IUCrData

ISSN 2414-3146

Cannabidiol revisited

Tobias Mayr,[a] Tobias Grassl,[a] Nikolaus Korber,[a] Volker Christoffel[b] and Michael Bodensteiner[c]*

[a]Institute for Inorganic Chemistry, University of Regensburg, 93040 Regensburg, Germany, [b]CSC - Christoffel Scientific Consulting, Bergstr. 11, 92369 Sengenthal, Germany, and [c]Central Analytics Service, X-ray Crystallography Dept., University of Regensburg, 93040 Regensburg, Germany. *Correspondence e-mail: michael.bodensteiner@ur.de

Received 4 February 2017
Accepted 17 February 2017

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

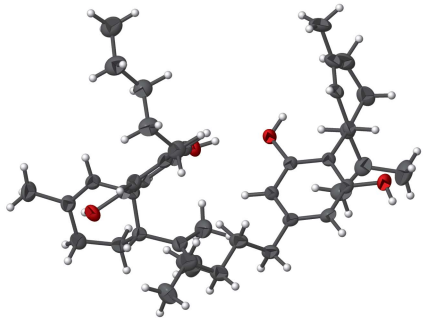
Keywords: crystal structure; cannabidiol; Cu $K\beta$ radiation; absolute structure.

CCDC reference: 1533487

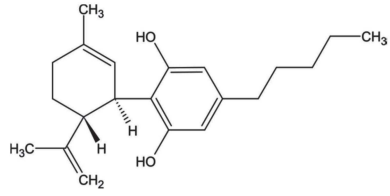
Structural data: full structural data are available from iucrdata.iucr.org

The crystal structure of cannabidiol, $C_{21}H_{30}O_2$, {systematic name: 2-[(1*R*,6*R*)-3-methyl-6-(prop-1-en-2-yl)cyclohex-2-enyl]-5-pentylbenzene-1,3-diol}, was determined earlier by Jones *et al.* [(1977). *Acta Cryst.* B**33**, 3211–3214] and Ottersen & Rosenqvist [(1977). *Acta Chem. Scand.* **B31**, 749–755]. In both investigations, the absolute configuration is given as *R*,*R*, referring to Mechoulam *et al.* [(1967.*J. Am. Chem. Soc.* **89**, 4552–4554]. In the latter, the absolute configuration was identified by chemical means. Using the advantages of modern single-crystal X-ray diffractometers such as area detectors and high-intensity radiation sources, a high-quality structure determination including the absolute configuration was possible and is shown in this work. Furthermore, the rather uncommon Cu $K\beta$ wavelength radiation was applied for the structure determination, which confirmed the absolute structure to be *R*,*R*.

3D view



Chemical scheme



Structure description

The crystal structure of the title compound, Fig. 1, was determined in the course of investigations regarding the application of Cu $K\beta$ radiation in single-crystal X-ray crystallography. Hence, the crystal was measured with this rather uncommon wavelength radiation.

Differing in the freely rotatable unsaturated alkyl chain of the cannabidiol molecule, two molecules with different orientations of this side chain are found within the asymmetric unit (Fig. 1). These independent molecules are both found to have the *R*,*R* configuration, confirming earlier investigations by Jones *et al.* (1977), Ottersen & Rosenqvist (1977) and Mechoulam *et al.* (1967). In the latter, the absolute configuration was identified by chemical means.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{30}O_2$
M_r	314.45
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	123
a, b, c (Å)	10.4395 (1), 10.8739 (1), 16.7853 (2)
β (°)	95.448 (1)
V (Å^3)	1896.83 (3)
Z	4
Radiation type	Cu $K\beta$
μ (mm^{-1})	0.39
Crystal size (mm)	0.28 × 0.16 × 0.15
Data collection	
Diffractometer	Agilent GV1000, TitanS2
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.996, 0.997
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	85198, 10203, 9859
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.695
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.088, 1.03
No. of reflections	10203
No. of parameters	655
No. of restraints	1
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.13
Absolute structure	Flack x determined using 4335 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013
Absolute structure parameter	−0.03 (6)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

Synthesis and crystallization

A crystal of cannabidiol was used as obtained from Trigal Pharma GmbH, Felix-Mottl-Str. 32, Vienna, Austria. The compound was recrystallized from *n*-heptane. A suitable crystal was selected using standard preparation techniques for non air- and moisture-sensitive samples and mounted on a MiTiGen-loop using mineral oil.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

We thank Mathias Meyer and Paul Swepston together with the Rigaku Oxford Diffraction team for supporting the project with Cu $K\beta$ mirror optics as well as for experimental and technical advice.

