IUCrData

ISSN 2414-3146

Ethyl 5-[(ethoxycarbonyl)oxy]-5,5-diphenylpent-2-ynoate

Jorge A Cabezas* and Cristian Saul Campos Fernandez

Escuela de Quimica, Universidad de Costa Rica, 2060, San Pedro, San Jose, Costa Rica. *Correspondence e-mail: jorge.cabezas@ucr.ac.cr

Received 4 October 2019
Accepted 12 November 2019

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

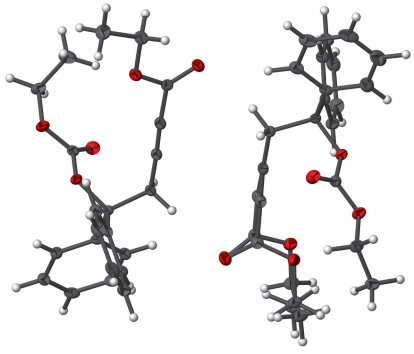
Keywords: crystal structure; homopropargyl alcohols; 1,3-dilithiopropyne; propargylation; alkynes.

CCDC reference: 1901024

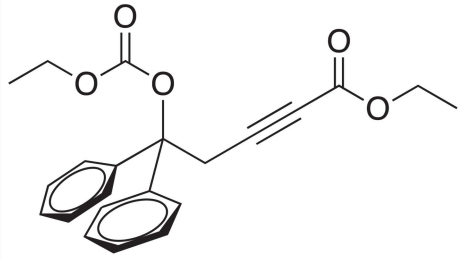
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{22}H_{22}O_5$, crystallizes with two molecules in the asymmetric unit, one of which shows disorder of its ethyl acetate group over two sets of sites in a 0.880 (2):0.120 (2) ratio. The C≡C distances in the two molecules are almost the same [1.1939 (16) and 1.199 (2) Å], but the Csp^3—C≡C angles differ somewhat [175.92 (12) and 172.53 (16)°]. In the crystal, several weak C—H···O interactions are seen.

3D view



Chemical scheme



Structure description

Highly functionalized homopropagyl alcohols are useful building blocks in organic synthesis (Kim *et al.*, 2017; Foley & Leighton, 2015; Francais *et al.*, 2010; Hosseyni *et al.*, 2016; Gao *et al.*, 2014; Trost & Rhee 2003; Yadav & Maiti, 2002). For instance, the title compound, where the homopropargyl alcohol is masked as a carbonate, might serve as an intermediate in the synthesis of α, β-unsaturated γ-lactones The aim of this work is to report the synthesis and crystal structure of the title compound.

The title compound crystallizes with two molecules in the asymmetric unit, as shown in Fig. 1. The bond distances for the *sp* carbon-atom triple bonds are almost the same [1.1939 (16) for C15≡C16 and 1.199 (2) Å for C34≡C35] but a greater difference is present between the bond angles of C14—C15≡C16 [175.92 (12)] and C33—C34≡C35 [172.53 (16)°]. There is disorder in the ethyl ester grouping of the C20 molecule and atoms C35, C36, O6 and C37 and their attached H atoms (if any) were modelled with two sets of sites in a 0.880 (2):0.120 (2) ratio. In the crystal, several weak C—H···O interactions occur (Table 1, Fig. 2).

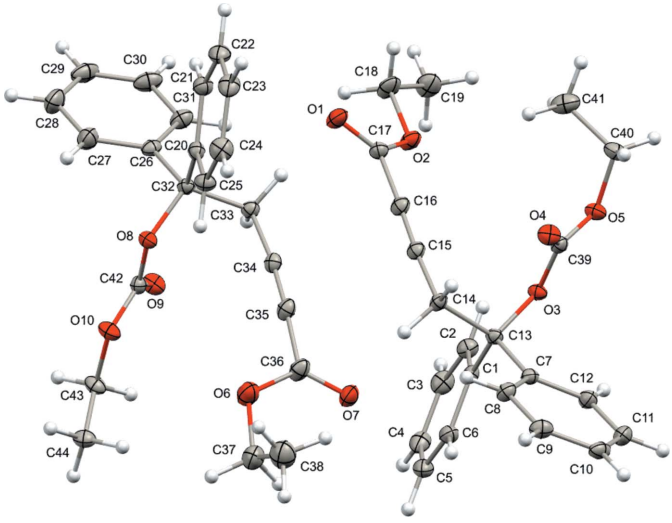


Figure 1
The title molecule with 50% probability ellipsoids. The minor disorder component for the C20 molecule is not shown.

Synthesis and crystallization

The title compound, **1**, was synthesized in a one-pot reaction by the treatment of propargyl bromide, **2**, with *n*-butyllithium in the presence of TMEDA at −78°C to generate the equivalent of 1,3-dilithiopropyne, **3**, followed by addition of benzophenone, **4**; and after stirring overnight, the reaction mixture was treated with ethyl chloroformate, **5**, according to the literature procedure (Cabezas *et al.*, 2001) (Fig. 3). The product was purified by column chromatography using hexane-ether mixtures, and then recrystallized from ethyl acetate solution.

Refinement

Crystal data, data collection and structure refinement are summarized in Table 2.

