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# (±)-*syn*-Isopropyl 4-(1,1,1,3,3,3-hexafluoropropan-2-yloxy)-1-hydroxy-3-methyl-2-(prop-1-ynyl)cyclopent-2-enecarboxylate

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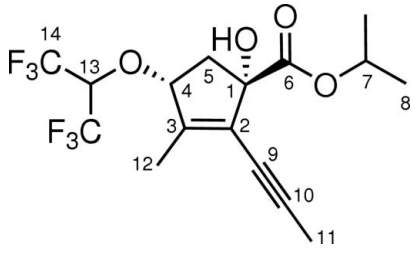
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.042; $wR$ factor = 0.106; data-to-parameter ratio = 13.2.

The title compound, $C_{16}H_{18}F_6O_4$, was obtained through an unprecedented one-pot reaction sequence involving a Gosteli–Claisen rearrangement and a cycloisomerization. The constitution and relative configuration were determined by single-crystal X-ray diffraction analysis. In the crystal, molecules are connected *via* O—H ··· O hydrogen bonds.

## Related literature

For the preparation, see: Neises & Steglich (1978); Hiersemann (2000). For details of the Gosteli–Claisen rearrangement, see: Gosteli (1972); Landor & Black (1965).




## Experimental

### Crystal data

$C_{16}H_{18}F_6O_4$
$M_r$ = 388.30
Triclinic, $P\overline{1}$
$a$ = 6.0166 (4) Å
$b$ = 11.9075 (6) Å
$c$ = 13.2798 (8) Å
$\alpha$ = 104.600 (5)°
$\beta$ = 91.775 (5)°
$\gamma$ = 96.955 (5)°
$V$ = 912.03 (10) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.14 mm$^{-1}$
$T$ = 173 K
0.42 × 0.20 × 0.18 mm

### Data collection

Oxford Diffraction Xcalibur S CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
$T_{min}$ = 0.944, $T_{max}$ = 0.975
7349 measured reflections
3175 independent reflections
2089 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
$S$ = 0.96
3175 reflections
240 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O3$^{i}$ | 0.84 | 2.10 | 2.8431 (17) | 148 |

Symmetry code: (i) $-x, -y, -z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2521).

## References

Gosteli, J. (1972). *Helv. Chim. Acta*, **55**, 451–460.
Hiersemann, M. (2000). *Synthesis*, pp. 1279–1290.
Landor, S. R. & Black, D. K. (1965). *J. Chem. Soc.* pp. 6784–6788.
Neises, B. & Steglich, W. (1978). *Angew. Chem.* **90**, 556–557.
Oxford Diffraction (2008). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o1660 [ **doi:10.1107/S1600536809023162** ]

# (±)-*syn*-Isopropyl 4-(1,1,1,3,3,3-hexafluoropropan-2-yloxy)-1-hydroxy-3-methyl-2-(prop-1-ynyl)cyclopent-2-enecarboxylate

**A. Gille, M. Schürmann, H. Preut and M. Hiersemann**

## Comment

For the preparation of the title compound (±)-(I) a propargyl vinyl ether (II) was synthesized from 2-butin-1-ol using an etherification with bromoacetic acid, an esterification under Steglich's conditions (Neises & Steglich, 1978) and an aldol condensation (Hiersemann, 2000). An unprecedented one-pot reaction sequence involving an uncatalyzed Gosteli-Claisen rearrangement (Gosteli, 1972) of the propargyl vinyl ether (II) (Landor & Black, 1965) and a cycloisomerization under incorporation of a solvent molecule provided (I) as a colourless solid. Fig. 1 depicts the constitution and relative configuration of the isolated diastereomer (I).

