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# Dehydroabietic acid

**Xiao-Ping Rao,* Zhan-Qian Song and Shi-Bin Shang**

Institute of Chemical Industry of Forest Products, Chinese Academy of Forestry, Nanjing 210042, People's Republic of China
Correspondence e-mail: rxping2001@163.com

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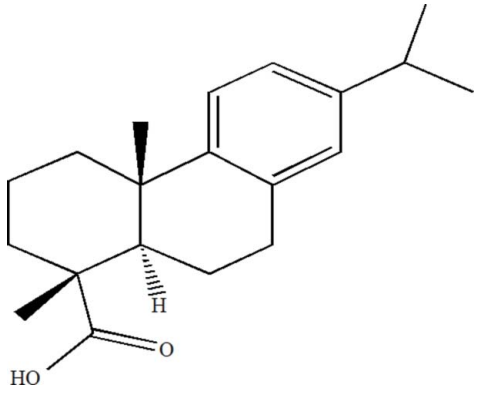
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.010 Å; $R$ factor = 0.077; $wR$ factor = 0.182; data-to-parameter ratio = 9.5.

The title compound [systematic name: (1*R*,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene-1-carboxylic acid], $C_{20}H_{28}O_2$, has been isolated from disproportionated rosin which is obtained by isomerizing gum rosin with a Pd-C catalyst.. Two crystallographically independent molecules exist in the asymmetric unit. In each molecule, there are three six-membered rings, which adopt planar, half-chair and chair conformations. The two cyclohexane rings form a *trans* ring junction with the two methyl groups in axial positions. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

## Related literature

For the synthesis and uses of dehydroabietic acid, see: Halbrook & Lawrence (1966); Jia *et al.* (2009); Piispanen *et al.* (2001); Rao *et al.* (2006); Rao, Song & He (2008); Rao, Song, He & Jia (2008); Sepulveda *et al.* (2005); Wada *et al.* (1985).




## Experimental

### Crystal data

$C_{20}H_{28}O_2$
$M_r$ = 300.42
Monoclinic, $P2_1$
$a$ = 11.738 (2) Å
$b$ = 11.875 (2) Å
$c$ = 13.654 (3) Å
$\beta$ = 107.50 (3)°
$V$ = 1815.1 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 K
0.40 × 0.20 × 0.20 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (*XCAD4*; Harms & Wocadlo, 1995)
$T_{min}$ = 0.973, $T_{max}$ = 0.986
3592 measured reflections
3417 independent reflections
2173 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.086
3 standard reflections
every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.182
$S$ = 1.00
3417 reflections
361 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.52 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$D$···O3$^{i}$ | 0.82 | 1.82 | 2.621 (8) | 165 |
| O4—H4$A$···O1$^{ii}$ | 0.82 | 1.79 | 2.598 (8) | 168 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); 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: AT2868).

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# supplementary materials

*Acta Cryst.* (2009). E**65**, o2402 [ **doi:10.1107/S1600536809035600** ]

# Dehydroabietic acid

**X.-P. Rao, Z.-Q. Song and S.-B. Shang**

## Comment

Dehydroabietic acid is an abietane diterpenic resin acid which can be easily obtained from Pinus resin or commercial disproportionated rosin (Halbrook & Lawrence, 1966). The tri-cyclic hydrophenanthrene structure of dehydroabietic acid has strong hydrophobicity, so it can be used as raw material for the synthesis of surfactants (Piispanen *et al.*, 2001; Jia *et al.*, 2009). Dehydroabietic acid is also widely used as starting material for design and synthesis of biological compounds (Sepulveda *et al.*, 2005; Rao, Song & He, 2008; Rao, Song He & Jia, 2008; Wada *et al.*, 1985). In this work, we describe the crystal structure of the title compound.

The overall geometry of the title compound (Fig. 1) is comparable to that found for dehydroabietic *N*-methyl anilide (Rao *et al.,* 2006) Two crystallorgraphic independent molecules exist in the asymmetric unit. In each molecule there are three six-membered rings, in which they form plannar, half-chair and chair conformations, respectively. The tricyclo phenanthrene structure of the title compound exhibited the same conformation with dehydroabietic *N*-methyl anilide. The two cyclohexane rings form a *trans* ring junction with two methyl groups in the same side of tricyclo phenanthrene structure. There are three chiral centers in each molecule, they exhibited R–, S– and R– configurations, respectively.

The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

## Experimental

The title compound wasere isolated from disproportionated rosin by recrystallization 5 times from acetone. Single crystals were grown from acetone.

## Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.96Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms, and C—H = 0.97–0.98Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for all other H atoms.

## Figures

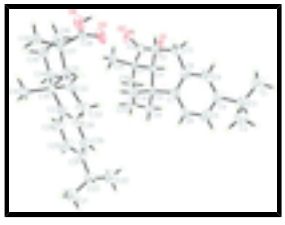


Fig. 1. The molecular structure of the title compound, showing the atom labelling scheme. H atoms are represented by spheres of arbitrary radius and displacement ellipsoids are drawn at the 30% probability level.