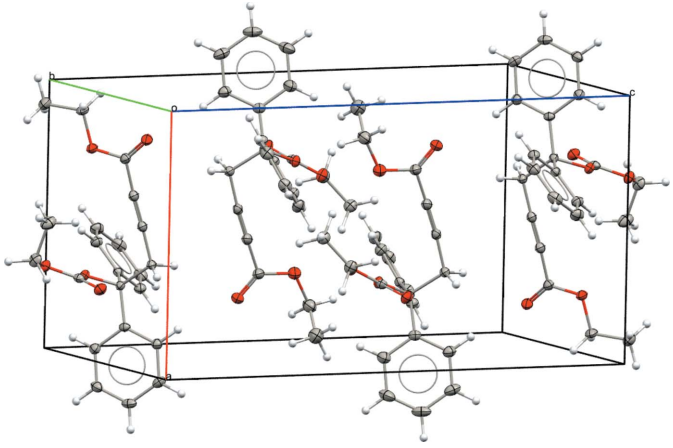


Figure 2
The crystal packing of the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O7	0.95	2.56	3.4079 (14)	149
C14—H14A···O7	0.99	2.61	3.4398 (14)	142
C18—H18A···O1^{i}	0.99	2.62	3.4119 (15)	137
C31—H31···O1	0.95	2.43	3.3617 (15)	169
C33—H33B···O1	0.99	2.61	3.3008 (13)	127

Symmetry code: (i) $-x+2, -y+1, -z+2$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{22}O_5$
M_r	366.39
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	9.9461 (4), 12.1510 (5), 16.8171 (7)
α, β, γ (°)	105.006 (1), 94.220 (1), 104.926 (1)
V (Å^3)	1875.34 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.22 × 0.17 × 0.13
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.721, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	54801, 8599, 7059
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.093, 1.02
No. of reflections	8599
No. of parameters	532
No. of restraints	186
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.25

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *shelXle* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

Acknowledgements

We thank Dr Vojtech Jancik for crystallographic assistance.

Funding information

We acknowledge the Vicerrectoría de Investigación (UCR) for financial support

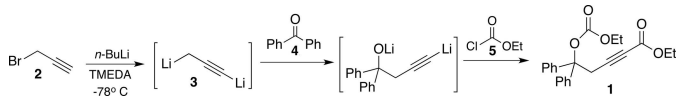


Figure 3
A synthetic scheme for the preparation of the title compound.

References

Bruker (2015). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cabezas, J. A., Pereira, A. & Amey, A. (2001). *Tetrahedron Lett.* **42**, 6819–6822.

Foley, C. N. & Leighton, J. L. (2015). *Org. Lett.* **17**, 5858–5861.

Francais, A., Leyva, A., Etxebarria-Jardi, G. & Ley, S. V. (2010). *Org. Lett.* **12**, 340–343.

Gao, P., Li, H. X., Hao, X. H., Jin, D. P., Chen, D. Q., Yan, X. B., Wu, X. X., Song, X. R., Liu, X. Y. & Liang, Y. M. (2014). *Org. Lett.* **16**, 6298–6301.

Hosseyni, S., Wojtas, L., Li, M. & Shi, X. (2016). *J. Am. Chem. Soc.* **138**, 3994–3997.

Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.

Kim, J., Jeong, W. & Rhee, Y. H. (2017). *Org. Lett.* **19**, 242–245.

Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Trost, B. M. & Rhee, Y. H. (2003). *J. Am. Chem. Soc.* **125**, 7482–7483.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Yadav, J. S. & Maiti, A. (2002). *Tetrahedron*, **58**, 4955–4961.

full crystallographic data

IUCrData (2019). **4**, x191526 [https://doi.org/10.1107/S2414314619015268]

Ethyl 5-[(ethoxycarbonyl)oxy]-5,5-diphenylpent-2-ynoate

Jorge A Cabezas and Cristian Saul Campos Fernandez

Ethyl 5-[(ethoxycarbonyl)oxy]-5,5-diphenylpent-2-ynoate

Crystal data

$C_{22}H_{22}O_5$
M_r = 366.39
Triclinic, $P\overline{1}$
a = 9.9461 (4) Å
b = 12.1510 (5) Å
c = 16.8171 (7) Å
α = 105.006 (1)°
β = 94.220 (1)°
γ = 104.926 (1)°
V = 1875.34 (13) Å^3
Z = 4
$F(000)$ = 776
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9767 reflections
θ = 2.8–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Block, light yellow
0.22 × 0.17 × 0.13 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: Incoatec Microsource
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.721, T_{max} = 0.746
54801 measured reflections
8599 independent reflections
7059 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −12→12
k = −15→15
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.093
S = 1.02
8599 reflections
532 parameters
186 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.5843P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

