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# 1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,-5,6,7,8,10,10a-dodecahydro-phenanthrene-1-carboxylic acid

**Zhen-Dong Zhao,* Yu-Xiang Chen, Yu-Min Wang and Liang-Wu Bi**

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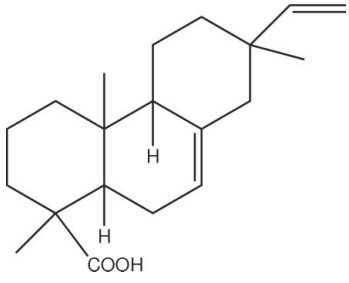
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.010 Å; $R$ factor = 0.069; $wR$ factor = 0.150; data-to-parameter ratio = 9.3.

The title compound, also known as isopimaric acid, $C_{20}H_{30}O_2$, was isolated from slash pine rosin. There are two unique molecules in the unit cell. The two cyclohexane rings have classical chair conformations. The cyclohexene ring represents a semi-chair. The molecular conformation is stabilized by weak intramolecular C—H···O hydrogen-bonding interactions. The molecules are dimerized through their carboxyl groups by O—H···O hydrogen bonds, forming $R_2^2(8)$ rings.

## Related literature

For physical and spectroscopic analysis, see: Baldwin *et al.* (1958); Harris & Sanderson (1948). For biological activities, see: Smith *et al.* (2005); Russo *et al.* (2007).




## Experimental

### Crystal data

$C_{20}H_{30}O_2$
$M_r$ = 302.44
Orthorhombic, $P2_12_12_1$
$a$ = 11.624 (2) Å
$b$ = 11.803 (2) Å
$c$ = 25.698 (5) Å
$V$ = 3525.7 (12) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 K
0.20 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.986, $T_{max}$ = 0.993
7031 measured reflections
6382 independent reflections
2613 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.088
3 standard reflections
every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.150
$S$ = 1.00
3598 reflections
385 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$B$···O4$^{i}$ | 0.82 | 1.84 | 2.653 (7) | 168 |
| O3—H3$D$···O1$^{ii}$ | 0.82 | 1.85 | 2.655 (7) | 168 |
| C11—H11$A$···O1 | 0.98 | 2.34 | 2.764 (7) | 105 |
| C32—H32$B$···O4 | 0.97 | 2.49 | 3.081 (9) | 119 |

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{5}{2}, -z+2$; (ii) $x-\frac{1}{2}, -y+\frac{5}{2}, -z+2$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2789).

## References

Baldwin, D. E., Loeblich, V. M. & Lawrence, R. V. (1958). *J. Org. Chem.* **23**, 25–26.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Harris, G. C. & Sanderson, T. F. (1948). *J. Am. Chem. Soc.* **70**, 2079–2081.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Russo, R., Loverme, J., Rana, G. L., D'Agostino, G., Sasso, O., Calignano, A. & Piomelli, D. (2007). *Eur. J. Pharmacol.* **566**, 117–119.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, E., Williamson, E., Zloh, M. & Gibbons, S. (2005). *Phytother. Res.* **19**, 538–542.

# supporting information

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## 1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,5,6,7,8,10,10a-dodecahydro-phenanthrene-1-carboxylic acid

**Zhen-Dong Zhao, Yu-Xiang Chen, Yu-Min Wang and Liang-Wu Bi**

### S1. Comment

The title compound has been isolated from slash pine rosin. It was identified as isopimaric acid on the basis of the comparison of its physical and spectral data with literature values (Baldwin *et al.*, 1958; Harris *et al.*, 1948). Isopimaric acid exhibits a wide range of biological activities such as antibacterial activity (Smith *et al.*, 2005), BK channels activity (Russo *et al.*, 2007). Although much attention has been paid to the bioactivities of isopimaric acid, the crystal structure of the title compound has not yet been reported. In this work, we describe the crystal structure of the title compound (I).

The molecular structure of (I) is shown in Fig. 1 and its crystal packing in Fig.2. The atoms of C11, C12 in the cyclohexene ring and the atoms C10, C14 in the conjoint cyclohexane ring are in the same plane. The atoms of C11, C12 and the atoms of C16, C17 in the cyclohexane ring are in another plane. The dihedral angle of the two planes is 134.4 degree. The three methyl groups attached to the cyclohexane rings are in axial positions and in the same direction. The molecular conformation is stabilized by C—H···O intra-molecular hydrogen bonding interactions (Table 1). The molecules are dimerized through their carboxyl groups by O—H···O hydrogen bonds, forming $R_2^2(8)$ rings (Fig. 2).

### S2. Experimental

A mixture of slash pine rosin (150 g), acetone (300 ml) and 2-amino-2-methyl-1-propanol (0.1 mol) was stirred at room temperature for 2 h and then filtrated. The residue was recrystallized with 95% ethanol. The crystal obtained from the solution was acidified by 5% hydrochloric acid solution and then dissolved in ether. The solution was washed with water until it was neutral, dryed with sodium sulfate and then concentrated. The residue was recrystallized with acetone and the title compound was obtained as colourless solid.

### S3. Refinement

All H atoms were placed geometrically with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and included in the refinement in riding motion approximation with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$ of the carrier atom. Because of no atom heavier than Si present in the structure, in the absence of significant anomalous dispersion effects, so Friedel pairs were averaged.

