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# Ethyl 3′-(2,4-dichlorophenyl)-5′-hydroxy-5′-methyl-4′,5′-dihydrospiro[fluorene-9,2′(3′*H*)-furan]-4′-carboxylate

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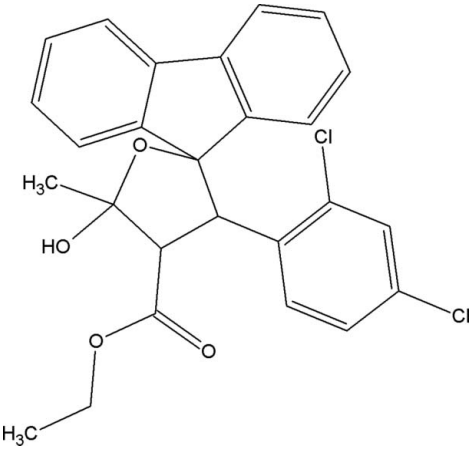
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.048; $wR$ factor = 0.151; data-to-parameter ratio = 18.3.

The furan ring and the five-membered fluorene unit in the title compound, $C_{26}H_{22}Cl_2O_4$, adopt envelope conformations. Intermolecular C—H···O interactions between symmetry-related molecules involving two C—H groups and an O atom as a bifurcated acceptor generate centrosymmetric hydrogen-bonded dimers with cyclic $R_2^2(16)$ and $R_2^2(8)$ ring motifs. A short C—H···Cl intramolecular contact occurs in the molecule.

## Related literature

For spiro compounds in pharmacologically active alkaloids, see: Cravotto *et al.* (2001). For the anticonvulsant activity of fluorene derivatives, see: Vanvakides *et al.* (2004). Fluorene derivatives, including polyfluorenes and oligofluorenes, are promising candidates for blue light-emitting materials in organic light-emitting devices (Muller *et al.*, 2003), organic phototransistors (Saragi *et al.*, 2004), non-linear optics (Kim *et al.*, 1998) and photochromic materials (Chun *et al.*, 2003). For the biological activity of furan derivatives and annulated furan derivatives and their use as precursors for the synthesis of natural products, see: Greve & Friedrichsen (2000). For hydrogen-bond motifs and ring puckering parameters, see: Bernstein *et al.* (1995); Cremer & Pople (1975); Nardelli (1983). For a related spiro-linked system, see: Feng *et al.* (2004).




## Experimental

### Crystal data

$C_{26}H_{22}Cl_2O_4$
$M_r$ = 469.34
Monoclinic, $C2/c$
$a$ = 28.6811 (13) Å
$b$ = 9.0600 (4) Å
$c$ = 17.4074 (8) Å
$\beta$ = 92.072 (3)°
$V$ = 4520.4 (4) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.32 mm$^{-1}$
$T$ = 293 K
0.25 × 0.20 × 0.20 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker 2004)
$T_{min}$ = 0.916, $T_{max}$ = 0.938
22504 measured reflections
5338 independent reflections
3663 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.056

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.151
$S$ = 1.04
5338 reflections
291 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2$A$···O3$^{i}$ | 0.98 | 2.37 | 3.331 (3) | 167 |
| C6—H6···O3$^{i}$ | 0.93 | 2.55 | 3.393 (3) | 151 |
| C3—H3···Cl1 | 0.98 | 2.57 | 3.082 (2) | 113 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2242).

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# supplementary materials

*Acta Cryst.* (2009). E**65**, o977-o978 [ **doi:10.1107/S1600536809011854** ]

# Ethyl 3'-(2,4-dichlorophenyl)-5'-hydroxy-5'-methyl-4',5'-dihydrospiro[fluorene-9,2'(3'*H*)-furan]-4'-carboxylate

**M. NizamMohideen, S. Thenmozhi, A. SubbiahPandi, G. Savitha and P. T. Perumal**

## Comment

Spiro compounds are often encountered in many pharmacologically active alkaloids (Cravotto *et al.*, 2001) and fluorene derivatives have been found to have anticonvulsant activity (Vanvakides *et al.*, 2004). In addition, fluorene derivatives, including polyfluorenes and oligofluorenes, have been studied extensively in recent years because they are very promising candidates for blue light-emitting materials in organic light-emitting devices (Muller *et al.*, 2003), organic phototransistors (Saragi *et al.*, 2004), nonlinear optics (Kim *et al.*, 1998) and photochromic materials (Chun *et al.*, 2003). Furan derivatives and annulated furan derivatives occur widely in nature and, along with their unnatural analogs, have been shown to have a wide range of biological activity as well as being important precursors for the synthesis of natural products (Greve & Friedrichsen, 2000). In view of these important properties, the crystal structure of the title compound, (I), has been determined.

in (I, Fig. 1) the C4-C5 and C4-C16 bond distances of the fluorene moiety are almost identical to the values reported in another spiro-linked system (Feng *et al.*, 2004).

