.0068 (18) | 0.0089 (17) |
| C2A | 0.100 (3) | 0.087 (3) | 0.059 (2) | -0.009 (2) | 0.004 (2) | 0.017 (2) |
| C3A | 0.075 (2) | 0.109 (3) | 0.0412 (17) | -0.015 (2) | 0.0014 (16) | 0.0091 (19) |
| C4A | 0.092 (3) | 0.086 (3) | 0.065 (2) | -0.002 (2) | 0.017 (2) | -0.010 (2) |
| C5A | 0.098 (3) | 0.070 (2) | 0.061 (2) | -0.0042 (19) | 0.011 (2) | 0.0109 (18) |
| C6A | 0.065 (2) | 0.068 (2) | 0.0467 (17) | -0.0039 (15) | 0.0049 (15) | 0.0042 (15) |
| C7A | 0.069 (2) | 0.0625 (17) | 0.0533 (18) | 0.0012 (15) | 0.0074 (16) | 0.0019 (15) |
| C8A | 0.064 (2) | 0.0695 (19) | 0.0497 (17) | -0.0039 (16) | 0.0066 (15) | 0.0044 (16) |
| C9A | 0.064 (2) | 0.0624 (18) | 0.0537 (19) | 0.0051 (15) | 0.0086 (15) | 0.0026 (15) |
| C10A | 0.072 (2) | 0.076 (2) | 0.0466 (17) | 0.0029 (16) | 0.0103 (16) | 0.0098 (16) |
| C11A | 0.069 (2) | 0.0607 (18) | 0.062 (2) | 0.0013 (15) | 0.0136 (18) | 0.0079 (16) |
| C12A | 0.069 (2) | 0.0587 (18) | 0.055 (2) | 0.0046 (14) | 0.0013 (16) | 0.0045 (14) |
| C13A | 0.065 (2) | 0.078 (2) | 0.0495 (17) | -0.0001 (16) | 0.0061 (15) | -0.0003 (16) |
| C14A | 0.072 (2) | 0.084 (2) | 0.053 (2) | 0.0041 (18) | 0.0064 (17) | -0.0032 (18) |
| C15A | 0.070 (2) | 0.104 (3) | 0.071 (2) | 0.003 (2) | 0.002 (2) | 0.002 (2) |
| C16A | 0.084 (3) | 0.129 (4) | 0.073 (3) | -0.009 (3) | -0.011 (2) | -0.019 (3) |
| C17A | 0.100 (4) | 0.166 (5) | 0.068 (3) | 0.010 (3) | -0.004 (3) | -0.040 (3) |

supplementary materials

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|------|-------------|-------------|-------------|--------------|--------------|--------------|
| C18A | 0.076 (3) | 0.132 (4) | 0.071 (3) | 0.009 (2) | 0.009 (2) | -0.026 (3) |
| C19A | 0.072 (2) | 0.0587 (19) | 0.059 (2) | 0.0005 (15) | 0.0010 (17) | -0.0020 (16) |
| C20A | 0.136 (5) | 0.101 (3) | 0.136 (5) | -0.009 (3) | -0.069 (4) | -0.003 (3) |
| F1B | 0.147 (3) | 0.147 (2) | 0.0525 (13) | 0.013 (2) | 0.0230 (15) | 0.0068 (14) |
| F2B | 0.118 (2) | 0.176 (3) | 0.0961 (19) | 0.005 (2) | -0.0205 (18) | 0.050 (2) |
