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4-(Decyloxy)phenyl 2-oxo-7-trifluoromethyl-2H-chromene-3-carboxylate

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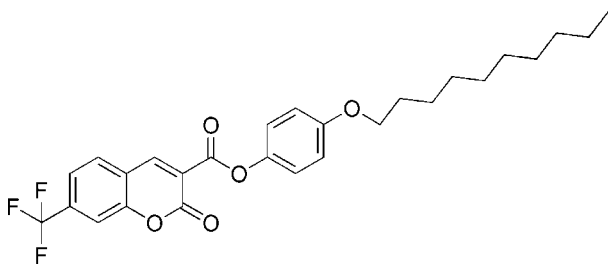
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 Key indicators: single-crystal X-ray study; $T = 99$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; disorder in main residue; R factor = 0.084; wR factor = 0.221; data-to-parameter ratio = 12.9.

The title compound, $\text{C}_{27}\text{H}_{29}\text{F}_3\text{O}_5$, is a liquid crystal (LC) and exhibits enantiotropic SmA phase transitions. In the crystal, the dihedral angle between the 2H-chromene ring system and the benzene ring is $62.97(2)^\circ$. The three F atoms of the $-\text{CF}_3$ group are disordered over two sets of sites with occupancy factors 0.71 (4):0.29 (4). In the crystal, pairs of $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds form inversion dimers and generate $R_2^2(10)$ rings. The structure also features $\text{C}-\text{H}\cdots\text{F}$ and $\text{C}-\text{H}\cdots\pi$ interactions along [100] and [010], respectively.

Related literature

For the synthesis and liquid crystal behaviour of the title compound, see: Mahadevan *et al.* (2013). For the biological activity of coumarins and their derivatives, see: Borges *et al.* (2005); Kontogiorgis & Hadjipavlou-Litina (2005) and for their industrial applications, see: Hejchman *et al.* (2011). For the structure of 4-(octyloxy)phenyl 2-oxo-2H-chromene-3-carboxylate, see: Palakshamurthy *et al.* (2013). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

| | |
|--|---|
| $\text{C}_{27}\text{H}_{29}\text{F}_3\text{O}_5$ | $V = 2571(5) \text{ \AA}^3$ |
| $M_r = 490.50$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 27.85(3) \text{ \AA}$ | $\mu = 0.10 \text{ mm}^{-1}$ |
| $b = 9.281(10) \text{ \AA}$ | $T = 99 \text{ K}$ |
| $c = 9.981(11) \text{ \AA}$ | $0.52 \times 0.42 \times 0.40 \text{ mm}$ |
| $\beta = 94.849(18)^\circ$ | |

Data collection

| | |
|---|--|
| Bruker APEXII diffractometer | 21163 measured reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2007) | 4448 independent reflections |
| $T_{\min} = 0.950$, $T_{\max} = 0.961$ | 2547 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.073$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.084$ | 344 parameters |
| $wR(F^2) = 0.221$ | H-atom parameters constrained |
| $S = 1.07$ | $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$ |
| 4448 reflections | $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$ |

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the C2–C7 and C12–C17 rings respectively.

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|-------------------------------------|--------------|--------------------|-------------|----------------------|
| C10—H10 \cdots O3 ⁱ | 0.95 | 2.56 | 3.396 (5) | 146 |
| C27—H27C \cdots F1 ⁱⁱ | 0.98 | 2.52 | 3.432 (13) | 154 |
| C6—H6 \cdots Cg2 ⁱ | 0.95 | 3.09 | 3.899 | 144 |
| C16—H16 \cdots Cg2 ⁱⁱⁱ | 0.95 | 3.33 | 4.120 | 142 |
| C3—H3 \cdots Cg1 ^{iv} | 0.95 | 3.43 | 4.325 | 163 |

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5309).

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supporting information

Acta Cryst. (2013). E69, o621–o622 [doi:10.1107/S1600536813008222]

4-(Decyloxy)phenyl 2-oxo-7-trifluoromethyl-2*H*-chromene-3-carboxylate

B. S. Palakshamurthy, H. C. Devarajegowda, H. T. Srinivasa, S. Sreenivasa and Vijithkumar

S1. Comment

The title compound is a liquid crystal (LC) exhibiting enantiotropic SmA phase transitions at 203.9(22.64) on heating and at 135.1(47.57) on cooling [The transition temperature in °C and the associated enthalpy values in kJ mol⁻¹ (in italics)] (Mahadevan *et al.*, 2013).

