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## Structure Reports

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## (2E)-3-[4-(Benzyloxy)phenyl]-1-(2,6-dichloro-3-fluorophenyl)prop-2-en-1-one

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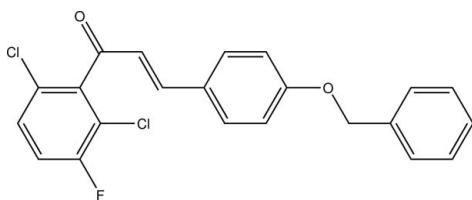
Received 4 November 2012; accepted 14 November 2012

Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.101; data-to-parameter ratio = 19.3.

In the title compound,  $\text{C}_{22}\text{H}_{15}\text{Cl}_2\text{FO}_2$ , a chalcone derivative featuring a threefold-halogenated aromatic substituent, the conformation about the  $\text{C}=\text{C}$  bond is *E*. In the crystal  $\text{C}-\text{H}\cdots\text{F}$  and  $\text{C}-\text{H}\cdots\text{Cl}$  contacts connect the molecules into undulating sheets parallel to (101). In addition,  $\text{C}-\text{H}\cdots\pi$  interactions are also present.

## Related literature

For background to possible applications of chalcones in pharmacy and industry, see: Lin *et al.* (2002); Modzelewska *et al.* (2006); Svetaz *et al.* (2004); Sarojini *et al.* (2006). For related structures, see: Yathirajan *et al.* (2006); Betz *et al.* (2012). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



## Experimental

## Crystal data

$\text{C}_{22}\text{H}_{15}\text{Cl}_2\text{FO}_2$   
 $M_r = 401.24$   
 Monoclinic,  $P2_1/c$   
 $a = 9.1977$  (2) Å  
 $b = 21.6887$  (4) Å  
 $c = 11.6072$  (2) Å  
 $\beta = 124.629$  (1)°

$V = 1905.29$  (6) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.36$  mm<sup>-1</sup>  
 $T = 200$  K  
 $0.40 \times 0.17 \times 0.14$  mm

## Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.681$ ,  $T_{\max} = 0.746$

17237 measured reflections  
 4712 independent reflections  
 3729 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.101$   
 $S = 1.02$   
 4712 reflections

244 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.40$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.27$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C21–C26 ring.

| $D-H\cdots A$                      | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C23–H23 $\cdots$ F1 <sup>i</sup>   | 0.95  | 2.55        | 3.375 (2)   | 145           |
| C34–H34 $\cdots$ Cl1 <sup>ii</sup> | 0.95  | 2.80        | 3.5462 (18) | 136           |
| C14–H14 $\cdots$ Cg <sup>iii</sup> | 0.95  | 2.51        | 3.297 (2)   | 140           |

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

Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT (Bruker, 2010); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 2012) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

ASP thanks the UOM for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2298).

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## supporting information

*Acta Cryst.* (2012). E68, o3386 [doi:10.1107/S1600536812046855]

**(2E)-3-[4-(Benzyloxy)phenyl]-1-(2,6-dichloro-3-fluorophenyl)prop-2-en-1-one**

**Aletti S. Praveen, Hemmige S. Yathirajan, Thomas Gerber, Benjamin van Brecht and Richard Betz**

**S1. Comment**

Chalcones are  $\alpha$ - $\beta$ -unsaturated ketones containing a reactive Michael system. Some substituted chalcones and their derivatives have been reported to possess interesting biological properties such as antitubercular (Lin *et al.*, 2002), anticancer (Modzelewska *et al.*, 2006) and antifungal (Svetaz *et al.*, 2004) activity. Chalcones also find application as organic nonlinear optical materials for their SHG conversion efficiency (Sarojini *et al.*, 2006). The crystal structures of some chalcones have been reported (Yathirajan *et al.*, 2006; Betz *et al.*, 2012). As part of our ongoing studies on chalcones, the title compound was synthesized.