**(1*R*,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl- 1,2,3,4,4a,9,10,10a-octahydrophenanthrene-1-carboxylic acid**

*Crystal data*

| | |
|---|---|
| $C_{20}H_{28}O_2$ | $F_{000}$ = 656 |
| $M_r$ = 300.42 | $D_x$ = 1.099 Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| Hall symbol: P 2yb | Cell parameters from 25 reflections |
| $a$ = 11.738 (2) Å | θ = 9–13º |
| $b$ = 11.875 (2) Å | μ = 0.07 mm$^{-1}$ |
| $c$ = 13.654 (3) Å | $T$ = 293 K |
| β = 107.50 (3)º | Block, white |
| $V$ = 1815.1 (6) Å$^3$ | 0.40 × 0.20 × 0.20 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $R_{int}$ = 0.086 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 25.2º |
| Monochromator: graphite | $\theta_{min}$ = 1.6º |
| $T$ = 293 K | $h$ = 0→14 |
| ω/2θ scans | $k$ = 0→14 |
| Absorption correction: ψ scan (XCAD4; Harms & Wocadlo, 1995) | $l$ = −16→15 |
| $T_{min}$ = 0.973, $T_{max}$ = 0.986 | 3 standard reflections |
| 3592 measured reflections | every 200 reflections |
| 3417 independent reflections | intensity decay: 1% |
| 2173 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.077 | H-atom parameters constrained |
| $wR(F^2)$ = 0.182 | $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.6P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.00 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3417 reflections | $\Delta\rho_{max}$ = 0.24 e Å$^{-3}$ |
| 361 parameters | $\Delta\rho_{min}$ = −0.52 e Å$^{-3}$ |
| 3 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.7903 (4) | 0.0770 (5) | 0.5980 (5) | 0.111 (2) |
| O2 | 0.6194 (4) | −0.0132 (5) | 0.5509 (4) | 0.0981 (18) |
| H2D | 0.6584 | −0.0487 | 0.5206 | 0.147* |
| C1 | 1.0216 (7) | 0.7319 (9) | 0.7227 (6) | 0.105 |
| H1A | 1.0473 | 0.6592 | 0.7068 | 0.158* |
| H1B | 1.0879 | 0.7832 | 0.7385 | 0.158* |
| H1C | 0.9594 | 0.7594 | 0.6646 | 0.158* |
| C2 | 0.9229 (7) | 0.8140 (7) | 0.8668 (6) | 0.094 |
| H2A | 0.8972 | 0.7796 | 0.9202 | 0.141* |
| H2B | 0.8559 | 0.8493 | 0.8180 | 0.141* |
| H2C | 0.9827 | 0.8697 | 0.8961 | 0.141* |
| C3 | 0.9762 (7) | 0.7223 (7) | 0.8115 (5) | 0.089 |
| H3A | 1.0471 | 0.6976 | 0.8660 | 0.107* |
| C4 | 0.8968 (8) | 0.6219 (7) | 0.7975 (7) | 0.094 (2) |
| C5 | 0.9072 (8) | 0.5624 (8) | 0.8875 (7) | 0.104 (3) |
| H5A | 0.9543 | 0.5913 | 0.9499 | 0.125* |
| C6 | 0.8482 (6) | 0.4603 (7) | 0.8856 (5) | 0.079 (2) |
| H6A | 0.8596 | 0.4206 | 0.9465 | 0.095* |
| C7 | 0.7739 (5) | 0.4168 (5) | 0.7964 (4) | 0.0580 (15) |
| C8 | 0.7635 (6) | 0.4722 (6) | 0.7055 (5) | 0.0660 (16) |
| C9 | 0.8238 (7) | 0.5731 (6) | 0.7076 (6) | 0.085 (2) |
| H9A | 0.8151 | 0.6101 | 0.6457 | 0.102* |
| C10 | 0.7026 (5) | 0.3060 (5) | 0.7991 (4) | 0.0554 (15) |
| C11 | 0.6944 (4) | 0.2442 (5) | 0.6962 (4) | 0.0463 (13) |
| H11A | 0.7771 | 0.2384 | 0.6946 | 0.056* |
| C12 | 0.6319 (5) | 0.3137 (6) | 0.6046 (4) | 0.0636 (16) |
| H12A | 0.5501 | 0.3259 | 0.6042 | 0.076* |
| H12B | 0.6299 | 0.2722 | 0.5429 | 0.076* |
| C13 | 0.6904 (7) | 0.4267 (7) | 0.6020 (5) | 0.085 (2) |
| H13A | 0.6285 | 0.4810 | 0.5704 | 0.101* |
| H13B | 0.7419 | 0.4203 | 0.5585 | 0.101* |
| C14 | 0.7656 (6) | 0.2326 (6) | 0.8889 (4) | 0.0674 (17) |
| H14A | 0.7627 | 0.2688 | 0.9518 | 0.081* |

