

4-(4-Fluorobenzoyl)-3-phenyl-3,4-dihydronaphthalen-1(2H)-one

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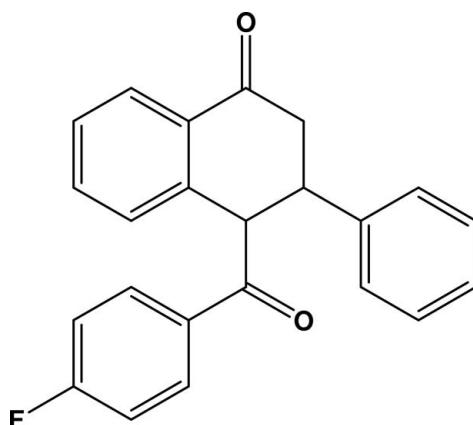
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Key indicators: single-crystal X-ray study; $T = 295\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 17.0.

In the title compound, $\text{C}_{23}\text{H}_{17}\text{FO}_2$, the cyclohexenone ring has an envelope conformation, the flap atom being the C atom to which the phenyl ring is attached. The 4-fluorobenzoyl ring and the phenyl ring are inclined to one another by $28.77(7)^\circ$, and by $52.00(7)$ and $44.77(7)^\circ$, respectively, to the aromatic ring fused to the cyclohexenone ring. In the crystal, molecules are linked via $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a two-dimensional network lying parallel to (100).

Related literature

For the domino reaction as an important tool in the construction of structurally complicated molecules, see: Zhao *et al.* (2012). For Pd-catalysed cascade reactions, see: Wang & Hu (2011); Yu & Hu (2012). For the use of condensed polycyclic compounds as synthetic building blocks, pharmacophores and electroluminescent materials, see: Rixson *et al.* (2012). For cross-coupling reactions of aryl halides with olefins and diynes, see: Hu *et al.* (2010, 2009).



Experimental

Crystal data

| | |
|-----------------------------------------|------------------------------------------|
| $\text{C}_{23}\text{H}_{17}\text{FO}_2$ | $V = 1726.1(2)\text{ \AA}^3$ |
| $M_r = 344.37$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 8.0063(6)\text{ \AA}$ | $\mu = 0.09\text{ mm}^{-1}$ |
| $b = 10.6688(8)\text{ \AA}$ | $T = 295\text{ K}$ |
| $c = 20.3796(15)\text{ \AA}$ | $0.35 \times 0.32 \times 0.29\text{ mm}$ |
| $\beta = 97.458(1)^\circ$ | |

Data collection

| | |
|-------------------------------------------------------------------|----------------------------------------|
| Bruker SMART APEX CCD diffractometer | 14632 measured reflections |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2000) | 3987 independent reflections |
| $T_{\min} = 0.969$, $T_{\max} = 0.974$ | 3176 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.025$ |

Refinement

| | |
|---------------------------------|-----------------------------------------------|
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | 235 parameters |
| $wR(F^2) = 0.117$ | H-atom parameters constrained |
| $S = 1.03$ | $\Delta\rho_{\max} = 0.23\text{ e \AA}^{-3}$ |
| 3987 reflections | $\Delta\rho_{\min} = -0.28\text{ e \AA}^{-3}$ |

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|-----------------------------------|--------------|--------------------|-------------|----------------------|
| C14—H14 \cdots O1 ⁱ | 0.93 | 2.53 | 3.425 (2) | 161 |
| C10—H10 \cdots O2 ⁱⁱ | 0.98 | 2.51 | 3.1427 (15) | 123 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2398).