The C=C bond in the Michael system is (*E*)-configured. The least-squares planes defined by the respective carbon atoms of the two terminal aromatic moieties intersect at an angle of 48.50 (10) ° and enclose angles of 62.82 (11) ° and 74.58 (8) ° with the least-squares plane defined by the carbon atoms of the central phenyl group. The larger of the latter two angles is created by the halogenated phenyl moiety (Fig 1).

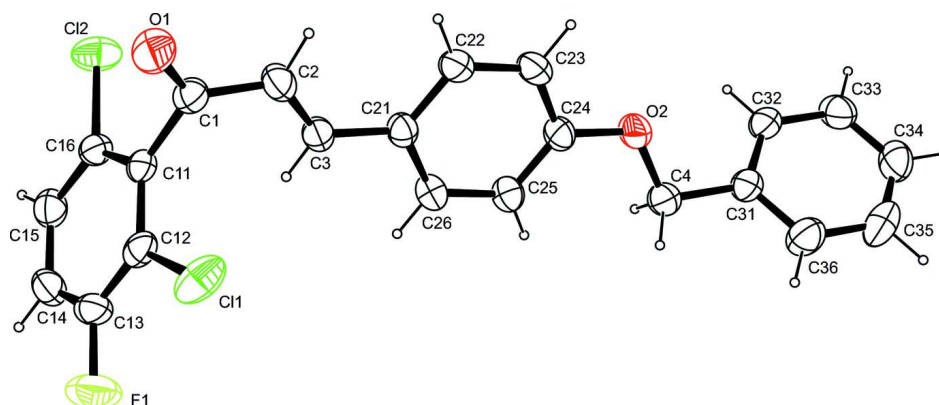
In the crystal, intermolecular C–H $\cdots$ F and C–H $\cdots$ Cl contacts whose range invariably falls by more than 0.1 Å below the sum of van-der-Waals radii of the corresponding atoms are observed. These contacts are exclusively supported by hydrogen atoms on the central as well as the terminal non-halogenated phenyl group and connect the molecules to undulated sheets parallel to [1 0 1]. In addition, C–H $\cdots$  $\pi$  interactions are present. Details about metrical parameters of these contacts as well as information about their symmetry can be found in Table 1. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the C–H $\cdots$ F as well as the C–H $\cdots$ Cl contacts necessitate a  $C^1_1(11)C^1_1(17)$  descriptor on the unary level. The shortest intercentroid distance between two aromatic systems was found at 4.5552 (12) Å and is apparent between the central as well as the halogenated phenyl moiety in neighbouring molecules.

**S2. Experimental**

To a stirred solution of 1-(2,6-dichloro-3-fluorophenyl)ethanone (1 g, 4.8 mmol) and 4-(benzyloxy)benzaldehyde (1.01 g, 4.8 mmol) in ethanol (10 ml), powdered KOH (0.40 g, 7.2 mmol) was added at 273 K. The reaction mixture was stirred at room temperature for 2 h. After completion of the reaction, the mixture was poured into ice cold water, acidified with HCl (1.5 N) until the pH value was approximately 3. The solid that precipitated was filtered and dried to afford the title compound as off-white solid, yield: 1.8 g (95%). Single crystals suitable for the X-ray diffraction study were grown from a mixture of toluene and acetone (*v:v* = 1:1) by slow evaporation at room temperature.

**S3. Refinement**

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic and vinylic carbon atoms, C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to  $1.2U_{\text{eq}}(\text{C})$ .