| | | | | |
|---|---|---|---|---|
| H14B | 0.8489 | 0.2255 | 0.8917 | 0.081* |
| C15 | 0.7105 (7) | 0.1152 (6) | 0.8826 (5) | 0.084 (2) |
| H15A | 0.7548 | 0.0714 | 0.9418 | 0.101* |
| H15B | 0.6288 | 0.1217 | 0.8844 | 0.101* |
| C16 | 0.7117 (6) | 0.0560 (6) | 0.7883 (6) | 0.081 (2) |
| H16A | 0.6743 | −0.0170 | 0.7869 | 0.097* |
| H16B | 0.7940 | 0.0434 | 0.7903 | 0.097* |
| C17 | 0.6479 (4) | 0.1187 (6) | 0.6895 (4) | 0.0549 (15) |
| C19 | 0.5766 (5) | 0.3435 (7) | 0.8062 (6) | 0.085 (2) |
| H19A | 0.5293 | 0.2780 | 0.8080 | 0.128* |
| H19B | 0.5369 | 0.3884 | 0.7473 | 0.128* |
| H19C | 0.5867 | 0.3869 | 0.8675 | 0.128* |
| C18 | 0.6865 (5) | 0.0604 (5) | 0.6094 (5) | 0.0623 (16) |
| C20 | 0.5112 (5) | 0.1113 (6) | 0.6643 (6) | 0.082 (2) |
| H20A | 0.4874 | 0.0336 | 0.6609 | 0.123* |
| H20B | 0.4741 | 0.1468 | 0.5994 | 0.123* |
| H20C | 0.4866 | 0.1487 | 0.7169 | 0.123* |
| O3 | 0.2917 (4) | 0.3532 (5) | 0.5604 (4) | 0.0990 (19) |
| O4 | 0.1213 (4) | 0.4452 (5) | 0.5126 (4) | 0.0960 (18) |
| H4A | 0.1581 | 0.4809 | 0.4806 | 0.144* |
| C21 | 0.4859 (7) | 0.2433 (8) | 1.2965 (6) | 0.098 |
| H21A | 0.5408 | 0.2628 | 1.3620 | 0.148* |
| H21B | 0.4750 | 0.1631 | 1.2926 | 0.148* |
| H21C | 0.4105 | 0.2793 | 1.2887 | 0.148* |
| C22 | 0.6600 (6) | 0.2601 (9) | 1.2160 (6) | 0.107 |
| H22A | 0.7119 | 0.2770 | 1.2834 | 0.160* |
| H22B | 0.6806 | 0.3068 | 1.1664 | 0.160* |
| H22C | 0.6689 | 0.1824 | 1.2004 | 0.160* |
| C23 | 0.5353 (6) | 0.2817 (7) | 1.2122 (5) | 0.085 |
| H23A | 0.5325 | 0.3640 | 1.2170 | 0.102* |
| C24 | 0.4502 (6) | 0.2576 (9) | 1.1044 (6) | 0.093 (2) |
| C25 | 0.4036 (8) | 0.1542 (8) | 1.0763 (7) | 0.112 (3) |
| H25A | 0.4219 | 0.0958 | 1.1239 | 0.134* |
| C26 | 0.3299 (7) | 0.1337 (7) | 0.9789 (7) | 0.108 (3) |
| H26A | 0.3076 | 0.0599 | 0.9597 | 0.129* |
| C27 | 0.2870 (5) | 0.2205 (6) | 0.9074 (5) | 0.0695 (19) |
| C28 | 0.3299 (6) | 0.3285 (6) | 0.9408 (5) | 0.0709 (18) |
| C29 | 0.4126 (6) | 0.3443 (6) | 1.0387 (5) | 0.0747 (19) |
| H29A | 0.4419 | 0.4162 | 1.0586 | 0.090* |
| C30 | 0.1924 (5) | 0.1988 (6) | 0.8056 (5) | 0.0650 (18) |
| C31 | 0.2050 (5) | 0.2939 (5) | 0.7340 (4) | 0.0540 (14) |
| H31A | 0.2888 | 0.2904 | 0.7352 | 0.065* |
| C32 | 0.1918 (7) | 0.4097 (6) | 0.7754 (5) | 0.079 (2) |
| H32A | 0.1163 | 0.4150 | 0.7905 | 0.095* |
| H32B | 0.1927 | 0.4665 | 0.7246 | 0.095* |
| C33 | 0.2942 (7) | 0.4292 (6) | 0.8722 (5) | 0.084 (2) |
| H33A | 0.3630 | 0.4545 | 0.8529 | 0.101* |
| H33B | 0.2722 | 0.4894 | 0.9110 | 0.101* |
| C34 | 0.2114 (6) | 0.0853 (6) | 0.7582 (5) | 0.0736 (19) |