## Experimental

The title compound (I) was synthesized according to the following procedure. A solution of (II) (0.044 g, 0.2 mmol) in dry hexafluoroisopropanol (0.6 ml) was stirred at 333 K for 23 h. The solvent was removed under reduced pressure. Flash chromatography (isohexane/ethyl acetate 100/1 to 50/1 to 20/1) afforded (I) as a single diastereomer (0.026 g, 0.07 mmol, 34%) and 37% of the starting material (II). Single crystals of (I) were obtained by vapor diffusion recrystallization technique from isohexane and ethyl acetate to yield colorless cuboids: mp 385 K; $R_f$ 0.32 (cyclohexane/ethyl acetate 5/1); $^1$H NMR ($CDCl_3$, 400 MHz, δ): 1.23 (d, *J* = 6.3 Hz, 3H, 8-H), 1.26 (d, *J* = 6.3 Hz, 3H, 8-H), 1.91 (s, 3H, 12-H), 2.01 (s, 3H, 11-H), 2.03 (dd, *J* = 13.6, 5.9 Hz, 1H, 5-H), 2.86 (dd, *J* = 13.6, 7.0 Hz, 1H, 5-H), 3.76 (br.s, 1H, –OH), 4.23 (sep, *J* = 5.9 Hz, 1H, 13-H), 4.67 (t, *J* = 5.9 Hz, 1H, 4-H), 5.06 (sep, *J* = 6.3 Hz, 1H, 7-H); $^{19}$F NMR ($CDCl_3$, 282 MHz, δ): (-77.0)-(-76.7) (m, 6 F); $^{13}$C NMR ($CDCl_3$, 101 MHz, δ): 4.8 (11-$CH_3$), 13.3 (12-$CH_3$), 21.7 (8-$CH_3$), 21.8 (8-$CH_3$), 42.7 (5-$CH_2$), 70.6 (7-CH), 71.6 (9-C), 76.0 (sep, *J* = 32.6 Hz, 13-CH), 83.1 (1-C), 88.9 (4-CH), 94.5 (10-C), 121.2 (q, *J* = 283.0 Hz, 14-C), 121.8 (q, *J* = 283.0 Hz, 14-C), 126.5 (2-C), 146.9 (3-C), 173.6 (6-C); IR (cm$^{-1}$): 3485(br,s) (ν O—H, OH in H-bridges), 2990(m) 2940(m) 2920(m) ($\nu_{as,s}$ C—H, $CH_2$, $CH_3$, CH), 2230(w) (ν C≡C), 1715(s) (ν C=O, ester), 1635(m) (ν C=C), 1380(m) ($\delta_s$ C—H, $CH_3$), 1295(s) (ν C—O, ester), 1240(s) 1230(s) 1220(s) (ν C—F), 1185(s) 1120(s) 1105(s) (ν C—O, ether); Anal. Calcd. for $C_{16}H_{18}F_6O_4$: C,49.5; H, 4.7; Found: C, 49.1; H, 4.7; M = 388.30 g/mol.

## Figures

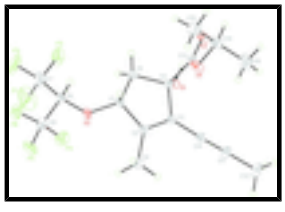


Fig. 1. The molecular structure of (I) with displacement ellipsoids shown at the 30% probability level.

## (±)-*syn*-Isopropyl 4-(1,1,1,3,3,3-hexafluoropropan-2-yloxy)-1-hydroxy- 3-methyl-2-(prop-1-ynyl)cyclopent-2-enecarboxylate

### *Crystal data*

| | |
|---|---|
| $C_{16}H_{18}F_6O_4$ | $Z = 2$ |
| $M_r = 388.30$ | $F_{000} = 400$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.414$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 6.0166$ (4) Å | Cell parameters from 3408 reflections |
| $b = 11.9075$ (6) Å | $\theta = 2.7$–$25.0^\circ$ |
| $c = 13.2798$ (8) Å | $\mu = 0.14$ mm$^{-1}$ |
| $\alpha = 104.600$ (5)° | $T = 173$ K |
| $\beta = 91.775$ (5)° | Block, colourless |
| $\gamma = 96.955$ (5)° | 0.42 × 0.20 × 0.18 mm |
| $V = 912.03$ (10) Å$^3$ | |