The benzene ring is planar with the largest displacement observed being -0.014 (1) Å for atom C22. The deviations of the atoms Cl1 and Cl2 from the least-squares plane of the phenyl rings are -0.114 (1) and 0.015 (1) Å, respectively.

The five membered fluorene moiety adopts an envelope conformation (flap atom C4) with a pseudo-twofold axis passing through the C4-C5 bond. The puckering parameters (Cremer & Pople, 1975) and the lowest displacement asymmetry parameters (Nardelli, 1983) for this ring are $q_2$ = 0.107 (2) Å, φ = 355.0 (1)° and $\Delta_S$(C4) is 1.3 (1)°. The tetrahydrofuran ring also adopts an envelope conformation (flap atom C2) with a pseudo-twofold axis passing through the C2-C3 bond. The puckering parameters (Cremer & Pople, 1975) and the lowest displacement asymmetry parameters (Nardelli, 1983) for this ring are $q_2$ = 0.388 (2) Å, φ = 252.1 (2)° and $\Delta_S$(C2) is 2.1 (2)°.

Carbonyl atom O3 acts as a intermolecuar bifurcated acceptor with both C2 and C6 (Table 1 and Fig. 2) from a symmetry-related molecule to form centrosymmetric hydrogen bonded dimers with cyclic $R_2^2(16)$ and $R_2^2(8)$ (Bernstein, *et al.*, 1995) ring systems, respectively. The structure is further stabilized by C—H···π interactions involing rings C26- H26C···*Cg*1 (*Cg*1 is the centroid of the C11—C16 ring).

## Experimental

To a stirred mixture of 9-(2,4-Dichloro-benzylidene)-9*H*-fluorene (1.0 mmol), ethylacetoacetate (1.0 mmol) and $NaHCO_3$ (3.0 mmol) in acetonitrile (10 ml), ceric ammonium nitrate (2.5 mmol) dissolved in acetonitrile (5 ml) was added dropwise at 0 ° under $N_2$. The reaction mixture was stirred until completion of the reaction as monitored by TLC. Water was added to the mixture and the product was extracted with ethyl acetate (2 × 20 ml) and then dried over anhydrous $Na_2SO_4$. Removal of the solvent under reduced pressure gave a crude product, which was purified by column chromatography on silica gel,

with ethyl acetate-hexane (4:6) as eluent to afford a pure product in 79% yield. Single crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethylacetate.

### Refinement

All H atoms were positioned geometrically, with O—H = 0.82 and C—H = 0.93–0.98 Å and constrained to ride on their parent atoms, with $U_{iso}$(H) = $xU_{eq}$(C, N), where $x$ = 1.5 for methyl H and $x$ = 1.2 for all H atoms.

### Figures

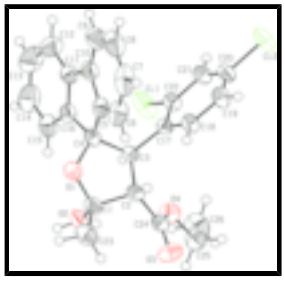

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme for. Displacement ellipsoids are drawn at the 50% probability level.

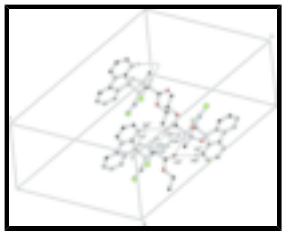

Fig. 2. The crystal packing of compound (I), showing the $R_2^2(16)$ and $R_2^2(8)$ rings. Hydrogen bonding is shown as dashed lines. H atoms not involved in the hydrogen bonding have been omitted for clarity. [Symmetry codes: -$x$ + 1/2, -$y$ + 1/2, -$z$]

### Ethyl 3'-(2,4-dichlorophenyl)-5'-hydroxy-5'-methyl-4',5'- dihydrospiro[fluorene-9,2'(3'*H*)-furan]-4'-carboxylate

*Crystal data*

| | |
|---|---|
| $C_{26}H_{22}Cl_2O_4$ | $F_{000}$ = 1952 |
| $M_r$ = 469.34 | $D_x$ = 1.379 Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -C 2yc | Cell parameters from 7176 reflections |
| $a$ = 28.6811 (13) Å | θ = 2.5–25º |
| $b$ = 9.0600 (4) Å | μ = 0.32 mm$^{-1}$ |
| $c$ = 17.4074 (8) Å | $T$ = 293 K |
| β = 92.072 (3)º | Prismatic, yellow |
| $V$ = 4520.4 (4) Å$^3$ | 0.25 × 0.20 × 0.20 mm |
| $Z$ = 8 | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 5338 independent reflections |
| Radiation source: fine-focus sealed tube | 3663 reflections with $I > 2\sigma(I)$ |