Organic compounds with a 2*H*-chromene ring system and their derivatives display a wide range of biological activities such as antiviral (Borges *et al.*, 2005) and anti-inflammatory (Kontogiorgis *et al.*, 2005) activity. They also display photochemical and photophysical properties, acting as molecular fluorescent sensors, laser dyes and have many industrial applications (Hejchman *et al.*, 2011). Keeping this in mind we report here the structure of the 4-(decyloxy)phenyl 7-(trifluoromethyl)-2-oxo-2*H*-chromene-3-carboxylate(I), and its comparison with 4-(octyloxy)phenyl 2-oxo-2*H*-chromene-3-carboxylate(II) (Palakshamurthy *et al.*, 2013).

The asymmetric unit of 4-(decyloxy)phenyl 7-(trifluoromethyl)-2-oxo-2*H*-chromene-3-carboxylate is shown in Fig.1. The three F atoms of the –CF₃ group are disordered over two sets of sites with occupancy factors 0.71 (4):0.29 (4). The dihedral angles between the 2*H*-chromene ring and the benzene ring are 62.97 (2)° and 21.11 (1)° in the compounds I and II respectively. The crystal structure is characterized by intermolecular C10—H10⋯O3 hydrogen bonds that form inversion dimers and generate a *R*²₂(10) ring pattern (Bernstein *et al.*, 1995). C27—H27⋯F1 hydrogen bonds then link the dimers into chains along *a*. The structure is further stabilized by C3—H3⋯Cg1, C6—H6⋯Cg2 and C16—H16⋯Cg2 interactions, Table 1.

S2. Experimental

A mixture of 7-(trifluoromethyl)-2-oxo-2*H*-chromene-3-carboxylic acid (0.100 g, 0.1 mmol), 4-(decyloxy) phenol (0.100 g, 0.1 mmol) dicyclohexylcarbodiimide (DCC) (0.100 g, 0.1 mmol) and catalytic quantity of DMAP (*N,N*-dimethyl amino pyridine) were stirred at room temperature for 48hrs in dry dichloromethane. Progress of the reaction was monitored by TLC (ethyl acetate: pet ether 2:8). After the completion of the reaction, the reaction mass was diluted with water and extracted into dichloromethane (25 ml). The organic layer was washed with water and dried over anhydrous sodium sulfate. The crude product thus obtained was purified by column chromatography using ethyl acetate: petroleum ether (2:8) as eluent followed by recrystallization from ethanol. A single crystal suitable for X-ray diffraction was grown from ethanol.

S3. Refinement

The H atoms bound to carbon were positioned with idealized geometry using a riding model with $d(\text{C-H}) = 0.93\text{--}0.99 \text{ \AA}$. All C–H atoms were refined with isotropic displacement parameters set to 1.2–1.5 $U_{\text{eq}}(\text{C})$. The F1, F2, and F3 fluorine atoms of the –CF₃ group were disordered over two sites and refined with site occupancy factors 0.71 (4):0.29 (4). The crystals were not of high quality which accounts for the high uncertainties in the lengths of the unit cell axes and the

relatively high residuals.

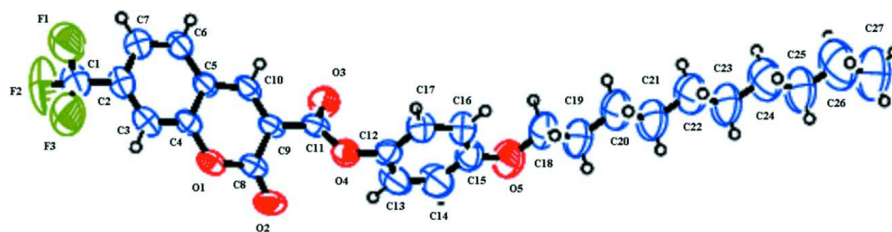


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. Only the major component of the disordered CF_3 group is shown.