### *Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur S CCD diffractometer | 3175 independent reflections |
| Radiation source: Enhance (Mo) X-ray Source | 2089 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.026$ |
| Detector resolution: 16.0560 pixels mm$^{-1}$ | $\theta_{max} = 25.0^\circ$ |
| $T = 173$ K | $\theta_{min} = 2.7^\circ$ |
| ω scans | $h = -7 \rightarrow 7$ |
| Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008) | $k = -13 \rightarrow 14$ |
| $T_{min} = 0.944$, $T_{max} = 0.975$ | $l = -15 \rightarrow 11$ |
| 7349 measured reflections | |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | H-atom parameters constrained |
| $wR(F^2) = 0.106$ | $w = 1/[\sigma^2(F_o^2) + (0.0599P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.96$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 3175 reflections | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| 240 parameters | $\Delta\rho_{min} = -0.29$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. CrysAlis RED (Oxford Diffraction 2008) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 0.8004 (4) | 0.56391 (17) | 0.16252 (16) | 0.1130 (8) |
| F2 | 1.0918 (3) | 0.49063 (18) | 0.10772 (12) | 0.1036 (7) |
| F3 | 0.7665 (3) | 0.39187 (15) | 0.06173 (11) | 0.0842 (6) |
| F4 | 1.0556 (2) | 0.43622 (12) | 0.40071 (10) | 0.0537 (4) |
| F5 | 0.9672 (2) | 0.59099 (11) | 0.36511 (11) | 0.0631 (5) |
| F6 | 1.2566 (2) | 0.51846 (13) | 0.30212 (11) | 0.0608 (4) |
| O1 | 0.0648 (2) | 0.15814 (11) | 0.13523 (10) | 0.0286 (4) |
| H1 | 0.0264 | 0.1079 | 0.0786 | 0.043* |
| O2 | 0.6874 (2) | 0.38842 (11) | 0.26452 (11) | 0.0310 (4) |
| O3 | 0.1934 (2) | −0.05252 (12) | 0.05543 (11) | 0.0406 (4) |
| O4 | 0.4173 (2) | −0.03491 (12) | 0.19701 (11) | 0.0391 (4) |
| C1 | 0.6115 (3) | 0.26537 (16) | 0.25431 (15) | 0.0243 (5) |
| H1A | 0.7429 | 0.2231 | 0.2609 | 0.029* |
| C2 | 0.4539 (3) | 0.25339 (16) | 0.33674 (15) | 0.0243 (5) |
| C3 | 0.2714 (3) | 0.17686 (16) | 0.29615 (15) | 0.0231 (4) |
| C4 | 0.2677 (3) | 0.13463 (16) | 0.17825 (14) | 0.0227 (4) |
| C5 | 0.4762 (3) | 0.20733 (17) | 0.15116 (15) | 0.0293 (5) |
| H5A | 0.5661 | 0.1562 | 0.1028 | 0.035* |
| H5B | 0.4311 | 0.2673 | 0.1177 | 0.035* |
| C6 | 0.9030 (3) | 0.41341 (17) | 0.23280 (16) | 0.0299 (5) |
| H6 | 0.9684 | 0.3384 | 0.2098 | 0.036* |
| C7 | 0.8883 (5) | 0.4666 (2) | 0.1413 (2) | 0.0605 (8) |
| C8 | 1.0461 (4) | 0.4918 (2) | 0.32484 (18) | 0.0397 (6) |
| C9 | 0.5107 (3) | 0.31553 (18) | 0.44862 (15) | 0.0367 (6) |
| H9A | 0.3943 | 0.2902 | 0.4912 | 0.055* |
| H9B | 0.5193 | 0.4002 | 0.4574 | 0.055* |
| H9C | 0.6557 | 0.2970 | 0.4708 | 0.055* |
| C10 | 0.0936 (3) | 0.13487 (17) | 0.35228 (15) | 0.0256 (5) |
| C11 | −0.0581 (3) | 0.09773 (18) | 0.39548 (16) | 0.0318 (5) |
| C12 | −0.2433 (4) | 0.0527 (2) | 0.44912 (19) | 0.0486 (6) |