Monochromator: graphite
$T$ = 293 K
ω and φ scans
Absorption correction: Multi-scan (SADABS; Bruker 2004)
$T_{min}$ = 0.916, $T_{max}$ = 0.938
22504 measured reflections
$R_{int}$ = 0.056
$\theta_{max}$ = 27.8º
$\theta_{min}$ = 1.4º
$h$ = −36→37
$k$ = −11→11
$l$ = −21→22

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.151
$S$ = 1.04
5338 reflections
291 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 2.471P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$
Extinction correction: none

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.06337 (2) | −0.03027 (7) | 0.10063 (4) | 0.0663 (2) |
| Cl2 | 0.00329 (2) | 0.39144 (8) | −0.09056 (4) | 0.0652 (2) |
| O1 | 0.21156 (5) | 0.19309 (19) | 0.21266 (8) | 0.0476 (4) |
| O2 | 0.23543 (6) | −0.03879 (18) | 0.17193 (10) | 0.0550 (4) |
| H2 | 0.2506 | −0.0562 | 0.2117 | 0.082* |
| O3 | 0.25673 (6) | 0.04006 (18) | −0.01270 (10) | 0.0540 (4) |
| O4 | 0.18812 (5) | −0.06733 (17) | 0.00380 (9) | 0.0455 (4) |
| C1 | 0.23639 (7) | 0.1143 (2) | 0.15672 (13) | 0.0411 (5) |
| C2 | 0.20777 (6) | 0.1382 (2) | 0.08249 (11) | 0.0331 (4) |
| H2A | 0.2135 | 0.2389 | 0.0645 | 0.040* |
| C3 | 0.15806 (6) | 0.1308 (2) | 0.10976 (11) | 0.0301 (4) |