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

Refinement. All H atoms were located initially by difference Fourier synthesis and then relocated to idealized locations for refinement as riding atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.81699 (8)	0.40080 (8)	0.89228 (5)	0.02350 (19)	
C1	0.27160 (11)	−0.01485 (10)	0.83753 (7)	0.0148 (2)	
O2	0.77716 (8)	0.29149 (7)	0.98210 (5)	0.01976 (18)	
C2	0.38374 (12)	−0.03498 (10)	0.88071 (7)	0.0189 (2)	
H2	0.436404	0.023699	0.929750	0.023*	
O3	0.28485 (8)	0.14591 (7)	0.95391 (5)	0.01491 (16)	
C3	0.41879 (13)	−0.14048 (11)	0.85231 (8)	0.0231 (3)	
H3	0.494787	−0.153900	0.882312	0.028*	
O4	0.25585 (8)	0.32945 (7)	0.96811 (5)	0.02070 (18)	
C4	0.34348 (13)	−0.22619 (10)	0.78046 (8)	0.0234 (3)	
H4	0.368287	−0.297905	0.760986	0.028*	
O5	0.34124 (8)	0.27211 (7)	1.07449 (5)	0.01890 (17)	
C5	0.23196 (13)	−0.20709 (10)	0.73711 (8)	0.0222 (2)	
H5	0.180079	−0.265766	0.687853	0.027*	
C6	0.19596 (12)	−0.10203 (10)	0.76568 (7)	0.0184 (2)	
H6	0.118989	−0.089543	0.735906	0.022*	
C7	0.07917 (11)	0.08796 (9)	0.84860 (7)	0.0144 (2)	
C8	0.01672 (12)	0.12322 (10)	0.78644 (7)	0.0178 (2)	
H8	0.072736	0.159065	0.751442	0.021*	
O8	0.70969 (8)	0.34032 (7)	0.55046 (5)	0.01559 (16)	
C9	−0.12827 (12)	0.10579 (10)	0.77571 (7)	0.0202 (2)	
H9	−0.170739	0.129795	0.733164	0.024*	
O9	0.74620 (9)	0.16970 (7)	0.56704 (5)	0.02059 (18)	
C11	−0.14925 (12)	0.01679 (11)	0.88751 (7)	0.0210 (2)	
H11	−0.205526	−0.019704	0.922125	0.025*	
C10	−0.21110 (12)	0.05387 (10)	0.82637 (7)	0.0204 (2)	
H10	−0.309631	0.043666	0.819345	0.024*	
O10	0.63254 (9)	0.18044 (7)	0.44917 (5)	0.01925 (17)	
C13	0.23688 (11)	0.10354 (9)	0.86421 (6)	0.0138 (2)	
C12	−0.00532 (12)	0.03314 (10)	0.89790 (7)	0.0184 (2)	
H12	0.036339	0.006570	0.939264	0.022*	
C14	0.32186 (11)	0.18946 (10)	0.81982 (7)	0.0153 (2)	
H14A	0.310036	0.147821	0.759660	0.018*	
H14B	0.283466	0.258206	0.825833	0.018*	
C16	0.59229 (12)	0.27350 (10)	0.88425 (7)	0.0174 (2)	
C15	0.47165 (11)	0.23273 (10)	0.85321 (7)	0.0160 (2)	
C17	0.73983 (11)	0.32921 (10)	0.91892 (7)	0.0167 (2)	
C18	0.92621 (12)	0.33846 (12)	1.01716 (8)	0.0260 (3)	
H18A	0.952370	0.426111	1.039938	0.031*	
H18B	0.984914	0.318049	0.973701	0.031*	
C19	0.94828 (14)	0.28291 (13)	1.08508 (8)	0.0295 (3)	
H19A	0.917726	0.196104	1.062224	0.044*	

H19B	0.893208	0.307004	1.128952	0.044*	
H19C	1.048331	0.309309	1.108453	0.044*	
C20	0.73795 (11)	0.52715 (10)	0.64707 (7)	0.0153 (2)	
C21	0.82971 (12)	0.62948 (10)	0.70272 (7)	0.0190 (2)	
H21	0.915297	0.625844	0.729476	0.023*	
C22	0.79681 (13)	0.73670 (10)	0.71929 (8)	0.0228 (3)	
H22	0.859448	0.805802	0.757828	0.027*	
C23	0.67345 (13)	0.74343 (11)	0.68001 (8)	0.0230 (3)	
H23	0.650630	0.816698	0.692060	0.028*	
C24	0.58307 (13)	0.64285 (11)	0.62293 (8)	0.0232 (3)	
H24	0.499279	0.647760	0.594908	0.028*	
C25	0.61448 (12)	0.53500 (10)	0.60654 (7)	0.0190 (2)	
H25	0.551812	0.466300	0.567642	0.023*	
C26	0.92255 (11)	0.41887 (9)	0.65338 (7)	0.0163 (2)	
C27	1.00433 (13)	0.44168 (12)	0.59285 (8)	0.0259 (3)	
H27	0.961109	0.445615	0.541776	0.031*	
C28	1.14856 (14)	0.45879 (12)	0.60613 (9)	0.0319 (3)	
H28	1.203468	0.474016	0.564177	0.038*	
C29	1.21224 (13)	0.45368 (11)	0.68038 (9)	0.0291 (3)	
H29	1.310988	0.465572	0.689682	0.035*	
C30	1.13185 (13)	0.43123 (11)	0.74099 (9)	0.0267 (3)	
H30	1.175639	0.427590	0.792012	0.032*	
C31	0.98712 (12)	0.41386 (10)	0.72797 (8)	0.0217 (2)	
H31	0.932545	0.398593	0.770051	0.026*	
C32	0.76597 (11)	0.40682 (9)	0.63736 (7)	0.0141 (2)	
C33	0.68114 (11)	0.34551 (10)	0.69605 (7)	0.0150 (2)	
H33A	0.719010	0.280635	0.703897	0.018*	
H33B	0.692982	0.404137	0.751197	0.018*	
C34	0.53171 (12)	0.29679 (10)	0.66250 (7)	0.0158 (2)	
O7	0.17896 (8)	0.13603 (8)	0.61612 (5)	0.02321 (19)	
O6	0.26944 (9)	0.16917 (9)	0.50205 (6)	0.0206 (2)	0.880 (2)
C35	0.4135 (2)	0.2486 (3)	0.62745 (19)	0.0171 (5)	0.880 (2)
C36	0.27374 (17)	0.17945 (14)	0.58293 (10)	0.0169 (4)	0.880 (2)
C37	0.13761 (18)	0.09582 (16)	0.44892 (11)	0.0230 (4)	0.880 (2)
H37A	0.099484	0.025072	0.468075	0.028*	0.880 (2)
H37B	0.155490	0.067932	0.391025	0.028*	0.880 (2)
C38	0.03124 (16)	0.16443 (14)	0.45078 (9)	0.0276 (3)	0.880 (2)
H38A	0.008023	0.186784	0.507262	0.041*	0.880 (2)
H38B	−0.054173	0.114859	0.412056	0.041*	0.880 (2)
H38C	0.070539	0.236371	0.434222	0.041*	0.880 (2)
O6A	0.2498 (7)	0.2460 (7)	0.5284 (4)	0.0205 (12)	0.120 (2)
C35A	0.4113 (15)	0.266 (3)	0.6382 (17)	0.0171 (5)	0.120 (2)
C36A	0.2675 (13)	0.2105 (12)	0.5967 (8)	0.0185 (14)	0.120 (2)
C37A	0.1103 (11)	0.1935 (9)	0.4795 (6)	0.0259 (15)	0.120 (2)
H37C	0.091324	0.245720	0.446239	0.031*	0.120 (2)
H37D	0.038483	0.185552	0.517038	0.031*	0.120 (2)
C38A	0.1017 (16)	0.0761 (13)	0.4239 (9)	0.031 (3)	0.120 (2)
H38D	0.176341	0.083822	0.389005	0.046*	0.120 (2)