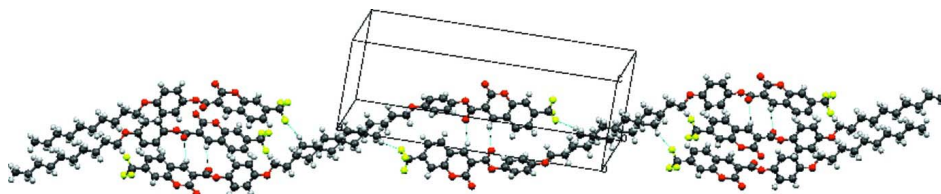


Figure 2

Crystal packing of the title compound with hydrogen bonds drawn as dashed lines.

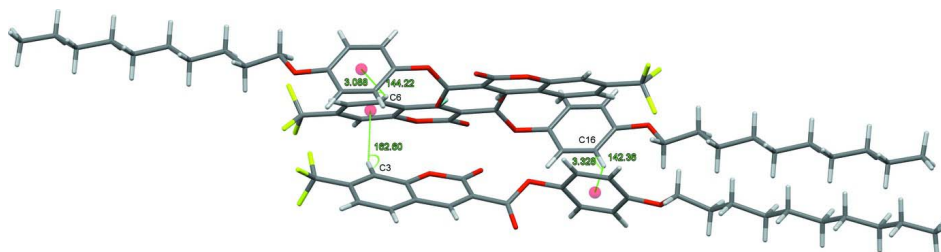


Figure 3

Packing of the title compound. $\text{C—H}\cdots\pi$ interactions are shown as dashed lines.

4-(Decyloxy)phenyl 2-oxo-7-trifluoromethyl-2*H*-chromene-3-carboxylate

Crystal data

$\text{C}_{27}\text{H}_{29}\text{F}_3\text{O}_5$

$M_r = 490.50$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 27.85\ (3)\ \text{\AA}$

$b = 9.281\ (10)\ \text{\AA}$

$c = 9.981\ (11)\ \text{\AA}$

$\beta = 94.849\ (18)^\circ$

$V = 2571\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 1032$

prism

$D_x = 1.267\ \text{Mg m}^{-3}$

Melting point: 418 K

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

Cell parameters from 2547 reflections

$\theta = 2.2\text{--}25^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 99\ \text{K}$

Prism, colourless

$0.52 \times 0.42 \times 0.40\ \text{mm}$

Data collection

| | |
|--|--|
| Bruker APEXII diffractometer | 21163 measured reflections 4448 independent reflections |
| Radiation source: fine-focus sealed tube | 2547 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{\text{int}} = 0.073$ |
| φ and ω scans | $\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.2^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2007) | $h = -33 \rightarrow 33$ $k = -11 \rightarrow 11$ $l = -11 \rightarrow 11$ |
| $T_{\text{min}} = 0.950$, $T_{\text{max}} = 0.961$ | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.084$ | H-atom parameters constrained |
| $wR(F^2) = 0.221$ | $w = 1/[\sigma^2(F_o^2) + (0.0928P)^2 + 0.7006P]$ |
| $S = 1.07$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 4448 reflections | $(\Delta/\sigma)_{\text{max}} = 0.004$ |
| 344 parameters | $\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$ |
| 0 constraints | |
| Primary atom site location: structure-invariant direct methods | |

Special details

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|-------------|-------------|----------------------------------|-----------|
| C25 | 1.0692 (2) | 0.6151 (7) | -0.3351 (7) | 0.150 (2) | |
| H25A | 1.0867 | 0.6580 | -0.2543 | 0.179* | |
| H25B | 1.0716 | 0.5093 | -0.3241 | 0.179* | |
| C24 | 1.0184 (2) | 0.6518 (7) | -0.3292 (7) | 0.153 (2) | |
| H24A | 1.0162 | 0.7579 | -0.3381 | 0.184* | |
| H24B | 1.0012 | 0.6110 | -0.4113 | 0.184* | |
| C2 | 0.31670 (15) | 0.6261 (4) | 0.2849 (4) | 0.0741 (11) | |
| C1 | 0.26593 (19) | 0.6458 (7) | 0.3228 (6) | 0.0935 (13) | |
| C27 | 1.1456 (2) | 0.6184 (9) | -0.4560 (9) | 0.208 (4) | |
| H27A | 1.1556 | 0.6545 | -0.5417 | 0.312* | |
| H27B | 1.1502 | 0.5138 | -0.4514 | 0.312* | |
| H27C | 1.1652 | 0.6643 | -0.3816 | 0.312* | |
| C26 | 1.0960 (3) | 0.6514 (10) | -0.4462 (8) | 0.213 (4) | |
| H26A | 1.0783 | 0.6082 | -0.5265 | 0.256* | |
| H26B | 1.0931 | 0.7571 | -0.4572 | 0.256* | |
| F1 | 0.2363 (3) | 0.694 (2) | 0.2215 (10) | 0.137 (6) | 0.71 (4) |