| | | | | |
|---|---|---|---|---|
| H3 | 0.1508 | 0.0267 | 0.1186 | 0.036* |
| C4 | 0.16289 (7) | 0.2080 (2) | 0.19036 (12) | 0.0349 (4) |
| C5 | 0.14777 (8) | 0.3679 (2) | 0.19083 (12) | 0.0401 (5) |
| C6 | 0.16812 (10) | 0.4898 (2) | 0.15801 (13) | 0.0536 (6) |
| H6 | 0.1963 | 0.4821 | 0.1336 | 0.064* |
| C7 | 0.14497 (14) | 0.6249 (3) | 0.16271 (16) | 0.0709 (9) |
| H7 | 0.1579 | 0.7085 | 0.1409 | 0.085* |
| C8 | 0.10342 (13) | 0.6362 (3) | 0.19909 (17) | 0.0722 (9) |
| H8 | 0.0883 | 0.7269 | 0.2005 | 0.087* |
| C9 | 0.08401 (11) | 0.5172 (3) | 0.23298 (16) | 0.0628 (7) |
| H9 | 0.0561 | 0.5263 | 0.2581 | 0.075* |
| C10 | 0.10643 (8) | 0.3822 (2) | 0.22951 (12) | 0.0438 (5) |
| C11 | 0.09519 (8) | 0.2392 (3) | 0.26364 (13) | 0.0442 (5) |
| C12 | 0.05912 (10) | 0.1956 (4) | 0.30938 (16) | 0.0650 (7) |
| H12 | 0.0350 | 0.2601 | 0.3201 | 0.078* |
| C13 | 0.05993 (12) | 0.0536 (4) | 0.33873 (18) | 0.0767 (9) |
| H13 | 0.0359 | 0.0223 | 0.3693 | 0.092* |
| C14 | 0.09547 (12) | −0.0415 (3) | 0.32353 (16) | 0.0690 (8) |
| H14 | 0.0959 | −0.1349 | 0.3457 | 0.083* |
| C15 | 0.13054 (9) | −0.0010 (3) | 0.27593 (14) | 0.0505 (6) |
| H15 | 0.1540 | −0.0672 | 0.2639 | 0.061* |
| C16 | 0.13012 (7) | 0.1400 (2) | 0.24653 (11) | 0.0378 (5) |
| C17 | 0.12017 (6) | 0.1925 (2) | 0.05700 (11) | 0.0300 (4) |
| C18 | 0.12700 (7) | 0.3165 (2) | 0.01180 (12) | 0.0369 (5) |
| H18 | 0.1564 | 0.3597 | 0.0125 | 0.044* |
| C19 | 0.09199 (8) | 0.3780 (2) | −0.03393 (13) | 0.0431 (5) |
| H19 | 0.0978 | 0.4604 | −0.0640 | 0.052* |
| C20 | 0.04839 (7) | 0.3160 (2) | −0.03454 (12) | 0.0405 (5) |
| C21 | 0.03967 (7) | 0.1925 (2) | 0.00810 (12) | 0.0413 (5) |
| H21 | 0.0101 | 0.1506 | 0.0074 | 0.050* |
| C22 | 0.07576 (7) | 0.1313 (2) | 0.05219 (12) | 0.0355 (4) |
| C23 | 0.28555 (8) | 0.1735 (3) | 0.15764 (15) | 0.0597 (7) |
| H23A | 0.3002 | 0.1568 | 0.2073 | 0.089* |
| H23B | 0.2848 | 0.2774 | 0.1471 | 0.089* |
| H23C | 0.3029 | 0.1239 | 0.1192 | 0.089* |
| C24 | 0.22049 (7) | 0.0327 (2) | 0.01982 (12) | 0.0356 (4) |
| C25 | 0.19678 (9) | −0.1675 (3) | −0.05956 (14) | 0.0541 (6) |
| H25A | 0.2275 | −0.2115 | −0.0531 | 0.065* |
| H25B | 0.1952 | −0.1145 | −0.1080 | 0.065* |
| C26 | 0.16055 (11) | −0.2826 (3) | −0.05889 (18) | 0.0685 (8) |
| H26A | 0.1637 | −0.3381 | −0.0120 | 0.103* |
| H26B | 0.1641 | −0.3474 | −0.1019 | 0.103* |
| H26C | 0.1303 | −0.2373 | −0.0623 | 0.103* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0460 (4) | 0.0596 (4) | 0.0915 (5) | −0.0247 (3) | −0.0215 (3) | 0.0354 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Cl2 | 0.0504 (4) | 0.0691 (4) | 0.0743 (5) | 0.0082 (3) | −0.0234 (3) | 0.0176 (3) |
| O1 | 0.0330 (8) | 0.0669 (10) | 0.0423 (9) | −0.0021 (7) | −0.0079 (7) | −0.0085 (7) |
| O2 | 0.0522 (10) | 0.0538 (10) | 0.0579 (10) | 0.0060 (8) | −0.0121 (8) | 0.0140 (8) |
| O3 | 0.0441 (10) | 0.0515 (10) | 0.0677 (11) | −0.0063 (7) | 0.0194 (8) | −0.0078 (8) |
| O4 | 0.0390 (8) | 0.0434 (8) | 0.0540 (9) | −0.0071 (7) | −0.0003 (7) | −0.0137 (7) |
| C1 | 0.0308 (11) | 0.0483 (12) | 0.0439 (12) | −0.0006 (9) | −0.0042 (9) | 0.0008 (9) |
| C2 | 0.0267 (10) | 0.0317 (10) | 0.0406 (11) | −0.0033 (7) | −0.0025 (8) | 0.0009 (8) |
| C3 | 0.0268 (10) | 0.0263 (9) | 0.0368 (10) | −0.0033 (7) | −0.0030 (8) | 0.0011 (7) |
| C4 | 0.0318 (10) | 0.0345 (10) | 0.0381 (11) | −0.0020 (8) | −0.0041 (8) | 0.0015 (8) |
| C5 | 0.0517 (13) | 0.0346 (11) | 0.0333 (10) | −0.0043 (9) | −0.0074 (9) | −0.0035 (8) |
| C6 | 0.0795 (18) | 0.0389 (12) | 0.0423 (13) | −0.0168 (12) | −0.0022 (12) | −0.0051 (10) |
| C7 | 0.130 (3) | 0.0322 (13) | 0.0493 (15) | −0.0157 (15) | −0.0189 (17) | 0.0005 (11) |
| C8 | 0.110 (3) | 0.0433 (15) | 0.0613 (17) | 0.0191 (15) | −0.0230 (18) | −0.0063 (13) |
| C9 | 0.0715 (18) | 0.0554 (16) | 0.0603 (16) | 0.0205 (13) | −0.0138 (14) | −0.0078 (13) |
| C10 | 0.0489 (13) | 0.0430 (12) | 0.0387 (11) | 0.0062 (10) | −0.0081 (10) | −0.0037 (9) |
| C11 | 0.0400 (12) | 0.0532 (13) | 0.0392 (12) | −0.0001 (10) | −0.0028 (10) | −0.0026 (10) |
| C12 | 0.0506 (15) | 0.085 (2) | 0.0606 (17) | 0.0010 (14) | 0.0145 (13) | −0.0025 (15) |
| C13 | 0.071 (2) | 0.092 (2) | 0.069 (2) | −0.0250 (18) | 0.0225 (16) | 0.0101 (17) |
| C14 | 0.089 (2) | 0.0612 (17) | 0.0574 (17) | −0.0222 (16) | 0.0069 (16) | 0.0162 (13) |
| C15 | 0.0611 (16) | 0.0435 (13) | 0.0467 (13) | −0.0029 (11) | −0.0020 (12) | 0.0064 (10) |
| C16 | 0.0388 (11) | 0.0412 (11) | 0.0330 (10) | −0.0042 (9) | −0.0033 (9) | 0.0019 (8) |
| C17 | 0.0281 (9) | 0.0277 (9) | 0.0340 (10) | −0.0013 (7) | −0.0029 (8) | −0.0039 (7) |
| C18 | 0.0336 (11) | 0.0337 (10) | 0.0430 (11) | −0.0071 (8) | −0.0048 (9) | 0.0018 (9) |
| C19 | 0.0471 (13) | 0.0347 (11) | 0.0469 (12) | −0.0054 (9) | −0.0079 (10) | 0.0077 (9) |
| C20 | 0.0370 (11) | 0.0400 (11) | 0.0434 (12) | 0.0052 (9) | −0.0107 (9) | −0.0004 (9) |
| C21 | 0.0309 (11) | 0.0431 (11) | 0.0493 (13) | −0.0056 (9) | −0.0070 (9) | −0.0004 (10) |
| C22 | 0.0317 (10) | 0.0322 (10) | 0.0424 (11) | −0.0061 (8) | −0.0030 (9) | 0.0027 (8) |
| C23 | 0.0327 (12) | 0.0820 (18) | 0.0634 (16) | −0.0086 (12) | −0.0113 (11) | −0.0038 (14) |
| C24 | 0.0340 (11) | 0.0305 (10) | 0.0419 (11) | 0.0004 (8) | −0.0020 (9) | 0.0041 (8) |
| C25 | 0.0615 (16) | 0.0469 (13) | 0.0534 (14) | 0.0042 (12) | −0.0065 (12) | −0.0132 (11) |
| C26 | 0.078 (2) | 0.0479 (15) | 0.0779 (19) | −0.0077 (13) | −0.0150 (16) | −0.0140 (13) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl1—C22 | 1.733 (2) | C11—C16 | 1.386 (3) |
| Cl2—C20 | 1.732 (2) | C11—C12 | 1.386 (3) |
| O1—C1 | 1.420 (3) | C12—C13 | 1.384 (4) |
| O1—C4 | 1.442 (2) | C12—H12 | 0.9300 |
| O2—C1 | 1.412 (3) | C13—C14 | 1.368 (4) |
| O2—H2 | 0.8200 | C13—H13 | 0.9300 |
| O3—C24 | 1.203 (3) | C14—C15 | 1.376 (4) |
| O4—C24 | 1.320 (2) | C14—H14 | 0.9300 |
| O4—C25 | 1.456 (3) | C15—C16 | 1.376 (3) |
| C1—C23 | 1.508 (3) | C15—H15 | 0.9300 |
| C1—C2 | 1.521 (3) | C17—C22 | 1.389 (3) |
| C2—C24 | 1.506 (3) | C17—C18 | 1.389 (3) |
| C2—C3 | 1.520 (3) | C18—C19 | 1.377 (3) |
| C2—H2A | 0.9800 | C18—H18 | 0.9300 |
| C3—C17 | 1.505 (3) | C19—C20 | 1.371 (3) |

