

Crystal structure of (Z)-3-[5-chloro-2-(prop-2-ynyloxy)phenyl]-3-hydroxy-1-[4-(trifluoromethyl)phenyl]prop-2-en-1-one

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The title compound, C₁₉H₁₂ClF₃O₃, obtained by the photochemical transformation of 2-[5-chloro-2-(prop-2-ynyloxy)benzoyl]-3-[4-(trifluoromethyl)phenyl]oxirane adopts a *Z* conformation with respect to the enolic C=C double bond. The dihedral angle between the benzene rings is 12.25 (16)° and an intramolecular O—H···O hydrogen bond closes an *S*(6) ring. An intramolecular C—H···O interaction also leads to an *S*(6) ring. In the crystal, very weak C—H···O interactions and short Cl···Cl contacts [3.3221 (16) Å] are seen, as well as weak aromatic π – π stacking interactions [centroid–centroid separation = 3.879 (2) Å].

Keywords: crystal structure; hydroxy enone; tautomerisation; hydrogen bonding; photo-isomerisation.

CCDC reference: 1403411

1. Related literature

For background to 1,3-diketones, see: Andrae *et al.* (1997); Crouse *et al.* (1989); Diana *et al.* (1978); Nishiyama *et al.* (2002); Sheikh *et al.* (2009, 2013); Tchertanov & Mouscadet (2007).

2. Experimental

2.1. Crystal data

C₁₉H₁₂ClF₃O₃
M_r = 380.74
 Triclinic, *P* $\bar{1}$
a = 8.2203 (12) Å
b = 9.3822 (14) Å
c = 12.3140 (18) Å
 α = 90.150 (2)°
 β = 109.201 (2)°
 γ = 106.212 (2)°
V = 856.5 (2) Å³
Z = 2
 Mo *K* α radiation
 μ = 0.27 mm⁻¹
T = 273 K
 0.34 × 0.29 × 0.14 mm

2.2. Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.912, *T_{max}* = 0.962
 8272 measured reflections
 3016 independent reflections
 2503 reflections with *I* > 2 σ (*I*)
R_{int} = 0.022

2.3. Refinement

R[*F*² > 2 σ (*F*²)] = 0.068
wR(*F*²) = 0.191
S = 1.10
 3003 reflections
 235 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max}$ = 0.52 e Å⁻³
 $\Delta\rho_{\min}$ = -0.35 e Å⁻³

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

| <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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| O2—H24···O1 | 0.82 | 1.79 | 2.529 (3) | 149 |
| C11—H11···O3 | 0.93 | 2.09 | 2.747 (3) | 126 |
| C7—H7B···O1 ⁱ | 0.97 | 2.62 | 3.476 (4) | 147 |

Symmetry code: (i) *x*, *y* + 1, *z*.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7451).

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supporting information

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Crystal structure of (Z)-3-[5-chloro-2-(prop-2-ynyloxy)phenyl]-3-hydroxy-1-[4-(trifluoromethyl)phenyl]prop-2-en-1-one

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S1. Chemical context

The functionalized β -hydroxyenones (or 1,3-diketones) are medicinal significant compounds showing antiviral (Diana *et al.*, 1978), insecticidal (Crouse *et al.*, 1989), anti-sunscreen (Andrae *et al.*, 1997), antioxidant (Nishiyama *et al.*, 2002), and more important HIV-1 Integrase (IN) inhibitor (Tchertanov & Mouscadet, 2007) activity. Also, 1,3-diketones are the building blocks for the synthesis of core heterocycles (Sheikh *et al.*, 2009), and biologically important metal complexes (Sheikh *et al.*, 2013). Our interest in the catalyst free photochemical organic transformation led us to synthesize the title compound and we report herein on its crystal structure (Fig. 1).