H38E	0.009810	0.043031	0.388383	0.046*	0.120 (2)
H38F	0.113397	0.022896	0.457184	0.046*	0.120 (2)
C39	0.29032 (11)	0.25774 (10)	0.99560 (7)	0.0157 (2)	
C40	0.38139 (13)	0.39366 (10)	1.12861 (7)	0.0221 (2)	
H40A	0.378894	0.393053	1.187235	0.026*	
H40B	0.313650	0.435068	1.114143	0.026*	
C41	0.52676 (13)	0.45870 (11)	1.11907 (9)	0.0285 (3)	
H41A	0.526216	0.467934	1.062873	0.043*	
H41B	0.592009	0.413303	1.127947	0.043*	
H41C	0.556999	0.537323	1.160174	0.043*	
C42	0.70128 (11)	0.22392 (10)	0.52654 (7)	0.0156 (2)	
C43	0.61129 (12)	0.05383 (10)	0.41182 (7)	0.0196 (2)	
H43A	0.587446	0.009968	0.453347	0.024*	
H43B	0.697986	0.039704	0.391396	0.024*	
C44	0.49251 (12)	0.01266 (10)	0.34075 (7)	0.0209 (2)	
H44A	0.516001	0.058708	0.301126	0.031*	
H44B	0.406551	0.024303	0.362050	0.031*	
H44C	0.477412	−0.071917	0.312674	0.031*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0173 (4)	0.0282 (5)	0.0221 (4)	−0.0020 (3)	−0.0007 (3)	0.0120 (4)
C1	0.0136 (5)	0.0152 (5)	0.0168 (5)	0.0033 (4)	0.0045 (4)	0.0067 (4)
O2	0.0137 (4)	0.0241 (4)	0.0211 (4)	0.0017 (3)	−0.0010 (3)	0.0105 (3)
C2	0.0170 (5)	0.0187 (6)	0.0214 (6)	0.0053 (4)	0.0012 (4)	0.0070 (5)
O3	0.0157 (4)	0.0148 (4)	0.0131 (4)	0.0039 (3)	−0.0009 (3)	0.0034 (3)
C3	0.0209 (6)	0.0223 (6)	0.0310 (7)	0.0097 (5)	0.0046 (5)	0.0122 (5)
O4	0.0212 (4)	0.0190 (4)	0.0229 (4)	0.0081 (3)	0.0009 (3)	0.0060 (3)
C4	0.0248 (6)	0.0158 (6)	0.0325 (7)	0.0077 (5)	0.0114 (5)	0.0084 (5)
O5	0.0208 (4)	0.0172 (4)	0.0155 (4)	0.0038 (3)	−0.0017 (3)	0.0019 (3)
C5	0.0224 (6)	0.0175 (6)	0.0226 (6)	0.0018 (5)	0.0054 (5)	0.0023 (5)
C6	0.0159 (5)	0.0192 (6)	0.0188 (6)	0.0031 (4)	0.0020 (4)	0.0053 (5)
C7	0.0123 (5)	0.0131 (5)	0.0159 (5)	0.0029 (4)	0.0010 (4)	0.0020 (4)
C8	0.0152 (5)	0.0183 (5)	0.0202 (6)	0.0035 (4)	0.0014 (4)	0.0075 (5)
O8	0.0189 (4)	0.0138 (4)	0.0133 (4)	0.0048 (3)	0.0007 (3)	0.0029 (3)
C9	0.0173 (6)	0.0191 (6)	0.0232 (6)	0.0062 (4)	−0.0028 (4)	0.0053 (5)
O9	0.0237 (4)	0.0195 (4)	0.0203 (4)	0.0101 (3)	0.0006 (3)	0.0056 (3)
C11	0.0167 (6)	0.0226 (6)	0.0209 (6)	0.0017 (5)	0.0055 (4)	0.0049 (5)
C10	0.0119 (5)	0.0190 (6)	0.0254 (6)	0.0038 (4)	0.0006 (4)	−0.0007 (5)
O10	0.0259 (4)	0.0146 (4)	0.0159 (4)	0.0070 (3)	−0.0010 (3)	0.0021 (3)
C13	0.0127 (5)	0.0151 (5)	0.0126 (5)	0.0027 (4)	−0.0002 (4)	0.0044 (4)
C12	0.0172 (5)	0.0208 (6)	0.0165 (5)	0.0038 (4)	0.0011 (4)	0.0060 (4)
C14	0.0125 (5)	0.0173 (5)	0.0167 (5)	0.0026 (4)	0.0008 (4)	0.0077 (4)
C16	0.0173 (6)	0.0176 (5)	0.0184 (5)	0.0042 (4)	0.0029 (4)	0.0077 (4)
C15	0.0164 (6)	0.0156 (5)	0.0168 (5)	0.0041 (4)	0.0032 (4)	0.0064 (4)
C17	0.0152 (5)	0.0178 (5)	0.0161 (5)	0.0039 (4)	0.0013 (4)	0.0041 (4)
C18	0.0128 (6)	0.0373 (7)	0.0255 (6)	0.0000 (5)	−0.0031 (5)	0.0139 (6)

