

## Cycloart-24-ene-3 $\beta$ ,26-diol from the leaves of *Aglaiia exima*

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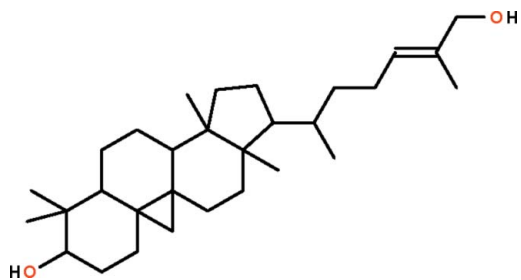
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.055;  $wR$  factor = 0.143; data-to-parameter ratio = 8.4.

Cycloart-24-ene-3 $\beta$ ,26-diol,  $\text{C}_{30}\text{H}_{50}\text{O}_2$ , isolated from the leaves of *Aglaiia exima*, has three six-membered rings fused together that adopt chair conformations. There are two independent molecules in the asymmetric unit.  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond interactions between the hydroxyl groups in the 3 $\beta$  and 26 positions lead to the formation of a layer structure parallel to (10 $\bar{2}$ ).

### Related literature

For the spectroscopic characterization of the title compound, see: Anjaneyulu *et al.* (1985, 1994); Parveen *et al.* (1990); Takahashi & Takani (1975).



### Experimental

#### Crystal data

$\text{C}_{30}\text{H}_{50}\text{O}_2$

$M_r = 442.70$

Monoclinic,  $P2_1$   
 $a = 9.9950$  (14) Å  
 $b = 7.512$  (1) Å  
 $c = 34.820$  (5) Å  
 $\beta = 91.029$  (2)°  
 $V = 2614.0$  (6) Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.07$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.45 \times 0.15 \times 0.02$  mm

#### Data collection

Bruker SMART APEX  
 diffractometer  
 16958 measured reflections

4970 independent reflections  
 3318 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.085$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$   
 $wR(F^2) = 0.143$   
 $S = 0.96$   
 4970 reflections  
 593 parameters

1 restraint  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                     | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$   | 0.84  | 2.11        | 2.807 (5)   | 140           |
| $\text{O2}-\text{H2}\cdots\text{O3}^{\text{ii}}$  | 0.84  | 1.97        | 2.784 (5)   | 162           |
| $\text{O3}-\text{H3}\cdots\text{O4}^{\text{iii}}$ | 0.84  | 2.00        | 2.747 (5)   | 148           |
| $\text{O4}-\text{H4}\cdots\text{O1}$              | 0.84  | 1.89        | 2.722 (4)   | 173           |

Symmetry codes: (i)  $-x + 1, y + \frac{1}{2}, -z + 1$ ; (ii)  $x - 2, y - 1, z - 1$ ; (iii)  $-x + 3, y + \frac{1}{2}, -z + 2$ .

Data collection: APEX2 software (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2187).

### References

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**supplementary materials**

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## Cycloart-24-ene-3 $\beta$ ,26-diol from the leaves of *Aglaia exima*

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### Comment

Cycloart-24-ene-3 $\beta$ ,26-diol (Scheme I) has been isolated from the leaves of *Garcinia magnostana* (Parveen *et al.*, 1990), the stem-bark of *Mangifera indica* (Anjaneyulu *et al.*, 1985, 1994) and from the wood of *Schizandar nigra* (Takahashi & Takani, 1975). The structure was elucidated in the studies by spectroscopic methods. The absolute configuration in the present crystal structure analysis is assumed to be that reported in the studies. Cycloart-24-ene-3 $\beta$ ,26-diol (Fig. 1) in this study was isolated from the leaves of *Aglaia exima*.

### Experimental

The leaves of *Algaia exima* were collected from Kampung Kepayang, Pahang, Malaysia. The leaves (1 kg) were extracted at room temperature with *n*-hexane successively; the insoluble material was removed by filtration after four days. The solvent was evaporated to dryness to give a residue of 25 g; a 15 g portion was subjected to column chromatography over silica gel at a gradient mixture of *n*-hexane and ethyl acetate. Of the 123 fractions, fraction 92 gave a colorless solid, identified by solution NMR, as cycloart-24-ene-3 $\beta$ ,26-diol. Single crystals were obtained by recrystallization from ethyl acetate.

### Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 1.00 Å,  $U(\text{H})$  1.2 to 1.5 $U(\text{C})$ ] and were included in the refinement in the riding model approximation. The hydroxy H-atoms were similarly placed [O—H 0.84 Å] and their temperature factor similarly tied.

Some 4185 Friedel pairs were merged.

### Figures

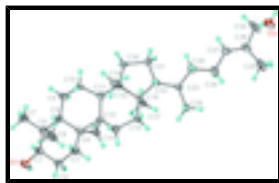


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of one of the two independent molecules of  $\text{C}_{30}\text{H}_{50}\text{O}_2$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

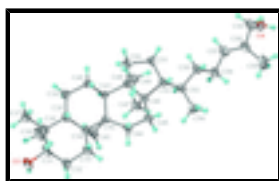


Fig. 2. Thermal ellipsoid plot (Barbour, 2001) of the second independent molecule of  $\text{C}_{30}\text{H}_{50}\text{O}_2$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