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supplementary materials

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4-(4-Fluorobenzoyl)-3-phenyl-3,4-dihydronaphthalen-1(2H)-one

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Comment

Domino reaction as an important tool to construct structurally complicate molecule due to high atom economy and environmental benefits (Zhao *et al.*, 2012). Pd-catalyzed cascade reactions have become an efficient protocol of modern organic synthesis chemistry (Wang *et al.*, 2011; Yu *et al.*, 2012). Condensed polycyclic compounds are playing increasingly important roles as synthetic building blocks, pharmacophores, and electroluminescence materials (Rixson *et al.*, 2012). We have reported some novel cross-coupling reactions of aryl halides with the olefins and diynes (Hu *et al.*, 2009; 2010). The reaction of bromobenzene with 1-(2-((4-fluorophenyl)ethynyl)phenyl)prop-2-en-1-one, in the presence of Pd(II) acetate and triphenylphosphine, in *DMF* at 418 K for 19 h, gave the unexpected title product.

The crystal structure data of molecule, C₃₁H₃₀N₂O, reveals that all the bond lengths and angles have normal values. The titled molecule contains three phenyl ring and one six-membered carbon ring with a boat conformation. One phenyl ring and the *cis*-fused cyclohexene ring are common side. All the rings are not coplanar (Fig. 1). In the molecule there are two chiral carbon atoms, C9 and C10, but the crystal is a racemic system due to lacking of the chiral separation. In the crystal packing, there are weak intermolecular C–H···O interactions C14–H14···O1ⁱ which forms 1-D chain were formed between neighboring molecules along *c* axis (Fig. 2). Symmetry code: (i) *x*, -*y*+1/2, *z*-1/2.

Experimental

An oven-dried Schlenk flask was evacuated, filled with nitrogen, and then charged with 1-(2-((4-fluorophenyl)ethynyl)phenyl)prop-2-en-1-one (2.51 g, 10 mmol), bromobenzene (1.72 g, 11 mmol), tributylamine (3 ml), PPh₃ (52.5 mg, 0.2 mmol), Pd(OAc)₂ (24 mg, 0.1 mol), and *DMF* (10 ml) to give a yellow solution. The reaction mixture was heated at 418 K with stirring. The reaction mixture was cooled to room temperature after 19 h and the resultant yellow-orange mixture was diluted with *Et₂O* (10 ml). The mixture was washed with H₂O (15 ml) and the aqueous layer was extracted with *Et₂O* (20 ml). The combined organic layers were dried (MgSO₄), filtered, and concentrated *in vacuo*. The crude product was purified by flash column chromatography on silica gel (petroleum ester : *EtOAc* = 9 : 1) and recrystallized from *EtOAc*, yield 2.45 g (71%). Colourless crystals suitable for X-ray diffraction were obtained by recrystallization from a solution of the title compound from ethyl acetate over a period of one week.

Refinement

All H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 Å–0.98 Å with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Computing details

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT (Bruker, 2000); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication:

SHELXTL (Sheldrick, 2008).