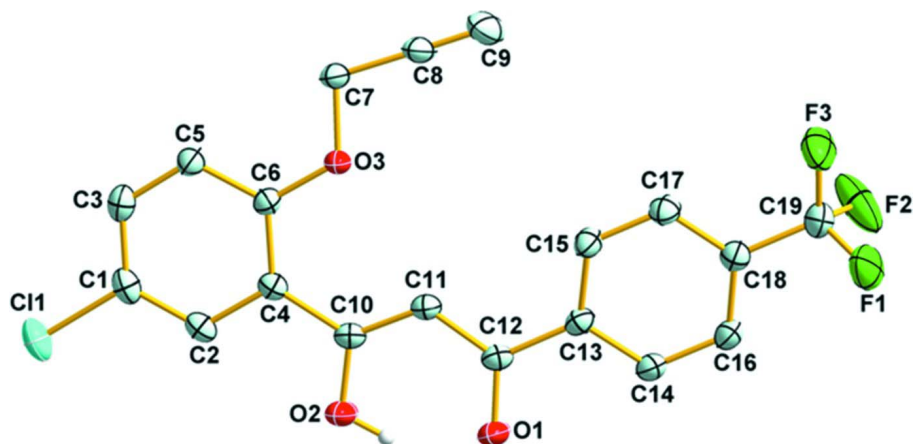
S2. Synthesis and crystallization

A deoxygenated solution of 2-{5-chloro-2-(prop-2-ynyloxy)benzoyl}-3-{4-(trifluoromethyl)phenyl} oxirane (2.0 mmol, 760 mg) in acetonitrile (100 ml) contained in a Pyrex glass vessel was purged with N₂ for 30 min. and then irradiated under N₂ atmosphere with light from a 125W Hg-vapor lamp for 90 min. The removal of solvent under reduced pressure yielded a gummy mass that was chromatographed over a silica gel column. The column was eluted with increasing proportion of ethyl acetate in pet ether-ethyl acetate mixture to obtain the desired photoproduct. In addition to this, another photoproduct 8-chloro-2-(4-trifluoromethylphenyl) benzo[b]furo[2,3-e]oxepin-10(4H)-one was also separated from the photolysate. The resulting pale yellow solid was filtered, washed several times with pet ether and then dried in vacuo overnight to yield the desired (Z)-3-{5-chloro-2-(prop-2-ynyloxy)phenyl}-1-{4-(trifluoromethyl)phenyl}-3-hydroxyprop-2-en-1-one, (267 mg, 35% yield). The pale yellow rectangular crystals, suitable for X-ray structure analysis, were obtained by recrystallization from ethanol by slow evaporation at room temperature after several days (m.p. 383–385 K).

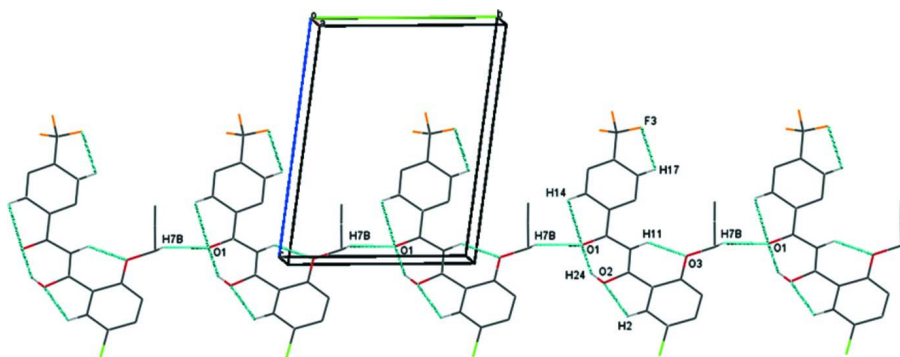
FT—IR (KBr) ν : 3418 (-OH), 2150 (C \equiv C), 1713 (C=O), 1605 (C=C) cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ , ppm: 8.08 (2H, d, J = 8.2 Hz, H-3'', 5''), 7.95 (1H, d, J = 2.7 Hz, H-6'), 7.73 (2H, d, J = 8.3 Hz, H-2'', 6''), 7.44 (1H, dd, J = 8.8, 2.7 Hz, H-4'), 7.23 (1H, s, H-2), 7.03 (1H, d, J = 8.8 Hz, H-3'), 4.82 (2H, d, J = 2.4 Hz, OCH₂-1'''), 2.63 (1H, t, J = 2.3 Hz, \equiv CH-3'''); ¹³C NMR (100 MHz, CDCl₃): δ , ppm: 57.00, 99.21, 114.67, 122.34, 125.05, 125.63, 126.58, 127.40, 127.62, 130.30, 132.84, 133.54, 133.86, 138.72, 154.99, 183.24, 183.69.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All non-hydrogen atoms were refined anisotropically. The positions of the hydrogen atoms were fixed according to a riding model and were refined isotropically.