## Cycloart-24-ene-3 $\beta$ ,26-diol

### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{30}H_{50}O_2$              | $F(000) = 984$  |
| $M_r = 442.70$                 | $D_x = 1.125 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1$             | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: P 2yb             | Cell parameters from 1946 reflections                   |
| $a = 9.9950 (14) \text{ \AA}$  | $\theta = 2.3\text{--}21.1^\circ$                       |
| $b = 7.512 (1) \text{ \AA}$    | $\mu = 0.07 \text{ mm}^{-1}$                            |
| $c = 34.820 (5) \text{ \AA}$   | $T = 100 \text{ K}$                                     |
| $\beta = 91.029 (2)^\circ$     | Plate, colorless  |
| $V = 2614.0 (6) \text{ \AA}^3$ | $0.45 \times 0.15 \times 0.02 \text{ mm}$               |
| $Z = 4$                        |   |

### Data collection

|  |  |
|--|--|
| Bruker SMART APEX diffractometer         | 3318 reflections with $I > 2\sigma(I)$                                 |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.085$   |
| graphite                                 | $\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 1.8^\circ$ |
| $\omega$ scans                           | $h = -11 \rightarrow 11$   |
| 16958 measured reflections               | $k = -8 \rightarrow 8$   |
| 4970 independent reflections             | $l = -36 \rightarrow 41$   |

### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.143$               | H-atom parameters constrained                                  |
| $S = 0.96$                      | $w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 0.1056P]$              |
| 4970 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 593 parameters                  | $(\Delta/\sigma)_{\text{max}} = 0.001$                         |
| 1 restraint                     | $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$            |
|                                 | $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$           |

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|    | <i>x</i>    | <i>y</i>   | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|-------------|------------|--------------|----------------------------------|
| O1 | 0.8196 (3)  | 0.5009 (4) | 0.73269 (10) | 0.0366 (9)                       |
| H1 | 0.8842      | 0.5089     | 0.7177       | 0.055*                           |
| O2 | -0.0926 (3) | 0.0538 (5) | 0.28107 (9)  | 0.0421 (9)                       |
| H2 | -0.1424     | -0.0187    | 0.2693       | 0.063*                           |

|      |            |            |              |             |
|------|------------|------------|--------------|-------------|
| O3   | 1.7903 (3) | 0.7649 (5) | 1.24335 (10) | 0.0485 (10) |
| H3   | 1.8534     | 0.7927     | 1.2289       | 0.073*      |
| O4   | 0.9518 (4) | 0.2801 (5) | 0.78237 (9)  | 0.0436 (9)  |
| H4   | 0.9051     | 0.3438     | 0.7675       | 0.065*      |
| C1   | 0.6990 (5) | 0.4641 (6) | 0.71105 (14) | 0.0298 (12) |
| H1a  | 0.6246     | 0.4576     | 0.7298       | 0.036*      |
| C2   | 0.6698 (5) | 0.6209 (6) | 0.68482 (13) | 0.0278 (11) |
| H2A  | 0.7463     | 0.6387     | 0.6676       | 0.033*      |
| H2B  | 0.6603     | 0.7297     | 0.7006       | 0.033*      |
| C3   | 0.5413 (4) | 0.5941 (6) | 0.66033 (14) | 0.0280 (11) |
| H3A  | 0.4626     | 0.5951     | 0.6772       | 0.034*      |
| H3B  | 0.5315     | 0.6933     | 0.6418       | 0.034*      |
| C4   | 0.5463 (4) | 0.4196 (6) | 0.63883 (13) | 0.0254 (11) |
| C5   | 0.5667 (5) | 0.2676 (6) | 0.66768 (13) | 0.0267 (11) |
| H5   | 0.4961     | 0.2843     | 0.6874       | 0.032*      |
| C6   | 0.7025 (5) | 0.2847 (7) | 0.68988 (14) | 0.0339 (12) |
| C7   | 0.7133 (6) | 0.1353 (7) | 0.71945 (17) | 0.0524 (18) |
| H7A  | 0.7288     | 0.0220     | 0.7063       | 0.079*      |
| H7B  | 0.6300     | 0.1282     | 0.7338       | 0.079*      |
| H7C  | 0.7881     | 0.1598     | 0.7373       | 0.079*      |
| C8   | 0.8233 (4) | 0.2766 (7) | 0.66345 (15) | 0.0433 (14) |
| H8A  | 0.8166     | 0.1704     | 0.6472       | 0.065*      |
| H8B  | 0.9058     | 0.2709     | 0.6790       | 0.065*      |
| H8C  | 0.8248     | 0.3832     | 0.6472       | 0.065*      |
| C9   | 0.5395 (5) | 0.0842 (6) | 0.64985 (14) | 0.0320 (12) |
| H9A  | 0.6117     | 0.0537     | 0.6319       | 0.038*      |
| H9B  | 0.5382     | -0.0074    | 0.6703       | 0.038*      |
| C10  | 0.4054 (4) | 0.0867 (6) | 0.62840 (13) | 0.0292 (11) |
| H10A | 0.3357     | 0.1359     | 0.6453       | 0.035*      |
| H10B | 0.3793     | -0.0362    | 0.6213       | 0.035*      |
| C11  | 0.4152 (4) | 0.2002 (6) | 0.59238 (13) | 0.0238 (11) |
| H11  | 0.4885     | 0.1462     | 0.5771       | 0.029*      |
| C12  | 0.4619 (4) | 0.3924 (6) | 0.60191 (13) | 0.0226 (10) |
| C13  | 0.6114 (4) | 0.4216 (6) | 0.60022 (12) | 0.0272 (11) |
| H13A | 0.6678     | 0.3184     | 0.5934       | 0.033*      |
| H13B | 0.6431     | 0.5375     | 0.5904       | 0.033*      |
| C14  | 0.3700 (4) | 0.5463 (6) | 0.58923 (13) | 0.0255 (11) |
| H14A | 0.4244     | 0.6562     | 0.5881       | 0.031*      |
| H14B | 0.3021     | 0.5640     | 0.6092       | 0.031*      |
| C15  | 0.2956 (4) | 0.5231 (6) | 0.54978 (12) | 0.0216 (10) |
| H15A | 0.2001     | 0.5536     | 0.5529       | 0.026*      |
| H15B | 0.3335     | 0.6082     | 0.5312       | 0.026*      |
| C16  | 0.3052 (4) | 0.3349 (5) | 0.53316 (12) | 0.0193 (10) |
| C17  | 0.4415 (4) | 0.3143 (6) | 0.51311 (13) | 0.0233 (10) |
| H17A | 0.5138     | 0.3503     | 0.5308       | 0.035*      |
| H17B | 0.4427     | 0.3899     | 0.4902       | 0.035*      |
| H17C | 0.4541     | 0.1897     | 0.5056       | 0.035*      |
| C18  | 0.2899 (4) | 0.1969 (6) | 0.56606 (13) | 0.0219 (10) |
| C19  | 0.1616 (4) | 0.2328 (6) | 0.58904 (13) | 0.0238 (10) |