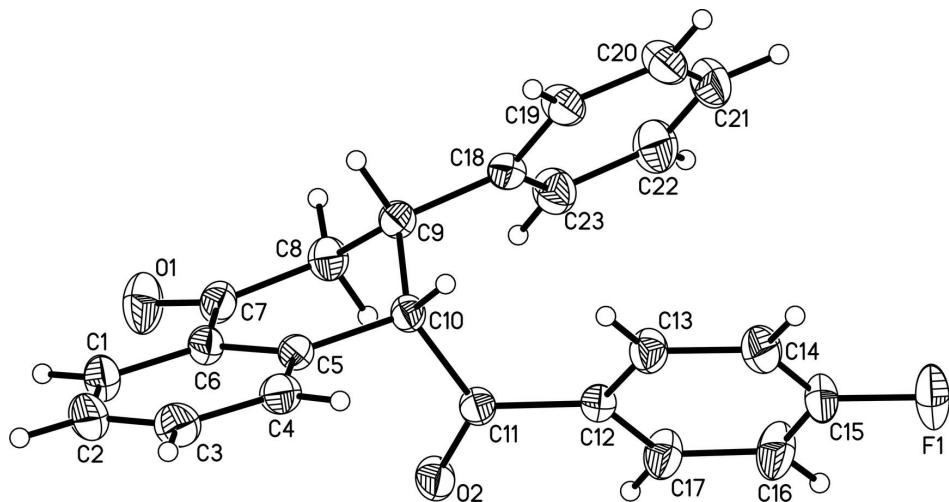


Figure 1

A view of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

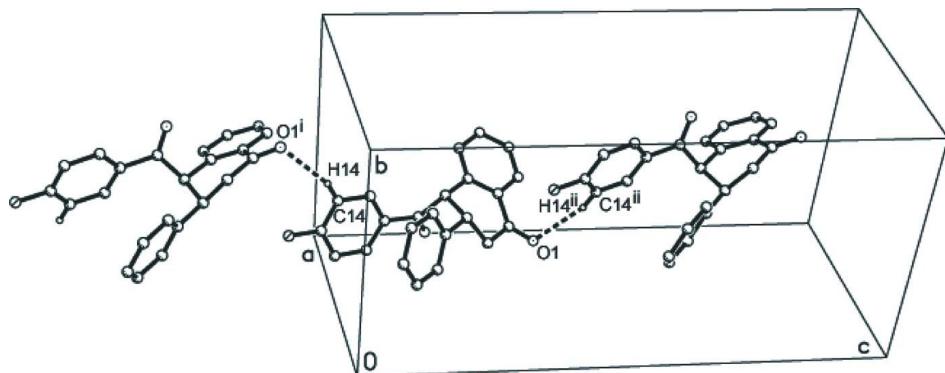
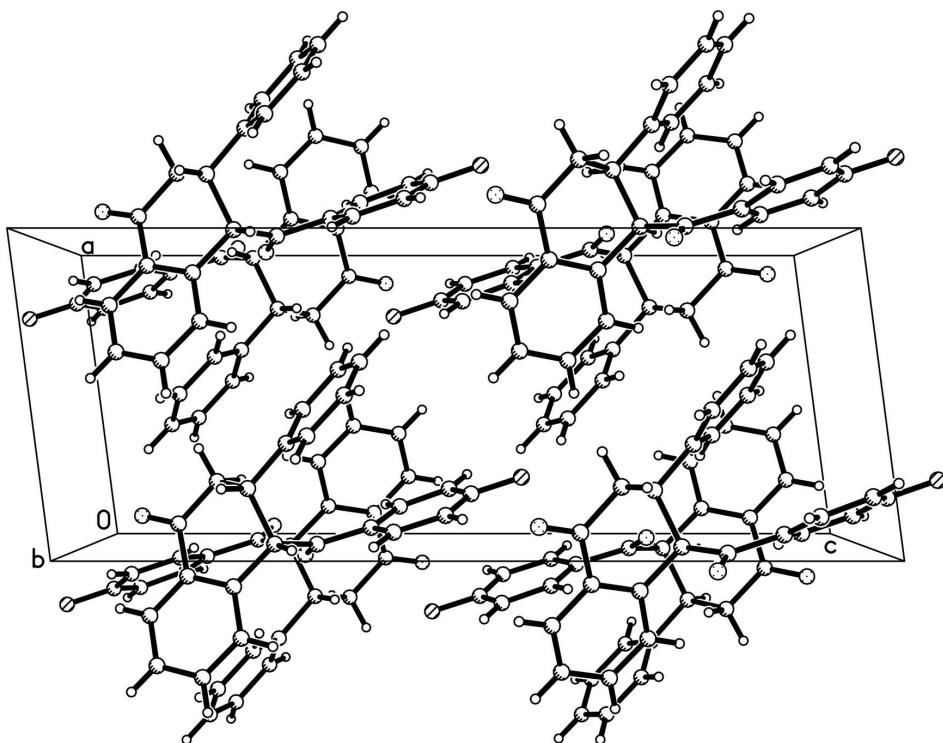


Figure 2

A view of 1-D chain along *c* axis. Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $x, -y+1/2, z+1/2$.

**Figure 3**

A view of the cell packing down *b* axis.