| O1B | 0.0784 (19) | 0.112 (2) | 0.090 (2) | 0.0279 (16) | 0.0214 (16) | 0.0041 (17) |
| O2B | 0.147 (3) | 0.084 (2) | 0.158 (4) | 0.013 (2) | -0.034 (3) | 0.021 (2) |
| O3B | 0.115 (2) | 0.084 (2) | 0.092 (2) | 0.0142 (17) | -0.0098 (19) | 0.0189 (16) |
| C1B | 0.094 (3) | 0.074 (2) | 0.060 (2) | -0.0007 (19) | 0.006 (2) | 0.0019 (18) |
| C2B | 0.100 (3) | 0.077 (2) | 0.057 (2) | -0.015 (2) | 0.004 (2) | 0.0165 (19) |
| C3B | 0.095 (3) | 0.098 (3) | 0.0426 (18) | -0.009 (2) | 0.0042 (19) | 0.0066 (18) |
| C4B | 0.111 (3) | 0.082 (3) | 0.065 (2) | 0.002 (2) | 0.004 (2) | -0.014 (2) |
| C5B | 0.092 (3) | 0.070 (2) | 0.076 (3) | -0.0006 (19) | 0.014 (2) | 0.0053 (19) |
| C6B | 0.070 (2) | 0.071 (2) | 0.0512 (19) | -0.0014 (16) | 0.0025 (16) | 0.0074 (16) |
| C7B | 0.073 (3) | 0.044 (3) | 0.053 (3) | -0.003 (2) | 0.007 (2) | 0.001 (2) |
| C7C | 0.081 (16) | 0.066 (15) | 0.042 (10) | -0.010 (14) | -0.001 (9) | -0.008 (12) |
| C8B | 0.0652 (19) | 0.0545 (15) | 0.0542 (19) | -0.0027 (13) | 0.0111 (16) | 0.0033 (14) |
| C9B | 0.069 (2) | 0.0576 (17) | 0.0533 (18) | -0.0107 (15) | 0.0103 (16) | 0.0011 (15) |
| C10B | 0.075 (2) | 0.073 (2) | 0.055 (2) | -0.0031 (17) | 0.0146 (17) | 0.0001 (17) |
| C11B | 0.076 (3) | 0.077 (2) | 0.067 (2) | 0.0070 (19) | 0.010 (2) | -0.0035 (19) |
| C12B | 0.069 (3) | 0.053 (3) | 0.063 (3) | 0.005 (2) | 0.015 (2) | 0.0016 (19) |
| C12C | 0.072 (13) | 0.036 (12) | 0.061 (11) | -0.001 (8) | 0.002 (9) | 0.008 (7) |
| C13B | 0.073 (2) | 0.070 (2) | 0.0530 (18) | -0.0125 (16) | 0.0081 (17) | 0.0045 (16) |
| C14B | 0.073 (2) | 0.089 (2) | 0.0526 (18) | -0.0039 (19) | 0.0089 (17) | 0.0108 (18) |
| C15B | 0.078 (3) | 0.102 (3) | 0.070 (3) | 0.001 (2) | 0.004 (2) | 0.009 (2) |
| C16B | 0.089 (3) | 0.110 (3) | 0.072 (3) | -0.001 (2) | -0.010 (2) | 0.025 (2) |
| C17B | 0.107 (4) | 0.171 (6) | 0.076 (3) | -0.018 (4) | 0.000 (3) | 0.052 (3) |
| C18B | 0.084 (3) | 0.158 (5) | 0.073 (3) | -0.014 (3) | 0.009 (2) | 0.046 (3) |
| C19B | 0.089 (3) | 0.085 (3) | 0.087 (3) | 0.014 (2) | 0.004 (2) | 0.006 (2) |