## supplementary materials

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|      |             |            |              |             |
|------|-------------|------------|--------------|-------------|
| H19A | 0.1465      | 0.1341     | 0.6068       | 0.036*      |
| H19B | 0.0849      | 0.2433     | 0.5712       | 0.036*      |
| H19C | 0.1721      | 0.3438     | 0.6036       | 0.036*      |
| C20  | 0.2706 (4)  | 0.0201 (5) | 0.54461 (12) | 0.0224 (10) |
| H20A | 0.2152      | -0.0627    | 0.5597       | 0.027*      |
| H20B | 0.3580      | -0.0368    | 0.5398       | 0.027*      |
| C21  | 0.1991 (4)  | 0.0684 (6) | 0.50638 (12) | 0.0216 (10) |
| H21A | 0.1076      | 0.0181     | 0.5056       | 0.026*      |
| H21B | 0.2491      | 0.0197     | 0.4845       | 0.026*      |
| C22  | 0.1935 (4)  | 0.2760 (6) | 0.50405 (12) | 0.0209 (10) |
| H22  | 0.1060      | 0.3136     | 0.5149       | 0.025*      |
| C23  | 0.1965 (4)  | 0.3444 (5) | 0.46270 (13) | 0.0221 (10) |
| H23  | 0.2832      | 0.3056     | 0.4515       | 0.027*      |
| C24  | 0.1913 (4)  | 0.5507 (6) | 0.46055 (13) | 0.0247 (10) |
| H24A | 0.2726      | 0.6004     | 0.4724       | 0.037*      |
| H24B | 0.1128      | 0.5938     | 0.4742       | 0.037*      |
| H24C | 0.1850      | 0.5880     | 0.4336       | 0.037*      |
| C25  | 0.0833 (4)  | 0.2608 (6) | 0.43826 (12) | 0.0249 (10) |
| H25A | 0.0895      | 0.1296     | 0.4406       | 0.030*      |
| H25B | -0.0035     | 0.2975     | 0.4490       | 0.030*      |
| C26  | 0.0835 (4)  | 0.3098 (6) | 0.39569 (12) | 0.0256 (11) |
| H26A | 0.0524      | 0.4342     | 0.3926       | 0.031*      |
| H26B | 0.1763      | 0.3032     | 0.3863       | 0.031*      |
| C27  | -0.0035 (4) | 0.1910 (7) | 0.37175 (13) | 0.0307 (12) |
| H27  | -0.0161     | 0.0740     | 0.3814       | 0.037*      |
| C28  | -0.0663 (4) | 0.2285 (7) | 0.33829 (13) | 0.0292 (11) |
| C29  | -0.0642 (6) | 0.4038 (7) | 0.31926 (16) | 0.0541 (17) |
| H29A | -0.0183     | 0.4900     | 0.3360       | 0.081*      |
| H29B | -0.1562     | 0.4436     | 0.3141       | 0.081*      |
| H29C | -0.0166     | 0.3942     | 0.2950       | 0.081*      |
| C30  | -0.1459 (5) | 0.0885 (8) | 0.31816 (15) | 0.0462 (15) |
| H30A | -0.1442     | -0.0222    | 0.3336       | 0.055*      |
| H30B | -0.2401     | 0.1278     | 0.3154       | 0.055*      |
| C31  | 1.6695 (5)  | 0.7436 (7) | 1.22082 (15) | 0.0388 (13) |
| H31  | 1.5927      | 0.7565     | 1.2386       | 0.047*      |
| C32  | 1.6570 (5)  | 0.8907 (6) | 1.19104 (14) | 0.0338 (12) |
| H32A | 1.7359      | 0.8877     | 1.1743       | 0.041*      |
| H32B | 1.6562      | 1.0074     | 1.2042       | 0.041*      |
| C33  | 1.5287 (5)  | 0.8723 (6) | 1.16604 (14) | 0.0314 (12) |
| H33A | 1.4487      | 0.8856     | 1.1822       | 0.038*      |
| H33B | 1.5262      | 0.9661     | 1.1461       | 0.038*      |
| C34  | 1.5290 (5)  | 0.6896 (6) | 1.14735 (14) | 0.0297 (12) |
| C35  | 1.5352 (5)  | 0.5445 (6) | 1.17769 (14) | 0.0320 (12) |
| H35  | 1.4598      | 0.5722     | 1.1952       | 0.038*      |
| C36  | 1.6625 (5)  | 0.5531 (7) | 1.20379 (14) | 0.0355 (13) |
| C37  | 1.6508 (6)  | 0.4222 (7) | 1.23772 (16) | 0.0543 (17) |
| H37A | 1.5632      | 0.4363     | 1.2495       | 0.082*      |
| H37B | 1.7215      | 0.4473     | 1.2568       | 0.082*      |
| H37C | 1.6603      | 0.2999     | 1.2284       | 0.082*      |