| | | | | | |
|------|--------------|-------------|-------------|-------------|----------|
| F2 | 0.2468 (5) | 0.5236 (12) | 0.361 (3) | 0.159 (9) | 0.71 (4) |
| F3 | 0.2620 (2) | 0.739 (2) | 0.4226 (19) | 0.145 (8) | 0.71 (4) |
| F1A | 0.2625 (6) | 0.621 (7) | 0.450 (2) | 0.154 (19) | 0.29 (4) |
| F2A | 0.2343 (8) | 0.556 (5) | 0.269 (5) | 0.147 (17) | 0.29 (4) |
| F3A | 0.2475 (14) | 0.766 (3) | 0.303 (7) | 0.18 (3) | 0.29 (4) |
| O1 | 0.43973 (9) | 0.6693 (2) | 0.4333 (2) | 0.0641 (7) | |
| O4 | 0.57932 (9) | 0.6725 (3) | 0.2856 (2) | 0.0709 (7) | |
| C10 | 0.46005 (13) | 0.5620 (3) | 0.1853 (3) | 0.0632 (10) | |
| H10 | 0.4667 | 0.5224 | 0.1011 | 0.076* | |
| C5 | 0.41071 (13) | 0.5803 (3) | 0.2142 (3) | 0.0589 (9) | |
| O2 | 0.51778 (10) | 0.6830 (3) | 0.4977 (2) | 0.0840 (8) | |
| C3 | 0.35544 (15) | 0.6606 (4) | 0.3744 (4) | 0.0694 (10) | |
| H3 | 0.3503 | 0.7004 | 0.4597 | 0.083* | |
| C8 | 0.48791 (14) | 0.6551 (3) | 0.4068 (3) | 0.0577 (9) | |
| C4 | 0.40217 (13) | 0.6371 (3) | 0.3396 (3) | 0.0576 (9) | |
| C9 | 0.49690 (13) | 0.5990 (3) | 0.2735 (3) | 0.0551 (8) | |
| O3 | 0.55682 (9) | 0.4843 (3) | 0.1529 (3) | 0.0868 (9) | |
| C6 | 0.37051 (14) | 0.5468 (4) | 0.1238 (4) | 0.0765 (11) | |
| H6 | 0.3755 | 0.5086 | 0.0378 | 0.092* | |
| C11 | 0.54679 (13) | 0.5768 (4) | 0.2315 (3) | 0.0611 (9) | |
| C7 | 0.32407 (15) | 0.5687 (4) | 0.1581 (4) | 0.0843 (12) | |
| H7 | 0.2973 | 0.5451 | 0.0965 | 0.101* | |
| C12 | 0.62703 (14) | 0.6568 (4) | 0.2462 (4) | 0.0668 (10) | |
| C17 | 0.64238 (15) | 0.7362 (4) | 0.1429 (4) | 0.0784 (11) | |
| H17 | 0.6206 | 0.8001 | 0.0944 | 0.094* | |
| C15 | 0.72109 (15) | 0.6315 (5) | 0.1777 (5) | 0.0847 (12) | |
| C16 | 0.68960 (15) | 0.7248 (4) | 0.1077 (4) | 0.0831 (12) | |
| H16 | 0.7000 | 0.7809 | 0.0360 | 0.100* | |
| O5 | 0.76854 (11) | 0.6082 (4) | 0.1512 (4) | 0.1166 (11) | |
| C13 | 0.65845 (18) | 0.5638 (5) | 0.3158 (4) | 0.0971 (14) | |
| H13 | 0.6480 | 0.5083 | 0.3878 | 0.117* | |
| C14 | 0.70536 (18) | 0.5511 (6) | 0.2811 (5) | 0.1108 (16) | |
| H14 | 0.7269 | 0.4863 | 0.3292 | 0.133* | |
| C19 | 0.83692 (17) | 0.6115 (6) | 0.0247 (5) | 0.1171 (17) | |
| H19A | 0.8573 | 0.6386 | 0.1070 | 0.141* | |
| H19B | 0.8350 | 0.5050 | 0.0225 | 0.141* | |
| C18 | 0.78703 (16) | 0.6698 (5) | 0.0363 (5) | 0.1025 (15) | |
| H18A | 0.7882 | 0.7760 | 0.0450 | 0.123* | |
| H18B | 0.7659 | 0.6451 | -0.0453 | 0.123* | |
| C20 | 0.86150 (18) | 0.6598 (6) | -0.0933 (6) | 0.1232 (18) | |
| H20A | 0.8609 | 0.7665 | -0.0952 | 0.148* | |
| H20B | 0.8425 | 0.6257 | -0.1754 | 0.148* | |
| C23 | 0.99056 (18) | 0.6129 (7) | -0.2183 (6) | 0.134 (2) | |
| H23A | 1.0081 | 0.6518 | -0.1357 | 0.161* | |
| H23B | 0.9920 | 0.5066 | -0.2107 | 0.161* | |
| C21 | 0.91271 (18) | 0.6116 (7) | -0.1003 (6) | 0.1294 (19) | |
| H21A | 0.9313 | 0.6457 | -0.0174 | 0.155* | |
| H21B | 0.9128 | 0.5050 | -0.0969 | 0.155* | |