4-(4-Fluorobenzoyl)-3-phenyl-3,4-dihydronaphthalen-1(2*H*)-one

Crystal data

C₂₃H₁₇FO₂

M_r = 344.37

Monoclinic, *P*2₁/*c*

Hall symbol: -P 2ybc

a = 8.0063 (6) Å

b = 10.6688 (8) Å

c = 20.3796 (15) Å

β = 97.458 (1)°

V = 1726.1 (2) Å³

Z = 4

F(000) = 720

D_x = 1.325 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 2731 reflections

θ = 2.1–23.6°

μ = 0.09 mm⁻¹

T = 295 K

Block, colourless

0.35 × 0.32 × 0.29 mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: sealed tube

Graphite monochromator

φ- and ω-scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2000)

*T*_{min} = 0.969, *T*_{max} = 0.974

14632 measured reflections

3987 independent reflections

3176 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.025

θ_{max} = 27.6°, θ_{min} = 2.0°

h = -10→10

k = -13→13

l = -26→24

*Refinement*Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.044$$

$$wR(F^2) = 0.117$$

$$S = 1.03$$

3987 reflections

235 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.44P]$$

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

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

$$\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C1 | 1.2122 (2) | 0.13272 (15) | 0.42049 (7) | 0.0558 (4) |
| H1 | 1.1940 | 0.0940 | 0.4598 | 0.067* |
| C2 | 1.3639 (2) | 0.18874 (18) | 0.41545 (8) | 0.0638 (5) |
| H2 | 1.4479 | 0.1890 | 0.4515 | 0.077* |
| C3 | 1.39238 (19) | 0.24486 (16) | 0.35687 (8) | 0.0590 (4) |
| H3 | 1.4957 | 0.2826 | 0.3536 | 0.071* |
| C4 | 1.26809 (17) | 0.24528 (14) | 0.30301 (7) | 0.0474 (3) |
| H4 | 1.2892 | 0.2820 | 0.2635 | 0.057* |
| C5 | 1.11203 (16) | 0.19136 (12) | 0.30728 (6) | 0.0380 (3) |
| C6 | 1.08446 (17) | 0.13349 (12) | 0.36657 (6) | 0.0429 (3) |
| C7 | 0.92053 (19) | 0.07364 (12) | 0.37339 (6) | 0.0447 (3) |
| C8 | 0.78176 (17) | 0.08569 (13) | 0.31679 (7) | 0.0450 (3) |
| H8A | 0.6749 | 0.0903 | 0.3343 | 0.054* |
| H8B | 0.7800 | 0.0109 | 0.2896 | 0.054* |
| C9 | 0.79881 (15) | 0.19977 (12) | 0.27346 (6) | 0.0379 (3) |
| H9 | 0.7963 | 0.2734 | 0.3021 | 0.045* |
| C10 | 0.97521 (15) | 0.19869 (11) | 0.24892 (6) | 0.0346 (3) |
| H10 | 0.9888 | 0.2775 | 0.2255 | 0.041* |
| C11 | 0.98542 (16) | 0.09084 (11) | 0.20047 (6) | 0.0362 (3) |
| C12 | 0.92979 (15) | 0.11179 (11) | 0.12871 (6) | 0.0363 (3) |
| C13 | 0.93535 (19) | 0.22788 (13) | 0.09884 (7) | 0.0466 (3) |
| H13 | 0.9760 | 0.2966 | 0.1241 | 0.056* |
| C14 | 0.8814 (2) | 0.24341 (16) | 0.03199 (7) | 0.0573 (4) |
| H14 | 0.8879 | 0.3210 | 0.0117 | 0.069* |
| C15 | 0.8189 (2) | 0.14184 (17) | -0.00295 (7) | 0.0574 (4) |
| C16 | 0.8094 (2) | 0.02478 (18) | 0.02434 (8) | 0.0660 (5) |