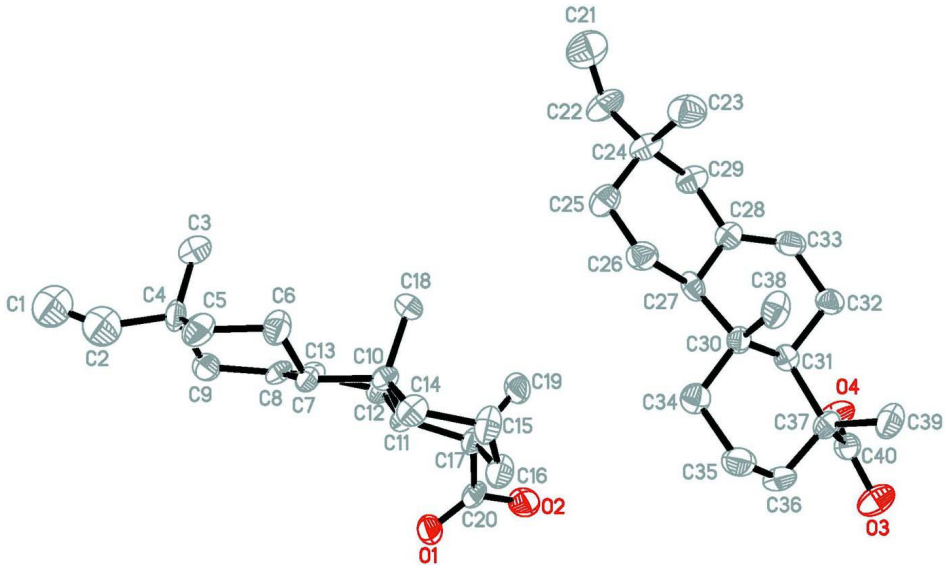


**Figure 1**

A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

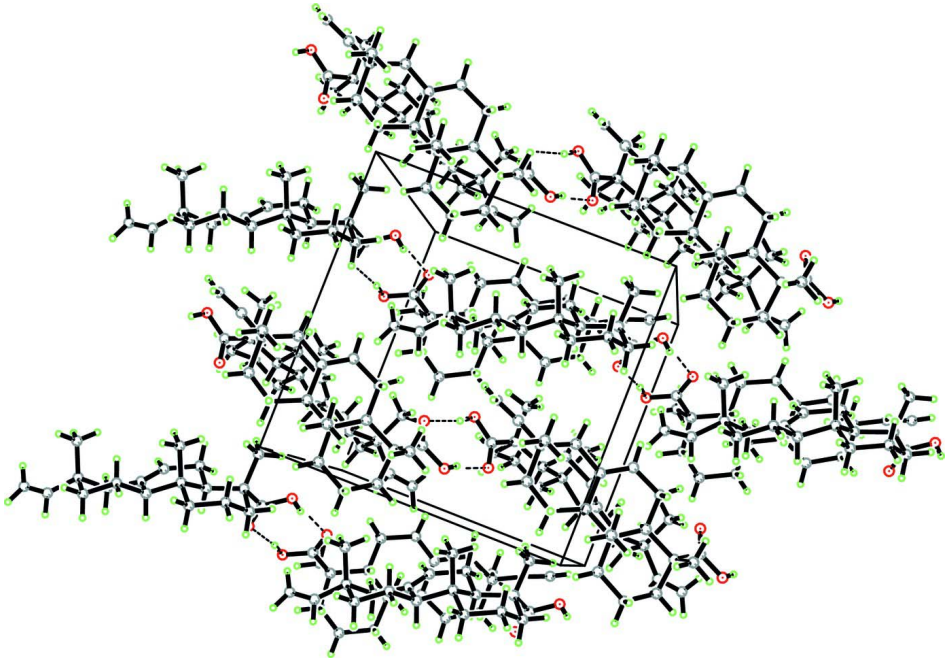

**Figure 2**

A view of the packing of the molecules dimerized through their carboxyl groups by O—H···O hydrogen bonds, forming $R_2^2(8)$ rings.

### 1,4a,7-Trimethyl-7-vinyl-1,2,3,4,4a,4b,5,6,7,8,10,10a- dodecahydrophenanthrene-1-carboxylic acid

*Crystal data*

$C_{20}H_{30}O_2$
$M_r$ = 302.44
Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab
$a$ = 11.624 (2) Å
$b$ = 11.803 (2) Å

$c$ = 25.698 (5) Å
$V$ = 3525.7 (12) Å$^3$
$Z$ = 8
$F$(000) = 1328
$D_x$ = 1.140 Mg m$^{-3}$
Melting point: 436 K
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å

Cell parameters from 25 reflections
$\theta$ = 9–12°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 K
Quadrate, colourless
0.20 × 0.10 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.986, $T_{max}$ = 0.993
7031 measured reflections

6382 independent reflections
2613 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.088
$\theta_{max}$ = 25.3°, $\theta_{min}$ = 1.6°
$h$ = 0→13
$k$ = 0→14
$l$ = −30→30
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.150
$S$ = 1.00
3598 reflections
385 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

*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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.7921 (4) | 0.8907 (4) | 0.96412 (19) | 0.0796 (17) |
| O2 | 0.6441 (4) | 1.0009 (4) | 0.97857 (19) | 0.0816 (16) |
| H2B | 0.6765 | 1.0386 | 0.9561 | 0.122* |
| C1 | 0.9481 (9) | 0.1236 (8) | 1.0193 (3) | 0.127 |
| H1A | 0.8843 | 0.0857 | 1.0060 | 0.152* |
| H1B | 1.0204 | 0.0899 | 1.0178 | 0.152* |
| C2 | 0.9370 (8) | 0.2172 (8) | 1.0385 (3) | 0.110 |
| H2A | 1.0080 | 0.2437 | 1.0499 | 0.132* |
| C3 | 0.7282 (7) | 0.2534 (6) | 1.0540 (3) | 0.088 (3) |