| C20B | 0.134 (5) | 0.130 (4) | 0.142 (6) | -0.020 (4) | -0.060 (5) | 0.029 (4) |

Geometric parameters (Å, °)

| | | | |
|----------|-----------|----------|-----------|
| F1A—C3A | 1.378 (4) | O3B—C19B | 1.346 (6) |
| F2A—C16A | 1.368 (5) | O3B—C20B | 1.461 (7) |
| O1A—C11A | 1.227 (4) | C1B—C2B | 1.384 (6) |
| O2A—C19A | 1.210 (4) | C1B—C6B | 1.388 (6) |
| O3A—C19A | 1.312 (4) | C1B—H1BA | 0.9300 |
| O3A—C20A | 1.474 (6) | C2B—C3B | 1.352 (6) |
| C1A—C6A | 1.387 (5) | C2B—H2BA | 0.9300 |
| C1A—C2A | 1.388 (6) | C3B—C4B | 1.370 (7) |
| C1A—H1AA | 0.9300 | C4B—C5B | 1.373 (6) |
| C2A—C3A | 1.353 (7) | C4B—H4BA | 0.9300 |
| C2A—H2AA | 0.9300 | C5B—C6B | 1.359 (6) |
| C3A—C4A | 1.360 (6) | C5B—H5BA | 0.9300 |
| C4A—C5A | 1.392 (6) | C6B—C7B | 1.516 (6) |
| C4A—H4AA | 0.9300 | C6B—C7C | 1.69 (3) |
| C5A—C6A | 1.381 (6) | C7B—C12B | 1.532 (8) |
| C5A—H5AA | 0.9300 | C7B—C8B | 1.551 (7) |

| | | | |
|---------------|-----------|---------------|------------|
| C6A—C7A | 1.519 (5) | C7B—H7BA | 0.9800 |
| C7A—C8A | 1.521 (5) | C7C—C12C | 1.46 (4) |
| C7A—C12A | 1.535 (5) | C7C—C8B | 1.46 (3) |
| C7A—H7AA | 0.9800 | C7C—H7CA | 0.9800 |
| C8A—C9A | 1.498 (5) | C8B—C9B | 1.514 (5) |
| C8A—H8AA | 0.9700 | C8B—H8BA | 0.9700 |
| C8A—H8AB | 0.9700 | C8B—H8BB | 0.9700 |
| C9A—C10A | 1.350 (5) | C9B—C10B | 1.346 (5) |
| C9A—C13A | 1.482 (5) | C9B—C13B | 1.477 (5) |
| C10A—C11A | 1.430 (5) | C10B—C11B | 1.445 (6) |
| C10A—H10A | 0.9300 | C10B—H10B | 0.9300 |
| C11A—C12A | 1.531 (5) | C11B—C12B | 1.548 (6) |
| C12A—C19A | 1.509 (5) | C11B—C12C | 1.64 (2) |
| C12A—H12A | 0.9800 | C12B—C19B | 1.496 (7) |
| C13A—C14A | 1.383 (5) | C12B—H12B | 0.9800 |
| C13A—C18A | 1.404 (6) | C12C—C19B | 1.685 (19) |
| C14A—C15A | 1.389 (6) | C12C—H12C | 0.9800 |
| C14A—H14A | 0.9300 | C13B—C14B | 1.380 (5) |
| C15A—C16A | 1.370 (7) | C13B—C18B | 1.401 (6) |
| C15A—H15A | 0.9300 | C14B—C15B | 1.374 (6) |
| C16A—C17A | 1.362 (7) | C14B—H14B | 0.9300 |
| C17A—C18A | 1.378 (7) | C15B—C16B | 1.363 (6) |
| C17A—H17A | 0.9300 | C15B—H15B | 0.9300 |