|      |            |            |              |             |
|------|------------|------------|--------------|-------------|
| C38  | 1.7893 (5) | 0.5113 (8) | 1.18178 (16) | 0.0503 (16) |
| H38A | 1.8032     | 0.6031     | 1.1623       | 0.075*      |
| H38B | 1.7800     | 0.3949     | 1.1693       | 0.075*      |
| H38C | 1.8661     | 0.5090     | 1.1997       | 0.075*      |
| C39  | 1.5035 (5) | 0.3581 (6) | 1.16203 (15) | 0.0354 (13) |
| H39A | 1.5792     | 0.3149     | 1.1466       | 0.043*      |
| H39B | 1.4911     | 0.2745     | 1.1837       | 0.043*      |
| C40  | 1.3760 (5) | 0.3636 (6) | 1.13696 (14) | 0.0319 (12) |
| H40A | 1.3481     | 0.2408     | 1.1303       | 0.038*      |
| H40B | 1.3029     | 0.4203     | 1.1514       | 0.038*      |
| C41  | 1.4014 (4) | 0.4681 (6) | 1.10048 (13) | 0.0246 (11) |
| H41  | 1.4767     | 0.4060     | 1.0875       | 0.030*      |
| C42  | 1.4523 (4) | 0.6587 (6) | 1.10937 (14) | 0.0268 (11) |
| C43  | 1.6032 (4) | 0.6776 (7) | 1.10967 (13) | 0.0331 (12) |
| H43A | 1.6412     | 0.7887     | 1.0992       | 0.040*      |
| H43B | 1.6569     | 0.5692     | 1.1050       | 0.040*      |
| C44  | 1.3665 (4) | 0.8139 (6) | 1.09347 (13) | 0.0272 (11) |
| H44A | 1.4231     | 0.9221     | 1.0933       | 0.033*      |
| H44B | 1.2933     | 0.8358     | 1.1116       | 0.033*      |
| C45  | 1.3034 (4) | 0.7912 (6) | 1.05290 (13) | 0.0252 (10) |
| H45A | 1.2085     | 0.8285     | 1.0535       | 0.030*      |
| H45B | 1.3499     | 0.8711     | 1.0349       | 0.030*      |
| C46  | 1.3107 (4) | 0.5989 (6) | 1.03765 (12) | 0.0209 (10) |
| C47  | 1.4503 (4) | 0.5699 (6) | 1.02068 (13) | 0.0263 (11) |
| H47A | 1.5190     | 0.6088     | 1.0393       | 0.039*      |
| H47B | 1.4580     | 0.6391     | 0.9970       | 0.039*      |
| H47C | 1.4627     | 0.4432     | 1.0150       | 0.039*      |
| C48  | 1.2828 (4) | 0.4665 (6) | 1.07123 (13) | 0.0245 (11) |
| C49  | 1.1492 (4) | 0.5086 (6) | 1.09087 (13) | 0.0280 (11) |
| H49A | 1.1295     | 0.4152     | 1.1096       | 0.042*      |
| H49B | 1.0771     | 0.5138     | 1.0714       | 0.042*      |
| H49C | 1.1562     | 0.6236     | 1.1040       | 0.042*      |
| C50  | 1.2662 (4) | 0.2873 (6) | 1.05027 (13) | 0.0260 (11) |
| H50A | 1.2061     | 0.2073     | 1.0646       | 0.031*      |
| H50B | 1.3539     | 0.2278     | 1.0474       | 0.031*      |
| C51  | 1.2046 (4) | 0.3346 (6) | 1.01051 (14) | 0.0282 (11) |
| H51A | 1.2587     | 0.2819     | 0.9899       | 0.034*      |
| H51B | 1.1124     | 0.2872     | 1.0082       | 0.034*      |
| C52  | 1.2033 (4) | 0.5400 (6) | 1.00700 (13) | 0.0239 (10) |
| H52  | 1.1146     | 0.5814     | 1.0164       | 0.029*      |
| C53  | 1.2148 (4) | 0.6056 (6) | 0.96550 (13) | 0.0248 (11) |
| H53  | 1.3036     | 0.5663     | 0.9558       | 0.030*      |
| C54  | 1.2084 (5) | 0.8091 (6) | 0.96208 (14) | 0.0322 (12) |
| H54A | 1.2865     | 0.8616     | 0.9752       | 0.048*      |
| H54B | 1.1265     | 0.8526     | 0.9740       | 0.048*      |
| H54C | 1.2083     | 0.8430     | 0.9349       | 0.048*      |
| C55  | 1.1061 (4) | 0.5187 (6) | 0.94001 (12) | 0.0289 (11) |
| H55A | 1.1120     | 0.3879     | 0.9431       | 0.035*      |
| H55B | 1.0174     | 0.5565     | 0.9493       | 0.035*      |