**Figure 1**

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

**(2E)-3-[4-(Benzyloxy)phenyl]-1-(2,6-dichloro-3-fluorophenyl)prop-2-en-1-one**

*Crystal data*

$C_{22}H_{15}Cl_2FO_2$

$M_r = 401.24$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.1977 (2) \text{ \AA}$

$b = 21.6887 (4) \text{ \AA}$

$c = 11.6072 (2) \text{ \AA}$

$\beta = 124.629 (1)^\circ$

$V = 1905.29 (6) \text{ \AA}^3$

$Z = 4$

$F(000) = 824$

$D_x = 1.399 \text{ Mg m}^{-3}$

Melting point = 369–367 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8954 reflections

$\theta = 2.5\text{--}28.3^\circ$

$\mu = 0.36 \text{ mm}^{-1}$

$T = 200 \text{ K}$

Cube, white

$0.40 \times 0.17 \times 0.14 \text{ mm}$

*Data collection*

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.681$ ,  $T_{\max} = 0.746$

17237 measured reflections

4712 independent reflections

3729 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.3^\circ$

$h = -12 \rightarrow 12$

$k = -20 \rightarrow 28$

$l = -15 \rightarrow 15$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.101$

$S = 1.02$

4712 reflections

244 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.8279P]$

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

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

$\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.27 \text{ e \AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| C11 | 1.07669 (6)  | 0.33062 (2)   | 0.86095 (4)   | 0.05144 (14)                     |
| C12 | 1.60001 (7)  | 0.17443 (2)   | 1.21644 (5)   | 0.05090 (14)                     |
| F1  | 1.35724 (19) | 0.38832 (5)   | 0.85238 (13)  | 0.0634 (3)                       |
| O1  | 1.20670 (18) | 0.23629 (6)   | 1.15009 (13)  | 0.0490 (3)                       |
| O2  | 0.66727 (17) | -0.00811 (5)  | 0.41450 (12)  | 0.0422 (3)                       |
| C1  | 1.2218 (2)   | 0.22034 (7)   | 1.05687 (16)  | 0.0338 (3)                       |
| C2  | 1.1208 (2)   | 0.16972 (8)   | 0.96163 (17)  | 0.0384 (4)                       |
| H2  | 1.0530       | 0.1445        | 0.9813        | 0.046*                           |
| C3  | 1.1186 (2)   | 0.15692 (7)   | 0.84785 (16)  | 0.0319 (3)                       |
| H3  | 1.1970       | 0.1800        | 0.8360        | 0.038*                           |
| C4  | 0.6360 (2)   | -0.00076 (8)  | 0.27948 (17)  | 0.0397 (4)                       |
| H4A | 0.7452       | -0.0099       | 0.2848        | 0.048*                           |
| H4B | 0.6001       | 0.0422        | 0.2465        | 0.048*                           |
| C11 | 1.3486 (2)   | 0.25530 (6)   | 1.03696 (14)  | 0.0283 (3)                       |
| C12 | 1.2938 (2)   | 0.30701 (7)   | 0.95115 (15)  | 0.0323 (3)                       |
| C13 | 1.4134 (3)   | 0.33938 (7)   | 0.93831 (17)  | 0.0394 (4)                       |
| C14 | 1.5873 (3)   | 0.32290 (8)   | 1.01107 (18)  | 0.0421 (4)                       |