| | | | | |
|------|------------|------------|-------------|-----------|
| C22 | 0.9393 (2) | 0.6543 (7) | -0.2135 (6) | 0.137 (2) |
| H22A | 0.9377 | 0.7607 | -0.2197 | 0.165* |
| H22B | 0.9216 | 0.6160 | -0.2960 | 0.165* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C25 | 0.088 (4) | 0.175 (6) | 0.191 (6) | 0.014 (4) | 0.044 (4) | 0.019 (5) |
| C24 | 0.103 (4) | 0.190 (6) | 0.172 (6) | 0.028 (4) | 0.051 (4) | 0.016 (5) |
| C2 | 0.076 (3) | 0.068 (2) | 0.081 (3) | 0.002 (2) | 0.025 (2) | 0.011 (2) |
| C1 | 0.090 (4) | 0.099 (4) | 0.095 (4) | 0.004 (3) | 0.026 (3) | 0.011 (3) |
| C27 | 0.108 (5) | 0.243 (9) | 0.282 (10) | 0.012 (5) | 0.066 (6) | 0.009 (8) |
| C26 | 0.137 (7) | 0.279 (10) | 0.235 (9) | 0.037 (6) | 0.088 (6) | 0.023 (8) |
| F1 | 0.097 (4) | 0.197 (15) | 0.118 (6) | 0.045 (6) | 0.021 (4) | 0.027 (6) |
| F2 | 0.103 (8) | 0.120 (6) | 0.26 (2) | 0.014 (5) | 0.083 (12) | 0.075 (10) |
| F3 | 0.094 (4) | 0.190 (15) | 0.158 (11) | 0.008 (6) | 0.043 (5) | -0.069 (11) |
| F1A | 0.106 (11) | 0.25 (5) | 0.117 (13) | 0.009 (18) | 0.061 (9) | 0.032 (18) |
| F2A | 0.083 (10) | 0.15 (3) | 0.21 (3) | -0.011 (12) | 0.038 (14) | -0.03 (2) |
| F3A | 0.18 (2) | 0.12 (2) | 0.27 (6) | 0.079 (18) | 0.10 (4) | 0.07 (3) |
| O1 | 0.0811 (17) | 0.0674 (15) | 0.0456 (13) | 0.0026 (12) | 0.0160 (12) | -0.0070 (11) |
| O4 | 0.0756 (17) | 0.0752 (16) | 0.0639 (15) | -0.0077 (13) | 0.0181 (12) | -0.0240 (13) |
| C10 | 0.083 (3) | 0.067 (2) | 0.0423 (19) | -0.0066 (18) | 0.0218 (19) | -0.0059 (16) |
| C5 | 0.072 (2) | 0.059 (2) | 0.0467 (19) | -0.0125 (17) | 0.0157 (18) | -0.0017 (15) |
| O2 | 0.089 (2) | 0.115 (2) | 0.0480 (14) | 0.0021 (16) | 0.0037 (14) | -0.0223 (14) |