| | | | | |
|---|---|---|---|---|
| H34A | 0.2947 | 0.0796 | 0.7609 | 0.088* |
| H34B | 0.1953 | 0.0252 | 0.8004 | 0.088* |
| C35 | 0.1367 (6) | 0.0670 (7) | 0.6509 (5) | 0.087 (2) |
| H35A | 0.0534 | 0.0631 | 0.6486 | 0.105* |
| H35B | 0.1580 | −0.0043 | 0.6266 | 0.105* |
| C36 | 0.1528 (6) | 0.1607 (7) | 0.5810 (5) | 0.078 (2) |
| H36A | 0.0986 | 0.1477 | 0.5127 | 0.094* |
| H36B | 0.2336 | 0.1571 | 0.5763 | 0.094* |
| C37 | 0.1309 (5) | 0.2781 (6) | 0.6151 (5) | 0.0650 (17) |
| C39 | −0.0057 (5) | 0.3022 (8) | 0.5926 (5) | 0.087 (2) |
| H39A | −0.0457 | 0.2913 | 0.5208 | 0.130* |
| H39B | −0.0169 | 0.3784 | 0.6112 | 0.130* |
| H39C | −0.0384 | 0.2516 | 0.6320 | 0.130* |
| C38 | 0.1848 (5) | 0.3630 (6) | 0.5582 (5) | 0.0688 (19) |
| C40 | 0.0688 (5) | 0.1971 (8) | 0.8262 (5) | 0.090 (3) |
| H40A | 0.0568 | 0.2672 | 0.8567 | 0.136* |
| H40B | 0.0668 | 0.1363 | 0.8719 | 0.136* |
| H40C | 0.0066 | 0.1868 | 0.7625 | 0.136* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.076 (3) | 0.120 (5) | 0.161 (5) | −0.044 (3) | 0.070 (3) | −0.075 (4) |
| O2 | 0.058 (3) | 0.116 (4) | 0.124 (4) | −0.022 (3) | 0.032 (3) | −0.060 (4) |
| C1 | 0.105 | 0.105 | 0.105 | 0.000 | 0.032 | 0.000 |
| C2 | 0.094 | 0.094 | 0.094 | 0.000 | 0.028 | 0.000 |
| C3 | 0.089 | 0.089 | 0.089 | 0.000 | 0.027 | 0.000 |
| C4 | 0.131 (7) | 0.069 (5) | 0.094 (6) | −0.045 (5) | 0.052 (5) | −0.031 (5) |
| C5 | 0.127 (7) | 0.088 (6) | 0.113 (7) | −0.054 (5) | 0.060 (5) | −0.055 (6) |
| C6 | 0.094 (5) | 0.094 (6) | 0.060 (4) | −0.027 (4) | 0.037 (4) | −0.015 (4) |
| C7 | 0.066 (4) | 0.055 (4) | 0.057 (4) | −0.007 (3) | 0.026 (3) | −0.008 (3) |
| C8 | 0.073 (4) | 0.057 (4) | 0.065 (4) | 0.003 (3) | 0.015 (3) | 0.005 (3) |
| C9 | 0.105 (5) | 0.070 (5) | 0.092 (5) | −0.007 (4) | 0.051 (4) | 0.022 (4) |
| C10 | 0.048 (3) | 0.065 (4) | 0.059 (4) | 0.000 (3) | 0.024 (3) | −0.008 (3) |
| C11 | 0.040 (3) | 0.056 (3) | 0.046 (3) | 0.002 (3) | 0.017 (2) | −0.009 (3) |
| C12 | 0.065 (3) | 0.068 (4) | 0.053 (3) | −0.008 (3) | 0.010 (3) | −0.006 (3) |
| C13 | 0.084 (5) | 0.091 (5) | 0.075 (5) | −0.004 (4) | 0.019 (4) | 0.020 (4) |
| C14 | 0.076 (4) | 0.075 (4) | 0.052 (3) | −0.009 (4) | 0.020 (3) | 0.000 (3) |
| C15 | 0.105 (5) | 0.079 (5) | 0.070 (4) | −0.018 (4) | 0.027 (4) | 0.005 (4) |
| C16 | 0.081 (5) | 0.058 (4) | 0.108 (6) | −0.008 (4) | 0.036 (4) | 0.005 (4) |
| C17 | 0.031 (2) | 0.079 (4) | 0.058 (3) | −0.006 (3) | 0.018 (2) | −0.009 (3) |
| C19 | 0.068 (4) | 0.089 (5) | 0.115 (6) | −0.003 (4) | 0.053 (4) | −0.025 (5) |
| C18 | 0.050 (3) | 0.057 (4) | 0.083 (4) | 0.001 (3) | 0.025 (3) | −0.008 (4) |
| C20 | 0.056 (3) | 0.086 (5) | 0.114 (6) | −0.020 (4) | 0.041 (4) | −0.036 (5) |
| O3 | 0.052 (2) | 0.135 (5) | 0.122 (4) | 0.028 (3) | 0.043 (3) | 0.071 (4) |
| O4 | 0.062 (3) | 0.112 (4) | 0.117 (4) | 0.016 (3) | 0.032 (3) | 0.066 (4) |
| C21 | 0.098 | 0.098 | 0.098 | 0.000 | 0.030 | 0.000 |
| C22 | 0.107 | 0.107 | 0.107 | 0.000 | 0.032 | 0.000 |