C19	0.0224 (6)	0.0400 (8)	0.0258 (7)	0.0054 (6)	−0.0029 (5)	0.0144 (6)
C20	0.0164 (5)	0.0156 (5)	0.0153 (5)	0.0050 (4)	0.0042 (4)	0.0058 (4)
C21	0.0172 (5)	0.0185 (6)	0.0202 (6)	0.0042 (4)	0.0019 (4)	0.0049 (5)
C22	0.0243 (6)	0.0153 (6)	0.0256 (6)	0.0026 (5)	0.0051 (5)	0.0033 (5)
C23	0.0281 (6)	0.0167 (6)	0.0289 (6)	0.0101 (5)	0.0094 (5)	0.0091 (5)
C24	0.0234 (6)	0.0242 (6)	0.0266 (6)	0.0115 (5)	0.0026 (5)	0.0106 (5)
C25	0.0196 (6)	0.0173 (5)	0.0191 (6)	0.0049 (4)	−0.0001 (4)	0.0050 (4)
C26	0.0143 (5)	0.0121 (5)	0.0216 (6)	0.0039 (4)	0.0020 (4)	0.0033 (4)
C27	0.0203 (6)	0.0305 (7)	0.0258 (6)	0.0042 (5)	0.0050 (5)	0.0089 (5)
C28	0.0201 (6)	0.0345 (7)	0.0376 (8)	0.0037 (5)	0.0124 (5)	0.0063 (6)
C29	0.0137 (6)	0.0211 (6)	0.0471 (8)	0.0046 (5)	0.0026 (5)	0.0017 (6)
C30	0.0181 (6)	0.0227 (6)	0.0369 (7)	0.0045 (5)	−0.0049 (5)	0.0085 (5)
C31	0.0172 (6)	0.0205 (6)	0.0270 (6)	0.0037 (5)	0.0010 (5)	0.0086 (5)
C32	0.0135 (5)	0.0141 (5)	0.0133 (5)	0.0027 (4)	0.0001 (4)	0.0032 (4)
C33	0.0137 (5)	0.0162 (5)	0.0152 (5)	0.0040 (4)	0.0011 (4)	0.0051 (4)
C34	0.0176 (5)	0.0158 (5)	0.0168 (5)	0.0065 (4)	0.0045 (4)	0.0072 (4)
O7	0.0169 (4)	0.0292 (5)	0.0237 (4)	0.0031 (3)	0.0045 (3)	0.0111 (4)
O6	0.0144 (4)	0.0276 (6)	0.0172 (5)	0.0016 (4)	0.0014 (3)	0.0067 (4)
C35	0.0172 (6)	0.0184 (12)	0.0182 (11)	0.0069 (5)	0.0042 (5)	0.0074 (8)
C36	0.0145 (6)	0.0179 (8)	0.0198 (8)	0.0068 (6)	0.0024 (5)	0.0059 (6)
C37	0.0173 (9)	0.0275 (9)	0.0179 (9)	0.0005 (7)	−0.0004 (6)	0.0027 (7)
C38	0.0198 (8)	0.0376 (9)	0.0235 (7)	0.0063 (6)	−0.0009 (6)	0.0084 (6)
O6A	0.0176 (19)	0.023 (2)	0.0191 (19)	0.0049 (18)	−0.0009 (17)	0.0056 (17)
C35A	0.0172 (6)	0.0184 (12)	0.0182 (11)	0.0069 (5)	0.0042 (5)	0.0074 (8)
C36A	0.018 (2)	0.021 (2)	0.018 (2)	0.007 (2)	0.002 (2)	0.008 (2)
C37A	0.019 (2)	0.032 (2)	0.022 (2)	0.003 (2)	−0.003 (2)	0.006 (2)
C38A	0.023 (4)	0.037 (4)	0.021 (4)	−0.002 (4)	−0.002 (4)	0.002 (4)
C39	0.0111 (5)	0.0167 (5)	0.0177 (5)	0.0023 (4)	0.0023 (4)	0.0039 (4)
C40	0.0255 (6)	0.0176 (6)	0.0179 (6)	0.0051 (5)	−0.0007 (5)	−0.0016 (5)
C41	0.0249 (6)	0.0213 (6)	0.0349 (7)	0.0014 (5)	−0.0050 (5)	0.0086 (5)
C42	0.0143 (5)	0.0163 (5)	0.0165 (5)	0.0049 (4)	0.0041 (4)	0.0039 (4)
C43	0.0249 (6)	0.0144 (5)	0.0188 (6)	0.0077 (5)	0.0020 (5)	0.0019 (4)
C44	0.0224 (6)	0.0157 (6)	0.0219 (6)	0.0037 (5)	0.0006 (5)	0.0032 (5)