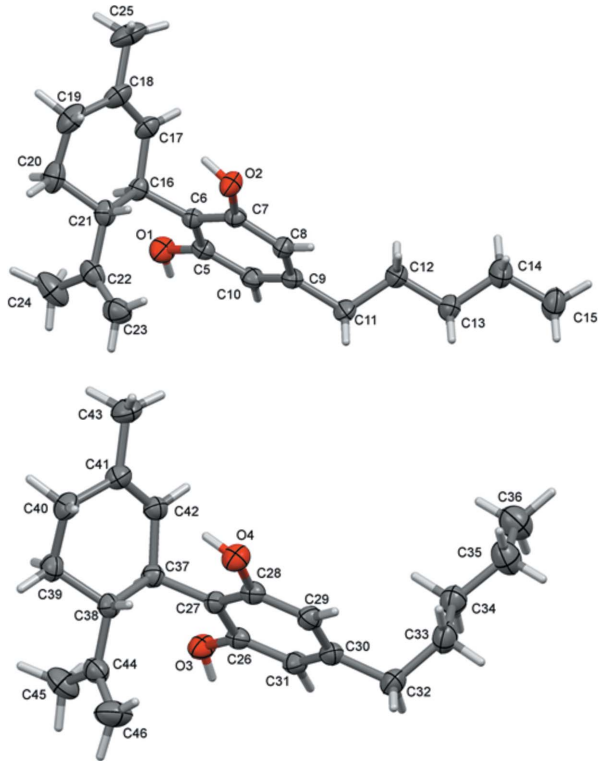


Figure 1
The molecular structure of the two independent molecules of cannabidiol, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

Funding information

Funding for this research was provided by: Rigaku Oxford Diffraction.

References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Jones, P. G., Falvello, L., Kennard, O., Sheldrick, G. M. & Mechoulam, R. (1977). *Acta Cryst.* B**33**, 3211–3214.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mechoulam, R., Braun, P. & Gaoni, Y. (1967). *J. Am. Chem. Soc.* **89**, 4552–4554.
Ottersen, T. & Rosenqvist, E. (1977). *Acta Chem. Scand.* **31b**, 749–755.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Rigaku OD (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2017). **2**, x170276 [https://doi.org/10.1107/S2414314617002760]

Cannabidiol revisited

Tobias Mayr, Tobias Grassl, Nikolaus Korber, Volker Christoffel and Michael Bodensteiner

2-[(1*R*,6*R*)-3-Methyl-6-(prop-1-en-2-yl)cyclohex-2-enyl]-5-pentylbenzene-1,3-diol

Crystal data

$C_{21}H_{30}O_2$
M_r = 314.45
Monoclinic, $P2_1$
a = 10.4395 (1) Å
b = 10.8739 (1) Å
c = 16.7853 (2) Å
β = 95.448 (1)°
V = 1896.83 (3) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.101 Mg m^{-3}
Melting point = 339–340 K
Cu $K\beta$ radiation, λ = 1.39222 Å
Cell parameters from 54306 reflections
θ = 3.6–74.8°
μ = 0.39 mm^{-1}
T = 123 K
Prism, clear colourless
0.28 × 0.16 × 0.15 mm

Data collection

Agilent GV1000, TitanS2 diffractometer
Radiation source: gradient vaccum rotating-anode X-ray tube, GV1000 (Cu) X-ray Source
Mirror monochromator
Detector resolution: 4.1818 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2015)
T_{min} = 0.996, T_{max} = 0.997
85198 measured reflections
10203 independent reflections
9859 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 75.3°, θ_{min} = 2.4°
h = −14→14
k = −15→14
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.088
S = 1.03
10203 reflections
655 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.1553P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Absolute structure: Flack x determined using 4335 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013
Absolute structure parameter: −0.03 (6)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.45873 (9)	0.27113 (11)	0.68801 (7)	0.0350 (2)
O3	0.22910 (10)	0.31909 (11)	0.75679 (7)	0.0361 (2)
O4	−0.01007 (11)	0.64952 (12)	0.85698 (7)	0.0370 (2)
O1	0.89037 (10)	0.39186 (12)	0.65474 (7)	0.0401 (2)
C7	0.56191 (12)	0.35049 (13)	0.70250 (8)	0.0280 (2)
C27	0.11199 (12)	0.47899 (13)	0.81028 (7)	0.0276 (2)
C28	0.06220 (12)	0.59824 (14)	0.80142 (8)	0.0298 (2)
C26	0.18235 (11)	0.43560 (13)	0.74901 (7)	0.0285 (2)
C6	0.68036 (12)	0.32428 (13)	0.67315 (7)	0.0272 (2)
C5	0.77497 (12)	0.41511 (14)	0.68514 (8)	0.0297 (2)
C29	0.08240 (13)	0.67195 (14)	0.73595 (9)	0.0337 (3)
C41	−0.10203 (14)	0.40126 (16)	0.96293 (9)	0.0360 (3)
C37	0.09170 (12)	0.39998 (14)	0.88271 (7)	0.0290 (2)
C9	0.63810 (13)	0.54375 (14)	0.75803 (8)	0.0317 (3)
C16	0.70534 (12)	0.20447 (14)	0.63181 (8)	0.0301 (2)
C8	0.54143 (13)	0.45499 (14)	0.74650 (8)	0.0309 (2)
C10	0.75383 (13)	0.52378 (14)	0.72519 (8)	0.0328 (3)
C38	0.16926 (12)	0.44801 (14)	0.96000 (8)	0.0309 (3)
C31	0.20157 (13)	0.50812 (15)	0.68246 (8)	0.0327 (3)
C42	−0.05071 (13)	0.39038 (15)	0.89332 (8)	0.0329 (3)
C30	0.15332 (13)	0.62697 (15)	0.67583 (8)	0.0334 (3)
C17	0.61190 (15)	0.18610 (16)	0.55797 (8)	0.0359 (3)
C18	0.56273 (18)	0.07793 (18)	0.53315 (9)	0.0432 (4)
C44	0.31309 (14)	0.43548 (17)	0.95708 (9)	0.0381 (3)
C21	0.70272 (14)	0.09214 (15)	0.68849 (9)	0.0352 (3)
C22	0.80132 (16)	0.10349 (17)	0.76024 (11)	0.0426 (3)
C11	0.61657 (16)	0.66019 (16)	0.80361 (10)	0.0390 (3)
C39	0.12060 (15)	0.38200 (17)	1.03236 (8)	0.0374 (3)
C32	0.17722 (17)	0.70713 (19)	0.60483 (10)	0.0434 (4)
C12	0.49874 (18)	0.73187 (17)	0.77039 (10)	0.0429 (3)
C33	0.0558 (2)	0.74656 (19)	0.55280 (11)	0.0475 (4)
C40	−0.01885 (16)	0.41677 (18)	1.04074 (9)	0.0408 (3)
C13	0.47857 (18)	0.85021 (16)	0.81437 (10)	0.0414 (3)
C23	0.7641 (2)	0.12371 (19)	0.83228 (11)	0.0498 (4)
C20	0.7152 (2)	−0.02482 (18)	0.63891 (13)	0.0494 (4)
C43	−0.24537 (17)	0.3974 (2)	0.96703 (12)	0.0503 (4)
C34	−0.01477 (18)	0.64082 (19)	0.50915 (11)	0.0449 (4)
C14	0.3597 (2)	0.9199 (2)	0.78214 (16)	0.0630 (6)
C25	0.4721 (3)	0.0678 (3)	0.45807 (12)	0.0607 (6)
C45	0.36871 (18)	0.3090 (2)	0.95561 (14)	0.0528 (5)
C46	0.38685 (19)	0.5344 (3)	0.95736 (16)	0.0603 (5)
C19	0.5994 (2)	−0.03921 (19)	0.57671 (13)	0.0540 (4)
C15	0.3401 (2)	1.0401 (2)	0.82410 (13)	0.0543 (5)
C35	−0.1338 (2)	0.6820 (2)	0.45576 (15)	0.0578 (5)
C24	0.9415 (2)	0.0943 (3)	0.74624 (18)	0.0663 (6)