| | | | |
|---|---|---|---|
| C3—C4 | 1.569 (3) | C19—H19 | 0.9300 |
| C3—H3 | 0.9800 | C20—C21 | 1.371 (3) |
| C4—C16 | 1.512 (3) | C21—C22 | 1.382 (3) |
| C4—C5 | 1.512 (3) | C21—H21 | 0.9300 |
| C5—C6 | 1.383 (3) | C22—Cl1 | 1.733 (2) |
| C5—C10 | 1.391 (3) | C23—H23A | 0.9600 |
| C6—C7 | 1.396 (4) | C23—H23B | 0.9600 |
| C6—H6 | 0.9300 | C23—H23C | 0.9600 |
| C7—C8 | 1.373 (4) | C25—C26 | 1.472 (4) |
| C7—H7 | 0.9300 | C25—H25A | 0.9700 |
| C8—C9 | 1.358 (4) | C25—H25B | 0.9700 |
| C8—H8 | 0.9300 | C26—H26A | 0.9600 |
| C9—C10 | 1.384 (3) | C26—H26B | 0.9600 |
| C9—H9 | 0.9300 | C26—H26C | 0.9600 |
| C10—C11 | 1.466 (3) | | |
| C1—O1—C4 | 111.55 (15) | C14—C13—H13 | 119.4 |
| C1—O2—H2 | 109.5 | C12—C13—H13 | 119.4 |
| C24—O4—C25 | 116.72 (17) | C13—C14—C15 | 121.0 (3) |
| O2—C1—O1 | 110.58 (18) | C13—C14—H14 | 119.5 |
| O2—C1—C23 | 111.83 (19) | C15—C14—H14 | 119.5 |
| O1—C1—C23 | 107.81 (18) | C14—C15—C16 | 118.4 (3) |
| O2—C1—C2 | 106.62 (17) | C14—C15—H15 | 120.8 |
| O1—C1—C2 | 104.00 (16) | C16—C15—H15 | 120.8 |
| C23—C1—C2 | 115.72 (19) | C15—C16—C11 | 121.2 (2) |
| C24—C2—C3 | 116.91 (16) | C15—C16—C4 | 128.4 (2) |
| C24—C2—C1 | 112.78 (17) | C11—C16—C4 | 110.23 (18) |
| C3—C2—C1 | 102.23 (16) | C22—C17—C18 | 115.87 (18) |
| C24—C2—H2A | 108.2 | C22—C17—C3 | 121.93 (17) |
| C3—C2—H2A | 108.2 | C18—C17—C3 | 122.18 (17) |
| C1—C2—H2A | 108.2 | C19—C18—C17 | 122.68 (19) |
| C17—C3—C2 | 117.24 (16) | C19—C18—H18 | 118.7 |
| C17—C3—C4 | 114.73 (15) | C17—C18—H18 | 118.7 |
| C2—C3—C4 | 101.89 (15) | C20—C19—C18 | 119.0 (2) |
| C17—C3—H3 | 107.5 | C20—C19—H19 | 120.5 |
| C2—C3—H3 | 107.5 | C18—C19—H19 | 120.5 |
| C4—C3—H3 | 107.5 | C19—C20—C21 | 120.98 (19) |
| O1—C4—C16 | 113.98 (16) | C19—C20—Cl2 | 120.33 (17) |
| O1—C4—C5 | 111.29 (16) | C21—C20—Cl2 | 118.68 (16) |
| C16—C4—C5 | 101.64 (17) | C20—C21—C22 | 118.69 (19) |
| O1—C4—C3 | 104.61 (15) | C20—C21—H21 | 120.7 |
| C16—C4—C3 | 111.10 (16) | C22—C21—H21 | 120.7 |
| C5—C4—C3 | 114.56 (16) | C21—C22—C17 | 122.71 (18) |
| C6—C5—C10 | 120.4 (2) | C21—C22—Cl1 | 116.49 (15) |
| C6—C5—C4 | 129.6 (2) | C17—C22—Cl1 | 120.78 (15) |
| C10—C5—C4 | 109.98 (18) | C21—C22—Cl1 | 116.49 (15) |
| C5—C6—C7 | 117.9 (3) | C17—C22—Cl1 | 120.78 (15) |
| C5—C6—H6 | 121.1 | C1—C23—H23A | 109.5 |
| C7—C6—H6 | 121.1 | C1—C23—H23B | 109.5 |
| C8—C7—C6 | 121.0 (3) | H23A—C23—H23B | 109.5 |