| | | | | |
|---|---|---|---|---|
| H3A | 0.7268 | 0.1988 | 1.0817 | 0.132* |
| H3B | 0.7082 | 0.2170 | 1.0218 | 0.132* |
| H3C | 0.6738 | 0.3126 | 1.0612 | 0.132* |
| C4 | 0.8463 (7) | 0.3031 (6) | 1.0497 (3) | 0.064 (2) |
| C5 | 0.8871 (7) | 0.3611 (6) | 1.0991 (3) | 0.076 (2) |
| H5A | 0.9652 | 0.3881 | 1.0942 | 0.091* |
| H5B | 0.8874 | 0.3070 | 1.1275 | 0.091* |
| C6 | 0.8086 (6) | 0.4615 (5) | 1.1131 (3) | 0.069 (2) |
| H6A | 0.7309 | 0.4342 | 1.1187 | 0.083* |
| H6B | 0.8352 | 0.4957 | 1.1452 | 0.083* |
| C7 | 0.8077 (5) | 0.5512 (5) | 1.0699 (2) | 0.0449 (16) |
| H7A | 0.8864 | 0.5808 | 1.0676 | 0.054* |
| C8 | 0.7810 (6) | 0.4997 (5) | 1.0180 (3) | 0.0496 (17) |
| C9 | 0.8475 (6) | 0.3926 (5) | 1.0056 (3) | 0.066 (2) |
| H9A | 0.9267 | 0.4127 | 0.9981 | 0.080* |
| H9B | 0.8154 | 0.3588 | 0.9744 | 0.080* |
| C10 | 0.7276 (6) | 0.6559 (5) | 1.0837 (2) | 0.0461 (16) |
| C11 | 0.7174 (6) | 0.7258 (5) | 1.0326 (2) | 0.0469 (16) |
| H11A | 0.7965 | 0.7339 | 1.0199 | 0.056* |
| C12 | 0.6560 (6) | 0.6608 (5) | 0.9911 (2) | 0.0579 (19) |
| H12A | 0.6586 | 0.7027 | 0.9586 | 0.070* |
| H12B | 0.5759 | 0.6510 | 1.0007 | 0.070* |
| C13 | 0.7126 (6) | 0.5439 (5) | 0.9835 (3) | 0.0599 (19) |
| H13A | 0.6972 | 0.5035 | 0.9533 | 0.072* |
| C14 | 0.7876 (6) | 0.7254 (5) | 1.1253 (2) | 0.0577 (18) |
| H14A | 0.8674 | 0.7364 | 1.1154 | 0.069* |
| H14B | 0.7864 | 0.6839 | 1.1579 | 0.069* |
| C15 | 0.7310 (7) | 0.8406 (5) | 1.1333 (3) | 0.071 (2) |
| H15A | 0.7709 | 0.8811 | 1.1607 | 0.086* |
| H15B | 0.6518 | 0.8298 | 1.1441 | 0.086* |
| C16 | 0.7336 (7) | 0.9111 (6) | 1.0836 (3) | 0.071 (2) |
| H16A | 0.8129 | 0.9255 | 1.0739 | 0.085* |
| H16B | 0.6968 | 0.9835 | 1.0900 | 0.085* |
| C17 | 0.6704 (6) | 0.8497 (5) | 1.0371 (2) | 0.0523 (18) |
| C18 | 0.6102 (5) | 0.6120 (6) | 1.1030 (3) | 0.0601 (19) |
| H18A | 0.5611 | 0.6751 | 1.1109 | 0.090* |
| H18B | 0.6211 | 0.5670 | 1.1337 | 0.090* |
| H18C | 0.5752 | 0.5665 | 1.0764 | 0.090* |
| C19 | 0.5398 (6) | 0.8563 (6) | 1.0466 (3) | 0.071 (2) |
| H19A | 0.5171 | 0.9341 | 1.0500 | 0.106* |
| H19B | 0.5210 | 0.8162 | 1.0780 | 0.106* |
| H19C | 0.4999 | 0.8225 | 1.0178 | 0.106* |
| C20 | 0.7059 (7) | 0.9151 (5) | 0.9899 (3) | 0.0554 (19) |
| O3 | 0.3506 (4) | 1.4855 (4) | 1.11796 (19) | 0.0782 (16) |
| H3D | 0.3267 | 1.5163 | 1.0915 | 0.117* |
| O4 | 0.2231 (4) | 1.3579 (4) | 1.09334 (19) | 0.0715 (15) |
| C21 | 0.0730 (7) | 0.5800 (8) | 1.1914 (4) | 0.112 (3) |
| H21A | 0.0778 | 0.5751 | 1.2275 | 0.135* |