| | | | | | | |
|---|---|---|---|---|---|---|
| C23 | 0.085 | 0.085 | 0.085 | 0.000 | 0.026 | 0.000 |
| C24 | 0.081 (5) | 0.107 (7) | 0.078 (5) | −0.006 (5) | 0.006 (4) | 0.001 (5) |
| C25 | 0.139 (8) | 0.084 (6) | 0.081 (5) | −0.014 (6) | −0.015 (5) | 0.021 (5) |
| C26 | 0.102 (6) | 0.074 (5) | 0.113 (7) | −0.022 (5) | −0.017 (5) | 0.029 (5) |
| C27 | 0.058 (4) | 0.076 (5) | 0.076 (4) | 0.010 (3) | 0.023 (3) | 0.031 (4) |
| C28 | 0.076 (4) | 0.072 (5) | 0.067 (4) | 0.016 (4) | 0.024 (3) | 0.002 (4) |
| C29 | 0.091 (5) | 0.069 (5) | 0.067 (4) | 0.008 (4) | 0.028 (4) | −0.009 (4) |
| C30 | 0.040 (3) | 0.088 (5) | 0.073 (4) | 0.002 (3) | 0.025 (3) | 0.017 (4) |
| C31 | 0.049 (3) | 0.058 (4) | 0.060 (3) | 0.014 (3) | 0.023 (3) | 0.010 (3) |
| C32 | 0.089 (5) | 0.077 (5) | 0.084 (5) | 0.020 (4) | 0.043 (4) | 0.018 (4) |
| C33 | 0.122 (6) | 0.055 (4) | 0.076 (5) | 0.017 (4) | 0.031 (4) | 0.000 (4) |
| C34 | 0.066 (4) | 0.061 (4) | 0.090 (5) | −0.011 (3) | 0.018 (4) | 0.011 (4) |
| C35 | 0.079 (5) | 0.083 (5) | 0.087 (5) | −0.027 (4) | 0.005 (4) | 0.013 (4) |
| C36 | 0.059 (4) | 0.094 (6) | 0.077 (4) | −0.011 (4) | 0.015 (3) | −0.005 (4) |
| C37 | 0.038 (3) | 0.084 (5) | 0.070 (4) | 0.005 (3) | 0.011 (3) | 0.019 (4) |
| C39 | 0.039 (3) | 0.125 (6) | 0.097 (5) | 0.010 (4) | 0.020 (3) | 0.032 (5) |
| C38 | 0.042 (3) | 0.099 (5) | 0.067 (4) | 0.007 (3) | 0.019 (3) | 0.029 (4) |
| C40 | 0.052 (3) | 0.138 (7) | 0.088 (4) | −0.011 (4) | 0.032 (3) | 0.042 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C18 | 1.289 (7) | O3—C38 | 1.252 (6) |
| O2—C18 | 1.281 (7) | O4—C38 | 1.271 (8) |
| O2—H2D | 0.8200 | O4—H4A | 0.8200 |
| C1—C3 | 1.469 (7) | C21—C23 | 1.507 (10) |
| C1—H1A | 0.9600 | C21—H21A | 0.9600 |
| C1—H1B | 0.9600 | C21—H21B | 0.9600 |
| C1—H1C | 0.9600 | C21—H21C | 0.9600 |
| C2—C3 | 1.559 (10) | C22—C23 | 1.472 (7) |
| C2—H2A | 0.9600 | C22—H22A | 0.9600 |
| C2—H2B | 0.9600 | C22—H22B | 0.9600 |
| C2—H2C | 0.9600 | C22—H22C | 0.9600 |
| C3—C4 | 1.490 (10) | C23—C24 | 1.538 (10) |
| C3—H3A | 0.9800 | C23—H23A | 0.9800 |
| C4—C5 | 1.391 (12) | C24—C29 | 1.350 (10) |
| C4—C9 | 1.395 (10) | C24—C25 | 1.352 (12) |
| C5—C6 | 1.393 (10) | C25—C26 | 1.373 (11) |
| C5—H5A | 0.9300 | C25—H25A | 0.9300 |
| C6—C7 | 1.368 (8) | C26—C27 | 1.405 (9) |
| C6—H6A | 0.9300 | C26—H26A | 0.9300 |
| C7—C8 | 1.377 (8) | C27—C28 | 1.404 (10) |
| C7—C10 | 1.567 (9) | C27—C30 | 1.518 (9) |
| C8—C9 | 1.388 (9) | C28—C29 | 1.408 (9) |
| C8—C13 | 1.516 (9) | C28—C33 | 1.498 (10) |
| C9—H9A | 0.9300 | C29—H29A | 0.9300 |
| C10—C14 | 1.504 (9) | C30—C31 | 1.530 (8) |
| C10—C11 | 1.562 (7) | C30—C34 | 1.540 (10) |
| C10—C19 | 1.576 (7) | C30—C40 | 1.558 (7) |
| C11—C12 | 1.494 (8) | C31—C32 | 1.513 (9) |