Geometric parameters (Å, º)

O1—C17	1.2062 (14)	C23—C24	1.3866 (18)
C1—C6	1.3933 (16)	C23—H23	0.9500
C1—C2	1.3943 (15)	C24—C25	1.3883 (16)
C1—C13	1.5279 (15)	C24—H24	0.9500
O2—C17	1.3261 (13)	C25—H25	0.9500
O2—C18	1.4603 (14)	C26—C27	1.3879 (17)
C2—C3	1.3895 (16)	C26—C31	1.3896 (16)
C2—H2	0.9500	C26—C32	1.5230 (15)
O3—C39	1.3431 (13)	C27—C28	1.3878 (18)
O3—C13	1.4608 (13)	C27—H27	0.9500
C3—C4	1.3852 (18)	C28—C29	1.381 (2)
C3—H3	0.9500	C28—H28	0.9500

O4—C39	1.1957 (13)	C29—C30	1.380 (2)
C4—C5	1.3845 (18)	C29—H29	0.9500
C4—H4	0.9500	C30—C31	1.3921 (16)
O5—C39	1.3353 (13)	C30—H30	0.9500
O5—C40	1.4545 (13)	C31—H31	0.9500
C5—C6	1.3899 (16)	C32—C33	1.5558 (15)
C5—H5	0.9500	C33—C34	1.4588 (15)
C6—H6	0.9500	C33—H33A	0.9900
C7—C8	1.3912 (15)	C33—H33B	0.9900
C7—C12	1.3919 (15)	C34—C35A	1.166 (13)
C7—C13	1.5251 (14)	C34—C35	1.199 (2)
C8—C9	1.3934 (16)	O7—C36	1.2052 (18)
C8—H8	0.9500	O7—C36A	1.219 (12)
O8—C42	1.3450 (13)	O6—C36	1.3297 (18)
O8—C32	1.4620 (13)	O6—C37	1.4577 (18)
C9—C10	1.3843 (17)	C35—C36	1.455 (2)
C9—H9	0.9500	C37—C38	1.504 (2)
O9—C42	1.1986 (14)	C37—H37A	0.9900
C11—C12	1.3850 (16)	C37—H37B	0.9900
C11—C10	1.3887 (17)	C38—H38A	0.9800
C11—H11	0.9500	C38—H38B	0.9800
C10—H10	0.9500	C38—H38C	0.9800
O10—C42	1.3292 (13)	O6A—C36A	1.343 (12)
O10—C43	1.4556 (13)	O6A—C37A	1.453 (11)
C13—C14	1.5504 (14)	C35A—C36A	1.448 (14)
C12—H12	0.9500	C37A—C38A	1.468 (14)
C14—C15	1.4601 (15)	C37A—H37C	0.9900
C14—H14A	0.9900	C37A—H37D	0.9900
C14—H14B	0.9900	C38A—H38D	0.9800
C16—C15	1.1939 (16)	C38A—H38E	0.9800
C16—C17	1.4524 (15)	C38A—H38F	0.9800
C18—C19	1.4989 (17)	C40—C41	1.5006 (18)
C18—H18A	0.9900	C40—H40A	0.9900
C18—H18B	0.9900	C40—H40B	0.9900
C19—H19A	0.9800	C41—H41A	0.9800
C19—H19B	0.9800	C41—H41B	0.9800
C19—H19C	0.9800	C41—H41C	0.9800
C20—C21	1.3935 (16)	C43—C44	1.5008 (16)
C20—C25	1.3973 (15)	C43—H43A	0.9900
C20—C32	1.5281 (15)	C43—H43B	0.9900
C21—C22	1.3882 (16)	C44—H44A	0.9800
C21—H21	0.9500	C44—H44B	0.9800
C22—C23	1.3817 (18)	C44—H44C	0.9800
C22—H22	0.9500		

C6—C1—C2	118.89 (10)	C28—C27—H27	119.6
C6—C1—C13	119.69 (10)	C26—C27—H27	119.6
C2—C1—C13	121.30 (10)	C29—C28—C27	119.97 (12)

