organic compounds

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

Annika Gille, Markus Schürmann, Hans Preut* and Martin Hiersemann

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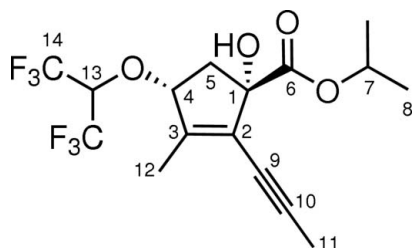
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The title compound, $C_{16}H_{18}F_6O_4$, was obtained through an unprecedented one-pot reaction sequence involving a Gosteli–Claisen rearrangement and a cycloisomerization. The constitution and relative configuration were determined by single-crystal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

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

Data collection

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

Refinement

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$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

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Hiersemann, M. (2000). *Synthesis*, pp. 1279–1290.
Landor, S. R. & Black, D. K. (1965). *J. Chem. Soc.* pp. 6784–6788.
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supporting information

(±)-*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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S1. 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).

S2. 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) (v O—H, OH in H-bridges), 2990(m) 2940(m) 2920(m) ($v_{as,s}$ C—H, CH$_2$, CH$_3$, CH), 2230(w) (v C≡C), 1715(s) (v C=O, ester), 1635(m) (v C=C), 1380(m) (δ_s C—H, CH$_3$), 1295(s) (v C—O, ester), 1240(s) 1230(s) 1220(s) (v C—F), 1185(s) 1120(s) 1105(s) (v C—O, ether); Anal. Calcd. for C$_{16}$H$_{18}$F$_6$O$_4$: C,49.5; H, 4.7; Found: C, 49.1; H, 4.7; M = 388.30 g/mol.
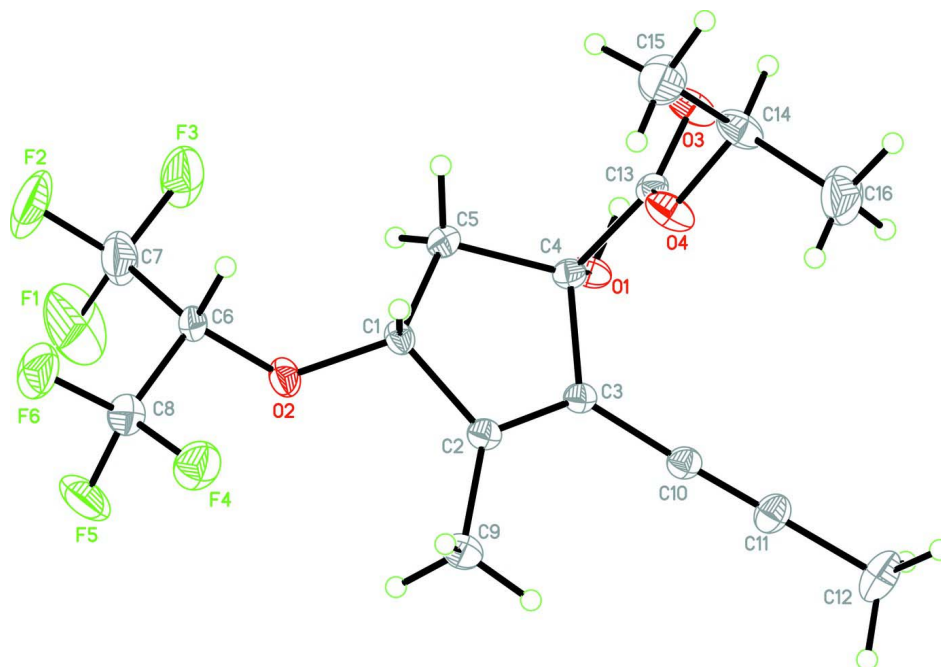
Figure 1

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

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Crystal data

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$M_r = 388.30$	$F(000) = 400$
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	$\theta = 2.7–25.0°$
$c = 13.2798$ (8) Å	$\mu = 0.14$ mm^{-1}
$\alpha = 104.600$ (5)°	$T = 173$ K
$\beta = 91.775$ (5)°	Block, colourless
$\gamma = 96.955$ (5)°	$0.42 \times 0.20 \times 0.18$ mm
$V = 912.03$ (10) Å^3	

Data collection

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

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.042$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.106$	
$S = 0.96$	H-atom parameters constrained
3175 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2]$
240 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.27$ e Å^{-3}
	$\Delta\rho_{min} = -0.29$ e Å^{-3}

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, º)

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, º)

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*.