C36	−0.1995 (2)	0.5757 (3)	0.40974 (17)	0.0636 (6)
H40A	−0.025 (2)	0.504 (2)	1.0609 (14)	0.043 (5)*
H38	0.1488 (18)	0.538 (2)	0.9650 (11)	0.027 (4)*
H8	0.459 (2)	0.466 (2)	0.7688 (13)	0.042 (5)*
H33A	−0.005 (3)	0.789 (3)	0.5860 (17)	0.062 (7)*
H39A	0.177 (2)	0.401 (2)	1.0813 (13)	0.041 (5)*
H21	0.617 (2)	0.089 (2)	0.7084 (12)	0.033 (5)*
H33B	0.085 (3)	0.807 (3)	0.5148 (17)	0.063 (8)*
H19A	0.527 (3)	−0.071 (3)	0.6008 (16)	0.060 (7)*
H42	−0.107 (2)	0.374 (2)	0.8461 (12)	0.035 (5)*
H32A	0.235 (3)	0.665 (3)	0.5677 (16)	0.055 (7)*
H37	0.1249 (18)	0.315 (2)	0.8712 (11)	0.030 (4)*
H16	0.7917 (18)	0.210 (2)	0.6121 (11)	0.030 (4)*
H39B	0.127 (2)	0.290 (3)	1.0247 (14)	0.046 (6)*
H40B	−0.055 (2)	0.365 (3)	1.0835 (15)	0.050 (6)*
H17	0.591 (2)	0.264 (3)	0.5272 (15)	0.047 (6)*
H31	0.251 (2)	0.476 (2)	0.6408 (13)	0.043 (5)*
H45A	0.356 (4)	0.256 (4)	1.004 (2)	0.092 (11)*
H20A	0.798 (3)	−0.018 (3)	0.6132 (16)	0.060 (7)*
H34A	−0.042 (2)	0.578 (3)	0.5498 (15)	0.050 (6)*
H10	0.823 (2)	0.585 (2)	0.7311 (13)	0.039 (5)*
H11A	0.608 (2)	0.639 (3)	0.8615 (15)	0.050 (6)*
H13A	0.550 (3)	0.902 (3)	0.8117 (18)	0.069 (8)*
H1	0.943 (3)	0.444 (3)	0.6727 (15)	0.054 (7)*
H13B	0.473 (3)	0.835 (3)	0.8723 (17)	0.058 (7)*
H29	0.049 (2)	0.756 (2)	0.7327 (13)	0.043 (6)*
H24A	0.961 (3)	0.150 (3)	0.7046 (19)	0.070 (8)*
H24B	0.993 (3)	0.106 (3)	0.793 (2)	0.077 (9)*
H3	0.290 (2)	0.307 (2)	0.7284 (14)	0.046 (6)*
H35A	−0.111 (3)	0.746 (3)	0.4193 (18)	0.063 (8)*
H35B	−0.190 (3)	0.721 (3)	0.4888 (19)	0.068 (8)*
H46A	0.483 (3)	0.524 (3)	0.9616 (18)	0.071 (9)*
H43A	−0.283 (3)	0.469 (3)	0.9898 (19)	0.074 (9)*
H12A	0.416 (3)	0.679 (3)	0.7686 (19)	0.073 (9)*
H46B	0.344 (4)	0.613 (4)	0.958 (2)	0.090 (11)*
H12B	0.499 (3)	0.750 (3)	0.7153 (19)	0.070 (8)*
H15A	0.342 (3)	1.031 (3)	0.883 (2)	0.080 (10)*
H19B	0.617 (3)	−0.104 (3)	0.537 (2)	0.074 (9)*
H32B	0.223 (3)	0.781 (3)	0.6257 (17)	0.064 (8)*
H14A	0.365 (4)	0.935 (4)	0.720 (3)	0.113 (14)*
H34B	0.048 (2)	0.596 (3)	0.4745 (15)	0.052 (6)*
H20B	0.721 (3)	−0.098 (3)	0.6747 (16)	0.057 (7)*
H43B	−0.293 (3)	0.386 (3)	0.9146 (17)	0.060 (7)*
H23A	0.676 (3)	0.132 (3)	0.8403 (16)	0.059 (7)*
H23B	0.818 (3)	0.130 (3)	0.8784 (17)	0.061 (7)*
H2	0.475 (2)	0.223 (2)	0.6496 (14)	0.044 (5)*
H4	−0.036 (3)	0.593 (3)	0.8864 (16)	0.054 (7)*
H43C	−0.264 (3)	0.331 (3)	1.0030 (19)	0.073 (9)*