## supplementary materials

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|      |            |            |              |             |
|------|------------|------------|--------------|-------------|
| C56  | 1.1143 (4) | 0.5629 (7) | 0.89736 (12) | 0.0287 (11) |
| H56A | 1.0759     | 0.6826     | 0.8927       | 0.034*      |
| H56B | 1.2094     | 0.5658     | 0.8899       | 0.034*      |
| C57  | 1.0402 (5) | 0.4284 (7) | 0.87294 (13) | 0.0347 (12) |
| H57  | 1.0464     | 0.3083     | 0.8814       | 0.042*      |
| C58  | 0.9668 (4) | 0.4553 (7) | 0.84101 (13) | 0.0341 (12) |
| C59  | 0.9430 (6) | 0.6328 (8) | 0.82220 (16) | 0.0490 (16) |
| H59A | 0.9726     | 0.7280     | 0.8396       | 0.074*      |
| H59B | 0.8474     | 0.6469     | 0.8163       | 0.074*      |
| H59C | 0.9936     | 0.6394     | 0.7984       | 0.074*      |
| C60  | 0.9049 (5) | 0.2981 (8) | 0.82061 (14) | 0.0441 (14) |
| H60A | 0.9263     | 0.1882     | 0.8352       | 0.053*      |
| H60B | 0.8064     | 0.3121     | 0.8199       | 0.053*      |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$  | $U^{33}$  | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-----------|-----------|--------------|--------------|--------------|
| O1  | 0.0353 (19) | 0.030 (2) | 0.044 (2) | -0.0015 (16) | -0.0179 (17) | -0.0067 (17) |
| O2  | 0.048 (2)   | 0.046 (2) | 0.033 (2) | -0.0128 (18) | 0.0014 (17)  | -0.0135 (18) |
| O3  | 0.050 (2)   | 0.044 (2) | 0.050 (2) | -0.001 (2)   | -0.0276 (19) | -0.009 (2)   |
| O4  | 0.061 (2)   | 0.041 (2) | 0.028 (2) | 0.0001 (19)  | -0.0149 (17) | 0.0036 (17)  |
| C1  | 0.026 (3)   | 0.026 (3) | 0.037 (3) | -0.003 (2)   | -0.011 (2)   | 0.000 (2)    |
| C2  | 0.031 (3)   | 0.024 (3) | 0.029 (3) | -0.004 (2)   | -0.003 (2)   | -0.002 (2)   |
| C3  | 0.024 (2)   | 0.023 (3) | 0.037 (3) | 0.004 (2)    | -0.005 (2)   | 0.000 (2)    |
| C4  | 0.023 (2)   | 0.022 (3) | 0.030 (3) | 0.001 (2)    | -0.008 (2)   | -0.006 (2)   |
| C5  | 0.035 (3)   | 0.015 (2) | 0.029 (3) | 0.000 (2)    | -0.011 (2)   | -0.001 (2)   |
| C6  | 0.033 (3)   | 0.028 (3) | 0.040 (3) | 0.002 (2)    | -0.014 (2)   | 0.002 (2)    |
| C7  | 0.071 (4)   | 0.024 (3) | 0.061 (4) | -0.008 (3)   | -0.042 (3)   | 0.007 (3)    |
| C8  | 0.029 (3)   | 0.043 (3) | 0.057 (3) | 0.013 (2)    | -0.018 (2)   | -0.021 (3)   |
| C9  | 0.038 (3)   | 0.019 (3) | 0.038 (3) | -0.001 (2)   | -0.015 (2)   | 0.000 (2)    |
| C10 | 0.030 (2)   | 0.017 (2) | 0.040 (3) | -0.007 (2)   | -0.014 (2)   | 0.002 (2)    |
| C11 | 0.021 (2)   | 0.023 (3) | 0.028 (3) | 0.0027 (19)  | -0.006 (2)   | -0.001 (2)   |
| C12 | 0.020 (2)   | 0.020 (2) | 0.028 (3) | -0.0056 (18) | -0.005 (2)   | 0.000 (2)    |
| C13 | 0.023 (2)   | 0.029 (3) | 0.030 (3) | -0.005 (2)   | -0.004 (2)   | -0.005 (2)   |
| C14 | 0.023 (2)   | 0.018 (2) | 0.035 (3) | 0.0005 (19)  | -0.003 (2)   | -0.001 (2)   |
| C15 | 0.022 (2)   | 0.018 (2) | 0.025 (3) | 0.0038 (18)  | -0.001 (2)   | -0.006 (2)   |
| C16 | 0.010 (2)   | 0.022 (2) | 0.026 (3) | 0.0001 (17)  | 0.0004 (18)  | -0.003 (2)   |
| C17 | 0.016 (2)   | 0.022 (2) | 0.032 (3) | -0.0010 (19) | 0.0014 (19)  | -0.001 (2)   |
| C18 | 0.017 (2)   | 0.017 (2) | 0.032 (3) | -0.0007 (18) | -0.0018 (19) | 0.000 (2)    |
| C19 | 0.020 (2)   | 0.023 (3) | 0.028 (3) | -0.0021 (18) | -0.0008 (19) | -0.001 (2)   |
| C20 | 0.022 (2)   | 0.014 (2) | 0.031 (3) | -0.0048 (18) | 0.000 (2)    | 0.003 (2)    |
| C21 | 0.017 (2)   | 0.020 (2) | 0.027 (3) | -0.0037 (19) | 0.0011 (19)  | -0.002 (2)   |
| C22 | 0.017 (2)   | 0.024 (2) | 0.022 (2) | 0.0003 (19)  | 0.0012 (18)  | -0.001 (2)   |
| C23 | 0.017 (2)   | 0.019 (2) | 0.030 (3) | -0.0030 (18) | -0.0026 (19) | -0.005 (2)   |
| C24 | 0.022 (2)   | 0.026 (3) | 0.027 (3) | -0.004 (2)   | -0.005 (2)   | 0.002 (2)    |
| C25 | 0.020 (2)   | 0.028 (3) | 0.027 (3) | -0.0027 (19) | 0.0003 (19)  | -0.002 (2)   |
| C26 | 0.017 (2)   | 0.032 (3) | 0.028 (3) | -0.001 (2)   | -0.0001 (19) | -0.002 (2)   |
| C27 | 0.034 (3)   | 0.033 (3) | 0.025 (3) | -0.016 (2)   | 0.005 (2)    | -0.001 (2)   |