| | | | | |
|---|---|---|---|---|
| H12A | −0.2256 | −0.0270 | 0.4525 | 0.073* |
| H12B | −0.3858 | 0.0518 | 0.4109 | 0.073* |
| H12C | −0.2428 | 0.1032 | 0.5200 | 0.073* |
| C13 | 0.2847 (3) | 0.00474 (17) | 0.13700 (15) | 0.0237 (5) |
| C14 | 0.4642 (4) | −0.15683 (18) | 0.16071 (17) | 0.0392 (6) |
| H14 | 0.3778 | −0.1949 | 0.0926 | 0.047* |
| C15 | 0.7079 (4) | −0.1520 (2) | 0.1452 (2) | 0.0602 (8) |
| H15A | 0.7475 | −0.1045 | 0.0963 | 0.090* |
| H15B | 0.7441 | −0.2315 | 0.1168 | 0.090* |
| H15C | 0.7929 | −0.1168 | 0.2121 | 0.090* |
| C16 | 0.3840 (5) | −0.2186 (2) | 0.2411 (2) | 0.0710 (9) |
| H16A | 0.4691 | −0.1819 | 0.3078 | 0.107* |
| H16B | 0.4064 | −0.3011 | 0.2184 | 0.107* |
| H16C | 0.2241 | −0.2131 | 0.2495 | 0.107* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.179 (2) | 0.0561 (12) | 0.1164 (16) | 0.0058 (12) | −0.0384 (15) | 0.0547 (11) |
| F2 | 0.1085 (14) | 0.1364 (17) | 0.0573 (11) | −0.0682 (13) | 0.0018 (10) | 0.0494 (11) |
| F3 | 0.1084 (12) | 0.0924 (13) | 0.0418 (9) | −0.0499 (10) | −0.0191 (9) | 0.0316 (8) |
| F4 | 0.0516 (8) | 0.0668 (10) | 0.0425 (8) | −0.0019 (7) | −0.0024 (6) | 0.0191 (7) |
| F5 | 0.0721 (9) | 0.0333 (8) | 0.0679 (10) | 0.0027 (7) | 0.0029 (8) | −0.0145 (7) |
| F6 | 0.0357 (8) | 0.0757 (10) | 0.0675 (10) | −0.0151 (7) | 0.0060 (7) | 0.0220 (8) |
| O1 | 0.0311 (7) | 0.0296 (8) | 0.0227 (8) | 0.0091 (6) | −0.0052 (6) | 0.0008 (6) |
| O2 | 0.0303 (8) | 0.0201 (7) | 0.0417 (9) | 0.0021 (6) | 0.0045 (6) | 0.0069 (6) |
| O3 | 0.0513 (9) | 0.0306 (8) | 0.0333 (9) | 0.0110 (7) | −0.0156 (8) | −0.0043 (7) |
| O4 | 0.0503 (9) | 0.0288 (8) | 0.0346 (9) | 0.0142 (7) | −0.0144 (7) | −0.0006 (7) |
| C1 | 0.0238 (10) | 0.0183 (10) | 0.0301 (11) | 0.0032 (8) | 0.0018 (9) | 0.0050 (8) |
| C2 | 0.0275 (10) | 0.0239 (10) | 0.0219 (11) | 0.0061 (8) | 0.0003 (9) | 0.0058 (8) |
| C3 | 0.0251 (10) | 0.0231 (10) | 0.0214 (10) | 0.0055 (8) | 0.0020 (8) | 0.0051 (8) |
| C4 | 0.0215 (10) | 0.0265 (10) | 0.0198 (10) | 0.0046 (8) | 0.0000 (8) | 0.0050 (8) |
| C5 | 0.0327 (11) | 0.0312 (11) | 0.0228 (11) | −0.0001 (9) | 0.0052 (9) | 0.0065 (9) |
| C6 | 0.0355 (11) | 0.0199 (10) | 0.0337 (12) | −0.0011 (8) | 0.0092 (10) | 0.0073 (9) |
| C7 | 0.0795 (19) | 0.0498 (18) | 0.0488 (17) | −0.0232 (15) | −0.0063 (15) | 0.0228 (14) |
| C8 | 0.0365 (13) | 0.0399 (14) | 0.0404 (14) | −0.0015 (10) | 0.0045 (11) | 0.0089 (11) |
| C9 | 0.0404 (12) | 0.0375 (13) | 0.0276 (12) | 0.0012 (10) | −0.0024 (10) | 0.0022 (10) |
| C10 | 0.0291 (11) | 0.0266 (11) | 0.0219 (11) | 0.0053 (9) | −0.0001 (9) | 0.0070 (9) |
| C11 | 0.0309 (12) | 0.0387 (13) | 0.0294 (12) | 0.0042 (10) | 0.0011 (10) | 0.0157 (10) |
| C12 | 0.0402 (13) | 0.0657 (17) | 0.0502 (16) | 0.0040 (12) | 0.0107 (12) | 0.0342 (13) |
| C13 | 0.0183 (9) | 0.0294 (11) | 0.0221 (11) | 0.0012 (8) | 0.0025 (9) | 0.0052 (9) |
| C14 | 0.0536 (14) | 0.0256 (12) | 0.0372 (13) | 0.0147 (10) | −0.0072 (11) | 0.0026 (10) |
| C15 | 0.0656 (17) | 0.0441 (15) | 0.078 (2) | 0.0211 (13) | 0.0294 (15) | 0.0188 (14) |
| C16 | 0.079 (2) | 0.0405 (15) | 0.100 (2) | 0.0080 (14) | 0.0324 (18) | 0.0276 (16) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| F1—C7 | 1.302 (3) | C5—H5A | 0.9900 |
| F2—C7 | 1.339 (3) | C5—H5B | 0.9900 |