H25A	0.443 (3)	0.156 (4)	0.438 (2)	0.075 (9)*
H15B	0.415 (3)	1.097 (4)	0.815 (2)	0.082 (10)*
H36A	−0.142 (3)	0.538 (3)	0.3720 (17)	0.063 (8)*
H25B	0.512 (3)	0.020 (3)	0.4207 (19)	0.069 (8)*
H25C	0.401 (3)	0.024 (3)	0.4684 (19)	0.073 (9)*
H45B	0.463 (3)	0.312 (3)	0.9515 (18)	0.068 (8)*
H11B	0.692 (3)	0.713 (3)	0.8057 (18)	0.072 (8)*
H45C	0.331 (3)	0.264 (3)	0.914 (2)	0.078 (9)*
H36B	−0.222 (3)	0.515 (4)	0.448 (2)	0.076 (9)*
H14B	0.280 (5)	0.877 (5)	0.788 (3)	0.125 (16)*
H36C	−0.284 (4)	0.611 (4)	0.377 (2)	0.088 (11)*
H24C	0.955 (4)	0.003 (4)	0.723 (2)	0.093 (11)*
H15C	0.261 (4)	1.085 (4)	0.808 (2)	0.098 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0289 (4)	0.0337 (5)	0.0438 (5)	−0.0053 (4)	0.0102 (4)	−0.0110 (4)
O3	0.0349 (5)	0.0343 (6)	0.0409 (5)	0.0056 (4)	0.0121 (4)	−0.0004 (4)
O4	0.0416 (5)	0.0330 (6)	0.0380 (5)	0.0046 (4)	0.0113 (4)	−0.0038 (4)
O1	0.0277 (4)	0.0454 (7)	0.0481 (6)	−0.0063 (4)	0.0074 (4)	−0.0059 (5)
C7	0.0276 (5)	0.0280 (6)	0.0281 (5)	−0.0018 (4)	0.0016 (4)	−0.0003 (4)
C27	0.0256 (5)	0.0302 (6)	0.0271 (5)	−0.0014 (4)	0.0027 (4)	−0.0013 (4)
C28	0.0273 (5)	0.0313 (7)	0.0309 (5)	−0.0008 (5)	0.0034 (4)	−0.0028 (5)
C26	0.0237 (5)	0.0316 (7)	0.0302 (5)	−0.0003 (4)	0.0027 (4)	−0.0026 (5)
C6	0.0277 (5)	0.0279 (6)	0.0258 (5)	0.0000 (4)	0.0018 (4)	0.0007 (4)
C5	0.0264 (5)	0.0331 (7)	0.0292 (5)	−0.0011 (5)	0.0003 (4)	0.0028 (5)
C29	0.0312 (6)	0.0311 (7)	0.0386 (6)	−0.0008 (5)	0.0028 (5)	0.0025 (5)
C41	0.0335 (6)	0.0392 (8)	0.0361 (6)	−0.0019 (5)	0.0077 (5)	0.0030 (6)
C37	0.0297 (5)	0.0301 (7)	0.0272 (5)	−0.0006 (5)	0.0032 (4)	−0.0002 (4)
C9	0.0332 (6)	0.0290 (7)	0.0314 (6)	0.0017 (5)	−0.0052 (5)	−0.0027 (5)
C16	0.0300 (6)	0.0299 (7)	0.0312 (5)	0.0007 (5)	0.0075 (4)	−0.0016 (5)
C8	0.0294 (5)	0.0315 (7)	0.0315 (5)	0.0020 (5)	0.0014 (4)	−0.0037 (5)
C10	0.0300 (6)	0.0306 (7)	0.0361 (6)	−0.0033 (5)	−0.0049 (5)	−0.0003 (5)
C38	0.0311 (6)	0.0330 (7)	0.0282 (5)	−0.0004 (5)	0.0016 (4)	−0.0004 (5)
C31	0.0277 (5)	0.0415 (8)	0.0294 (5)	−0.0034 (5)	0.0061 (4)	−0.0021 (5)
C42	0.0314 (6)	0.0358 (7)	0.0315 (6)	−0.0042 (5)	0.0026 (5)	0.0016 (5)
C30	0.0295 (6)	0.0394 (8)	0.0311 (6)	−0.0056 (5)	0.0015 (4)	0.0042 (5)
C17	0.0409 (7)	0.0392 (8)	0.0285 (6)	−0.0005 (6)	0.0084 (5)	−0.0053 (5)
C18	0.0503 (8)	0.0465 (10)	0.0342 (6)	−0.0056 (7)	0.0112 (6)	−0.0129 (6)
C44	0.0331 (6)	0.0473 (9)	0.0332 (6)	0.0020 (6)	−0.0008 (5)	−0.0012 (6)
C21	0.0356 (6)	0.0301 (7)	0.0405 (7)	0.0012 (5)	0.0070 (5)	0.0011 (5)
C22	0.0397 (7)	0.0357 (8)	0.0513 (8)	0.0028 (6)	−0.0013 (6)	0.0103 (7)
C11	0.0403 (7)	0.0319 (8)	0.0426 (7)	0.0032 (6)	−0.0069 (6)	−0.0098 (6)
C39	0.0414 (7)	0.0435 (9)	0.0271 (5)	0.0016 (6)	0.0020 (5)	0.0031 (5)
C32	0.0429 (7)	0.0482 (10)	0.0393 (7)	−0.0094 (7)	0.0051 (6)	0.0112 (7)
C12	0.0493 (8)	0.0349 (8)	0.0423 (8)	0.0082 (6)	−0.0073 (6)	−0.0079 (6)
C33	0.0572 (9)	0.0442 (10)	0.0403 (8)	−0.0047 (7)	0.0011 (7)	0.0111 (7)