| | | | | |
|---|---|---|---|---|
| H21B | 0.0414 | 0.5207 | 1.1724 | 0.135* |
| C22 | 0.1091 (7) | 0.6666 (6) | 1.1685 (4) | 0.087 (3) |
| H22A | 0.1017 | 0.6661 | 1.1325 | 0.104* |
| C23 | 0.1430 (6) | 0.7841 (7) | 1.2495 (3) | 0.090 (3) |
| H23A | 0.0621 | 0.7856 | 1.2569 | 0.134* |
| H23B | 0.1774 | 0.8540 | 1.2606 | 0.134* |
| H23C | 0.1781 | 0.7222 | 1.2678 | 0.134* |
| C24 | 0.1616 (7) | 0.7689 (6) | 1.1909 (3) | 0.064 (2) |
| C25 | 0.2897 (6) | 0.7684 (5) | 1.1787 (3) | 0.067 (2) |
| H25A | 0.3248 | 0.7052 | 1.1968 | 0.081* |
| H25B | 0.2993 | 0.7553 | 1.1417 | 0.081* |
| C26 | 0.3541 (6) | 0.8752 (5) | 1.1930 (3) | 0.065 (2) |
| H26A | 0.4335 | 0.8676 | 1.1819 | 0.078* |
| H26B | 0.3539 | 0.8831 | 1.2306 | 0.078* |
| C27 | 0.3037 (5) | 0.9826 (5) | 1.1690 (2) | 0.0489 (17) |
| H27A | 0.3218 | 0.9779 | 1.1318 | 0.059* |
| C28 | 0.1740 (6) | 0.9838 (6) | 1.1720 (2) | 0.0519 (18) |
| C29 | 0.1117 (6) | 0.8732 (5) | 1.1632 (3) | 0.066 (2) |
| H29A | 0.1107 | 0.8581 | 1.1261 | 0.080* |
| H29B | 0.0325 | 0.8825 | 1.1743 | 0.080* |
| C30 | 0.3612 (5) | 1.0954 (5) | 1.1883 (3) | 0.0486 (17) |
| C31 | 0.2935 (5) | 1.1915 (5) | 1.1639 (2) | 0.0467 (17) |
| H31A | 0.2870 | 1.1711 | 1.1270 | 0.056* |
| C32 | 0.1699 (6) | 1.1945 (6) | 1.1837 (3) | 0.0574 (19) |
| H32A | 0.1690 | 1.2197 | 1.2196 | 0.069* |
| H32B | 0.1255 | 1.2480 | 1.1633 | 0.069* |
| C33 | 0.1167 (6) | 1.0799 (6) | 1.1800 (3) | 0.067 (2) |
| H33A | 0.0372 | 1.0752 | 1.1834 | 0.081* |
| C34 | 0.4849 (5) | 1.0947 (6) | 1.1698 (3) | 0.061 (2) |
| H34A | 0.4870 | 1.0785 | 1.1328 | 0.074* |
| H34B | 0.5266 | 1.0351 | 1.1877 | 0.074* |
| C35 | 0.5446 (6) | 1.2105 (6) | 1.1803 (3) | 0.074 (2) |
| H35A | 0.5418 | 1.2283 | 1.2171 | 0.089* |
| H35B | 0.6246 | 1.2073 | 1.1696 | 0.089* |
| C36 | 0.4813 (5) | 1.3000 (6) | 1.1494 (3) | 0.061 (2) |
| H36A | 0.4862 | 1.2810 | 1.1127 | 0.073* |
| H36B | 0.5199 | 1.3721 | 1.1543 | 0.073* |
| C37 | 0.3535 (6) | 1.3142 (5) | 1.1640 (2) | 0.0509 (17) |
| C38 | 0.3546 (6) | 1.0973 (5) | 1.2479 (2) | 0.067 (2) |
| H38A | 0.3887 | 1.1660 | 1.2607 | 0.101* |
| H38B | 0.3954 | 1.0334 | 1.2618 | 0.101* |
| H38C | 0.2755 | 1.0938 | 1.2586 | 0.101* |
| C39 | 0.3395 (7) | 1.3764 (5) | 1.2144 (3) | 0.075 (2) |
| H39A | 0.3785 | 1.4480 | 1.2124 | 0.112* |
| H39B | 0.3719 | 1.3322 | 1.2422 | 0.112* |
| H39C | 0.2592 | 1.3889 | 1.2210 | 0.112* |
| C40 | 0.3037 (6) | 1.3882 (6) | 1.1227 (3) | 0.0529 (18) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.085 (4) | 0.058 (3) | 0.096 (4) | 0.016 (3) | 0.042 (3) | 0.013 (3) |
| O2 | 0.082 (4) | 0.073 (3) | 0.089 (4) | 0.007 (3) | 0.022 (3) | 0.030 (3) |
| C1 | 0.137 | 0.107 | 0.137 | 0.000 | 0.000 | 0.000 |
| C2 | 0.110 | 0.110 | 0.110 | 0.000 | 0.000 | 0.000 |
| C3 | 0.093 (7) | 0.057 (5) | 0.114 (7) | −0.008 (5) | 0.022 (6) | 0.012 (5) |
| C4 | 0.079 (6) | 0.043 (4) | 0.070 (5) | 0.026 (4) | 0.023 (5) | 0.006 (4) |
| C5 | 0.094 (6) | 0.058 (5) | 0.076 (5) | 0.002 (5) | −0.002 (5) | 0.019 (5) |
| C6 | 0.087 (6) | 0.056 (4) | 0.064 (5) | 0.017 (4) | 0.005 (4) | 0.005 (4) |
| C7 | 0.049 (4) | 0.035 (3) | 0.051 (4) | 0.001 (3) | 0.000 (3) | 0.000 (3) |
| C8 | 0.063 (4) | 0.031 (3) | 0.054 (4) | −0.004 (4) | −0.001 (4) | 0.014 (3) |
| C9 | 0.060 (5) | 0.057 (4) | 0.082 (5) | −0.007 (4) | 0.012 (4) | −0.015 (4) |
| C10 | 0.052 (4) | 0.041 (3) | 0.046 (4) | −0.002 (4) | −0.003 (3) | 0.005 (3) |
| C11 | 0.057 (4) | 0.036 (3) | 0.048 (4) | −0.004 (3) | −0.004 (4) | 0.004 (3) |
| C12 | 0.073 (5) | 0.049 (4) | 0.052 (4) | 0.004 (4) | −0.005 (4) | 0.015 (3) |
| C13 | 0.073 (5) | 0.047 (4) | 0.059 (5) | −0.012 (4) | −0.004 (4) | −0.005 (4) |
| C14 | 0.070 (5) | 0.059 (4) | 0.044 (4) | 0.000 (4) | −0.009 (4) | 0.005 (4) |
| C15 | 0.101 (6) | 0.055 (4) | 0.058 (5) | 0.020 (5) | 0.000 (4) | −0.004 (4) |
| C16 | 0.088 (6) | 0.056 (4) | 0.068 (5) | 0.010 (5) | 0.005 (4) | −0.002 (4) |
| C17 | 0.072 (5) | 0.039 (4) | 0.045 (4) | 0.003 (4) | 0.008 (4) | −0.003 (3) |
| C18 | 0.052 (4) | 0.062 (5) | 0.066 (5) | −0.001 (4) | 0.019 (4) | 0.000 (4) |
| C19 | 0.072 (5) | 0.062 (5) | 0.078 (5) | 0.025 (4) | 0.016 (4) | 0.022 (4) |
| C20 | 0.070 (5) | 0.033 (4) | 0.064 (5) | 0.008 (4) | −0.002 (4) | 0.018 (4) |
| O3 | 0.097 (4) | 0.057 (3) | 0.080 (4) | −0.014 (3) | −0.028 (3) | 0.019 (3) |
| O4 | 0.075 (3) | 0.057 (3) | 0.083 (3) | −0.011 (3) | −0.036 (3) | 0.025 (3) |
| C21 | 0.121 (8) | 0.099 (7) | 0.116 (8) | −0.031 (7) | −0.020 (7) | −0.016 (7) |
| C22 | 0.092 (7) | 0.055 (5) | 0.113 (8) | −0.025 (5) | −0.032 (6) | 0.006 (5) |
| C23 | 0.096 (6) | 0.091 (6) | 0.082 (6) | −0.002 (6) | 0.001 (5) | 0.023 (5) |
| C24 | 0.075 (6) | 0.052 (5) | 0.065 (5) | −0.016 (4) | −0.012 (4) | 0.010 (4) |
| C25 | 0.075 (6) | 0.055 (5) | 0.072 (5) | −0.005 (4) | −0.017 (5) | −0.004 (4) |
| C26 | 0.051 (4) | 0.065 (5) | 0.079 (5) | −0.003 (4) | −0.004 (4) | 0.007 (4) |
| C27 | 0.045 (4) | 0.062 (4) | 0.039 (4) | 0.022 (4) | 0.003 (3) | 0.009 (3) |
| C28 | 0.061 (5) | 0.050 (4) | 0.044 (4) | 0.002 (4) | −0.008 (3) | 0.007 (4) |
| C29 | 0.073 (5) | 0.060 (5) | 0.066 (5) | −0.013 (4) | −0.007 (4) | 0.004 (4) |
| C30 | 0.045 (4) | 0.045 (4) | 0.056 (4) | 0.002 (3) | 0.000 (4) | −0.005 (3) |
| C31 | 0.043 (4) | 0.053 (4) | 0.044 (4) | 0.001 (3) | −0.003 (3) | −0.004 (3) |
| C32 | 0.058 (5) | 0.057 (5) | 0.057 (4) | 0.003 (4) | 0.012 (4) | 0.014 (4) |
| C33 | 0.055 (5) | 0.065 (5) | 0.082 (6) | 0.000 (4) | 0.016 (4) | 0.023 (5) |
| C34 | 0.060 (5) | 0.062 (5) | 0.062 (5) | −0.002 (4) | −0.005 (4) | −0.003 (4) |
| C35 | 0.056 (5) | 0.073 (5) | 0.092 (6) | −0.006 (4) | 0.007 (5) | 0.011 (5) |
| C36 | 0.053 (5) | 0.052 (4) | 0.076 (5) | −0.015 (4) | −0.010 (4) | 0.014 (4) |
| C37 | 0.060 (4) | 0.045 (4) | 0.048 (4) | −0.004 (4) | −0.009 (4) | −0.001 (3) |
| C38 | 0.103 (6) | 0.055 (4) | 0.043 (4) | 0.011 (5) | −0.008 (4) | −0.004 (4) |
| C39 | 0.104 (6) | 0.051 (4) | 0.070 (5) | 0.001 (5) | 0.000 (5) | −0.011 (4) |
| C40 | 0.050 (4) | 0.052 (5) | 0.057 (4) | 0.003 (4) | −0.002 (4) | 0.003 (4) |