| H14 | 1.6678       | 0.3463        | 1.0022        | 0.050*                           |
| C15 | 1.6452 (2)   | 0.27204 (8)   | 1.09749 (17)  | 0.0393 (4)                       |
| H15 | 1.7658       | 0.2601        | 1.1489        | 0.047*                           |
| C16 | 1.5253 (2)   | 0.23868 (7)   | 1.10837 (15)  | 0.0316 (3)                       |
| C21 | 1.0085 (2)   | 0.11127 (7)   | 0.73997 (16)  | 0.0316 (3)                       |
| C22 | 0.8987 (2)   | 0.06930 (8)   | 0.74900 (17)  | 0.0397 (4)                       |
| H22 | 0.9000       | 0.0682        | 0.8314        | 0.048*                           |
| C23 | 0.7894 (3)   | 0.02991 (8)   | 0.64018 (18)  | 0.0423 (4)                       |
| H23 | 0.7164       | 0.0018        | 0.6482        | 0.051*                           |
| C24 | 0.7851 (2)   | 0.03105 (7)   | 0.51816 (16)  | 0.0343 (3)                       |
| C25 | 0.8940 (2)   | 0.07133 (7)   | 0.50716 (17)  | 0.0340 (3)                       |
| H25 | 0.8936       | 0.0719        | 0.4252        | 0.041*                           |
| C26 | 1.0036 (2)   | 0.11089 (7)   | 0.61807 (17)  | 0.0338 (3)                       |
| H26 | 1.0775       | 0.1386        | 0.6102        | 0.041*                           |
| C31 | 0.4920 (2)   | -0.04454 (7)  | 0.18024 (16)  | 0.0351 (3)                       |
| C32 | 0.5074 (2)   | -0.10705 (8)  | 0.21101 (17)  | 0.0392 (4)                       |
| H32 | 0.6079       | -0.1217       | 0.2969        | 0.047*                           |
| C33 | 0.3791 (3)   | -0.14809 (9)  | 0.11877 (18)  | 0.0459 (4)                       |
| H33 | 0.3918       | -0.1907       | 0.1414        | 0.055*                           |
| C34 | 0.2328 (3)   | -0.12764 (10) | -0.00587 (19) | 0.0499 (5)                       |
| H34 | 0.1447       | -0.1561       | -0.0695       | 0.060*                           |
| C35 | 0.2144 (3)   | -0.06611 (11) | -0.03803 (19) | 0.0570 (5)                       |
| H35 | 0.1131       | -0.0519       | -0.1240       | 0.068*                           |
| C36 | 0.3439 (3)   | -0.02427 (9)  | 0.05494 (19)  | 0.0489 (4)                       |
| H36 | 0.3302       | 0.0183        | 0.0321        | 0.059*                           |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$   | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|------------|--------------|--------------|--------------|
| C11 | 0.0470 (3)  | 0.0639 (3)  | 0.0343 (2) | 0.0235 (2)   | 0.01765 (19) | 0.00794 (19) |
| C12 | 0.0530 (3)  | 0.0481 (3)  | 0.0527 (3) | 0.0169 (2)   | 0.0307 (2)   | 0.0229 (2)   |
| F1  | 0.0982 (10) | 0.0362 (6)  | 0.0626 (7) | 0.0085 (6)   | 0.0497 (7)   | 0.0160 (5)   |
| O1  | 0.0587 (8)  | 0.0596 (8)  | 0.0430 (7) | -0.0113 (7)  | 0.0375 (7)   | -0.0124 (6)  |
| O2  | 0.0528 (7)  | 0.0349 (6)  | 0.0353 (6) | -0.0162 (5)  | 0.0228 (6)   | -0.0089 (5)  |