| | | | | |
|-----|--------------|--------------|--------------|------------|
| H16 | 0.7648 | -0.0426 | -0.0011 | 0.079* |
| C17 | 0.8681 (2) | 0.00996 (14) | 0.09066 (7) | 0.0522 (4) |
| H17 | 0.8662 | -0.0689 | 0.1100 | 0.063* |
| C18 | 0.65496 (16) | 0.21523 (12) | 0.21773 (6) | 0.0400 (3) |
| C19 | 0.62893 (18) | 0.33103 (14) | 0.18694 (8) | 0.0496 (3) |
| H19 | 0.6977 | 0.3983 | 0.2016 | 0.060* |
| C20 | 0.5021 (2) | 0.34751 (17) | 0.13476 (8) | 0.0608 (4) |
| H20 | 0.4877 | 0.4252 | 0.1141 | 0.073* |
| C21 | 0.3973 (2) | 0.24981 (19) | 0.11325 (8) | 0.0655 (5) |
| H21 | 0.3120 | 0.2612 | 0.0782 | 0.079* |
| C22 | 0.4193 (2) | 0.13585 (18) | 0.14369 (9) | 0.0648 (5) |
| H22 | 0.3475 | 0.0698 | 0.1297 | 0.078* |
| C23 | 0.54788 (19) | 0.11786 (15) | 0.19539 (8) | 0.0524 (4) |
| H23 | 0.5624 | 0.0395 | 0.2153 | 0.063* |
| F1 | 0.76491 (16) | 0.15640 (12) | -0.06863 (5) | 0.0895 (4) |
| O1 | 0.89843 (17) | 0.01466 (11) | 0.42256 (5) | 0.0676 (3) |
| O2 | 1.02932 (15) | -0.01260 (9) | 0.22076 (5) | 0.0544 (3) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| C1 | 0.0642 (10) | 0.0602 (9) | 0.0388 (8) | 0.0110 (7) | -0.0090 (7) | -0.0028 (6) |
| C2 | 0.0542 (9) | 0.0783 (11) | 0.0523 (9) | 0.0133 (8) | -0.0180 (7) | -0.0134 (8) |
| C3 | 0.0405 (7) | 0.0705 (10) | 0.0628 (10) | 0.0041 (7) | -0.0052 (7) | -0.0164 (8) |
| C4 | 0.0418 (7) | 0.0519 (8) | 0.0474 (8) | 0.0032 (6) | 0.0013 (6) | -0.0090 (6) |
| C5 | 0.0411 (6) | 0.0355 (6) | 0.0356 (6) | 0.0058 (5) | -0.0025 (5) | -0.0072 (5) |
| C6 | 0.0510 (8) | 0.0405 (7) | 0.0349 (6) | 0.0072 (6) | -0.0033 (5) | -0.0044 (5) |
| C7 | 0.0633 (9) | 0.0361 (7) | 0.0339 (7) | 0.0038 (6) | 0.0028 (6) | -0.0010 (5) |