*Geometric parameters (Å, º)*

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| O1—C20 | 1.235 (7) | O3—C40 | 1.278 (7) |
| O2—C20 | 1.276 (7) | O3—H3D | 0.8200 |
| O2—H2B | 0.8200 | O4—C40 | 1.256 (7) |
| C1—C2 | 1.217 (8) | C21—C22 | 1.252 (10) |
| C1—H1A | 0.9300 | C21—H21A | 0.9300 |
| C1—H1B | 0.9300 | C21—H21B | 0.9300 |
| C2—C4 | 1.491 (10) | C22—C24 | 1.470 (9) |
| C2—H2A | 0.9300 | C22—H22A | 0.9300 |
| C3—C4 | 1.496 (9) | C23—C24 | 1.532 (10) |
| C3—H3A | 0.9600 | C23—H23A | 0.9600 |
| C3—H3B | 0.9600 | C23—H23B | 0.9600 |
| C3—H3C | 0.9600 | C23—H23C | 0.9600 |
| C4—C5 | 1.518 (10) | C24—C25 | 1.522 (10) |
| C4—C9 | 1.549 (9) | C24—C29 | 1.536 (9) |
| C5—C6 | 1.538 (9) | C25—C26 | 1.511 (8) |
| C5—H5A | 0.9700 | C25—H25A | 0.9700 |
| C5—H5B | 0.9700 | C25—H25B | 0.9700 |
| C6—C7 | 1.534 (8) | C26—C27 | 1.528 (8) |
| C6—H6A | 0.9700 | C26—H26A | 0.9700 |
| C6—H6B | 0.9700 | C26—H26B | 0.9700 |
| C7—C8 | 1.498 (8) | C27—C28 | 1.510 (8) |
| C7—C10 | 1.587 (8) | C27—C30 | 1.570 (8) |
| C7—H7A | 0.9800 | C27—H27A | 0.9800 |
| C8—C13 | 1.299 (8) | C28—C33 | 1.331 (8) |
| C8—C9 | 1.516 (8) | C28—C29 | 1.510 (8) |
| C9—H9A | 0.9700 | C29—H29A | 0.9700 |
| C9—H9B | 0.9700 | C29—H29B | 0.9700 |
| C10—C14 | 1.517 (8) | C30—C34 | 1.514 (8) |
| C10—C18 | 1.542 (8) | C30—C31 | 1.516 (8) |
| C10—C11 | 1.555 (7) | C30—C38 | 1.536 (8) |
| C11—C12 | 1.496 (8) | C31—C32 | 1.525 (8) |
| C11—C17 | 1.565 (8) | C31—C37 | 1.607 (8) |
| C11—H11A | 0.9800 | C31—H31A | 0.9800 |
| C12—C13 | 1.541 (8) | C32—C33 | 1.491 (9) |
| C12—H12A | 0.9700 | C32—H32A | 0.9700 |
| C12—H12B | 0.9700 | C32—H32B | 0.9700 |
| C13—H13A | 0.9300 | C33—H33A | 0.9300 |
| C14—C15 | 1.524 (8) | C34—C35 | 1.556 (8) |
| C14—H14A | 0.9700 | C34—H34A | 0.9700 |
| C14—H14B | 0.9700 | C34—H34B | 0.9700 |
| C15—C16 | 1.525 (9) | C35—C36 | 1.514 (9) |
| C15—H15A | 0.9700 | C35—H35A | 0.9700 |
| C15—H15B | 0.9700 | C35—H35B | 0.9700 |
| C16—C17 | 1.578 (9) | C36—C37 | 1.541 (9) |
| C16—H16A | 0.9700 | C36—H36A | 0.9700 |
| C16—H16B | 0.9700 | C36—H36B | 0.9700 |