**Figure 1**

Molecular structure of the title compound showing displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

A view along the *a* axis of the inter- and intramolecular hydrogen bonds in the title compound (shown as dashed lines, see Table 1 for details). Hydrogen atoms not involved in hydrogen bonding are omitted for clarity.

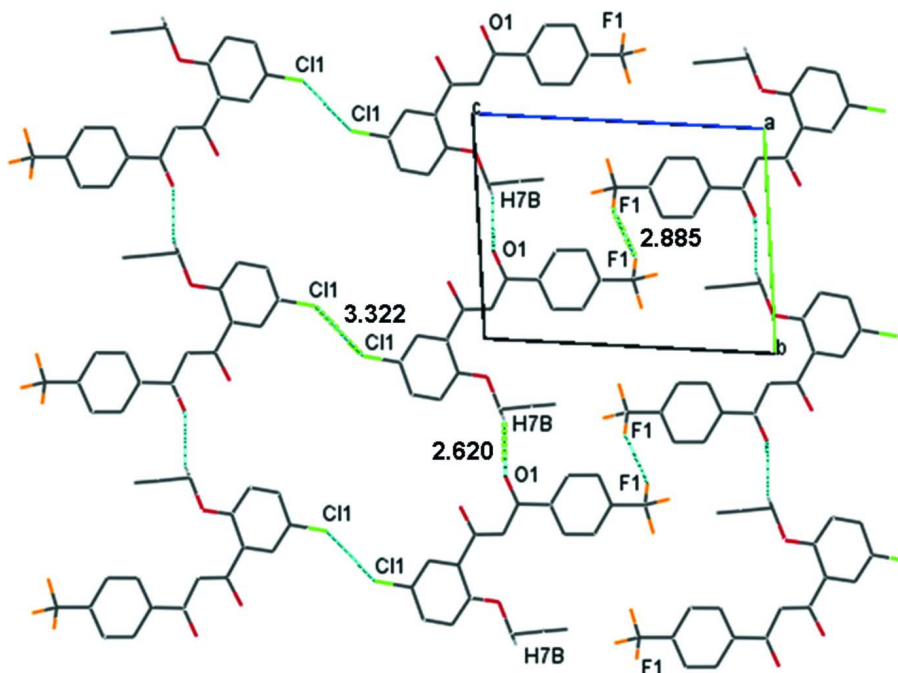


Figure 3

A view along the *a* axis showing F...F and Cl...Cl contact distances (dashed lines). Hydrogen atoms not involved in the interactions are excluded for clarity.

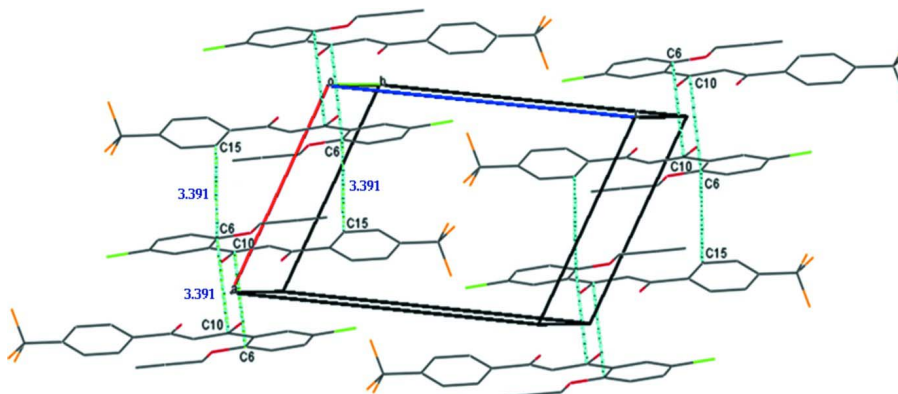


Figure 4

A partial view along *b* axis of the π - π interactions (dashed lines) in the crystal packing of the title compound. All hydrogen atoms are omitted for clarity.