| | | | |
|---|---|---|---|
| C8—C7—H7 | 119.5 | C1—C23—H23C | 109.5 |
| C6—C7—H7 | 119.5 | H23A—C23—H23C | 109.5 |
| C9—C8—C7 | 121.2 (3) | H23B—C23—H23C | 109.5 |
| C9—C8—H8 | 119.4 | O3—C24—O4 | 123.5 (2) |
| C7—C8—H8 | 119.4 | O3—C24—C2 | 122.80 (19) |
| C8—C9—C10 | 119.0 (3) | O4—C24—C2 | 113.70 (17) |
| C8—C9—H9 | 120.5 | O4—C25—C26 | 107.2 (2) |
| C10—C9—H9 | 120.5 | O4—C25—H25A | 110.3 |
| C9—C10—C5 | 120.6 (2) | C26—C25—H25A | 110.3 |
| C9—C10—C11 | 130.8 (2) | O4—C25—H25B | 110.3 |
| C5—C10—C11 | 108.50 (19) | C26—C25—H25B | 110.3 |
| C16—C11—C12 | 119.9 (2) | H25A—C25—H25B | 108.5 |
| C16—C11—C10 | 108.35 (19) | C25—C26—H26A | 109.5 |
| C12—C11—C10 | 131.6 (2) | C25—C26—H26B | 109.5 |
| C13—C12—C11 | 118.3 (3) | H26A—C26—H26B | 109.5 |
| C13—C12—H12 | 120.8 | C25—C26—H26C | 109.5 |
| C11—C12—H12 | 120.8 | H26A—C26—H26C | 109.5 |
| C14—C13—C12 | 121.1 (3) | H26B—C26—H26C | 109.5 |
| C4—O1—C1—O2 | −89.2 (2) | C10—C11—C12—C13 | −174.5 (3) |
| C4—O1—C1—C23 | 148.26 (19) | C11—C12—C13—C14 | 0.4 (5) |
| C4—O1—C1—C2 | 24.9 (2) | C12—C13—C14—C15 | −2.7 (5) |
| O2—C1—C2—C24 | −48.0 (2) | C13—C14—C15—C16 | 2.7 (4) |
| O1—C1—C2—C24 | −164.86 (16) | C14—C15—C16—C11 | −0.5 (3) |
| C23—C1—C2—C24 | 77.1 (2) | C14—C15—C16—C4 | −176.5 (2) |
| O2—C1—C2—C3 | 78.44 (19) | C12—C11—C16—C15 | −1.9 (3) |
| O1—C1—C2—C3 | −38.47 (19) | C10—C11—C16—C15 | 175.3 (2) |
| C23—C1—C2—C3 | −156.49 (19) | C12—C11—C16—C4 | 174.9 (2) |
| C24—C2—C3—C17 | −73.5 (2) | C10—C11—C16—C4 | −8.0 (2) |
| C1—C2—C3—C17 | 162.79 (16) | O1—C4—C16—C15 | −52.6 (3) |
| C24—C2—C3—C4 | 160.35 (16) | C5—C4—C16—C15 | −172.4 (2) |
| C1—C2—C3—C4 | 36.69 (18) | C3—C4—C16—C15 | 65.3 (3) |
| C1—O1—C4—C16 | 120.28 (19) | O1—C4—C16—C11 | 131.00 (19) |
| C1—O1—C4—C5 | −125.50 (19) | C5—C4—C16—C11 | 11.2 (2) |
| C1—O1—C4—C3 | −1.3 (2) | C3—C4—C16—C11 | −111.1 (2) |
| C17—C3—C4—O1 | −150.41 (16) | C2—C3—C17—C22 | 145.93 (19) |
| C2—C3—C4—O1 | −22.69 (18) | C4—C3—C17—C22 | −94.6 (2) |
| C17—C3—C4—C16 | 86.2 (2) | C2—C3—C17—C18 | −35.9 (3) |
| C2—C3—C4—C16 | −146.11 (16) | C4—C3—C17—C18 | 83.6 (2) |
| C17—C3—C4—C5 | −28.3 (2) | C22—C17—C18—C19 | 1.4 (3) |
| C2—C3—C4—C5 | 99.43 (19) | C3—C17—C18—C19 | −176.88 (19) |
| O1—C4—C5—C6 | 49.6 (3) | C17—C18—C19—C20 | 0.7 (3) |
| C16—C4—C5—C6 | 171.4 (2) | C18—C19—C20—C21 | −1.5 (3) |
| C3—C4—C5—C6 | −68.8 (3) | C18—C19—C20—Cl2 | 179.31 (17) |
| O1—C4—C5—C10 | −132.24 (18) | C19—C20—C21—C22 | 0.2 (3) |
| C16—C4—C5—C10 | −10.5 (2) | Cl2—C20—C21—C22 | 179.34 (16) |
| C3—C4—C5—C10 | 109.4 (2) | C20—C21—C22—C17 | 2.1 (3) |
| C10—C5—C6—C7 | −2.3 (3) | C20—C21—C22—Cl1 | −176.59 (17) |
| C4—C5—C6—C7 | 175.7 (2) | C20—C21—C22—Cl1 | −176.59 (17) |
| C5—C6—C7—C8 | 0.3 (4) | C18—C17—C22—C21 | −2.8 (3) |