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|---|---|---|---|
| C17—C20 | 1.496 (9) | C37—C40 | 1.491 (8) |
| C17—C19 | 1.540 (8) | C37—C39 | 1.498 (8) |
| C18—H18A | 0.9600 | C38—H38A | 0.9600 |
| C18—H18B | 0.9600 | C38—H38B | 0.9600 |
| C18—H18C | 0.9600 | C38—H38C | 0.9600 |
| C19—H19A | 0.9600 | C39—H39A | 0.9600 |
| C19—H19B | 0.9600 | C39—H39B | 0.9600 |
| C19—H19C | 0.9600 | C39—H39C | 0.9600 |
| | | | |
| C20—O2—H2B | 109.5 | C40—O3—H3D | 109.5 |
| C2—C1—H1A | 120.0 | C22—C21—H21A | 120.0 |
| C2—C1—H1B | 120.0 | C22—C21—H21B | 120.0 |
| H1A—C1—H1B | 120.0 | H21A—C21—H21B | 120.0 |
| C1—C2—C4 | 140.4 (11) | C21—C22—C24 | 128.7 (9) |
| C1—C2—H2A | 109.8 | C21—C22—H22A | 115.6 |
| C4—C2—H2A | 109.8 | C24—C22—H22A | 115.6 |
| C4—C3—H3A | 109.5 | C24—C23—H23A | 109.5 |
| C4—C3—H3B | 109.5 | C24—C23—H23B | 109.5 |
| H3A—C3—H3B | 109.5 | H23A—C23—H23B | 109.5 |
| C4—C3—H3C | 109.5 | C24—C23—H23C | 109.5 |
| H3A—C3—H3C | 109.5 | H23A—C23—H23C | 109.5 |
| H3B—C3—H3C | 109.5 | H23B—C23—H23C | 109.5 |
| C2—C4—C3 | 113.4 (7) | C22—C24—C25 | 108.8 (7) |
| C2—C4—C5 | 104.3 (7) | C22—C24—C23 | 115.0 (7) |
| C3—C4—C5 | 113.7 (6) | C25—C24—C23 | 109.9 (6) |
| C2—C4—C9 | 108.4 (6) | C22—C24—C29 | 108.7 (6) |
| C3—C4—C9 | 109.2 (6) | C25—C24—C29 | 106.1 (6) |
| C5—C4—C9 | 107.5 (6) | C23—C24—C29 | 108.0 (6) |
| C4—C5—C6 | 111.0 (6) | C26—C25—C24 | 115.6 (6) |
| C4—C5—H5A | 109.4 | C26—C25—H25A | 108.4 |
| C6—C5—H5A | 109.4 | C24—C25—H25A | 108.4 |
| C4—C5—H5B | 109.4 | C26—C25—H25B | 108.4 |
| C6—C5—H5B | 109.4 | C24—C25—H25B | 108.4 |
| H5A—C5—H5B | 108.0 | H25A—C25—H25B | 107.5 |
| C7—C6—C5 | 111.5 (5) | C25—C26—C27 | 113.8 (6) |
| C7—C6—H6A | 109.3 | C25—C26—H26A | 108.8 |
| C5—C6—H6A | 109.3 | C27—C26—H26A | 108.8 |
| C7—C6—H6B | 109.3 | C25—C26—H26B | 108.8 |
| C5—C6—H6B | 109.3 | C27—C26—H26B | 108.8 |
| H6A—C6—H6B | 108.0 | H26A—C26—H26B | 107.7 |
| C8—C7—C6 | 111.4 (5) | C28—C27—C26 | 111.7 (6) |
| C8—C7—C10 | 113.1 (5) | C28—C27—C30 | 113.6 (5) |
| C6—C7—C10 | 112.3 (5) | C26—C27—C30 | 114.4 (5) |
| C8—C7—H7A | 106.5 | C28—C27—H27A | 105.4 |
| C6—C7—H7A | 106.5 | C26—C27—H27A | 105.4 |
| C10—C7—H7A | 106.5 | C30—C27—H27A | 105.4 |
| C13—C8—C7 | 124.8 (6) | C33—C28—C29 | 121.3 (6) |
| C13—C8—C9 | 120.2 (7) | C33—C28—C27 | 121.0 (7) |