| C3 | 0.086 (3) | 0.064 (2) | 0.061 (2) | 0.004 (2) | 0.025 (2) | 0.0030 (17) |
| C8 | 0.078 (3) | 0.0537 (19) | 0.0421 (19) | 0.0018 (17) | 0.0112 (18) | -0.0019 (15) |
| C4 | 0.076 (2) | 0.0484 (18) | 0.050 (2) | -0.0019 (16) | 0.0143 (18) | 0.0045 (15) |
| C9 | 0.077 (2) | 0.0517 (18) | 0.0377 (18) | -0.0050 (16) | 0.0124 (17) | -0.0009 (14) |
| O3 | 0.0876 (19) | 0.0923 (19) | 0.0828 (18) | -0.0046 (15) | 0.0212 (15) | -0.0405 (15) |
| C6 | 0.081 (3) | 0.091 (3) | 0.060 (2) | -0.017 (2) | 0.019 (2) | -0.006 (2) |
| C11 | 0.080 (3) | 0.062 (2) | 0.0414 (18) | -0.0022 (19) | 0.0086 (17) | -0.0041 (16) |
| C7 | 0.078 (3) | 0.102 (3) | 0.074 (3) | -0.013 (2) | 0.011 (2) | -0.001 (2) |
| C12 | 0.071 (2) | 0.070 (2) | 0.059 (2) | 0.002 (2) | 0.0062 (19) | -0.0184 (19) |
| C17 | 0.078 (3) | 0.072 (2) | 0.086 (3) | 0.002 (2) | 0.011 (2) | 0.004 (2) |
| C15 | 0.069 (3) | 0.096 (3) | 0.090 (3) | 0.002 (2) | 0.012 (2) | -0.013 (3) |
| C16 | 0.070 (3) | 0.081 (3) | 0.100 (3) | -0.005 (2) | 0.017 (2) | 0.005 (2) |
| O5 | 0.076 (2) | 0.146 (3) | 0.129 (3) | 0.0163 (19) | 0.013 (2) | 0.006 (2) |
| C13 | 0.105 (4) | 0.118 (4) | 0.070 (3) | 0.015 (3) | 0.019 (2) | 0.016 (3) |
| C14 | 0.094 (4) | 0.144 (4) | 0.096 (3) | 0.037 (3) | 0.011 (3) | 0.018 (3) |
| C19 | 0.067 (3) | 0.150 (5) | 0.135 (4) | 0.004 (3) | 0.016 (3) | 0.000 (4) |
| C18 | 0.077 (3) | 0.105 (3) | 0.128 (4) | -0.007 (3) | 0.026 (3) | -0.015 (3) |
| C20 | 0.085 (3) | 0.138 (4) | 0.151 (5) | 0.009 (3) | 0.035 (3) | -0.007 (4) |
| C23 | 0.077 (3) | 0.161 (5) | 0.166 (5) | 0.012 (3) | 0.028 (3) | 0.015 (4) |
| C21 | 0.076 (3) | 0.160 (5) | 0.154 (5) | 0.011 (3) | 0.022 (3) | 0.013 (4) |
| C22 | 0.091 (4) | 0.171 (5) | 0.154 (5) | 0.020 (4) | 0.036 (4) | 0.003 (4) |