| C18A—H18A | 0.9300 | C16B—C17B | 1.335 (7) |
| C20A—H20A | 0.9600 | C17B—C18B | 1.368 (7) |
| C20A—H20B | 0.9600 | C17B—H17B | 0.9300 |
| C20A—H20C | 0.9600 | C18B—H18B | 0.9300 |
| F1B—C3B | 1.363 (5) | C20B—H20D | 0.9600 |
| F2B—C16B | 1.359 (5) | C20B—H20E | 0.9600 |
| O1B—C11B | 1.227 (5) | C20B—H20F | 0.9600 |
| O2B—C19B | 1.160 (5) | | |
| C19A—O3A—C20A | 115.0 (3) | C6B—C5B—C4B | 121.7 (4) |
| C6A—C1A—C2A | 120.5 (4) | C6B—C5B—H5BA | 119.1 |
| C6A—C1A—H1AA | 119.8 | C4B—C5B—H5BA | 119.1 |
| C2A—C1A—H1AA | 119.8 | C5B—C6B—C1B | 118.4 (4) |
| C3A—C2A—C1A | 118.2 (4) | C5B—C6B—C7B | 116.5 (4) |
| C3A—C2A—H2AA | 120.9 | C1B—C6B—C7B | 125.1 (4) |
| C1A—C2A—H2AA | 120.9 | C5B—C6B—C7C | 137.7 (12) |
| C2A—C3A—C4A | 124.2 (4) | C1B—C6B—C7C | 102.7 (12) |
| C2A—C3A—F1A | 117.8 (4) | C7B—C6B—C7C | 23.8 (10) |
| C4A—C3A—F1A | 118.0 (4) | C6B—C7B—C12B | 112.7 (4) |
| C3A—C4A—C5A | 117.0 (4) | C6B—C7B—C8B | 110.3 (4) |
| C3A—C4A—H4AA | 121.5 | C12B—C7B—C8B | 109.4 (5) |
| C5A—C4A—H4AA | 121.5 | C6B—C7B—H7BA | 108.1 |
| C6A—C5A—C4A | 121.4 (4) | C12B—C7B—H7BA | 108.1 |
| C6A—C5A—H5AA | 119.3 | C8B—C7B—H7BA | 108.1 |
| C4A—C5A—H5AA | 119.3 | C12C—C7C—C8B | 115 (2) |
| C5A—C6A—C1A | 118.8 (3) | C12C—C7C—C6B | 103.7 (19) |
| C5A—C6A—C7A | 122.7 (3) | C8B—C7C—C6B | 105.8 (18) |

supplementary materials

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|----------------|-----------|----------------|------------|
| C1A—C6A—C7A | 118.5 (3) | C12C—C7C—H7CA | 110.6 |
| C6A—C7A—C8A | 111.9 (3) | C8B—C7C—H7CA | 110.6 |
| C6A—C7A—C12A | 112.4 (3) | C6B—C7C—H7CA | 110.6 |
| C8A—C7A—C12A | 110.5 (3) | C7C—C8B—C9B | 111.2 (10) |
| C6A—C7A—H7AA | 107.3 | C7C—C8B—C7B | 26.0 (12) |
| C8A—C7A—H7AA | 107.3 | C9B—C8B—C7B | 113.6 (3) |
| C12A—C7A—H7AA | 107.3 | C7C—C8B—H8BA | 130.1 |
| C9A—C8A—C7A | 113.6 (3) | C9B—C8B—H8BA | 108.8 |
| C9A—C8A—H8AA | 108.8 | C7B—C8B—H8BA | 108.8 |
| C7A—C8A—H8AA | 108.8 | C7C—C8B—H8BB | 86.3 |
| C9A—C8A—H8AB | 108.8 | C9B—C8B—H8BB | 108.8 |
| C7A—C8A—H8AB | 108.8 | C7B—C8B—H8BB | 108.8 |