C40	0.0433 (7)	0.0491 (10)	0.0311 (6)	−0.0003 (6)	0.0093 (5)	0.0014 (6)
C13	0.0501 (8)	0.0306 (8)	0.0427 (7)	0.0049 (6)	0.0006 (6)	−0.0046 (6)
C23	0.0590 (10)	0.0435 (10)	0.0448 (8)	−0.0022 (8)	−0.0065 (7)	0.0068 (7)
C20	0.0574 (10)	0.0300 (8)	0.0621 (10)	0.0052 (7)	0.0128 (8)	−0.0040 (7)
C43	0.0355 (7)	0.0689 (13)	0.0484 (8)	−0.0037 (8)	0.0136 (6)	0.0041 (9)
C34	0.0488 (8)	0.0426 (9)	0.0429 (8)	−0.0007 (7)	0.0022 (6)	0.0068 (7)
C14	0.0641 (12)	0.0452 (12)	0.0756 (14)	0.0181 (9)	−0.0141 (10)	−0.0167 (10)
C25	0.0731 (13)	0.0685 (15)	0.0401 (8)	−0.0155 (11)	0.0037 (9)	−0.0236 (9)
C45	0.0413 (8)	0.0558 (12)	0.0591 (10)	0.0145 (8)	−0.0063 (7)	−0.0074 (9)
C46	0.0366 (8)	0.0613 (14)	0.0833 (15)	−0.0080 (8)	0.0070 (9)	0.0000 (11)
C19	0.0686 (12)	0.0369 (10)	0.0577 (10)	−0.0088 (8)	0.0122 (9)	−0.0156 (8)
C15	0.0680 (12)	0.0384 (10)	0.0581 (10)	0.0133 (9)	0.0143 (9)	−0.0006 (8)
C35	0.0599 (11)	0.0511 (12)	0.0593 (11)	0.0042 (9)	−0.0112 (9)	0.0052 (9)
C24	0.0396 (9)	0.0783 (17)	0.0794 (15)	0.0108 (10)	−0.0016 (9)	0.0226 (14)
C36	0.0584 (12)	0.0607 (14)	0.0692 (13)	0.0005 (10)	−0.0070 (10)	0.0006 (11)

Geometric parameters (Å, º)