| C8 | 0.0474 (7) | 0.0448 (7) | 0.0424 (7) | -0.0022 (6) | 0.0048 (6) | 0.0037 (6) |
| C9 | 0.0398 (6) | 0.0359 (6) | 0.0370 (6) | 0.0018 (5) | 0.0014 (5) | -0.0020 (5) |
| C10 | 0.0396 (6) | 0.0301 (6) | 0.0327 (6) | 0.0003 (5) | -0.0004 (5) | -0.0008 (4) |
| C11 | 0.0406 (6) | 0.0316 (6) | 0.0353 (6) | -0.0001 (5) | 0.0007 (5) | -0.0007 (5) |
| C12 | 0.0383 (6) | 0.0373 (6) | 0.0329 (6) | 0.0014 (5) | 0.0028 (5) | -0.0008 (5) |
| C13 | 0.0598 (8) | 0.0407 (7) | 0.0382 (7) | -0.0004 (6) | 0.0020 (6) | 0.0027 (5) |
| C14 | 0.0731 (10) | 0.0558 (9) | 0.0422 (8) | 0.0100 (8) | 0.0050 (7) | 0.0127 (7) |
| C15 | 0.0601 (9) | 0.0781 (11) | 0.0311 (7) | 0.0101 (8) | -0.0048 (6) | 0.0025 (7) |
| C16 | 0.0846 (12) | 0.0691 (11) | 0.0407 (8) | -0.0139 (9) | -0.0061 (8) | -0.0110 (7) |
| C17 | 0.0715 (10) | 0.0449 (8) | 0.0387 (7) | -0.0079 (7) | 0.0013 (7) | -0.0022 (6) |
| C18 | 0.0362 (6) | 0.0441 (7) | 0.0395 (7) | 0.0054 (5) | 0.0036 (5) | -0.0030 (5) |
| C19 | 0.0455 (7) | 0.0466 (8) | 0.0550 (8) | 0.0065 (6) | -0.0002 (6) | 0.0026 (6) |
| C20 | 0.0554 (9) | 0.0670 (10) | 0.0586 (10) | 0.0192 (8) | 0.0014 (7) | 0.0122 (8) |
| C21 | 0.0489 (9) | 0.0930 (13) | 0.0505 (9) | 0.0118 (9) | -0.0088 (7) | -0.0001 (9) |
| C22 | 0.0524 (9) | 0.0777 (12) | 0.0597 (10) | -0.0067 (8) | -0.0104 (7) | -0.0119 (9) |
| C23 | 0.0505 (8) | 0.0510 (8) | 0.0533 (8) | -0.0019 (6) | -0.0032 (6) | -0.0033 (7) |
| F1 | 0.1108 (9) | 0.1147 (9) | 0.0360 (5) | 0.0106 (7) | -0.0174 (5) | 0.0072 (5) |
| O1 | 0.0949 (9) | 0.0638 (7) | 0.0418 (6) | -0.0137 (6) | 0.0003 (6) | 0.0132 (5) |
| O2 | 0.0832 (8) | 0.0338 (5) | 0.0426 (5) | 0.0118 (5) | -0.0052 (5) | 0.0003 (4) |