| H8AA—C8A—H8AB | 107.7 | H8BA—C8B—H8BB | 107.7 |
| C10A—C9A—C13A | 121.3 (3) | C10B—C9B—C13B | 122.3 (3) |
| C10A—C9A—C8A | 120.4 (3) | C10B—C9B—C8B | 119.9 (3) |
| C13A—C9A—C8A | 118.3 (3) | C13B—C9B—C8B | 117.8 (3) |
| C9A—C10A—C11A | 123.9 (3) | C9B—C10B—C11B | 124.0 (3) |
| C9A—C10A—H10A | 118.0 | C9B—C10B—H10B | 118.0 |
| C11A—C10A—H10A | 118.0 | C11B—C10B—H10B | 118.0 |
| O1A—C11A—C10A | 122.5 (3) | O1B—C11B—C10B | 122.3 (4) |
| O1A—C11A—C12A | 120.1 (4) | O1B—C11B—C12B | 120.0 (4) |
| C10A—C11A—C12A | 117.4 (3) | C10B—C11B—C12B | 117.5 (3) |
| C19A—C12A—C11A | 108.7 (3) | O1B—C11B—C12C | 119.8 (8) |
| C19A—C12A—C7A | 112.7 (3) | C10B—C11B—C12C | 109.5 (7) |
| C11A—C12A—C7A | 109.9 (3) | C12B—C11B—C12C | 34.0 (7) |
| C19A—C12A—H12A | 108.5 | C19B—C12B—C7B | 114.8 (5) |
| C11A—C12A—H12A | 108.5 | C19B—C12B—C11B | 109.7 (4) |
| C7A—C12A—H12A | 108.5 | C7B—C12B—C11B | 109.1 (4) |
| C14A—C13A—C18A | 118.8 (4) | C19B—C12B—H12B | 107.7 |
| C14A—C13A—C9A | 120.4 (3) | C7B—C12B—H12B | 107.7 |
| C18A—C13A—C9A | 120.8 (3) | C11B—C12B—H12B | 107.7 |
| C13A—C14A—C15A | 120.6 (4) | C7C—C12C—C11B | 99.1 (17) |
| C13A—C14A—H14A | 119.7 | C7C—C12C—C19B | 106.2 (19) |
| C15A—C14A—H14A | 119.7 | C11B—C12C—C19B | 97.0 (11) |
| C16A—C15A—C14A | 118.2 (4) | C7C—C12C—H12C | 117.1 |
| C16A—C15A—H15A | 120.9 | C11B—C12C—H12C | 117.1 |
| C14A—C15A—H15A | 120.9 | C19B—C12C—H12C | 117.1 |
| C17A—C16A—F2A | 118.5 (4) | C14B—C13B—C18B | 116.9 (4) |
| C17A—C16A—C15A | 123.3 (4) | C14B—C13B—C9B | 121.5 (3) |
| F2A—C16A—C15A | 118.2 (4) | C18B—C13B—C9B | 121.6 (4) |
| C16A—C17A—C18A | 118.2 (4) | C15B—C14B—C13B | 121.4 (4) |
| C16A—C17A—H17A | 120.9 | C15B—C14B—H14B | 119.3 |
| C18A—C17A—H17A | 120.9 | C13B—C14B—H14B | 119.3 |
| C17A—C18A—C13A | 120.8 (4) | C16B—C15B—C14B | 119.4 (4) |
| C17A—C18A—H18A | 119.6 | C16B—C15B—H15B | 120.3 |
| C13A—C18A—H18A | 119.6 | C14B—C15B—H15B | 120.3 |
| O2A—C19A—O3A | 123.3 (4) | C17B—C16B—F2B | 119.5 (4) |
| O2A—C19A—C12A | 123.4 (3) | C17B—C16B—C15B | 121.2 (4) |
| O3A—C19A—C12A | 113.2 (3) | F2B—C16B—C15B | 119.3 (4) |