Geometric parameters (Å, °)

| | | | |
|---------------|------------|------------|-----------|
| C25—C26 | 1.429 (8) | C9—C11 | 1.499 (5) |
| C25—C24 | 1.460 (8) | O3—C11 | 1.212 (4) |
| C25—H25A | 0.9900 | C6—C7 | 1.381 (5) |
| C25—H25B | 0.9900 | C6—H6 | 0.9500 |
| C24—C23 | 1.451 (7) | C7—H7 | 0.9500 |
| C24—H24A | 0.9900 | C12—C17 | 1.365 (5) |
| C24—H24B | 0.9900 | C12—C13 | 1.375 (5) |
| C2—C3 | 1.379 (5) | C17—C16 | 1.394 (5) |
| C2—C7 | 1.404 (5) | C17—H17 | 0.9500 |
| C2—C1 | 1.506 (6) | C15—C14 | 1.375 (6) |
| C1—F3A | 1.234 (17) | C15—C16 | 1.380 (6) |
| C1—F1A | 1.300 (18) | C15—O5 | 1.387 (5) |
| C1—F2A | 1.30 (2) | C16—H16 | 0.9500 |
| C1—F2 | 1.323 (11) | O5—C18 | 1.417 (5) |
| C1—F1 | 1.329 (9) | C13—C14 | 1.385 (6) |
| C1—F3 | 1.332 (9) | C13—H13 | 0.9500 |
| C27—C26 | 1.426 (9) | C14—H14 | 0.9500 |
| C27—H27A | 0.9800 | C19—C20 | 1.481 (7) |
| C27—H27B | 0.9800 | C19—C18 | 1.505 (6) |
| C27—H27C | 0.9800 | C19—H19A | 0.9900 |
| C26—H26A | 0.9900 | C19—H19B | 0.9900 |
| C26—H26B | 0.9900 | C18—H18A | 0.9900 |
| O1—C4 | 1.376 (4) | C18—H18B | 0.9900 |
| O1—C8 | 1.396 (4) | C20—C21 | 1.502 (7) |
| O4—C11 | 1.349 (4) | C20—H20A | 0.9900 |
| O4—C12 | 1.425 (4) | C20—H20B | 0.9900 |
| C10—C9 | 1.339 (5) | C23—C22 | 1.482 (7) |
| C10—C5 | 1.438 (5) | C23—H23A | 0.9900 |
| C10—H10 | 0.9500 | C23—H23B | 0.9900 |
| C5—C4 | 1.397 (4) | C21—C22 | 1.458 (7) |
| C5—C6 | 1.412 (5) | C21—H21A | 0.9900 |
| O2—C8 | 1.206 (4) | C21—H21B | 0.9900 |
| C3—C4 | 1.392 (5) | C22—H22A | 0.9900 |
| C3—H3 | 0.9500 | C22—H22B | 0.9900 |
| C8—C9 | 1.469 (4) | | |
| C26—C25—C24 | 123.3 (7) | C10—C9—C11 | 117.2 (3) |
| C26—C25—H25A | 106.5 | C8—C9—C11 | 122.3 (3) |
| C24—C25—H25A | 106.5 | C7—C6—C5 | 121.2 (4) |
| C26—C25—H25B | 106.5 | C7—C6—H6 | 119.4 |
| C24—C25—H25B | 106.5 | C5—C6—H6 | 119.4 |
| H25A—C25—H25B | 106.5 | O3—C11—O4 | 122.8 (3) |
| C23—C24—C25 | 123.7 (6) | O3—C11—C9 | 123.3 (3) |
| C23—C24—H24A | 106.4 | O4—C11—C9 | 113.9 (3) |
| C25—C24—H24A | 106.4 | C6—C7—C2 | 119.4 (4) |
| C23—C24—H24B | 106.4 | C6—C7—H7 | 120.3 |