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| C7—C8—C9 | 114.8 (6) | C29—C28—C27 | 117.6 (6) |
| C8—C9—C4 | 114.2 (5) | C28—C29—C24 | 116.2 (6) |
| C8—C9—H9A | 108.7 | C28—C29—H29A | 108.2 |
| C4—C9—H9A | 108.7 | C24—C29—H29A | 108.2 |
| C8—C9—H9B | 108.7 | C28—C29—H29B | 108.2 |
| C4—C9—H9B | 108.7 | C24—C29—H29B | 108.2 |
| H9A—C9—H9B | 107.6 | H29A—C29—H29B | 107.4 |
| C14—C10—C18 | 111.2 (6) | C34—C30—C31 | 111.6 (6) |
| C14—C10—C11 | 110.0 (5) | C34—C30—C38 | 111.1 (6) |
| C18—C10—C11 | 112.4 (5) | C31—C30—C38 | 112.0 (5) |
| C14—C10—C7 | 108.0 (5) | C34—C30—C27 | 107.5 (5) |
| C18—C10—C7 | 109.3 (5) | C31—C30—C27 | 106.5 (5) |
| C11—C10—C7 | 105.6 (5) | C38—C30—C27 | 107.9 (5) |
| C12—C11—C10 | 111.5 (5) | C30—C31—C32 | 111.6 (6) |
| C12—C11—C17 | 111.4 (5) | C30—C31—C37 | 116.7 (5) |
| C10—C11—C17 | 117.4 (5) | C32—C31—C37 | 112.7 (5) |
| C12—C11—H11A | 105.1 | C30—C31—H31A | 104.8 |
| C10—C11—H11A | 105.1 | C32—C31—H31A | 104.8 |
| C17—C11—H11A | 105.1 | C37—C31—H31A | 104.8 |
| C11—C12—C13 | 110.2 (5) | C33—C32—C31 | 110.4 (6) |
| C11—C12—H12A | 109.6 | C33—C32—H32A | 109.6 |
| C13—C12—H12A | 109.6 | C31—C32—H32A | 109.6 |
| C11—C12—H12B | 109.6 | C33—C32—H32B | 109.6 |
| C13—C12—H12B | 109.6 | C31—C32—H32B | 109.6 |
| H12A—C12—H12B | 108.1 | H32A—C32—H32B | 108.1 |
| C8—C13—C12 | 122.3 (6) | C28—C33—C32 | 125.2 (7) |
| C8—C13—H13A | 118.8 | C28—C33—H33A | 117.4 |
| C12—C13—H13A | 118.8 | C32—C33—H33A | 117.4 |
| C10—C14—C15 | 112.3 (6) | C30—C34—C35 | 111.4 (6) |
| C10—C14—H14A | 109.2 | C30—C34—H34A | 109.3 |
| C15—C14—H14A | 109.2 | C35—C34—H34A | 109.3 |
| C10—C14—H14B | 109.2 | C30—C34—H34B | 109.3 |
| C15—C14—H14B | 109.2 | C35—C34—H34B | 109.3 |
| H14A—C14—H14B | 107.9 | H34A—C34—H34B | 108.0 |
| C14—C15—C16 | 111.4 (6) | C36—C35—C34 | 107.8 (6) |
| C14—C15—H15A | 109.3 | C36—C35—H35A | 110.1 |
| C16—C15—H15A | 109.3 | C34—C35—H35A | 110.1 |
| C14—C15—H15B | 109.3 | C36—C35—H35B | 110.1 |
| C16—C15—H15B | 109.3 | C34—C35—H35B | 110.1 |
| H15A—C15—H15B | 108.0 | H35A—C35—H35B | 108.5 |
| C15—C16—C17 | 112.0 (6) | C35—C36—C37 | 114.6 (6) |
| C15—C16—H16A | 109.2 | C35—C36—H36A | 108.6 |
| C17—C16—H16A | 109.2 | C37—C36—H36A | 108.6 |
| C15—C16—H16B | 109.2 | C35—C36—H36B | 108.6 |
| C17—C16—H16B | 109.2 | C37—C36—H36B | 108.6 |
| H16A—C16—H16B | 107.9 | H36A—C36—H36B | 107.6 |
| C20—C17—C19 | 112.0 (6) | C40—C37—C39 | 106.6 (5) |
| C20—C17—C11 | 109.0 (5) | C40—C37—C36 | 105.3 (6) |

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| C19—C17—C11 | 113.8 (6) | C39—C37—C36 | 111.6 (6) |
| C20—C17—C16 | 104.3 (5) | C40—C37—C31 | 111.0 (5) |
| C19—C17—C16 | 108.4 (6) | C39—C37—C31 | 113.4 (6) |
| C11—C17—C16 | 108.8 (6) | C36—C37—C31 | 108.6 (5) |
| C10—C18—H18A | 109.5 | C30—C38—H38A | 109.5 |
| C10—C18—H18B | 109.5 | C30—C38—H38B | 109.5 |
| H18A—C18—H18B | 109.5 | H38A—C38—H38B | 109.5 |
| C10—C18—H18C | 109.5 | C30—C38—H38C | 109.5 |
| H18A—C18—H18C | 109.5 | H38A—C38—H38C | 109.5 |
| H18B—C18—H18C | 109.5 | H38B—C38—H38C | 109.5 |
| C17—C19—H19A | 109.5 | C37—C39—H39A | 109.5 |
| C17—C19—H19B | 109.5 | C37—C39—H39B | 109.5 |
| H19A—C19—H19B | 109.5 | H39A—C39—H39B | 109.5 |
| C17—C19—H19C | 109.5 | C37—C39—H39C | 109.5 |
| H19A—C19—H19C | 109.5 | H39A—C39—H39C | 109.5 |
| H19B—C19—H19C | 109.5 | H39B—C39—H39C | 109.5 |
| O1—C20—O2 | 121.3 (7) | O4—C40—O3 | 121.1 (7) |
| O1—C20—C17 | 122.6 (6) | O4—C40—C37 | 123.4 (7) |
| O2—C20—C17 | 116.1 (7) | O3—C40—C37 | 115.4 (6) |