(Z)-3-[5-Chloro-2-(prop-2-ynyloxy)phenyl]-3-hydroxy-1-[4-(trifluoromethyl)phenyl]prop-2-en-1-one

Crystal data

$C_{19}H_{12}ClF_3O_3$

$M_r = 380.74$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.2203$ (12) Å

$b = 9.3822$ (14) Å

$c = 12.3140$ (18) Å

$\alpha = 90.150$ (2)°

$\beta = 109.201$ (2)°

$\gamma = 106.212$ (2)°

$V = 856.5$ (2) Å³

$Z = 2$

$F(000) = 388.0$

$D_x = 1.476$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 3035 reflections
 $\theta = 2.3\text{--}25.6^\circ$
 $\mu = 0.27 \text{ mm}^{-1}$

$T = 273 \text{ K}$
 Block, colorless
 $0.34 \times 0.29 \times 0.14 \text{ mm}$

Data collection

Bruker SMART CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2000)
 $T_{\min} = 0.912$, $T_{\max} = 0.962$

8272 measured reflections
 3016 independent reflections
 2503 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -9 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.191$
 $S = 1.10$
 3003 reflections
 235 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0963P)^2 + 0.390P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.52 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.35 \text{ e \AA}^{-3}$

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)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C11 | 0.10694 (14) | 1.10578 (13) | 1.42182 (7) | 0.0913 (4) |
| O3 | 0.2888 (3) | 1.16996 (19) | 0.99885 (16) | 0.0600 (5) |
| O2 | 0.1279 (3) | 0.7541 (2) | 1.1146 (2) | 0.0766 (7) |
| H24 | 0.1237 | 0.6813 | 1.0756 | 0.115* |
| C13 | 0.2457 (4) | 0.7250 (3) | 0.8037 (2) | 0.0508 (6) |
| C12 | 0.2108 (4) | 0.7295 (3) | 0.9149 (2) | 0.0525 (7) |
| C4 | 0.1949 (3) | 1.0149 (3) | 1.1317 (2) | 0.0474 (6) |
| C2 | 0.1511 (4) | 1.0020 (3) | 1.2318 (2) | 0.0558 (7) |
| H2 | 0.1147 | 0.9080 | 1.2557 | 0.067* |
| C6 | 0.2471 (3) | 1.1588 (3) | 1.0975 (2) | 0.0485 (6) |
| C10 | 0.1852 (3) | 0.8758 (3) | 1.0691 (2) | 0.0493 (6) |
| C11 | 0.2284 (4) | 0.8668 (3) | 0.9711 (2) | 0.0531 (7) |