| | | | |
|---------------------|------------|---------------------|-------------|
| O3A—C20A—H20A | 109.5 | C16B—C17B—C18B | 120.3 (4) |
| O3A—C20A—H20B | 109.5 | C16B—C17B—H17B | 119.9 |
| H20A—C20A—H20B | 109.5 | C18B—C17B—H17B | 119.9 |
| O3A—C20A—H20C | 109.5 | C17B—C18B—C13B | 120.9 (4) |
| H20A—C20A—H20C | 109.5 | C17B—C18B—H18B | 119.5 |
| H20B—C20A—H20C | 109.5 | C13B—C18B—H18B | 119.5 |
| C19B—O3B—C20B | 116.5 (4) | O2B—C19B—O3B | 122.9 (5) |
| C2B—C1B—C6B | 120.8 (4) | O2B—C19B—C12B | 122.4 (5) |
| C2B—C1B—H1BA | 119.6 | O3B—C19B—C12B | 114.6 (4) |
| C6B—C1B—H1BA | 119.6 | O2B—C19B—C12C | 156.0 (9) |
| C3B—C2B—C1B | 118.5 (4) | O3B—C19B—C12C | 81.1 (8) |
| C3B—C2B—H2BA | 120.8 | C12B—C19B—C12C | 33.6 (7) |
| C1B—C2B—H2BA | 120.8 | O3B—C20B—H20D | 109.5 |
| C2B—C3B—F1B | 118.8 (4) | O3B—C20B—H20E | 109.5 |
| C2B—C3B—C4B | 122.1 (4) | H20D—C20B—H20E | 109.5 |
| F1B—C3B—C4B | 119.1 (4) | O3B—C20B—H20F | 109.5 |
| C3B—C4B—C5B | 118.4 (4) | H20D—C20B—H20F | 109.5 |
| C3B—C4B—H4BA | 120.8 | H20E—C20B—H20F | 109.5 |
| C5B—C4B—H4BA | 120.8 | | |
| C6A—C1A—C2A—C3A | 0.5 (6) | C7B—C6B—C7C—C12C | -46 (2) |
| C1A—C2A—C3A—C4A | -0.9 (7) | C5B—C6B—C7C—C8B | 43 (3) |
| C1A—C2A—C3A—F1A | 179.6 (4) | C1B—C6B—C7C—C8B | -123.0 (17) |
| C2A—C3A—C4A—C5A | 1.2 (7) | C7B—C6B—C7C—C8B | 75 (3) |
| F1A—C3A—C4A—C5A | -179.3 (4) | C12C—C7C—C8B—C9B | -48 (3) |
| C3A—C4A—C5A—C6A | -1.0 (7) | C6B—C7C—C8B—C9B | -161.5 (11) |
| C4A—C5A—C6A—C1A | 0.6 (6) | C12C—C7C—C8B—C7B | 53 (3) |
| C4A—C5A—C6A—C7A | 178.8 (4) | C6B—C7C—C8B—C7B | -61 (2) |
| C2A—C1A—C6A—C5A | -0.3 (6) | C6B—C7B—C8B—C7C | 85 (3) |
| C2A—C1A—C6A—C7A | -178.5 (4) | C12B—C7B—C8B—C7C | -40 (3) |
| C5A—C6A—C7A—C8A | -72.1 (5) | C6B—C7B—C8B—C9B | 175.0 (3) |
| C1A—C6A—C7A—C8A | 106.0 (4) | C12B—C7B—C8B—C9B | 50.5 (5) |
| C5A—C6A—C7A—C12A | 52.9 (5) | C7C—C8B—C9B—C10B | 7.0 (15) |
| C1A—C6A—C7A—C12A | -128.9 (4) | C7B—C8B—C9B—C10B | -21.0 (5) |
| C6A—C7A—C8A—C9A | 174.9 (3) | C7C—C8B—C9B—C13B | -174.2 (15) |
| C12A—C7A—C8A—C9A | 48.8 (4) | C7B—C8B—C9B—C13B | 157.7 (4) |