Geometric parameters (\AA , $\text{^{\circ}}$)

| | | | |
|-----------|-------------|-------------|-------------|
| C1—C2 | 1.369 (2) | C11—C12 | 1.4892 (17) |
| C1—C6 | 1.4009 (19) | C12—C13 | 1.3834 (18) |
| C1—H1 | 0.9300 | C12—C17 | 1.3880 (18) |
| C2—C3 | 1.381 (3) | C13—C14 | 1.3846 (19) |
| C2—H2 | 0.9300 | C13—H13 | 0.9300 |
| C3—C4 | 1.3825 (19) | C14—C15 | 1.356 (2) |
| C3—H3 | 0.9300 | C14—H14 | 0.9300 |
| C4—C5 | 1.3885 (19) | C15—F1 | 1.3610 (17) |
| C4—H4 | 0.9300 | C15—C16 | 1.373 (2) |
| C5—C6 | 1.3997 (19) | C16—C17 | 1.381 (2) |
| C5—C10 | 1.5108 (16) | C16—H16 | 0.9300 |
| C6—C7 | 1.482 (2) | C17—H17 | 0.9300 |
| C7—O1 | 1.2154 (17) | C18—C23 | 1.3857 (19) |
| C7—C8 | 1.4996 (19) | C18—C19 | 1.3891 (19) |
| C8—C9 | 1.5203 (18) | C19—C20 | 1.383 (2) |
| C8—H8A | 0.9700 | C19—H19 | 0.9300 |
| C8—H8B | 0.9700 | C20—C21 | 1.374 (3) |
| C9—C18 | 1.5180 (17) | C20—H20 | 0.9300 |
| C9—C10 | 1.5583 (17) | C21—C22 | 1.366 (3) |
| C9—H9 | 0.9800 | C21—H21 | 0.9300 |
| C10—C11 | 1.5253 (16) | C22—C23 | 1.388 (2) |
| C10—H10 | 0.9800 | C22—H22 | 0.9300 |
| C11—O2 | 1.2144 (15) | C23—H23 | 0.9300 |
| | | | |
| C2—C1—C6 | 120.20 (15) | O2—C11—C12 | 120.48 (11) |
| C2—C1—H1 | 119.9 | O2—C11—C10 | 120.17 (11) |
| C6—C1—H1 | 119.9 | C12—C11—C10 | 119.22 (10) |
| C1—C2—C3 | 120.11 (14) | C13—C12—C17 | 118.97 (12) |
| C1—C2—H2 | 119.9 | C13—C12—C11 | 122.95 (11) |
| C3—C2—H2 | 119.9 | C17—C12—C11 | 118.07 (11) |
| C2—C3—C4 | 120.35 (15) | C12—C13—C14 | 121.09 (14) |
| C2—C3—H3 | 119.8 | C12—C13—H13 | 119.5 |
| C4—C3—H3 | 119.8 | C14—C13—H13 | 119.5 |
| C3—C4—C5 | 120.65 (14) | C15—C14—C13 | 117.89 (15) |
| C3—C4—H4 | 119.7 | C15—C14—H14 | 121.1 |
| C5—C4—H4 | 119.7 | C13—C14—H14 | 121.1 |
| C4—C5—C6 | 118.74 (12) | C14—C15—F1 | 118.27 (15) |
| C4—C5—C10 | 119.71 (12) | C14—C15—C16 | 123.37 (14) |
| C6—C5—C10 | 121.53 (12) | F1—C15—C16 | 118.35 (15) |
| C5—C6—C1 | 119.92 (13) | C15—C16—C17 | 118.08 (15) |
| C5—C6—C7 | 120.78 (11) | C15—C16—H16 | 121.0 |
| C1—C6—C7 | 119.29 (13) | C17—C16—H16 | 121.0 |
| O1—C7—C6 | 121.72 (13) | C16—C17—C12 | 120.56 (14) |
| O1—C7—C8 | 120.39 (14) | C16—C17—H17 | 119.7 |
| C6—C7—C8 | 117.88 (11) | C12—C17—H17 | 119.7 |
| C7—C8—C9 | 113.70 (11) | C23—C18—C19 | 117.92 (13) |
| C7—C8—H8A | 108.8 | C23—C18—C9 | 122.78 (12) |
| C9—C8—H8A | 108.8 | C19—C18—C9 | 119.30 (12) |