|     |           |           |           |              |             |            |
|-----|-----------|-----------|-----------|--------------|-------------|------------|
| C28 | 0.022 (2) | 0.038 (3) | 0.028 (3) | -0.006 (2)   | 0.004 (2)   | -0.010 (2) |
| C29 | 0.069 (4) | 0.048 (4) | 0.045 (4) | 0.003 (3)    | -0.031 (3)  | -0.009 (3) |
| C30 | 0.036 (3) | 0.071 (4) | 0.031 (3) | -0.022 (3)   | -0.003 (2)  | -0.017 (3) |
| C31 | 0.036 (3) | 0.042 (3) | 0.038 (3) | 0.002 (2)    | -0.018 (2)  | -0.008 (3) |
| C32 | 0.039 (3) | 0.025 (3) | 0.037 (3) | 0.001 (2)    | -0.013 (2)  | -0.006 (2) |
| C33 | 0.030 (3) | 0.027 (3) | 0.037 (3) | 0.003 (2)    | -0.007 (2)  | -0.005 (2) |
| C34 | 0.033 (3) | 0.021 (3) | 0.035 (3) | 0.003 (2)    | -0.010 (2)  | -0.003 (2) |
| C35 | 0.036 (3) | 0.024 (3) | 0.036 (3) | 0.006 (2)    | -0.014 (2)  | -0.005 (2) |
| C36 | 0.038 (3) | 0.027 (3) | 0.040 (3) | 0.004 (2)    | -0.020 (2)  | -0.007 (2) |
| C37 | 0.077 (4) | 0.031 (3) | 0.053 (4) | 0.000 (3)    | -0.040 (3)  | 0.001 (3)  |
| C38 | 0.039 (3) | 0.047 (4) | 0.063 (4) | 0.013 (3)    | -0.024 (3)  | -0.025 (3) |
| C39 | 0.045 (3) | 0.021 (3) | 0.039 (3) | 0.002 (2)    | -0.018 (2)  | -0.004 (2) |
| C40 | 0.038 (3) | 0.021 (3) | 0.037 (3) | -0.003 (2)   | -0.014 (2)  | 0.000 (2)  |
| C41 | 0.024 (2) | 0.018 (2) | 0.032 (3) | 0.0019 (19)  | -0.008 (2)  | 0.000 (2)  |
| C42 | 0.024 (2) | 0.020 (2) | 0.036 (3) | -0.0025 (19) | -0.005 (2)  | 0.001 (2)  |
| C43 | 0.025 (2) | 0.034 (3) | 0.040 (3) | -0.002 (2)   | -0.003 (2)  | -0.005 (2) |
| C44 | 0.023 (2) | 0.025 (3) | 0.034 (3) | -0.001 (2)   | -0.005 (2)  | -0.002 (2) |
| C45 | 0.022 (2) | 0.022 (2) | 0.031 (3) | -0.002 (2)   | -0.003 (2)  | -0.006 (2) |
| C46 | 0.018 (2) | 0.025 (3) | 0.020 (2) | -0.0017 (19) | 0.0013 (18) | 0.000 (2)  |
| C47 | 0.016 (2) | 0.029 (3) | 0.034 (3) | 0.001 (2)    | 0.0000 (19) | -0.009 (2) |
| C48 | 0.020 (2) | 0.022 (3) | 0.031 (3) | -0.0010 (19) | -0.004 (2)  | 0.000 (2)  |
| C49 | 0.022 (2) | 0.031 (3) | 0.031 (3) | -0.0029 (19) | -0.001 (2)  | 0.000 (2)  |
| C50 | 0.022 (2) | 0.025 (3) | 0.030 (3) | -0.005 (2)   | -0.005 (2)  | -0.003 (2) |
| C51 | 0.021 (2) | 0.029 (3) | 0.034 (3) | -0.005 (2)   | -0.006 (2)  | 0.000 (2)  |
| C52 | 0.017 (2) | 0.024 (3) | 0.030 (3) | 0.0017 (19)  | -0.002 (2)  | -0.003 (2) |
| C53 | 0.016 (2) | 0.030 (3) | 0.028 (3) | -0.0027 (19) | 0.000 (2)   | -0.002 (2) |
| C54 | 0.032 (3) | 0.033 (3) | 0.031 (3) | -0.004 (2)   | -0.005 (2)  | 0.006 (2)  |
| C55 | 0.025 (2) | 0.035 (3) | 0.027 (3) | -0.005 (2)   | -0.003 (2)  | 0.000 (2)  |
| C56 | 0.026 (2) | 0.037 (3) | 0.023 (3) | -0.001 (2)   | -0.003 (2)  | -0.001 (2) |
| C57 | 0.034 (3) | 0.042 (3) | 0.028 (3) | -0.009 (2)   | -0.001 (2)  | 0.005 (2)  |
| C58 | 0.025 (2) | 0.052 (3) | 0.025 (3) | -0.009 (2)   | 0.003 (2)   | 0.005 (3)  |
| C59 | 0.053 (3) | 0.056 (4) | 0.038 (3) | 0.018 (3)    | -0.014 (3)  | -0.006 (3) |
| C60 | 0.042 (3) | 0.057 (4) | 0.032 (3) | -0.021 (3)   | -0.008 (2)  | 0.009 (3)  |