| | | | |
|---|---|---|---|
| C6—C7—C8—C9 | 1.4 (4) | C3—C17—C22—C21 | 175.45 (19) |
| C7—C8—C9—C10 | −0.9 (4) | C18—C17—C22—Cl1 | 175.82 (15) |
| C8—C9—C10—C5 | −1.1 (4) | C3—C17—C22—Cl1 | −5.9 (3) |
| C8—C9—C10—C11 | 176.4 (2) | C18—C17—C22—Cl1 | 175.82 (15) |
| C6—C5—C10—C9 | 2.7 (3) | C3—C17—C22—Cl1 | −5.9 (3) |
| C4—C5—C10—C9 | −175.6 (2) | C25—O4—C24—O3 | −3.3 (3) |
| C6—C5—C10—C11 | −175.3 (2) | C25—O4—C24—C2 | 176.29 (18) |
| C4—C5—C10—C11 | 6.4 (2) | C3—C2—C24—O3 | 170.8 (2) |
| C9—C10—C11—C16 | −176.7 (2) | C1—C2—C24—O3 | −71.1 (3) |
| C5—C10—C11—C16 | 1.0 (2) | C3—C2—C24—O4 | −8.7 (3) |
| C9—C10—C11—C12 | −0.1 (4) | C1—C2—C24—O4 | 109.3 (2) |
| C5—C10—C11—C12 | 177.7 (3) | C24—O4—C25—C26 | 171.0 (2) |
| C16—C11—C12—C13 | 1.9 (4) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2A···O3$^{i}$ | 0.98 | 2.37 | 3.331 (3) | 167 |
| C6—H6···O3$^{i}$ | 0.93 | 2.55 | 3.393 (3) | 151 |
| C3—H3···Cl1 | 0.98 | 2.57 | 3.082 (2) | 113 |
| C26—H26C···Cg1$^{ii}$ | 0.96 | 2.95 | 3.556 (1) | 122 |