| | | | |
|----------------|--------------|-----------------|--------------|
| C7—C8—H8B | 108.8 | C20—C19—C18 | 120.83 (15) |
| C9—C8—H8B | 108.8 | C20—C19—H19 | 119.6 |
| H8A—C8—H8B | 107.7 | C18—C19—H19 | 119.6 |
| C18—C9—C8 | 113.89 (11) | C21—C20—C19 | 120.40 (16) |
| C18—C9—C10 | 113.07 (10) | C21—C20—H20 | 119.8 |
| C8—C9—C10 | 109.51 (10) | C19—C20—H20 | 119.8 |
| C18—C9—H9 | 106.6 | C22—C21—C20 | 119.56 (15) |
| C8—C9—H9 | 106.6 | C22—C21—H21 | 120.2 |
| C10—C9—H9 | 106.6 | C20—C21—H21 | 120.2 |
| C5—C10—C11 | 112.10 (10) | C21—C22—C23 | 120.46 (16) |
| C5—C10—C9 | 110.03 (10) | C21—C22—H22 | 119.8 |
| C11—C10—C9 | 109.89 (10) | C23—C22—H22 | 119.8 |
| C5—C10—H10 | 108.2 | C18—C23—C22 | 120.82 (15) |
| C11—C10—H10 | 108.2 | C18—C23—H23 | 119.6 |
| C9—C10—H10 | 108.2 | C22—C23—H23 | 119.6 |
| | | | |
| C6—C1—C2—C3 | -0.8 (2) | C5—C10—C11—C12 | -148.71 (11) |
| C1—C2—C3—C4 | 0.2 (3) | C9—C10—C11—C12 | 88.60 (13) |
| C2—C3—C4—C5 | 1.1 (2) | O2—C11—C12—C13 | -156.49 (14) |
| C3—C4—C5—C6 | -1.8 (2) | C10—C11—C12—C13 | 27.68 (18) |
| C3—C4—C5—C10 | 176.91 (12) | O2—C11—C12—C17 | 24.2 (2) |
| C4—C5—C6—C1 | 1.10 (19) | C10—C11—C12—C17 | -151.59 (12) |
| C10—C5—C6—C1 | -177.54 (12) | C17—C12—C13—C14 | -0.3 (2) |
| C4—C5—C6—C7 | -179.46 (12) | C11—C12—C13—C14 | -179.61 (13) |
| C10—C5—C6—C7 | 1.90 (18) | C12—C13—C14—C15 | 1.7 (2) |
| C2—C1—C6—C5 | 0.2 (2) | C13—C14—C15—F1 | 179.74 (15) |
| C2—C1—C6—C7 | -179.26 (14) | C13—C14—C15—C16 | -1.2 (3) |
| C5—C6—C7—O1 | 174.25 (13) | C14—C15—C16—C17 | -0.5 (3) |
| C1—C6—C7—O1 | -6.3 (2) | F1—C15—C16—C17 | 178.48 (15) |
| C5—C6—C7—C8 | -4.57 (18) | C15—C16—C17—C12 | 1.9 (3) |
| C1—C6—C7—C8 | 174.87 (13) | C13—C12—C17—C16 | -1.5 (2) |
| O1—C7—C8—C9 | 156.38 (13) | C11—C12—C17—C16 | 177.80 (14) |
| C6—C7—C8—C9 | -24.80 (17) | C8—C9—C18—C23 | -18.97 (18) |
| C7—C8—C9—C18 | -177.69 (11) | C10—C9—C18—C23 | 106.89 (15) |
| C7—C8—C9—C10 | 54.60 (14) | C8—C9—C18—C19 | 161.63 (12) |
| C4—C5—C10—C11 | 87.24 (14) | C10—C9—C18—C19 | -72.51 (15) |
| C6—C5—C10—C11 | -94.14 (14) | C23—C18—C19—C20 | -1.3 (2) |
| C4—C5—C10—C9 | -150.15 (12) | C9—C18—C19—C20 | 178.14 (13) |
| C6—C5—C10—C9 | 28.47 (15) | C18—C19—C20—C21 | 1.2 (2) |
| C18—C9—C10—C5 | 176.40 (10) | C19—C20—C21—C22 | -0.1 (3) |
| C8—C9—C10—C5 | -55.42 (13) | C20—C21—C22—C23 | -0.9 (3) |
| C18—C9—C10—C11 | -59.70 (13) | C19—C18—C23—C22 | 0.2 (2) |
| C8—C9—C10—C11 | 68.48 (13) | C9—C18—C23—C22 | -179.17 (14) |
| C5—C10—C11—O2 | 35.45 (17) | C21—C22—C23—C18 | 0.9 (3) |
| C9—C10—C11—O2 | -87.23 (14) | | |

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

| $D\text{—H}\cdots A$ | $D\text{—H}$ | $H\cdots A$ | $D\cdots A$ | $D\text{—H}\cdots A$ |
|----------------------------------|--------------|-------------|-------------|----------------------|
| C14—H14 \cdots O1 ⁱ | 0.93 | 2.53 | 3.425 (2) | 161 |

supplementary materials

| | | | | |
|----------------------------|------|------|-------------|-----|
| C10—H10···O2 ⁱⁱ | 0.98 | 2.51 | 3.1427 (15) | 123 |
|----------------------------|------|------|-------------|-----|

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