| | | | |
|---|---|---|---|
| F3—C7 | 1.329 (3) | C6—C8 | 1.507 (3) |
| F4—C8 | 1.341 (3) | C6—C7 | 1.510 (3) |
| F5—C8 | 1.319 (2) | C6—H6 | 1.0000 |
| F6—C8 | 1.330 (2) | C9—H9A | 0.9800 |
| O1—C4 | 1.419 (2) | C9—H9B | 0.9800 |
| O1—H1 | 0.8400 | C9—H9C | 0.9800 |
| O2—C6 | 1.397 (2) | C10—C11 | 1.192 (3) |
| O2—C1 | 1.452 (2) | C11—C12 | 1.461 (3) |
| O3—C13 | 1.200 (2) | C12—H12A | 0.9800 |
| O4—C13 | 1.318 (2) | C12—H12B | 0.9800 |
| O4—C14 | 1.474 (2) | C12—H12C | 0.9800 |
| C1—C2 | 1.492 (3) | C14—C15 | 1.483 (3) |
| C1—C5 | 1.528 (3) | C14—C16 | 1.502 (4) |
| C1—H1A | 1.0000 | C14—H14 | 1.0000 |
| C2—C3 | 1.342 (2) | C15—H15A | 0.9800 |
| C2—C9 | 1.492 (3) | C15—H15B | 0.9800 |
| C3—C10 | 1.434 (3) | C15—H15C | 0.9800 |
| C3—C4 | 1.517 (3) | C16—H16A | 0.9800 |
| C4—C13 | 1.522 (3) | C16—H16B | 0.9800 |
| C4—C5 | 1.541 (3) | C16—H16C | 0.9800 |
| C4—O1—H1 | 109.5 | F5—C8—F4 | 106.86 (19) |
| C6—O2—C1 | 115.42 (14) | F6—C8—F4 | 106.70 (18) |
| C13—O4—C14 | 118.33 (14) | F5—C8—C6 | 113.48 (18) |
| O2—C1—C2 | 109.65 (14) | F6—C8—C6 | 112.73 (19) |
| O2—C1—C5 | 112.11 (17) | F4—C8—C6 | 109.13 (18) |
| C2—C1—C5 | 105.10 (14) | C2—C9—H9A | 109.5 |
| O2—C1—H1A | 110.0 | C2—C9—H9B | 109.5 |
| C2—C1—H1A | 110.0 | H9A—C9—H9B | 109.5 |
| C5—C1—H1A | 110.0 | C2—C9—H9C | 109.5 |
| C3—C2—C9 | 127.42 (19) | H9A—C9—H9C | 109.5 |
| C3—C2—C1 | 110.79 (16) | H9B—C9—H9C | 109.5 |
| C9—C2—C1 | 121.69 (16) | C11—C10—C3 | 177.6 (2) |
| C2—C3—C10 | 126.87 (18) | C10—C11—C12 | 179.6 (2) |
| C2—C3—C4 | 112.41 (17) | C11—C12—H12A | 109.5 |
| C10—C3—C4 | 120.72 (15) | C11—C12—H12B | 109.5 |
| O1—C4—C3 | 108.58 (14) | H12A—C12—H12B | 109.5 |
| O1—C4—C13 | 108.35 (13) | C11—C12—H12C | 109.5 |
| C3—C4—C13 | 114.56 (17) | H12A—C12—H12C | 109.5 |
| O1—C4—C5 | 112.55 (16) | H12B—C12—H12C | 109.5 |
| C3—C4—C5 | 103.23 (14) | O3—C13—O4 | 124.56 (18) |
| C13—C4—C5 | 109.61 (15) | O3—C13—C4 | 122.24 (17) |
| C1—C5—C4 | 106.17 (16) | O4—C13—C4 | 113.11 (15) |
| C1—C5—H5A | 110.5 | O4—C14—C15 | 107.02 (18) |
| C4—C5—H5A | 110.5 | O4—C14—C16 | 106.87 (18) |
| C1—C5—H5B | 110.5 | C15—C14—C16 | 115.0 (2) |
| C4—C5—H5B | 110.5 | O4—C14—H14 | 109.3 |
| H5A—C5—H5B | 108.7 | C15—C14—H14 | 109.3 |
| O2—C6—C8 | 108.60 (17) | C16—C14—H14 | 109.3 |
| O2—C6—C7 | 109.24 (18) | C14—C15—H15A | 109.5 |