| C7A—C8A—C9A—C10A | -20.7 (4) | C13B—C9B—C10B—C11B | -179.9 (4) |
| C7A—C8A—C9A—C13A | 159.1 (3) | C8B—C9B—C10B—C11B | -1.2 (5) |
| C13A—C9A—C10A—C11A | 179.0 (3) | C9B—C10B—C11B—O1B | 176.5 (4) |
| C8A—C9A—C10A—C11A | -1.1 (5) | C9B—C10B—C11B—C12B | -7.6 (6) |
| C9A—C10A—C11A—O1A | 175.3 (4) | C9B—C10B—C11B—C12C | 28.6 (9) |
| C9A—C10A—C11A—C12A | -6.8 (5) | C6B—C7B—C12B—C19B | 56.2 (7) |
| O1A—C11A—C12A—C19A | -23.2 (4) | C8B—C7B—C12B—C19B | 179.2 (4) |
| C10A—C11A—C12A—C19A | 158.8 (3) | C6B—C7B—C12B—C11B | 179.8 (4) |
| O1A—C11A—C12A—C7A | -147.0 (3) | C8B—C7B—C12B—C11B | -57.2 (6) |
| C10A—C11A—C12A—C7A | 35.0 (4) | O1B—C11B—C12B—C19B | -20.0 (6) |
| C6A—C7A—C12A—C19A | 57.7 (4) | C10B—C11B—C12B—C19B | 163.9 (4) |
| C8A—C7A—C12A—C19A | -176.5 (3) | C12C—C11B—C12B—C19B | 79.6 (13) |
| C6A—C7A—C12A—C11A | 179.2 (3) | O1B—C11B—C12B—C7B | -146.6 (5) |
| C8A—C7A—C12A—C11A | -55.1 (4) | C10B—C11B—C12B—C7B | 37.4 (6) |

supplementary materials

| | | | |
|---------------------|-------------|---------------------|-------------|
| C10A—C9A—C13A—C14A | -144.8 (4) | C12C—C11B—C12B—C7B | -47.0 (12) |
| C8A—C9A—C13A—C14A | 35.4 (5) | C8B—C7C—C12C—C11B | 71 (2) |
| C10A—C9A—C13A—C18A | 35.0 (6) | C6B—C7C—C12C—C11B | -173.6 (13) |
| C8A—C9A—C13A—C18A | -144.9 (4) | C8B—C7C—C12C—C19B | 171.4 (15) |
| C18A—C13A—C14A—C15A | -0.5 (6) | C6B—C7C—C12C—C19B | -74 (2) |
| C9A—C13A—C14A—C15A | 179.3 (4) | O1B—C11B—C12C—C7C | 152.4 (16) |
| C13A—C14A—C15A—C16A | 0.9 (7) | C10B—C11B—C12C—C7C | -58.7 (19) |
| C14A—C15A—C16A—C17A | -0.8 (8) | C12B—C11B—C12C—C7C | 51.8 (17) |
| C14A—C15A—C16A—F2A | 178.9 (4) | O1B—C11B—C12C—C19B | 44.7 (12) |
| F2A—C16A—C17A—C18A | -179.4 (6) | C10B—C11B—C12C—C19B | -166.5 (6) |
| C15A—C16A—C17A—C18A | 0.4 (10) | C12B—C11B—C12C—C19B | -55.9 (9) |
| C16A—C17A—C18A—C13A | 0.1 (9) | C10B—C9B—C13B—C14B | -160.8 (4) |
| C14A—C13A—C18A—C17A | 0.0 (8) | C8B—C9B—C13B—C14B | 20.5 (5) |
| C9A—C13A—C18A—C17A | -179.7 (5) | C10B—C9B—C13B—C18B | 20.0 (6) |
| C20A—O3A—C19A—O2A | 3.9 (7) | C8B—C9B—C13B—C18B | -158.7 (4) |