| C1  | 0.0340 (8)  | 0.0385 (8)  | 0.0293 (7) | -0.0024 (7)  | 0.0183 (6)   | -0.0012 (6)  |
| C2  | 0.0388 (9)  | 0.0404 (9)  | 0.0404 (8) | -0.0120 (7)  | 0.0251 (7)   | -0.0061 (7)  |
| C3  | 0.0299 (8)  | 0.0297 (7)  | 0.0336 (7) | -0.0026 (6)  | 0.0166 (6)   | -0.0014 (6)  |
| C4  | 0.0460 (9)  | 0.0328 (8)  | 0.0352 (8) | -0.0060 (7)  | 0.0200 (7)   | -0.0020 (7)  |
| C11 | 0.0337 (8)  | 0.0275 (7)  | 0.0219 (6) | -0.0026 (6)  | 0.0147 (6)   | -0.0044 (5)  |
| C12 | 0.0373 (8)  | 0.0321 (7)  | 0.0233 (7) | 0.0029 (6)   | 0.0147 (6)   | -0.0032 (6)  |
| C13 | 0.0610 (11) | 0.0252 (7)  | 0.0335 (8) | -0.0018 (7)  | 0.0277 (8)   | 0.0006 (6)   |
| C14 | 0.0498 (10) | 0.0384 (9)  | 0.0427 (9) | -0.0165 (8)  | 0.0291 (8)   | -0.0073 (7)  |
| C15 | 0.0339 (8)  | 0.0436 (9)  | 0.0362 (8) | -0.0060 (7)  | 0.0174 (7)   | -0.0049 (7)  |
| C16 | 0.0355 (8)  | 0.0292 (7)  | 0.0271 (7) | 0.0012 (6)   | 0.0160 (6)   | 0.0017 (6)   |
| C21 | 0.0304 (7)  | 0.0271 (7)  | 0.0334 (7) | -0.0014 (6)  | 0.0157 (6)   | -0.0019 (6)  |
| C22 | 0.0506 (10) | 0.0338 (8)  | 0.0338 (8) | -0.0099 (7)  | 0.0235 (8)   | -0.0017 (6)  |
| C23 | 0.0532 (11) | 0.0325 (8)  | 0.0405 (9) | -0.0151 (8)  | 0.0261 (8)   | -0.0026 (7)  |
| C24 | 0.0385 (9)  | 0.0235 (7)  | 0.0355 (8) | -0.0037 (6)  | 0.0178 (7)   | -0.0043 (6)  |
| C25 | 0.0351 (8)  | 0.0325 (8)  | 0.0365 (8) | -0.0018 (7)  | 0.0216 (7)   | -0.0052 (6)  |
| C26 | 0.0323 (8)  | 0.0316 (8)  | 0.0401 (8) | -0.0043 (6)  | 0.0221 (7)   | -0.0051 (6)  |
| C31 | 0.0375 (8)  | 0.0357 (8)  | 0.0298 (7) | -0.0025 (7)  | 0.0178 (7)   | -0.0019 (6)  |
| C32 | 0.0424 (9)  | 0.0380 (9)  | 0.0296 (8) | -0.0038 (7)  | 0.0160 (7)   | 0.0003 (6)   |
| C33 | 0.0602 (12) | 0.0400 (9)  | 0.0399 (9) | -0.0137 (9)  | 0.0299 (9)   | -0.0075 (7)  |
| C34 | 0.0494 (11) | 0.0637 (12) | 0.0352 (9) | -0.0186 (10) | 0.0232 (8)   | -0.0167 (8)  |
| C35 | 0.0410 (10) | 0.0772 (15) | 0.0328 (9) | 0.0045 (10)  | 0.0091 (8)   | -0.0019 (9)  |
| C36 | 0.0474 (11) | 0.0455 (10) | 0.0409 (9) | 0.0067 (8)   | 0.0175 (8)   | 0.0052 (8)   |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| C11—C12 | 1.7230 (17) | C15—H15 | 0.9500    |
| C12—C16 | 1.7344 (15) | C21—C26 | 1.389 (2) |
| F1—C13  | 1.3425 (18) | C21—C22 | 1.408 (2) |
| O1—C1   | 1.2167 (19) | C22—C23 | 1.376 (2) |
| O2—C24  | 1.3643 (18) | C22—H22 | 0.9500    |
| O2—C4   | 1.4327 (19) | C23—C24 | 1.395 (2) |
| C1—C2   | 1.457 (2)   | C23—H23 | 0.9500    |
| C1—C11  | 1.515 (2)   | C24—C25 | 1.388 (2) |
| C2—C3   | 1.338 (2)   | C25—C26 | 1.391 (2) |
| C2—H2   | 0.9500      | C25—H25 | 0.9500    |
| C3—C21  | 1.460 (2)   | C26—H26 | 0.9500    |
| C3—H3   | 0.9500      | C31—C36 | 1.383 (2) |
| C4—C31  | 1.500 (2)   | C31—C32 | 1.389 (2) |
| C4—H4A  | 0.9900      | C32—C33 | 1.377 (2) |
| C4—H4B  | 0.9900      | C32—H32 | 0.9500    |