O2—C7	1.3836 (16)	C11—H11B	0.98 (3)
O2—H2	0.86 (3)	C39—C40	1.523 (2)
O3—C26	1.3595 (18)	C39—H39A	0.98 (2)
O3—H3	0.84 (3)	C39—H39B	1.02 (3)
O4—C28	1.3723 (16)	C32—C33	1.531 (3)
O4—H4	0.85 (3)	C32—H32A	1.02 (3)
O1—C5	1.3756 (17)	C32—H32B	0.98 (3)
O1—H1	0.83 (3)	C12—C13	1.508 (2)
C7—C6	1.4031 (18)	C12—H12A	1.03 (3)
C7—C8	1.3829 (19)	C12—H12B	0.95 (3)
C27—C28	1.400 (2)	C33—C34	1.516 (3)
C27—C26	1.4011 (17)	C33—H33A	1.00 (3)
C27—C37	1.5198 (18)	C33—H33B	0.99 (3)
C28—C29	1.392 (2)	C40—H40A	1.01 (3)
C26—C31	1.3973 (19)	C40—H40B	1.01 (3)
C6—C5	1.3978 (18)	C13—C14	1.510 (3)
C6—C16	1.510 (2)	C13—H13A	0.94 (3)
C5—C10	1.388 (2)	C13—H13B	0.99 (3)
C29—C30	1.395 (2)	C23—H23A	0.95 (3)
C29—H29	0.98 (3)	C23—H23B	0.92 (3)
C41—C42	1.3361 (19)	C20—C19	1.529 (3)
C41—C40	1.508 (2)	C20—H20A	1.00 (3)
C41—C43	1.505 (2)	C20—H20B	1.00 (3)
C37—C38	1.5530 (17)	C43—H43A	0.96 (4)
C37—C42	1.5176 (18)	C43—H43B	0.98 (3)
C37—H37	1.01 (2)	C43—H43C	0.97 (4)
C9—C8	1.397 (2)	C34—C35	1.529 (3)
C9—C10	1.392 (2)	C34—H34A	1.02 (3)
C9—C11	1.507 (2)	C34—H34B	1.04 (3)
C16—C17	1.516 (2)	C14—C15	1.507 (3)

C16—C21	1.550 (2)	C14—H14A	1.06 (4)
C16—H16	0.991 (19)	C14—H14B	0.97 (5)
C8—H8	0.98 (2)	C25—H25A	1.05 (4)
C10—H10	0.98 (2)	C25—H25B	0.94 (3)
C38—C44	1.5131 (19)	C25—H25C	0.91 (4)
C38—C39	1.5380 (19)	C45—H45A	1.01 (4)
C38—H38	1.01 (2)	C45—H45B	1.00 (3)
C31—C30	1.388 (2)	C45—H45C	0.91 (4)
C31—H31	0.97 (2)	C46—H46A	1.01 (3)
C42—H42	0.96 (2)	C46—H46B	0.97 (4)
C30—C32	1.516 (2)	C19—H19A	0.95 (3)
C17—C18	1.334 (2)	C19—H19B	0.99 (3)
C17—H17	1.01 (3)	C15—H15A	1.00 (3)
C18—C25	1.506 (3)	C15—H15B	1.03 (4)
C18—C19	1.500 (3)	C15—H15C	0.97 (4)
C44—C45	1.494 (3)	C35—C36	1.517 (4)
C44—C46	1.323 (3)	C35—H35A	0.97 (3)
C21—C22	1.513 (2)	C35—H35B	0.94 (3)
C21—C20	1.532 (2)	C24—H24A	0.96 (3)
C21—H21	0.99 (2)	C24—H24B	0.91 (4)
C22—C23	1.323 (3)	C24—H24C	1.09 (4)
C22—C24	1.507 (3)	C36—H36A	1.00 (3)
C11—C12	1.517 (2)	C36—H36B	0.96 (4)
C11—H11A	1.01 (3)	C36—H36C	1.07 (4)

C7—O2—H2	107.9 (16)	C33—C32—H32B	109.2 (18)
C26—O3—H3	112.3 (18)	H32A—C32—H32B	106 (2)
C28—O4—H4	108.9 (19)	C11—C12—H12A	111.8 (19)
C5—O1—H1	108.4 (18)	C11—C12—H12B	112.7 (19)
O2—C7—C6	120.59 (12)	C13—C12—C11	113.86 (13)
C8—C7—O2	116.69 (12)	C13—C12—H12A	109.4 (18)
C8—C7—C6	122.72 (12)	C13—C12—H12B	108 (2)
C28—C27—C26	116.69 (12)	H12A—C12—H12B	100 (3)
C28—C27—C37	122.01 (11)	C32—C33—H33A	110.4 (16)
C26—C27—C37	121.29 (13)	C32—C33—H33B	105.3 (17)
O4—C28—C27	121.79 (13)	C34—C33—C32	113.65 (17)
O4—C28—C29	115.90 (13)	C34—C33—H33A	108.3 (18)
C29—C28—C27	122.32 (12)	C34—C33—H33B	111.2 (17)
O3—C26—C27	116.85 (12)	H33A—C33—H33B	108 (2)
O3—C26—C31	121.68 (12)	C41—C40—C39	111.66 (12)
C31—C26—C27	121.46 (13)	C41—C40—H40A	110.2 (13)
C7—C6—C16	122.20 (12)	C41—C40—H40B	109.2 (14)
C5—C6—C7	116.12 (12)	C39—C40—H40A	111.0 (13)
C5—C6—C16	121.68 (11)	C39—C40—H40B	110.6 (14)
O1—C5—C6	116.65 (13)	H40A—C40—H40B	103.9 (19)
O1—C5—C10	121.43 (13)	C12—C13—C14	113.65 (15)
C10—C5—C6	121.92 (12)	C12—C13—H13A	109.9 (19)
C28—C29—C30	119.97 (14)	C12—C13—H13B	111.2 (18)