| C20A—O3A—C19A—C12A | -175.3 (5) | C18B—C13B—C14B—C15B | -1.2 (7) |
| C11A—C12A—C19A—O2A | -84.5 (4) | C9B—C13B—C14B—C15B | 179.5 (4) |
| C7A—C12A—C19A—O2A | 37.7 (5) | C13B—C14B—C15B—C16B | 0.9 (7) |
| C11A—C12A—C19A—O3A | 94.8 (4) | C14B—C15B—C16B—C17B | 0.0 (8) |
| C7A—C12A—C19A—O3A | -143.1 (3) | C14B—C15B—C16B—F2B | 177.7 (4) |
| C6B—C1B—C2B—C3B | 0.1 (7) | F2B—C16B—C17B—C18B | -178.3 (6) |
| C1B—C2B—C3B—F1B | 179.5 (4) | C15B—C16B—C17B—C18B | -0.6 (10) |
| C1B—C2B—C3B—C4B | -1.2 (7) | C16B—C17B—C18B—C13B | 0.3 (10) |
| C2B—C3B—C4B—C5B | 1.5 (7) | C14B—C13B—C18B—C17B | 0.6 (8) |
| F1B—C3B—C4B—C5B | -179.2 (4) | C9B—C13B—C18B—C17B | 179.9 (5) |
| C3B—C4B—C5B—C6B | -0.8 (7) | C20B—O3B—C19B—O2B | 0.0 (8) |
| C4B—C5B—C6B—C1B | -0.1 (7) | C20B—O3B—C19B—C12B | 179.3 (5) |
| C4B—C5B—C6B—C7B | -178.9 (5) | C20B—O3B—C19B—C12C | 179.3 (8) |
| C4B—C5B—C6B—C7C | -165.1 (15) | C7B—C12B—C19B—O2B | -132.9 (6) |
| C2B—C1B—C6B—C5B | 0.5 (6) | C11B—C12B—C19B—O2B | 103.9 (6) |
| C2B—C1B—C6B—C7B | 179.1 (4) | C7B—C12B—C19B—O3B | 47.9 (6) |
| C2B—C1B—C6B—C7C | 170.2 (10) | C11B—C12B—C19B—O3B | -75.4 (5) |
| C5B—C6B—C7B—C12B | -150.1 (5) | C7B—C12B—C19B—C12C | 47.8 (13) |
| C1B—C6B—C7B—C12B | 31.3 (7) | C11B—C12B—C19B—C12C | -75.5 (13) |
| C7C—C6B—C7B—C12B | 53 (2) | C7C—C12C—C19B—O2B | -43 (3) |
| C5B—C6B—C7B—C8B | 87.4 (5) | C11B—C12C—C19B—O2B | 59 (2) |
| C1B—C6B—C7B—C8B | -91.2 (5) | C7C—C12C—C19B—O3B | 138.5 (18) |
| C7C—C6B—C7B—C8B | -69 (3) | C11B—C12C—C19B—O3B | -119.8 (9) |
| C5B—C6B—C7C—C12C | -78 (2) | C7C—C12C—C19B—C12B | -41.6 (15) |
| C1B—C6B—C7C—C12C | 115 (2) | C11B—C12C—C19B—C12B | 60.1 (10) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|-------|-------------|-------------|---------------|
| C8A—H8AB \cdots O2B ⁱ | 0.97 | 2.38 | 3.263 (5) | 151 |
| C8B—H8BB \cdots O2A ⁱⁱ | 0.97 | 2.57 | 3.404 (5) | 144 |
| C15B—H15B \cdots O1B ⁱⁱⁱ | 0.93 | 2.55 | 3.226 (6) | 130 |

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

Fig. 1

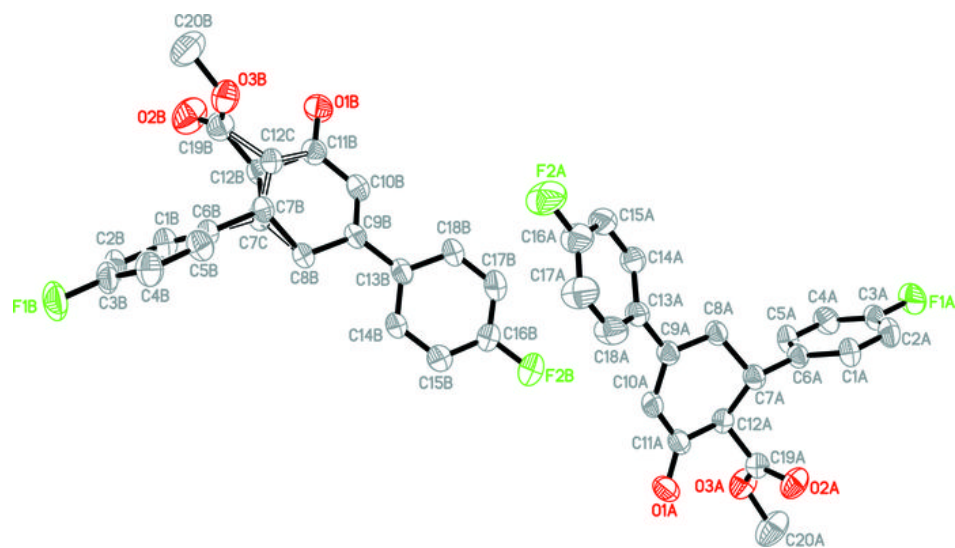


Fig. 2