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| C1—C2—C4—C3 | 24.1 (17) | C21—C22—C24—C25 | 107.4 (11) |
| C1—C2—C4—C5 | 148.3 (13) | C21—C22—C24—C23 | −16.4 (14) |
| C1—C2—C4—C9 | −97.4 (14) | C21—C22—C24—C29 | −137.6 (10) |
| C2—C4—C5—C6 | 174.2 (6) | C22—C24—C25—C26 | 172.6 (6) |
| C3—C4—C5—C6 | −61.8 (7) | C23—C24—C25—C26 | −60.7 (9) |
| C9—C4—C5—C6 | 59.2 (8) | C29—C24—C25—C26 | 55.8 (8) |
| C4—C5—C6—C7 | −60.6 (8) | C24—C25—C26—C27 | −55.4 (9) |
| C5—C6—C7—C8 | 52.2 (8) | C25—C26—C27—C28 | 42.9 (8) |
| C5—C6—C7—C10 | −179.8 (6) | C25—C26—C27—C30 | 173.7 (6) |
| C6—C7—C8—C13 | 137.7 (7) | C26—C27—C28—C33 | 144.3 (7) |
| C10—C7—C8—C13 | 10.1 (9) | C30—C27—C28—C33 | 13.1 (9) |
| C6—C7—C8—C9 | −46.6 (7) | C26—C27—C28—C29 | −38.6 (8) |
| C10—C7—C8—C9 | −174.3 (5) | C30—C27—C28—C29 | −169.8 (5) |
| C13—C8—C9—C4 | −135.3 (7) | C33—C28—C29—C24 | −138.2 (7) |
| C7—C8—C9—C4 | 48.8 (8) | C27—C28—C29—C24 | 44.7 (9) |
| C2—C4—C9—C8 | −165.8 (6) | C22—C24—C29—C28 | −166.6 (7) |
| C3—C4—C9—C8 | 70.2 (8) | C25—C24—C29—C28 | −49.7 (8) |
| C5—C4—C9—C8 | −53.6 (8) | C23—C24—C29—C28 | 68.1 (8) |
| C8—C7—C10—C14 | −159.8 (5) | C28—C27—C30—C34 | −165.1 (5) |
| C6—C7—C10—C14 | 73.1 (6) | C26—C27—C30—C34 | 65.1 (7) |
| C8—C7—C10—C18 | 79.1 (6) | C28—C27—C30—C31 | −45.4 (7) |
| C6—C7—C10—C18 | −48.0 (7) | C26—C27—C30—C31 | −175.2 (6) |
| C8—C7—C10—C11 | −42.0 (7) | C28—C27—C30—C38 | 75.0 (6) |
| C6—C7—C10—C11 | −169.2 (5) | C26—C27—C30—C38 | −54.8 (7) |
| C14—C10—C11—C12 | −178.6 (5) | C34—C30—C31—C32 | −178.2 (5) |
| C18—C10—C11—C12 | −54.0 (7) | C38—C30—C31—C32 | −52.9 (7) |
| C7—C10—C11—C12 | 65.1 (7) | C27—C30—C31—C32 | 64.8 (7) |
| C14—C10—C11—C17 | −48.3 (8) | C34—C30—C31—C37 | −46.6 (8) |

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|---|---|---|---|
| C18—C10—C11—C17 | 76.3 (7) | C38—C30—C31—C37 | 78.7 (7) |
| C7—C10—C11—C17 | −164.6 (5) | C27—C30—C31—C37 | −163.6 (5) |
| C10—C11—C12—C13 | −52.7 (7) | C30—C31—C32—C33 | −49.6 (7) |
| C17—C11—C12—C13 | 173.9 (5) | C37—C31—C32—C33 | 176.9 (5) |
| C7—C8—C13—C12 | 4.0 (10) | C29—C28—C33—C32 | −173.5 (7) |
| C9—C8—C13—C12 | −171.4 (6) | C27—C28—C33—C32 | 3.5 (11) |
| C11—C12—C13—C8 | 17.2 (9) | C31—C32—C33—C28 | 14.2 (10) |
| C18—C10—C14—C15 | −72.3 (7) | C31—C30—C34—C35 | 55.7 (8) |
| C11—C10—C14—C15 | 53.0 (8) | C38—C30—C34—C35 | −70.2 (7) |
| C7—C10—C14—C15 | 167.9 (5) | C27—C30—C34—C35 | 172.0 (5) |
| C10—C14—C15—C16 | −60.5 (8) | C30—C34—C35—C36 | −62.2 (8) |
| C14—C15—C16—C17 | 58.8 (9) | C34—C35—C36—C37 | 61.0 (8) |
| C12—C11—C17—C20 | −70.0 (7) | C35—C36—C37—C40 | −169.2 (6) |
| C10—C11—C17—C20 | 159.7 (6) | C35—C36—C37—C39 | 75.5 (8) |
| C12—C11—C17—C19 | 55.8 (7) | C35—C36—C37—C31 | −50.3 (7) |
| C10—C11—C17—C19 | −74.5 (8) | C30—C31—C37—C40 | 157.9 (6) |
| C12—C11—C17—C16 | 176.8 (5) | C32—C31—C37—C40 | −71.0 (7) |
| C10—C11—C17—C16 | 46.5 (8) | C30—C31—C37—C39 | −82.1 (7) |
| C15—C16—C17—C20 | −166.5 (6) | C32—C31—C37—C39 | 49.0 (8) |
| C15—C16—C17—C19 | 74.0 (7) | C30—C31—C37—C36 | 42.5 (7) |
| C15—C16—C17—C11 | −50.2 (8) | C32—C31—C37—C36 | 173.6 (5) |
| C19—C17—C20—O1 | −152.8 (7) | C39—C37—C40—O4 | −121.8 (7) |
| C11—C17—C20—O1 | −26.0 (10) | C36—C37—C40—O4 | 119.5 (7) |
| C16—C17—C20—O1 | 90.1 (8) | C31—C37—C40—O4 | 2.2 (9) |
| C19—C17—C20—O2 | 29.6 (9) | C39—C37—C40—O3 | 59.8 (8) |
| C11—C17—C20—O2 | 156.4 (6) | C36—C37—C40—O3 | −58.9 (7) |
| C16—C17—C20—O2 | −87.4 (7) | C31—C37—C40—O3 | −176.3 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*B*···O4$^{i}$ | 0.82 | 1.84 | 2.653 (7) | 168 |
| O3—H3*D*···O1$^{ii}$ | 0.82 | 1.85 | 2.655 (7) | 168 |
| C11—H11*A*···O1 | 0.98 | 2.34 | 2.764 (7) | 105 |
| C32—H32*B*···O4 | 0.97 | 2.49 | 3.081 (9) | 119 |

Symmetry codes: (i) *x*+1/2, −*y*+5/2, −*z*+2; (ii) *x*−1/2, −*y*+5/2, −*z*+2.