| | | | |
|---|---|---|---|
| C11—C17 | 1.581 (8) | C31—C37 | 1.605 (8) |
| C11—H11A | 0.9800 | C31—H31A | 0.9800 |
| C12—C13 | 1.511 (10) | C32—C33 | 1.514 (10) |
| C12—H12A | 0.9700 | C32—H32A | 0.9700 |
| C12—H12B | 0.9700 | C32—H32B | 0.9700 |
| C13—H13A | 0.9700 | C33—H33A | 0.9700 |
| C13—H13B | 0.9700 | C33—H33B | 0.9700 |
| C14—C15 | 1.529 (10) | C34—C35 | 1.480 (9) |
| C14—H14A | 0.9700 | C34—H34A | 0.9700 |
| C14—H14B | 0.9700 | C34—H34B | 0.9700 |
| C15—C16 | 1.471 (10) | C35—C36 | 1.515 (10) |
| C15—H15A | 0.9700 | C35—H35A | 0.9700 |
| C15—H15B | 0.9700 | C35—H35B | 0.9700 |
| C16—C17 | 1.524 (9) | C36—C37 | 1.516 (10) |
| C16—H16A | 0.9700 | C36—H36A | 0.9700 |
| C16—H16B | 0.9700 | C36—H36B | 0.9700 |
| C17—C18 | 1.477 (8) | C37—C38 | 1.522 (9) |
| C17—C20 | 1.540 (7) | C37—C39 | 1.566 (7) |
| 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—H20A | 0.9600 | C40—H40A | 0.9600 |
| C20—H20B | 0.9600 | C40—H40B | 0.9600 |
| C20—H20C | 0.9600 | C40—H40C | 0.9600 |
| C18—O2—H2D | 109.5 | C38—O4—H4A | 109.5 |
| C3—C1—H1A | 109.5 | C23—C21—H21A | 109.5 |
| C3—C1—H1B | 109.5 | C23—C21—H21B | 109.5 |
| H1A—C1—H1B | 109.5 | H21A—C21—H21B | 109.5 |
| C3—C1—H1C | 109.5 | C23—C21—H21C | 109.5 |
| H1A—C1—H1C | 109.5 | H21A—C21—H21C | 109.5 |
| H1B—C1—H1C | 109.5 | H21B—C21—H21C | 109.5 |
| C3—C2—H2A | 109.5 | C23—C22—H22A | 109.5 |
| C3—C2—H2B | 109.5 | C23—C22—H22B | 109.5 |
| H2A—C2—H2B | 109.5 | H22A—C22—H22B | 109.5 |
| C3—C2—H2C | 109.5 | C23—C22—H22C | 109.5 |
| H2A—C2—H2C | 109.5 | H22A—C22—H22C | 109.5 |
| H2B—C2—H2C | 109.5 | H22B—C22—H22C | 109.5 |
| C1—C3—C4 | 108.9 (7) | C22—C23—C21 | 121.9 (7) |
| C1—C3—C2 | 130.1 (7) | C22—C23—C24 | 110.7 (6) |
| C4—C3—C2 | 107.1 (6) | C21—C23—C24 | 112.7 (6) |
| C1—C3—H3A | 102.4 | C22—C23—H23A | 102.9 |
| C4—C3—H3A | 102.4 | C21—C23—H23A | 102.9 |
| C2—C3—H3A | 102.4 | C24—C23—H23A | 102.9 |
| C5—C4—C9 | 115.8 (7) | C29—C24—C25 | 118.7 (7) |
| C5—C4—C3 | 114.1 (7) | C29—C24—C23 | 118.8 (8) |
| C9—C4—C3 | 129.8 (7) | C25—C24—C23 | 122.1 (8) |
| C4—C5—C6 | 121.0 (7) | C24—C25—C26 | 121.3 (8) |
| C4—C5—H5A | 119.5 | C24—C25—H25A | 119.4 |
| C6—C5—H5A | 119.5 | C26—C25—H25A | 119.4 |