|               |              |                 |             |
|---------------|--------------|-----------------|-------------|
| C11—C16       | 1.388 (2)    | C33—C34         | 1.374 (3)   |
| C11—C12       | 1.390 (2)    | C33—H33         | 0.9500      |
| C12—C13       | 1.383 (2)    | C34—C35         | 1.370 (3)   |
| C13—C14       | 1.366 (3)    | C34—H34         | 0.9500      |
| C14—C15       | 1.378 (2)    | C35—C36         | 1.395 (3)   |
| C14—H14       | 0.9500       | C35—H35         | 0.9500      |
| C15—C16       | 1.383 (2)    | C36—H36         | 0.9500      |
|               |              |                 |             |
| C24—O2—C4     | 117.41 (12)  | C26—C21—C3      | 118.88 (14) |
| O1—C1—C2      | 122.91 (15)  | C22—C21—C3      | 123.48 (14) |
| O1—C1—C11     | 118.69 (14)  | C23—C22—C21     | 120.93 (15) |
| C2—C1—C11     | 118.39 (13)  | C23—C22—H22     | 119.5       |
| C3—C2—C1      | 123.41 (15)  | C21—C22—H22     | 119.5       |
| C3—C2—H2      | 118.3        | C22—C23—C24     | 120.38 (15) |
| C1—C2—H2      | 118.3        | C22—C23—H23     | 119.8       |
| C2—C3—C21     | 126.97 (15)  | C24—C23—H23     | 119.8       |
| C2—C3—H3      | 116.5        | O2—C24—C25      | 124.31 (14) |
| C21—C3—H3     | 116.5        | O2—C24—C23      | 115.75 (14) |
| O2—C4—C31     | 108.06 (13)  | C25—C24—C23     | 119.92 (14) |
| O2—C4—H4A     | 110.1        | C24—C25—C26     | 118.97 (14) |
| C31—C4—H4A    | 110.1        | C24—C25—H25     | 120.5       |
| O2—C4—H4B     | 110.1        | C26—C25—H25     | 120.5       |
| C31—C4—H4B    | 110.1        | C21—C26—C25     | 122.23 (15) |
| H4A—C4—H4B    | 108.4        | C21—C26—H26     | 118.9       |
| C16—C11—C12   | 117.55 (14)  | C25—C26—H26     | 118.9       |
| C16—C11—C1    | 121.37 (13)  | C36—C31—C32     | 118.46 (16) |
| C12—C11—C1    | 121.01 (14)  | C36—C31—C4      | 121.24 (16) |
| C13—C12—C11   | 119.96 (15)  | C32—C31—C4      | 120.27 (15) |
| C13—C12—C11   | 120.13 (12)  | C33—C32—C31     | 120.94 (16) |
| C11—C12—C11   | 119.91 (13)  | C33—C32—H32     | 119.5       |
| F1—C13—C14    | 119.16 (16)  | C31—C32—H32     | 119.5       |
| F1—C13—C12    | 119.19 (17)  | C34—C33—C32     | 120.26 (18) |
| C14—C13—C12   | 121.65 (15)  | C34—C33—H33     | 119.9       |
| C13—C14—C15   | 119.46 (16)  | C32—C33—H33     | 119.9       |
| C13—C14—H14   | 120.3        | C35—C34—C33     | 119.79 (17) |
| C15—C14—H14   | 120.3        | C35—C34—H34     | 120.1       |
| C14—C15—C16   | 119.14 (16)  | C33—C34—H34     | 120.1       |
| C14—C15—H15   | 120.4        | C34—C35—C36     | 120.27 (18) |
| C16—C15—H15   | 120.4        | C34—C35—H35     | 119.9       |
| C15—C16—C11   | 122.21 (14)  | C36—C35—H35     | 119.9       |