C28—C29—H29	120.1 (13)	C14—C13—H13A	108 (2)
C30—C29—H29	119.9 (13)	C14—C13—H13B	108.2 (17)
C42—C41—C40	121.44 (13)	H13A—C13—H13B	105 (2)
C42—C41—C43	121.36 (14)	C22—C23—H23A	121.3 (17)
C43—C41—C40	117.19 (13)	C22—C23—H23B	124.9 (18)
C27—C37—C38	112.15 (11)	H23A—C23—H23B	114 (2)
C27—C37—H37	106.6 (11)	C21—C20—H20A	106.8 (18)
C38—C37—H37	107.8 (11)	C21—C20—H20B	109.9 (16)
C42—C37—C27	110.20 (11)	C19—C20—C21	110.68 (15)
C42—C37—C38	111.34 (10)	C19—C20—H20A	111.8 (16)
C42—C37—H37	108.5 (11)	C19—C20—H20B	109.1 (16)
C8—C9—C11	120.68 (13)	H20A—C20—H20B	108 (2)
C10—C9—C8	118.71 (13)	C41—C43—H43A	116 (2)
C10—C9—C11	120.60 (14)	C41—C43—H43B	112.6 (16)
C6—C16—C17	111.32 (12)	C41—C43—H43C	107.7 (19)
C6—C16—C21	112.57 (11)	H43A—C43—H43B	106 (3)
C6—C16—H16	107.6 (12)	H43A—C43—H43C	104 (3)
C17—C16—C21	110.52 (12)	H43B—C43—H43C	111 (3)
C17—C16—H16	105.6 (11)	C33—C34—C35	113.08 (17)
C21—C16—H16	108.9 (12)	C33—C34—H34A	109.5 (14)
C7—C8—C9	119.71 (12)	C33—C34—H34B	108.6 (15)
C7—C8—H8	119.2 (14)	C35—C34—H34A	109.1 (14)
C9—C8—H8	121.1 (14)	C35—C34—H34B	109.3 (14)
C5—C10—C9	120.58 (13)	H34A—C34—H34B	107 (2)
C5—C10—H10	118.7 (13)	C13—C14—H14A	108 (2)
C9—C10—H10	120.7 (13)	C13—C14—H14B	114 (3)
C37—C38—H38	107.4 (11)	C15—C14—C13	114.30 (18)
C44—C38—C37	112.51 (11)	C15—C14—H14A	110 (3)
C44—C38—C39	112.76 (12)	C15—C14—H14B	103 (3)
C44—C38—H38	108.0 (11)	H14A—C14—H14B	107 (4)
C39—C38—C37	108.52 (11)	C18—C25—H25A	110.3 (18)
C39—C38—H38	107.4 (11)	C18—C25—H25B	108.3 (19)
C26—C31—H31	119.9 (15)	C18—C25—H25C	110 (2)
C30—C31—C26	120.74 (12)	H25A—C25—H25B	115 (3)
C30—C31—H31	119.4 (15)	H25A—C25—H25C	108 (3)
C41—C42—C37	125.11 (12)	H25B—C25—H25C	105 (3)
C41—C42—H42	118.5 (13)	C44—C45—H45A	116 (2)
C37—C42—H42	116.4 (12)	C44—C45—H45B	111 (2)
C29—C30—C32	120.33 (15)	C44—C45—H45C	112 (2)
C31—C30—C29	118.81 (13)	H45A—C45—H45B	106 (3)
C31—C30—C32	120.86 (14)	H45A—C45—H45C	103 (3)
C16—C17—H17	113.6 (14)	H45B—C45—H45C	109 (3)
C18—C17—C16	124.76 (16)	C44—C46—H46A	119 (2)
C18—C17—H17	121.6 (14)	C44—C46—H46B	117 (2)
C17—C18—C25	121.23 (19)	H46A—C46—H46B	124 (3)
C17—C18—C19	121.57 (16)	C18—C19—C20	113.24 (16)
C19—C18—C25	117.16 (18)	C18—C19—H19A	109.6 (18)
C45—C44—C38	118.15 (15)	C18—C19—H19B	109 (2)

C46—C44—C38	120.39 (17)	C20—C19—H19A	110.7 (16)
C46—C44—C45	121.46 (17)	C20—C19—H19B	110.3 (19)
C16—C21—H21	107.7 (13)	H19A—C19—H19B	103 (3)
C22—C21—C16	111.96 (13)	C14—C15—H15A	113 (2)
C22—C21—C20	114.41 (15)	C14—C15—H15B	108 (2)
C22—C21—H21	107.7 (12)	C14—C15—H15C	117 (3)
C20—C21—C16	108.26 (13)	H15A—C15—H15B	105 (3)
C20—C21—H21	106.5 (13)	H15A—C15—H15C	105 (3)
C23—C22—C21	120.27 (16)	H15B—C15—H15C	107 (3)
C23—C22—C24	121.83 (19)	C34—C35—H35A	110.3 (17)
C24—C22—C21	117.87 (18)	C34—C35—H35B	107.5 (19)
C9—C11—C12	113.57 (12)	C36—C35—C34	112.3 (2)
C9—C11—H11A	109.0 (16)	C36—C35—H35A	110.5 (18)
C9—C11—H11B	110.9 (19)	C36—C35—H35B	112 (2)
C12—C11—H11A	109.1 (14)	H35A—C35—H35B	104 (3)
C12—C11—H11B	110 (2)	C22—C24—H24A	110.2 (18)
H11A—C11—H11B	104 (2)	C22—C24—H24B	111 (2)
C38—C39—H39A	110.1 (14)	C22—C24—H24C	106 (2)
C38—C39—H39B	109.3 (14)	H24A—C24—H24B	113 (3)
C40—C39—C38	110.32 (12)	H24A—C24—H24C	106 (3)
C40—C39—H39A	111.6 (13)	H24B—C24—H24C	110 (3)
C40—C39—H39B	109.4 (14)	C35—C36—H36A	111.6 (18)
H39A—C39—H39B	105.9 (19)	C35—C36—H36B	108 (2)
C30—C32—C33	114.85 (14)	C35—C36—H36C	108 (2)
C30—C32—H32A	112.2 (16)	H36A—C36—H36B	110 (3)
C30—C32—H32B	107.4 (17)	H36A—C36—H36C	109 (3)
C33—C32—H32A	106.6 (15)	H36B—C36—H36C	110 (3)