| | | | |
|---|---|---|---|
| C7—C6—C5 | 121.7 (7) | C25—C26—C27 | 122.3 (8) |
| C7—C6—H6A | 119.2 | C25—C26—H26A | 118.9 |
| C5—C6—H6A | 119.2 | C27—C26—H26A | 118.9 |
| C6—C7—C8 | 118.9 (6) | C28—C27—C26 | 115.2 (6) |
| C6—C7—C10 | 119.9 (6) | C28—C27—C30 | 123.0 (6) |
| C8—C7—C10 | 121.3 (5) | C26—C27—C30 | 121.5 (7) |
| C7—C8—C9 | 119.2 (6) | C27—C28—C29 | 120.5 (6) |
| C7—C8—C13 | 122.8 (6) | C27—C28—C33 | 121.4 (6) |
| C9—C8—C13 | 118.0 (6) | C29—C28—C33 | 118.0 (7) |
| C8—C9—C4 | 123.4 (7) | C24—C29—C28 | 121.6 (7) |
| C8—C9—H9A | 118.3 | C24—C29—H29A | 119.2 |
| C4—C9—H9A | 118.3 | C28—C29—H29A | 119.2 |
| C14—C10—C11 | 110.1 (5) | C27—C30—C31 | 105.9 (5) |
| C14—C10—C7 | 111.7 (5) | C27—C30—C34 | 112.0 (5) |
| C11—C10—C7 | 105.2 (4) | C31—C30—C34 | 108.9 (5) |
| C14—C10—C19 | 110.7 (5) | C27—C30—C40 | 107.6 (5) |
| C11—C10—C19 | 112.5 (5) | C31—C30—C40 | 113.5 (5) |
| C7—C10—C19 | 106.4 (5) | C34—C30—C40 | 108.9 (6) |
| C12—C11—C10 | 112.0 (5) | C32—C31—C30 | 113.1 (5) |
| C12—C11—C17 | 113.5 (4) | C32—C31—C37 | 113.5 (5) |
| C10—C11—C17 | 114.9 (4) | C30—C31—C37 | 115.6 (5) |
| C12—C11—H11A | 105.1 | C32—C31—H31A | 104.4 |
| C10—C11—H11A | 105.1 | C30—C31—H31A | 104.4 |
| C17—C11—H11A | 105.1 | C37—C31—H31A | 104.4 |
| C11—C12—C13 | 113.5 (5) | C31—C32—C33 | 108.8 (6) |
| C11—C12—H12A | 108.9 | C31—C32—H32A | 109.9 |
| C13—C12—H12A | 108.9 | C33—C32—H32A | 109.9 |
| C11—C12—H12B | 108.9 | C31—C32—H32B | 109.9 |
| C13—C12—H12B | 108.9 | C33—C32—H32B | 109.9 |
| H12A—C12—H12B | 107.7 | H32A—C32—H32B | 108.3 |
| C12—C13—C8 | 115.4 (6) | C28—C33—C32 | 115.0 (7) |
| C12—C13—H13A | 108.4 | C28—C33—H33A | 108.5 |
| C8—C13—H13A | 108.4 | C32—C33—H33A | 108.5 |
| C12—C13—H13B | 108.4 | C28—C33—H33B | 108.5 |
| C8—C13—H13B | 108.4 | C32—C33—H33B | 108.5 |
| H13A—C13—H13B | 107.5 | H33A—C33—H33B | 107.5 |
| C10—C14—C15 | 112.8 (5) | C35—C34—C30 | 115.2 (6) |
| C10—C14—H14A | 109.0 | C35—C34—H34A | 108.5 |
| C15—C14—H14A | 109.0 | C30—C34—H34A | 108.5 |
| C10—C14—H14B | 109.0 | C35—C34—H34B | 108.5 |
| C15—C14—H14B | 109.0 | C30—C34—H34B | 108.5 |
| H14A—C14—H14B | 107.8 | H34A—C34—H34B | 107.5 |
| C16—C15—C14 | 111.7 (6) | C34—C35—C36 | 111.6 (6) |
| C16—C15—H15A | 109.3 | C34—C35—H35A | 109.3 |
| C14—C15—H15A | 109.3 | C36—C35—H35A | 109.3 |
| C16—C15—H15B | 109.3 | C34—C35—H35B | 109.3 |
| C14—C15—H15B | 109.3 | C36—C35—H35B | 109.3 |
| H15A—C15—H15B | 107.9 | H35A—C35—H35B | 108.0 |
| C15—C16—C17 | 114.2 (6) | C35—C36—C37 | 114.6 (6) |