| | | | | |
|-----|------------|------------|------------|-------------|
| H11 | 0.2707 | 0.9545 | 0.9408 | 0.064* |
| C15 | 0.3271 (4) | 0.8532 (3) | 0.7619 (3) | 0.0585 (7) |
| H15 | 0.3643 | 0.9449 | 0.8054 | 0.070* |
| C18 | 0.3000 (4) | 0.7117 (3) | 0.5930 (2) | 0.0575 (7) |
| C5 | 0.2558 (4) | 1.2823 (3) | 1.1638 (3) | 0.0602 (7) |
| H5 | 0.2906 | 1.3771 | 1.1410 | 0.072* |
| C17 | 0.3537 (4) | 0.8470 (3) | 0.6579 (3) | 0.0618 (7) |
| H17 | 0.4077 | 0.9339 | 0.6313 | 0.074* |
| C1 | 0.1610 (4) | 1.1259 (4) | 1.2959 (2) | 0.0620 (8) |
| F3 | 0.4185 (4) | 0.8295 (3) | 0.4541 (2) | 0.1194 (9) |
| C7 | 0.3400 (4) | 1.3122 (3) | 0.9602 (3) | 0.0611 (7) |
| H7A | 0.4485 | 1.3760 | 1.0179 | 0.073* |
| H7B | 0.2448 | 1.3590 | 0.9473 | 0.073* |
| C14 | 0.1936 (4) | 0.5899 (3) | 0.7376 (3) | 0.0633 (8) |
| H14 | 0.1400 | 0.5026 | 0.7641 | 0.076* |
| C3 | 0.2136 (4) | 1.2658 (4) | 1.2624 (3) | 0.0656 (8) |
| H3 | 0.2204 | 1.3491 | 1.3067 | 0.079* |
| C8 | 0.3725 (5) | 1.2911 (4) | 0.8531 (3) | 0.0750 (9) |
| C16 | 0.2202 (5) | 0.5826 (3) | 0.6329 (3) | 0.0686 (8) |
| H16 | 0.1844 | 0.4911 | 0.5893 | 0.082* |
| C19 | 0.3237 (5) | 0.7041 (4) | 0.4781 (3) | 0.0763 (9) |
| C9 | 0.3999 (8) | 1.2763 (5) | 0.7678 (4) | 0.1171 (17) |
| H9 | 0.4219 | 1.2645 | 0.6994 | 0.140* |
| F2 | 0.1707 (4) | 0.6729 (5) | 0.3933 (2) | 0.1649 (15) |
| F1 | 0.4002 (7) | 0.6064 (4) | 0.4652 (3) | 0.1871 (19) |
| O1 | 0.1593 (3) | 0.6070 (2) | 0.9541 (2) | 0.0759 (7) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.0980 (7) | 0.1272 (9) | 0.0637 (6) | 0.0341 (6) | 0.0464 (5) | 0.0084 (5) |
| O3 | 0.0908 (14) | 0.0395 (10) | 0.0603 (12) | 0.0176 (9) | 0.0411 (11) | 0.0111 (8) |
| O2 | 0.1181 (19) | 0.0490 (12) | 0.0843 (15) | 0.0281 (12) | 0.0595 (14) | 0.0234 (10) |
| C13 | 0.0497 (14) | 0.0436 (14) | 0.0584 (16) | 0.0184 (11) | 0.0138 (12) | 0.0050 (12) |
| C12 | 0.0546 (15) | 0.0452 (14) | 0.0606 (16) | 0.0198 (12) | 0.0194 (13) | 0.0107 (12) |
| C4 | 0.0428 (13) | 0.0516 (15) | 0.0478 (14) | 0.0153 (11) | 0.0147 (11) | 0.0084 (11) |
| C2 | 0.0511 (15) | 0.0652 (17) | 0.0518 (15) | 0.0155 (13) | 0.0199 (12) | 0.0142 (13) |
| C6 | 0.0479 (14) | 0.0473 (14) | 0.0502 (14) | 0.0137 (11) | 0.0168 (11) | 0.0048 (11) |
| C10 | 0.0491 (14) | 0.0440 (14) | 0.0553 (15) | 0.0151 (11) | 0.0173 (12) | 0.0134 (11) |
| C11 | 0.0618 (16) | 0.0408 (14) | 0.0579 (16) | 0.0137 (12) | 0.0235 (13) | 0.0104 (12) |
| C15 | 0.0625 (17) | 0.0463 (15) | 0.0667 (18) | 0.0101 (13) | 0.0272 (14) | -0.0019 (13) |
| C18 | 0.0556 (16) | 0.0593 (17) | 0.0560 (16) | 0.0204 (13) | 0.0145 (13) | 0.0020 (13) |
| C5 | 0.0659 (18) | 0.0521 (16) | 0.0641 (17) | 0.0166 (13) | 0.0252 (15) | 0.0005 (13) |
| C17 | 0.0632 (17) | 0.0519 (16) | 0.0696 (19) | 0.0092 (13) | 0.0284 (15) | 0.0047 (14) |
| C1 | 0.0552 (16) | 0.084 (2) | 0.0492 (15) | 0.0215 (15) | 0.0204 (13) | 0.0054 (14) |
| F3 | 0.152 (2) | 0.1049 (18) | 0.1022 (17) | -0.0047 (16) | 0.0797 (17) | -0.0060 (14) |
| C7 | 0.0781 (19) | 0.0425 (14) | 0.0730 (19) | 0.0208 (13) | 0.0367 (16) | 0.0161 (13) |
| C14 | 0.082 (2) | 0.0418 (15) | 0.0623 (18) | 0.0163 (14) | 0.0211 (15) | 0.0068 (13) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|--------------|
| C3 | 0.0672 (18) | 0.071 (2) | 0.0613 (18) | 0.0240 (15) | 0.0219 (15) | -0.0085 (15) |
| C8 | 0.109 (3) | 0.0563 (18) | 0.085 (2) | 0.0361 (18) | 0.057 (2) | 0.0315 (16) |
| C16 | 0.091 (2) | 0.0459 (16) | 0.0621 (18) | 0.0197 (15) | 0.0182 (16) | -0.0030 (13) |
| C19 | 0.094 (3) | 0.067 (2) | 0.062 (2) | 0.0193 (19) | 0.0233 (19) | -0.0047 (16) |
| C9 | 0.196 (5) | 0.104 (3) | 0.111 (3) | 0.073 (3) | 0.105 (4) | 0.051 (3) |
| F2 | 0.118 (2) | 0.248 (4) | 0.0599 (14) | -0.032 (2) | 0.0136 (14) | 0.0125 (18) |
| F1 | 0.372 (6) | 0.176 (3) | 0.126 (2) | 0.180 (4) | 0.148 (3) | 0.048 (2) |
| O1 | 0.1138 (18) | 0.0424 (11) | 0.0871 (15) | 0.0259 (11) | 0.0525 (14) | 0.0177 (10) |