| | | | |
|---------------|------------|---------------|-----------|
| C25—C24—H24B | 106.4 | C2—C7—H7 | 120.3 |
| H24A—C24—H24B | 106.5 | C17—C12—C13 | 119.6 (4) |
| C3—C2—C7 | 120.4 (4) | C17—C12—O4 | 120.8 (3) |
| C3—C2—C1 | 120.6 (4) | C13—C12—O4 | 119.6 (4) |
| C7—C2—C1 | 119.0 (4) | C12—C17—C16 | 120.8 (4) |
| F3A—C1—F1A | 104.7 (15) | C12—C17—H17 | 119.6 |
| F3A—C1—F2A | 104.9 (19) | C16—C17—H17 | 119.6 |
| F1A—C1—F2A | 100.8 (14) | C14—C15—C16 | 119.4 (4) |
| F3A—C1—F2 | 130.3 (12) | C14—C15—O5 | 115.4 (4) |
| F1A—C1—F2 | 60.1 (18) | C16—C15—O5 | 125.2 (4) |
| F2A—C1—F2 | 44.9 (18) | C15—C16—C17 | 119.5 (4) |
| F3A—C1—F1 | 50 (3) | C15—C16—H16 | 120.2 |
| F1A—C1—F1 | 135.3 (10) | C17—C16—H16 | 120.2 |
| F2A—C1—F1 | 63 (2) | C15—O5—C18 | 120.6 (4) |
| F2—C1—F1 | 105.6 (10) | C12—C13—C14 | 120.1 (4) |
| F3A—C1—F3 | 58 (3) | C12—C13—H13 | 120.0 |
| F1A—C1—F3 | 51 (2) | C14—C13—H13 | 120.0 |
| F2A—C1—F3 | 129.4 (10) | C15—C14—C13 | 120.6 (4) |
| F2—C1—F3 | 106.4 (8) | C15—C14—H14 | 119.7 |
| F1—C1—F3 | 105.3 (7) | C13—C14—H14 | 119.7 |
| F3A—C1—C2 | 117.1 (10) | C20—C19—C18 | 116.3 (5) |
| F1A—C1—C2 | 111.8 (10) | C20—C19—H19A | 108.2 |
| F2A—C1—C2 | 115.8 (9) | C18—C19—H19A | 108.2 |
| F2—C1—C2 | 112.2 (6) | C20—C19—H19B | 108.2 |
| F1—C1—C2 | 112.7 (5) | C18—C19—H19B | 108.2 |
| F3—C1—C2 | 113.9 (6) | H19A—C19—H19B | 107.4 |
| C26—C27—H27A | 109.5 | O5—C18—C19 | 108.5 (4) |
| C26—C27—H27B | 109.5 | O5—C18—H18A | 110.0 |
| H27A—C27—H27B | 109.5 | C19—C18—H18A | 110.0 |
| C26—C27—H27C | 109.5 | O5—C18—H18B | 110.0 |
| H27A—C27—H27C | 109.5 | C19—C18—H18B | 110.0 |
| H27B—C27—H27C | 109.5 | H18A—C18—H18B | 108.4 |
| C27—C26—C25 | 125.2 (8) | C19—C20—C21 | 116.8 (5) |
| C27—C26—H26A | 106.0 | C19—C20—H20A | 108.1 |
| C25—C26—H26A | 106.0 | C21—C20—H20A | 108.1 |
| C27—C26—H26B | 106.0 | C19—C20—H20B | 108.1 |
| C25—C26—H26B | 106.0 | C21—C20—H20B | 108.1 |
| H26A—C26—H26B | 106.3 | H20A—C20—H20B | 107.3 |
| C4—O1—C8 | 122.6 (3) | C24—C23—C22 | 122.6 (5) |
| C11—O4—C12 | 115.6 (3) | C24—C23—H23A | 106.7 |
| C9—C10—C5 | 122.1 (3) | C22—C23—H23A | 106.7 |
| C9—C10—H10 | 119.0 | C24—C23—H23B | 106.7 |
| C5—C10—H10 | 119.0 | C22—C23—H23B | 106.7 |
| C4—C5—C6 | 118.0 (3) | H23A—C23—H23B | 106.6 |
| C4—C5—C10 | 117.5 (3) | C22—C21—C20 | 120.2 (5) |
| C6—C5—C10 | 124.5 (3) | C22—C21—H21A | 107.3 |
| C2—C3—C4 | 119.9 (3) | C20—C21—H21A | 107.3 |
| C2—C3—H3 | 120.0 | C22—C21—H21B | 107.3 |

| | | | |
|-----------|-----------|---------------|-----------|
| C4—C3—H3 | 120.0 | C20—C21—H21B | 107.3 |
| O2—C8—O1 | 116.8 (3) | H21A—C21—H21B | 106.9 |
| O2—C8—C9 | 126.7 (3) | C21—C22—C23 | 120.6 (5) |
| O1—C8—C9 | 116.4 (3) | C21—C22—H22A | 107.2 |
| O1—C4—C3 | 118.0 (3) | C23—C22—H22A | 107.2 |
| O1—C4—C5 | 120.9 (3) | C21—C22—H22B | 107.2 |
| C3—C4—C5 | 121.1 (4) | C23—C22—H22B | 107.2 |
| C10—C9—C8 | 120.4 (3) | H22A—C22—H22B | 106.8 |

Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of the C2—C7 and C12—C17 rings respectively.

| <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> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| C10—H10...O3 ⁱ | 0.95 | 2.56 | 3.396 (5) | 146 |
| C27—H27C...F1 ⁱⁱ | 0.98 | 2.52 | 3.432 (13) | 154 |
| C6—H6...Cg2 ⁱ | 0.95 | 3.09 | 3.899 | 144 |
| C16—H16...Cg2 ⁱⁱⁱ | 0.95 | 3.33 | 4.120 | 142 |
| C3—H3...Cg1 ^{iv} | 0.95 | 3.43 | 4.325 | 163 |

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