O2—C7—C6—C5	174.74 (12)	C9—C11—C12—C13	−178.47 (15)
O2—C7—C6—C16	−5.95 (19)	C16—C6—C5—O1	2.37 (18)
O2—C7—C8—C9	−175.02 (12)	C16—C6—C5—C10	−178.04 (12)
O3—C26—C31—C30	−179.81 (12)	C16—C17—C18—C25	−178.89 (16)
O4—C28—C29—C30	−179.15 (13)	C16—C17—C18—C19	−1.5 (3)
O1—C5—C10—C9	−177.67 (13)	C16—C21—C22—C23	109.34 (19)
C7—C6—C5—O1	−178.32 (12)	C16—C21—C22—C24	−69.1 (2)
C7—C6—C5—C10	1.27 (18)	C16—C21—C20—C19	−63.94 (19)
C7—C6—C16—C17	59.51 (16)	C8—C7—C6—C5	−5.08 (19)
C7—C6—C16—C21	−65.22 (16)	C8—C7—C6—C16	174.22 (12)
C27—C28—C29—C30	0.7 (2)	C8—C9—C10—C5	−3.1 (2)
C27—C26—C31—C30	1.3 (2)	C8—C9—C11—C12	−55.2 (2)
C27—C37—C38—C44	−67.06 (16)	C10—C9—C8—C7	−0.6 (2)
C27—C37—C38—C39	167.46 (12)	C10—C9—C11—C12	123.46 (16)
C27—C37—C42—C41	−135.63 (17)	C38—C37—C42—C41	−10.5 (2)
C28—C27—C26—O3	−178.98 (11)	C38—C39—C40—C41	50.55 (19)
C28—C27—C26—C31	−0.02 (18)	C31—C30—C32—C33	118.00 (18)
C28—C27—C37—C38	−70.38 (15)	C42—C41—C40—C39	−16.2 (2)
C28—C27—C37—C42	54.26 (16)	C42—C37—C38—C44	168.94 (13)
C28—C29—C30—C31	0.6 (2)	C42—C37—C38—C39	43.46 (16)

C28—C29—C30—C32	−179.24 (13)	C30—C32—C33—C34	−66.3 (2)
C26—C27—C28—O4	178.87 (12)	C17—C16—C21—C22	176.17 (13)
C26—C27—C28—C29	−0.96 (18)	C17—C16—C21—C20	49.14 (16)
C26—C27—C37—C38	109.37 (14)	C17—C18—C19—C20	−11.9 (3)
C26—C27—C37—C42	−125.99 (13)	C44—C38—C39—C40	169.73 (14)
C26—C31—C30—C29	−1.5 (2)	C21—C16—C17—C18	−18.05 (19)
C26—C31—C30—C32	178.27 (13)	C21—C20—C19—C18	44.8 (2)
C6—C7—C8—C9	4.8 (2)	C22—C21—C20—C19	170.46 (16)
C6—C5—C10—C9	2.8 (2)	C11—C9—C8—C7	178.12 (13)
C6—C16—C17—C18	−143.92 (15)	C11—C9—C10—C5	178.21 (13)
C6—C16—C21—C22	−58.66 (16)	C11—C12—C13—C14	−178.8 (2)
C6—C16—C21—C20	174.31 (13)	C39—C38—C44—C45	57.14 (19)
C5—C6—C16—C17	−121.22 (13)	C39—C38—C44—C46	−121.6 (2)
C5—C6—C16—C21	114.05 (13)	C32—C33—C34—C35	−178.50 (17)
C29—C30—C32—C33	−62.2 (2)	C12—C13—C14—C15	−178.5 (2)
C37—C27—C28—O4	−1.37 (19)	C33—C34—C35—C36	177.4 (2)
C37—C27—C28—C29	178.80 (12)	C40—C41—C42—C37	−4.0 (3)
C37—C27—C26—O3	1.26 (18)	C20—C21—C22—C23	−127.02 (19)
C37—C27—C26—C31	−179.78 (11)	C20—C21—C22—C24	54.5 (2)
C37—C38—C44—C45	−66.00 (18)	C43—C41—C42—C37	176.55 (17)
C37—C38—C44—C46	115.25 (19)	C43—C41—C40—C39	163.23 (17)
C37—C38—C39—C40	−64.94 (16)	C25—C18—C19—C20	165.63 (17)