C17—O2—C18	114.95 (9)	C29—C28—H28	120.0
C3—C2—C1	120.29 (11)	C27—C28—H28	120.0
C3—C2—H2	119.9	C30—C29—C28	119.74 (12)
C1—C2—H2	119.9	C30—C29—H29	120.1
C39—O3—C13	119.10 (8)	C28—C29—H29	120.1
C4—C3—C2	120.33 (11)	C29—C30—C31	120.54 (12)
C4—C3—H3	119.8	C29—C30—H30	119.7
C2—C3—H3	119.8	C31—C30—H30	119.7
C5—C4—C3	119.85 (11)	C26—C31—C30	119.95 (12)
C5—C4—H4	120.1	C26—C31—H31	120.0
C3—C4—H4	120.1	C30—C31—H31	120.0
C39—O5—C40	115.48 (9)	O8—C32—C26	109.63 (8)
C4—C5—C6	119.97 (11)	O8—C32—C20	104.17 (8)
C4—C5—H5	120.0	C26—C32—C20	112.04 (9)
C6—C5—H5	120.0	O8—C32—C33	109.97 (8)
C5—C6—C1	120.67 (11)	C26—C32—C33	112.54 (9)
C5—C6—H6	119.7	C20—C32—C33	108.15 (9)
C1—C6—H6	119.7	C34—C33—C32	111.71 (9)
C8—C7—C12	119.15 (10)	C34—C33—H33A	109.3
C8—C7—C13	123.00 (10)	C32—C33—H33A	109.3
C12—C7—C13	117.84 (9)	C34—C33—H33B	109.3
C7—C8—C9	119.76 (11)	C32—C33—H33B	109.3
C7—C8—H8	120.1	H33A—C33—H33B	107.9
C9—C8—H8	120.1	C35A—C34—C33	175.3 (14)
C42—O8—C32	117.61 (8)	C35—C34—C33	172.53 (16)
C10—C9—C8	120.72 (11)	C36—O6—C37	116.13 (12)
C10—C9—H9	119.6	C34—C35—C36	174.5 (3)
C8—C9—H9	119.6	O7—C36—O6	126.34 (14)
C12—C11—C10	119.83 (11)	O7—C36—C35	123.46 (19)
C12—C11—H11	120.1	O6—C36—C35	110.17 (18)
C10—C11—H11	120.1	O6—C37—C38	111.08 (14)
C9—C10—C11	119.58 (10)	O6—C37—H37A	109.4
C9—C10—H10	120.2	C38—C37—H37A	109.4
C11—C10—H10	120.2	O6—C37—H37B	109.4
C42—O10—C43	115.04 (9)	C38—C37—H37B	109.4
O3—C13—C7	108.81 (8)	H37A—C37—H37B	108.0
O3—C13—C1	103.54 (8)	C37—C38—H38A	109.5
C7—C13—C1	112.21 (9)	C37—C38—H38B	109.5
O3—C13—C14	110.73 (8)	H38A—C38—H38B	109.5
C7—C13—C14	112.37 (9)	C37—C38—H38C	109.5
C1—C13—C14	108.83 (9)	H38A—C38—H38C	109.5
C11—C12—C7	120.93 (11)	H38B—C38—H38C	109.5
C11—C12—H12	119.5	C36A—O6A—C37A	114.6 (8)
C7—C12—H12	119.5	C34—C35A—C36A	170 (2)
C15—C14—C13	112.62 (9)	O7—C36A—O6A	124.6 (10)
C15—C14—H14A	109.1	O7—C36A—C35A	125.9 (15)
C13—C14—H14A	109.1	O6A—C36A—C35A	109.3 (15)
C15—C14—H14B	109.1	O6A—C37A—C38A	109.6 (10)

C13—C14—H14B	109.1	O6A—C37A—H37C	109.8
H14A—C14—H14B	107.8	C38A—C37A—H37C	109.8
C15—C16—C17	176.04 (12)	O6A—C37A—H37D	109.8
C16—C15—C14	175.92 (12)	C38A—C37A—H37D	109.8
O1—C17—O2	125.22 (10)	H37C—C37A—H37D	108.2
O1—C17—C16	122.99 (10)	C37A—C38A—H38D	109.5
O2—C17—C16	111.78 (9)	C37A—C38A—H38E	109.5
O2—C18—C19	106.98 (10)	H38D—C38A—H38E	109.5
O2—C18—H18A	110.3	C37A—C38A—H38F	109.5
C19—C18—H18A	110.3	H38D—C38A—H38F	109.5
O2—C18—H18B	110.3	H38E—C38A—H38F	109.5
C19—C18—H18B	110.3	O4—C39—O5	127.00 (10)
H18A—C18—H18B	108.6	O4—C39—O3	127.29 (10)
C18—C19—H19A	109.5	O5—C39—O3	105.70 (9)
C18—C19—H19B	109.5	O5—C40—C41	110.11 (10)
H19A—C19—H19B	109.5	O5—C40—H40A	109.6
C18—C19—H19C	109.5	C41—C40—H40A	109.6
H19A—C19—H19C	109.5	O5—C40—H40B	109.6
H19B—C19—H19C	109.5	C41—C40—H40B	109.6
C21—C20—C25	118.95 (10)	H40A—C40—H40B	108.2
C21—C20—C32	119.96 (10)	C40—C41—H41A	109.5
C25—C20—C32	120.84 (10)	C40—C41—H41B	109.5
C22—C21—C20	120.41 (11)	H41A—C41—H41B	109.5
C22—C21—H21	119.8	C40—C41—H41C	109.5
C20—C21—H21	119.8	H41A—C41—H41C	109.5
C23—C22—C21	120.34 (11)	H41B—C41—H41C	109.5
C23—C22—H22	119.8	O9—C42—O10	126.63 (10)
C21—C22—H22	119.8	O9—C42—O8	126.88 (10)
C22—C23—C24	119.75 (11)	O10—C42—O8	106.49 (9)
C22—C23—H23	120.1	O10—C43—C44	107.14 (9)
C24—C23—H23	120.1	O10—C43—H43A	110.3
C23—C24—C25	120.30 (11)	C44—C43—H43A	110.3
C23—C24—H24	119.9	O10—C43—H43B	110.3
C25—C24—H24	119.9	C44—C43—H43B	110.3
C24—C25—C20	120.23 (11)	H43A—C43—H43B	108.5
C24—C25—H25	119.9	C43—C44—H44A	109.5
C20—C25—H25	119.9	C43—C44—H44B	109.5
C27—C26—C31	119.08 (11)	H44A—C44—H44B	109.5
C27—C26—C32	118.58 (10)	C43—C44—H44C	109.5
C31—C26—C32	122.26 (10)	H44A—C44—H44C	109.5
C28—C27—C26	120.73 (12)	H44B—C44—H44C	109.5