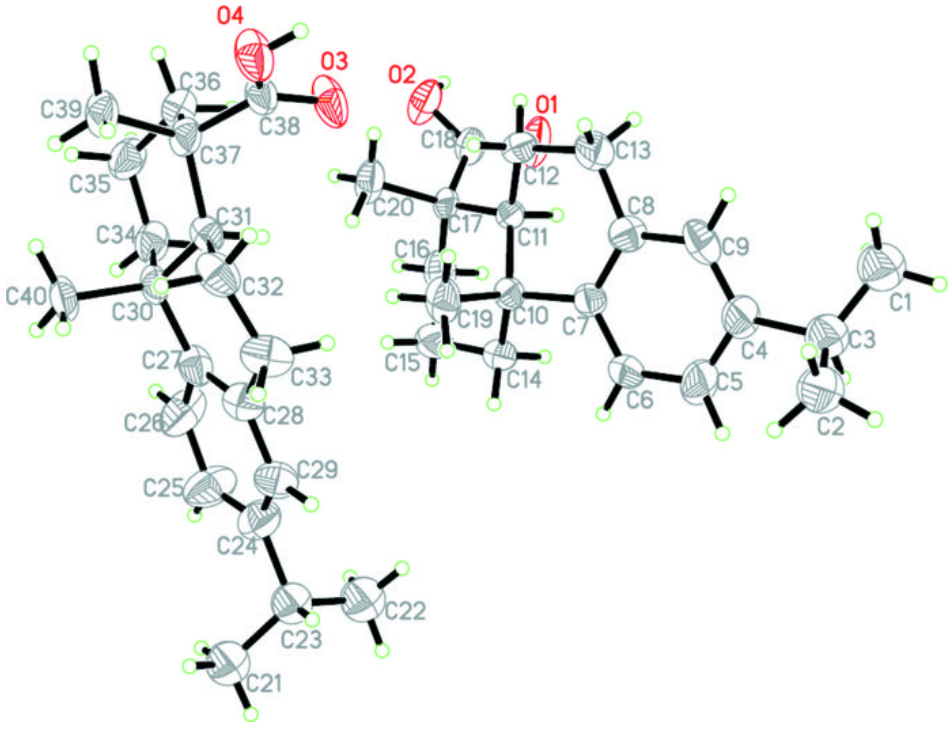
| | | | |
|---|---|---|---|
| C15—C16—H16A | 108.7 | C35—C36—H36A | 108.6 |
| C17—C16—H16A | 108.7 | C37—C36—H36A | 108.6 |
| C15—C16—H16B | 108.7 | C35—C36—H36B | 108.6 |
| C17—C16—H16B | 108.7 | C37—C36—H36B | 108.6 |
| H16A—C16—H16B | 107.6 | H36A—C36—H36B | 107.6 |
| C18—C17—C16 | 104.4 (5) | C36—C37—C38 | 108.5 (5) |
| C18—C17—C20 | 109.8 (5) | C36—C37—C39 | 111.5 (6) |
| C16—C17—C20 | 111.8 (5) | C38—C37—C39 | 109.7 (5) |
| C18—C17—C11 | 107.9 (4) | C36—C37—C31 | 109.2 (5) |
| C16—C17—C11 | 109.9 (5) | C38—C37—C31 | 104.9 (5) |
| C20—C17—C11 | 112.7 (5) | C39—C37—C31 | 112.9 (5) |
| C10—C19—H19A | 109.5 | C37—C39—H39A | 109.5 |
| C10—C19—H19B | 109.5 | C37—C39—H39B | 109.5 |
| H19A—C19—H19B | 109.5 | H39A—C39—H39B | 109.5 |
| C10—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 |
| O2—C18—O1 | 117.1 (6) | O3—C38—O4 | 121.3 (6) |
| O2—C18—C17 | 120.9 (5) | O3—C38—C37 | 119.6 (6) |
| O1—C18—C17 | 121.9 (6) | O4—C38—C37 | 119.1 (5) |
| C17—C20—H20A | 109.5 | C30—C40—H40A | 109.5 |
| C17—C20—H20B | 109.5 | C30—C40—H40B | 109.5 |
| H20A—C20—H20B | 109.5 | H40A—C40—H40B | 109.5 |
| C17—C20—H20C | 109.5 | C30—C40—H40C | 109.5 |
| H20A—C20—H20C | 109.5 | H40A—C40—H40C | 109.5 |
| H20B—C20—H20C | 109.5 | H40B—C40—H40C | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2D···O3$^{i}$ | 0.82 | 1.82 | 2.621 (8) | 165 |
| O4—H4A···O1$^{ii}$ | 0.82 | 1.79 | 2.598 (8) | 168 |
| C11—H11A···O1 | 0.98 | 2.36 | 2.813 (8) | 108 |
| C31—H31A···O3 | 0.98 | 2.51 | 2.933 (8) | 106 |
| C36—H36B···O3 | 0.97 | 2.45 | 2.870 (10) | 105 |

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

**Fig. 1**


O4
O3
O2
O1
C36
C39
C38
C18
C13
C37
C35
C12
C20
C17
C8
C31
C34
C11
C9
C16
C40
C32
C30
C19
C10
C7
C1
C4
C3
C27
C33
C15
C14
C5
C6
C2
C26
C28
C29
C25
C24
C22
C23
C21