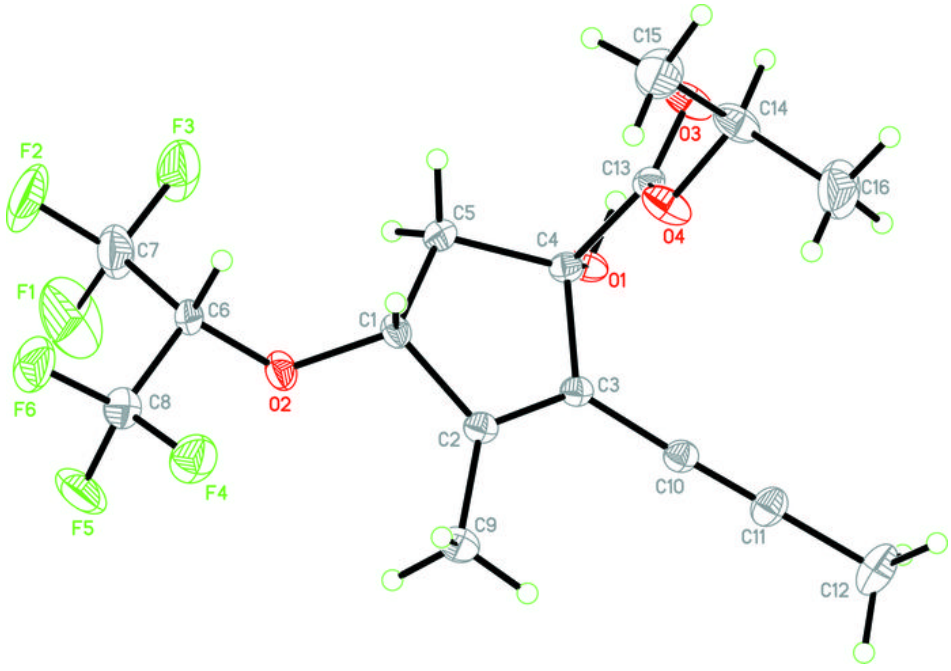
| | | | |
|---|---|---|---|
| C8—C6—C7 | 113.26 (19) | C14—C15—H15B | 109.5 |
| O2—C6—H6 | 108.5 | H15A—C15—H15B | 109.5 |
| C8—C6—H6 | 108.5 | C14—C15—H15C | 109.5 |
| C7—C6—H6 | 108.5 | H15A—C15—H15C | 109.5 |
| F1—C7—F3 | 108.0 (2) | H15B—C15—H15C | 109.5 |
| F1—C7—F2 | 106.8 (2) | C14—C16—H16A | 109.5 |
| F3—C7—F2 | 106.9 (2) | C14—C16—H16B | 109.5 |
| F1—C7—C6 | 113.5 (2) | H16A—C16—H16B | 109.5 |
| F3—C7—C6 | 110.3 (2) | C14—C16—H16C | 109.5 |
| F2—C7—C6 | 111.1 (2) | H16A—C16—H16C | 109.5 |
| F5—C8—F6 | 107.57 (18) | H16B—C16—H16C | 109.5 |
| C6—O2—C1—C2 | 149.56 (16) | O2—C6—C7—F1 | 60.4 (2) |
| C6—O2—C1—C5 | −94.11 (18) | C8—C6—C7—F1 | −60.8 (3) |
| O2—C1—C2—C3 | 133.92 (16) | O2—C6—C7—F3 | −60.9 (3) |
| C5—C1—C2—C3 | 13.2 (2) | C8—C6—C7—F3 | 177.9 (2) |
| O2—C1—C2—C9 | −49.5 (2) | O2—C6—C7—F2 | −179.27 (18) |
| C5—C1—C2—C9 | −170.23 (17) | C8—C6—C7—F2 | 59.6 (3) |
| C9—C2—C3—C10 | −2.3 (3) | O2—C6—C8—F5 | −58.6 (2) |
| C1—C2—C3—C10 | 173.99 (18) | C7—C6—C8—F5 | 63.0 (3) |