Geometric parameters (Å, °)

| | | | |
|-------------|-----------|-------------|-----------|
| C11—C1 | 1.745 (3) | C18—C17 | 1.377 (4) |
| O3—C6 | 1.364 (3) | C18—C16 | 1.382 (4) |
| O3—C7 | 1.416 (3) | C18—C19 | 1.494 (4) |
| O2—C10 | 1.310 (3) | C5—C3 | 1.366 (4) |
| O2—H24 | 0.8200 | C5—H5 | 0.9300 |
| C13—C14 | 1.384 (4) | C17—H17 | 0.9300 |
| C13—C15 | 1.393 (4) | C1—C3 | 1.372 (5) |
| C13—C12 | 1.491 (4) | F3—C19 | 1.312 (4) |
| C12—O1 | 1.265 (3) | C7—C8 | 1.452 (4) |
| C12—C11 | 1.409 (4) | C7—H7A | 0.9700 |
| C4—C2 | 1.390 (4) | C7—H7B | 0.9700 |
| C4—C6 | 1.408 (4) | C14—C16 | 1.381 (4) |
| C4—C10 | 1.484 (4) | C14—H14 | 0.9300 |
| C2—C1 | 1.371 (4) | C3—H3 | 0.9300 |
| C2—H2 | 0.9300 | C8—C9 | 1.161 (5) |
| C6—C5 | 1.388 (4) | C16—H16 | 0.9300 |
| C10—C11 | 1.373 (4) | C19—F1 | 1.283 (4) |
| C11—H11 | 0.9300 | C19—F2 | 1.299 (4) |
| C15—C17 | 1.372 (4) | C9—H9 | 0.9300 |
| C15—H15 | 0.9300 | | |
| C6—O3—C7 | 119.4 (2) | C6—C5—H5 | 119.7 |
| C10—O2—H24 | 109.5 | C15—C17—C18 | 119.8 (3) |
| C14—C13—C15 | 117.9 (3) | C15—C17—H17 | 120.1 |
| C14—C13—C12 | 119.7 (3) | C18—C17—H17 | 120.1 |
| C15—C13—C12 | 122.4 (2) | C2—C1—C3 | 120.8 (3) |
| O1—C12—C11 | 121.7 (3) | C2—C1—Cl1 | 119.7 (3) |
| O1—C12—C13 | 118.0 (2) | C3—C1—Cl1 | 119.5 (2) |
| C11—C12—C13 | 120.2 (2) | O3—C7—C8 | 107.8 (2) |
| C2—C4—C6 | 117.9 (2) | O3—C7—H7A | 110.1 |
| C2—C4—C10 | 117.5 (2) | C8—C7—H7A | 110.1 |
| C6—C4—C10 | 124.6 (2) | O3—C7—H7B | 110.1 |
| C1—C2—C4 | 120.9 (3) | C8—C7—H7B | 110.1 |
| C1—C2—H2 | 119.6 | H7A—C7—H7B | 108.5 |
| C4—C2—H2 | 119.6 | C16—C14—C13 | 121.0 (3) |
| O3—C6—C5 | 122.6 (2) | C16—C14—H14 | 119.5 |
| O3—C6—C4 | 117.4 (2) | C13—C14—H14 | 119.5 |