*Geometric parameters (Å, °)*

|        |           |          |           |
|--------|-----------|----------|-----------|
| O1—C1  | 1.437 (5) | C29—H29B | 0.9800    |
| O1—H1  | 0.8400    | C29—H29C | 0.9800    |
| O2—C30 | 1.430 (6) | C30—H30A | 0.9900    |
| O2—H2  | 0.8400    | C30—H30B | 0.9900    |
| O3—C31 | 1.437 (5) | C31—C32  | 1.519 (7) |
| O3—H3  | 0.8400    | C31—C36  | 1.550 (7) |
| O4—C60 | 1.426 (6) | C31—H31  | 1.0000    |
| O4—H4  | 0.8400    | C32—C33  | 1.543 (6) |
| C1—C2  | 1.516 (6) | C32—H32A | 0.9900    |
| C1—C6  | 1.537 (7) | C32—H32B | 0.9900    |
| C1—H1a | 1.0000    | C33—C34  | 1.519 (6) |
| C2—C3  | 1.542 (6) | C33—H33A | 0.9900    |
| C2—H2A | 0.9900    | C33—H33B | 0.9900    |

## supplementary materials

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|          |           |          |           |
|----------|-----------|----------|-----------|
| C2—H2B   | 0.9900    | C34—C35  | 1.518 (7) |
| C3—C4    | 1.510 (6) | C34—C43  | 1.521 (7) |
| C3—H3A   | 0.9900    | C34—C42  | 1.534 (6) |
| C3—H3B   | 0.9900    | C35—C39  | 1.534 (6) |
| C4—C13   | 1.504 (6) | C35—C36  | 1.551 (6) |
| C4—C5    | 1.532 (6) | C35—H35  | 1.0000    |
| C4—C12   | 1.538 (6) | C36—C38  | 1.525 (7) |
| C5—C9    | 1.534 (6) | C36—C37  | 1.543 (7) |
| C5—C6    | 1.555 (6) | C37—H37A | 0.9800    |
| C5—H5    | 1.0000    | C37—H37B | 0.9800    |
| C6—C8    | 1.533 (7) | C37—H37C | 0.9800    |
| C6—C7    | 1.525 (7) | C38—H38A | 0.9800    |
| C7—H7A   | 0.9800    | C38—H38B | 0.9800    |
| C7—H7B   | 0.9800    | C38—H38C | 0.9800    |
| C7—H7C   | 0.9800    | C39—C40  | 1.532 (6) |
| C8—H8A   | 0.9800    | C39—H39A | 0.9900    |
| C8—H8B   | 0.9800    | C39—H39B | 0.9900    |
| C8—H8C   | 0.9800    | C40—C41  | 1.519 (6) |
| C9—C10   | 1.523 (6) | C40—H40A | 0.9900    |
| C9—H9A   | 0.9900    | C40—H40B | 0.9900    |
| C9—H9B   | 0.9900    | C41—C48  | 1.549 (5) |
| C10—C11  | 1.521 (6) | C41—C42  | 1.549 (6) |
| C10—H10A | 0.9900    | C41—H41  | 1.0000    |
| C10—H10B | 0.9900    | C42—C43  | 1.514 (6) |
| C11—C18  | 1.539 (5) | C42—C44  | 1.544 (6) |
| C11—C12  | 1.552 (6) | C43—H43A | 0.9900    |
| C11—H11  | 1.0000    | C43—H43B | 0.9900    |
| C12—C13  | 1.513 (6) | C44—C45  | 1.546 (6) |
| C12—C14  | 1.536 (6) | C44—H44A | 0.9900    |
| C13—H13A | 0.9900    | C44—H44B | 0.9900    |
| C13—H13B | 0.9900    | C45—C46  | 1.541 (6) |
| C14—C15  | 1.560 (6) | C45—H45A | 0.9900    |
| C14—H14A | 0.9900    | C45—H45B | 0.9900    |
| C14—H14B | 0.9900    | C46—C47  | 1.541 (6) |
| C15—C16  | 1.531 (6) | C46—C52  | 1.564 (6) |
| C15—H15A | 0.9900    | C46—C48  | 1.564 (6) |
| C15—H15B | 0.9900    | C47—H47A | 0.9800    |
| C16—C17  | 1.550 (6) | C47—H47B | 0.9800    |
| C16—C18  | 1.554 (6) | C47—H47C | 0.9800    |
| C16—C22  | 1.558 (5) | C48—C50  | 1.539 (6) |
| C17—H17A | 0.9800    | C48—C49  | 1.543 (6) |
| C17—H17B | 0.9800    | C49—H49A | 0.9800    |
| C17—H17C | 0.9800    | C49—H49B | 0.9800    |
| C18—C20  | 1.534 (6) | C49—H49C | 0.9800    |
| C18—C19  | 1.548 (6) | C50—C51  | 1.546 (6) |
| C19—H19A | 0.9800    | C50—H50A | 0.9900    |
| C19—H19B | 0.9800    | C50—H50B | 0.9900    |
| C19—H19C | 0.9800    | C51—C52  | 1.548 (6) |
| C20—C21  | 1.542 (6) | C51—H51A | 0.9900    |