| C9—C2—C3—C4 | 178.06 (18) | O2—C6—C8—F6 | 178.82 (17) |
| C1—C2—C3—C4 | −5.6 (2) | C7—C6—C8—F6 | −59.6 (3) |
| C2—C3—C4—O1 | −123.92 (17) | O2—C6—C8—F4 | 60.5 (2) |
| C10—C3—C4—O1 | 56.4 (2) | C7—C6—C8—F4 | −178.0 (2) |
| C2—C3—C4—C13 | 114.83 (18) | C14—O4—C13—O3 | 2.1 (3) |
| C10—C3—C4—C13 | −64.8 (2) | C14—O4—C13—C4 | −174.52 (16) |
| C2—C3—C4—C5 | −4.3 (2) | O1—C4—C13—O3 | 25.9 (2) |
| C10—C3—C4—C5 | 176.06 (16) | C3—C4—C13—O3 | 147.25 (18) |
| O2—C1—C5—C4 | −134.32 (16) | C5—C4—C13—O3 | −97.3 (2) |
| C2—C1—C5—C4 | −15.27 (19) | O1—C4—C13—O4 | −157.37 (16) |
| O1—C4—C5—C1 | 128.89 (16) | C3—C4—C13—O4 | −36.0 (2) |
| C3—C4—C5—C1 | 12.02 (18) | C5—C4—C13—O4 | 79.5 (2) |
| C13—C4—C5—C1 | −110.46 (17) | C13—O4—C14—C15 | 115.9 (2) |
| C1—O2—C6—C8 | −119.38 (18) | C13—O4—C14—C16 | −120.4 (2) |
| C1—O2—C6—C7 | 116.65 (19) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O3$^{i}$ | 0.84 | 2.10 | 2.8431 (17) | 148 |

Symmetry codes: (i) −*x*, −*y*, −*z*.

**Fig. 1**


C15
C14
O3
C13
C16
F3
F2
C5
C4
O4
C7
O1
F1
C6
C1
C3
O2
F6
C8
C2
C10
F4
C9
C11
F5
C12