C6—C1—C2—C3	−0.11 (17)	C31—C26—C27—C28	0.28 (18)
C13—C1—C2—C3	−175.99 (10)	C32—C26—C27—C28	177.03 (11)
C1—C2—C3—C4	0.57 (18)	C26—C27—C28—C29	−0.3 (2)
C2—C3—C4—C5	−0.55 (18)	C27—C28—C29—C30	0.2 (2)
C3—C4—C5—C6	0.07 (18)	C28—C29—C30—C31	−0.10 (19)
C4—C5—C6—C1	0.39 (17)	C27—C26—C31—C30	−0.21 (17)

C2—C1—C6—C5	−0.37 (17)	C32—C26—C31—C30	−176.84 (10)
C13—C1—C6—C5	175.58 (10)	C29—C30—C31—C26	0.12 (18)
C12—C7—C8—C9	1.47 (17)	C42—O8—C32—C26	69.26 (11)
C13—C7—C8—C9	179.81 (10)	C42—O8—C32—C20	−170.67 (8)
C7—C8—C9—C10	0.12 (17)	C42—O8—C32—C33	−54.98 (11)
C8—C9—C10—C11	−1.20 (18)	C27—C26—C32—O8	40.24 (13)
C12—C11—C10—C9	0.68 (17)	C31—C26—C32—O8	−143.12 (10)
C39—O3—C13—C7	−70.88 (11)	C27—C26—C32—C20	−74.91 (13)
C39—O3—C13—C1	169.59 (8)	C31—C26—C32—C20	101.74 (12)
C39—O3—C13—C14	53.10 (12)	C27—C26—C32—C33	162.96 (10)
C8—C7—C13—O3	137.44 (10)	C31—C26—C32—C33	−20.40 (14)
C12—C7—C13—O3	−44.20 (12)	C21—C20—C32—O8	−148.68 (10)
C8—C7—C13—C1	−108.59 (12)	C25—C20—C32—O8	37.21 (13)
C12—C7—C13—C1	69.77 (12)	C21—C20—C32—C26	−30.25 (14)
C8—C7—C13—C14	14.44 (15)	C25—C20—C32—C26	155.64 (10)
C12—C7—C13—C14	−167.19 (10)	C21—C20—C32—C33	94.36 (11)
C6—C1—C13—O3	155.22 (9)	C25—C20—C32—C33	−79.75 (12)
C2—C1—C13—O3	−28.94 (13)	O8—C32—C33—C34	−37.14 (12)
C6—C1—C13—C7	38.05 (13)	C26—C32—C33—C34	−159.68 (9)
C2—C1—C13—C7	−146.10 (10)	C20—C32—C33—C34	76.01 (11)
C6—C1—C13—C14	−86.96 (12)	C37—O6—C36—O7	−1.5 (2)
C2—C1—C13—C14	88.89 (12)	C37—O6—C36—C35	176.50 (17)
C10—C11—C12—C7	0.93 (17)	C36—O6—C37—C38	81.27 (17)
C8—C7—C12—C11	−2.00 (17)	C37A—O6A—C36A—O7	3.5 (18)
C13—C7—C12—C11	179.57 (10)	C37A—O6A—C36A—C35A	178.1 (14)
O3—C13—C14—C15	40.91 (12)	C34—C35A—C36A—O7	95 (16)
C7—C13—C14—C15	162.82 (9)	C34—C35A—C36A—O6A	−79 (16)
C1—C13—C14—C15	−72.27 (11)	C36A—O6A—C37A—C38A	−83.3 (13)
C18—O2—C17—O1	−3.28 (17)	C40—O5—C39—O4	−11.57 (16)
C18—O2—C17—C16	176.62 (10)	C40—O5—C39—O3	168.94 (8)
C17—O2—C18—C19	−178.12 (10)	C13—O3—C39—O4	2.35 (16)
C25—C20—C21—C22	1.80 (17)	C13—O3—C39—O5	−178.17 (8)
C32—C20—C21—C22	−172.42 (10)	C39—O5—C40—C41	−83.02 (12)
C20—C21—C22—C23	−0.77 (18)	C43—O10—C42—O9	0.13 (16)
C21—C22—C23—C24	−0.87 (18)	C43—O10—C42—O8	−179.54 (8)
C22—C23—C24—C25	1.46 (18)	C32—O8—C42—O9	−6.90 (16)
C23—C24—C25—C20	−0.42 (18)	C32—O8—C42—O10	172.76 (8)
C21—C20—C25—C24	−1.20 (17)	C42—O10—C43—C44	160.11 (9)
C32—C20—C25—C24	172.96 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O7	0.95	2.56	3.4079 (14)	149
C14—H14*A*···O7	0.99	2.61	3.4398 (14)	142
C18—H18*A*···O1^{i}	0.99	2.62	3.4119 (15)	137

C31—H31···O1	0.95	2.43	3.3617 (15)	169
C33—H33*B*···O1	0.99	2.61	3.3008 (13)	127

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+2.