| C15—C16—C12   | 118.54 (13)  | C31—C36—C35     | 120.29 (18) |
| C11—C16—C12   | 119.25 (12)  | C31—C36—H36     | 119.9       |
| C26—C21—C22   | 117.55 (14)  | C35—C36—H36     | 119.9       |
|               |              |                 |             |
| O1—C1—C2—C3   | 170.87 (17)  | C2—C3—C21—C26   | 169.97 (17) |
| C11—C1—C2—C3  | -8.0 (2)     | C2—C3—C21—C22   | -6.4 (3)    |
| C1—C2—C3—C21  | -173.56 (16) | C26—C21—C22—C23 | -0.6 (3)    |
| C24—O2—C4—C31 | -175.17 (14) | C3—C21—C22—C23  | 175.83 (16) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| O1—C1—C11—C16   | 88.4 (2)     | C21—C22—C23—C24 | -0.2 (3)     |
| C2—C1—C11—C16   | -92.69 (18)  | C4—O2—C24—C25   | -9.4 (2)     |
| O1—C1—C11—C12   | -88.61 (19)  | C4—O2—C24—C23   | 169.19 (15)  |
| C2—C1—C11—C12   | 90.29 (18)   | C22—C23—C24—O2  | -177.49 (16) |
| C16—C11—C12—C13 | 0.7 (2)      | C22—C23—C24—C25 | 1.2 (3)      |
| C1—C11—C12—C13  | 177.82 (13)  | O2—C24—C25—C26  | 177.34 (15)  |
| C16—C11—C12—C11 | -179.34 (11) | C23—C24—C25—C26 | -1.2 (2)     |
| C1—C11—C12—C11  | -2.20 (19)   | C22—C21—C26—C25 | 0.6 (2)      |
| C11—C12—C13—F1  | 178.39 (13)  | C3—C21—C26—C25  | -176.04 (15) |
| C11—C12—C13—F1  | -1.6 (2)     | C24—C25—C26—C21 | 0.3 (2)      |
| C11—C12—C13—C14 | -1.7 (2)     | O2—C4—C31—C36   | 127.10 (17)  |
| C11—C12—C13—C14 | 178.27 (13)  | O2—C4—C31—C32   | -54.7 (2)    |
| F1—C13—C14—C15  | -178.82 (15) | C36—C31—C32—C33 | 0.4 (3)      |
| C12—C13—C14—C15 | 1.3 (3)      | C4—C31—C32—C33  | -177.76 (16) |
| C13—C14—C15—C16 | 0.1 (2)      | C31—C32—C33—C34 | -0.1 (3)     |
| C14—C15—C16—C11 | -1.2 (2)     | C32—C33—C34—C35 | -0.3 (3)     |
| C14—C15—C16—C12 | 179.46 (12)  | C33—C34—C35—C36 | 0.3 (3)      |
| C12—C11—C16—C15 | 0.8 (2)      | C32—C31—C36—C35 | -0.4 (3)     |
| C1—C11—C16—C15  | -176.37 (14) | C4—C31—C36—C35  | 177.73 (18)  |
| C12—C11—C16—C12 | -179.88 (11) | C34—C35—C36—C31 | 0.1 (3)      |
| C1—C11—C16—C12  | 2.99 (19)    |                 |              |

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

*Cg* is the centroid of the C21–C26 ring.

| <i>D</i> —H... <i>A</i>             | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------------------|-------------|---------------|-----------------------|-------------------------|
| C23—H23...F1 <sup>i</sup>           | 0.95        | 2.55          | 3.375 (2)             | 145                     |
| C34—H34...C11 <sup>ii</sup>         | 0.95        | 2.80          | 3.5462 (18)           | 136                     |
| C14—H14... <i>Cg</i> <sup>iii</sup> | 0.95        | 2.51          | 3.297 (2)             | 140                     |

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