| | | | |
|-----------------|------------|-----------------|------------|
| C5—C6—C4 | 120.0 (3) | C5—C3—C1 | 119.8 (3) |
| O2—C10—C11 | 120.1 (2) | C5—C3—H3 | 120.1 |
| O2—C10—C4 | 114.1 (2) | C1—C3—H3 | 120.1 |
| C11—C10—C4 | 125.9 (2) | C9—C8—C7 | 179.0 (4) |
| C10—C11—C12 | 122.4 (2) | C14—C16—C18 | 119.9 (3) |
| C10—C11—H11 | 118.8 | C14—C16—H16 | 120.1 |
| C12—C11—H11 | 118.8 | C18—C16—H16 | 120.1 |
| C17—C15—C13 | 121.4 (3) | F1—C19—F2 | 106.7 (4) |
| C17—C15—H15 | 119.3 | F1—C19—F3 | 105.4 (4) |
| C13—C15—H15 | 119.3 | F2—C19—F3 | 103.3 (3) |
| C17—C18—C16 | 120.0 (3) | F1—C19—C18 | 113.8 (3) |
| C17—C18—C19 | 120.2 (3) | F2—C19—C18 | 111.9 (3) |
| C16—C18—C19 | 119.8 (3) | F3—C19—C18 | 114.9 (3) |
| C3—C5—C6 | 120.6 (3) | C8—C9—H9 | 180.0 |
| C3—C5—H5 | 119.7 | | |
| | | | |
| C14—C13—C12—O1 | 10.5 (4) | C4—C6—C5—C3 | -0.2 (4) |
| C15—C13—C12—O1 | -170.5 (3) | C13—C15—C17—C18 | -0.4 (5) |
| C14—C13—C12—C11 | -167.0 (3) | C16—C18—C17—C15 | -0.2 (4) |
| C15—C13—C12—C11 | 12.0 (4) | C19—C18—C17—C15 | 178.4 (3) |
| C6—C4—C2—C1 | -0.9 (4) | C4—C2—C1—C3 | 0.3 (4) |
| C10—C4—C2—C1 | 178.5 (2) | C4—C2—C1—C11 | -179.5 (2) |
| C7—O3—C6—C5 | -1.2 (4) | C6—O3—C7—C8 | -178.7 (3) |
| C7—O3—C6—C4 | 179.3 (2) | C15—C13—C14—C16 | -0.6 (4) |
| C2—C4—C6—O3 | -179.7 (2) | C12—C13—C14—C16 | 178.4 (3) |
| C10—C4—C6—O3 | 1.0 (4) | C6—C5—C3—C1 | -0.4 (5) |
| C2—C4—C6—C5 | 0.8 (4) | C2—C1—C3—C5 | 0.4 (5) |
| C10—C4—C6—C5 | -178.5 (3) | C11—C1—C3—C5 | -179.9 (2) |
| C2—C4—C10—O2 | 2.7 (4) | O3—C7—C8—C9 | -158 (26) |
| C6—C4—C10—O2 | -178.0 (2) | C13—C14—C16—C18 | 0.1 (5) |
| C2—C4—C10—C11 | -177.9 (3) | C17—C18—C16—C14 | 0.3 (5) |
| C6—C4—C10—C11 | 1.3 (4) | C19—C18—C16—C14 | -178.2 (3) |
| O2—C10—C11—C12 | 1.0 (4) | C17—C18—C19—F1 | 130.3 (4) |
| C4—C10—C11—C12 | -178.2 (2) | C16—C18—C19—F1 | -51.1 (5) |
| O1—C12—C11—C10 | -2.2 (4) | C17—C18—C19—F2 | -108.6 (4) |
| C13—C12—C11—C10 | 175.2 (2) | C16—C18—C19—F2 | 69.9 (4) |
| C14—C13—C15—C17 | 0.7 (4) | C17—C18—C19—F3 | 8.7 (5) |
| C12—C13—C15—C17 | -178.3 (3) | C16—C18—C19—F3 | -172.7 (3) |
| O3—C6—C5—C3 | -179.7 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O2—H24 \cdots O1 | 0.82 | 1.79 | 2.529 (3) | 149 |
| C11—H11 \cdots O3 | 0.93 | 2.09 | 2.747 (3) | 126 |
| C7—H7B \cdots O1 ⁱ | 0.97 | 2.62 | 3.476 (4) | 147 |

Symmetry code: (i) *x*, *y*+1, *z*.