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

**Fig. 1**

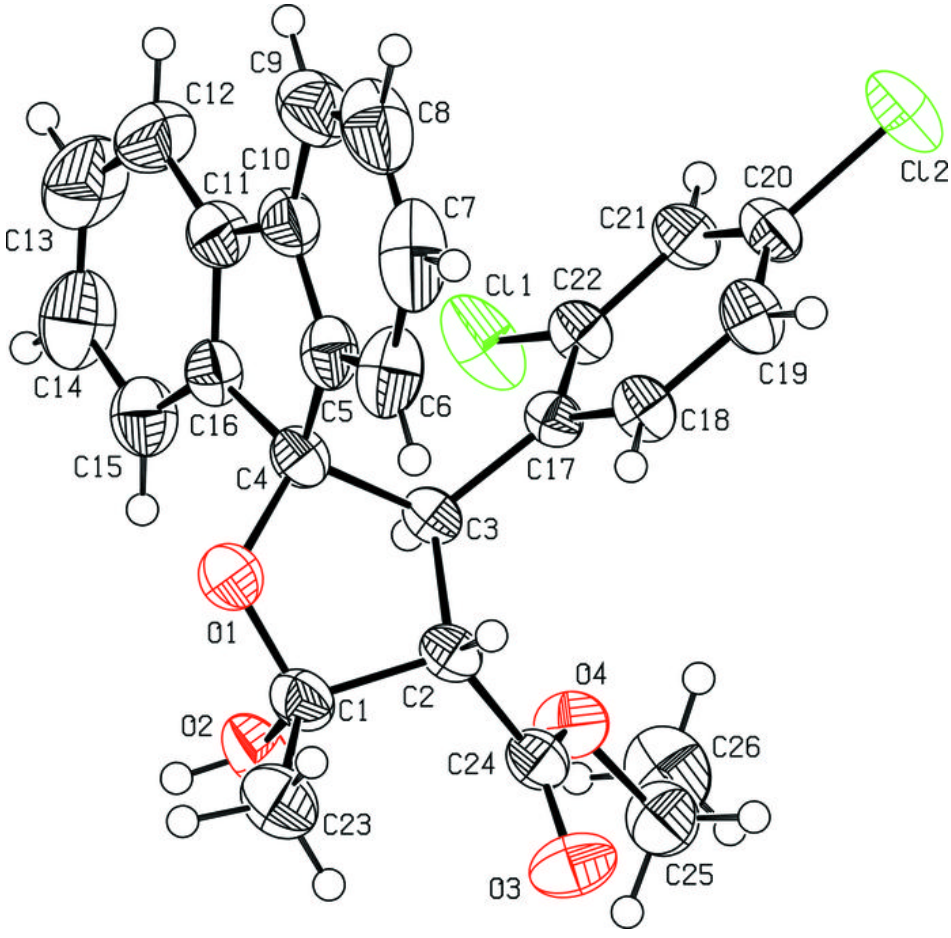
C9
C12
C8
C10
C11
C7
C13
C21
C20
Cl2
Cl1
C22
C19
C14
C5
C6
C18
C16
C15
C4
C17
C3
O1
O4
C1
C2
O2
C24
C26
C23
C25
O3

**Fig. 2**