|            |           |               |           |
|------------|-----------|---------------|-----------|
| C20—H20A   | 0.9900    | C51—H51B      | 0.9900    |
| C20—H20B   | 0.9900    | C52—C53       | 1.533 (6) |
| C21—C22    | 1.563 (6) | C52—H52       | 1.0000    |
| C21—H21A   | 0.9900    | C53—C54       | 1.535 (6) |
| C21—H21B   | 0.9900    | C53—C55       | 1.536 (6) |
| C22—C23    | 1.530 (6) | C53—H53       | 1.0000    |
| C22—H22    | 1.0000    | C54—H54A      | 0.9800    |
| C23—C25    | 1.538 (5) | C54—H54B      | 0.9800    |
| C23—C24    | 1.552 (6) | C54—H54C      | 0.9800    |
| C23—H23    | 1.0000    | C55—C56       | 1.526 (6) |
| C24—H24A   | 0.9800    | C55—H55A      | 0.9900    |
| C24—H24B   | 0.9800    | C55—H55B      | 0.9900    |
| C24—H24C   | 0.9800    | C56—C57       | 1.507 (6) |
| C25—C26    | 1.528 (6) | C56—H56A      | 0.9900    |
| C25—H25A   | 0.9900    | C56—H56B      | 0.9900    |
| C25—H25B   | 0.9900    | C57—C58       | 1.336 (6) |
| C26—C27    | 1.490 (6) | C57—H57       | 0.9500    |
| C26—H26A   | 0.9900    | C58—C59       | 1.503 (7) |
| C26—H26B   | 0.9900    | C58—C60       | 1.506 (7) |
| C27—C28    | 1.344 (6) | C59—H59A      | 0.9800    |
| C27—H27    | 0.9500    | C59—H59B      | 0.9800    |
| C28—C29    | 1.474 (7) | C59—H59C      | 0.9800    |
| C28—C30    | 1.486 (7) | C60—H60A      | 0.9900    |
| C29—H29A   | 0.9800    | C60—H60B      | 0.9900    |
| C1—O1—H1   | 109.5     | C28—C30—H30B  | 109.6     |
| C30—O2—H2  | 109.5     | H30A—C30—H30B | 108.1     |
| C31—O3—H3  | 109.5     | O3—C31—C32    | 110.5 (4) |
| C60—O4—H4  | 109.5     | O3—C31—C36    | 110.1 (4) |
| O1—C1—C2   | 108.5 (4) | C32—C31—C36   | 114.1 (4) |
| O1—C1—C6   | 113.2 (4) | O3—C31—H31    | 107.3     |
| C2—C1—C6   | 113.5 (4) | C32—C31—H31   | 107.3     |
| O1—C1—H1a  | 107.1     | C36—C31—H31   | 107.3     |
| C2—C1—H1a  | 107.1     | C31—C32—C33   | 112.1 (4) |
| C6—C1—H1a  | 107.1     | C31—C32—H32A  | 109.2     |
| C1—C2—C3   | 112.4 (4) | C33—C32—H32A  | 109.2     |
| C1—C2—H2A  | 109.1     | C31—C32—H32B  | 109.2     |
| C3—C2—H2A  | 109.1     | C33—C32—H32B  | 109.2     |
| C1—C2—H2B  | 109.1     | H32A—C32—H32B | 107.9     |
| C3—C2—H2B  | 109.1     | C34—C33—C32   | 108.4 (4) |
| H2A—C2—H2B | 107.8     | C34—C33—H33A  | 110.0     |
| C4—C3—C2   | 110.7 (4) | C32—C33—H33A  | 110.0     |
| C4—C3—H3A  | 109.5     | C34—C33—H33B  | 110.0     |
| C2—C3—H3A  | 109.5     | C32—C33—H33B  | 110.0     |
| C4—C3—H3B  | 109.5     | H33A—C33—H33B | 108.4     |
| C2—C3—H3B  | 109.5     | C35—C34—C33   | 110.5 (4) |
| H3A—C3—H3B | 108.1     | C35—C34—C43   | 122.9 (4) |
| C13—C4—C3  | 116.9 (4) | C33—C34—C43   | 115.4 (4) |
| C13—C4—C5  | 122.6 (4) | C35—C34—C42   | 120.3 (4) |
| C3—C4—C5   | 109.1 (4) | C33—C34—C42   | 120.1 (4) |

## supplementary materials

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|               |           |               |           |
|---------------|-----------|---------------|-----------|
| C13—C4—C12    | 59.6 (3)  | C43—C34—C42   | 59.4 (3)  |
| C3—C4—C12     | 120.5 (4) | C34—C35—C39   | 113.7 (4) |
| C5—C4—C12     | 120.9 (4) | C34—C35—C36   | 113.6 (4) |
| C4—C5—C9      | 112.6 (4) | C39—C35—C36   | 114.1 (4) |
| C4—C5—C6      | 111.7 (4) | C34—C35—H35   | 104.7     |
| C9—C5—C6      | 115.0 (4) | C39—C35—H35   | 104.7     |
| C4—C5—H5      | 105.6     | C36—C35—H35   | 104.7     |
| C9—C5—H5      | 105.6     | C38—C36—C37   | 109.2 (4) |
| C6—C5—H5      | 105.6     | C38—C36—C31   | 110.5 (4) |
| C8—C6—C7      | 109.2 (4) | C37—C36—C31   | 107.4 (4) |
| C8—C6—C1      | 110.3 (4) | C38—C36—C35   | 112.2 (4) |
| C7—C6—C1      | 108.9 (4) | C37—C36—C35   | 110.5 (4) |
| C8—C6—C5      | 112.9 (4) | C31—C36—C35   | 107.0 (4) |
| C7—C6—C5      | 109.0 (4) | C36—C37—H37A  | 109.5     |
| C1—C6—C5      | 106.5 (4) | C36—C37—H37B  | 109.5     |
| C6—C7—H7A     | 109.5     | H37A—C37—H37B | 109.5     |
| C6—C7—H7B     | 109.5     | C36—C37—H37C  | 109.5     |
| H7A—C7—H7B    | 109.5     | H37A—C37—H37C | 109.5     |
| C6—C7—H7C     | 109.5     | H37B—C37—H37C | 109.5     |
| H7A—C7—H7C    | 109.5     | C36—C38—H38A  | 109.5     |
| H7B—C7—H7C    | 109.5     | C36—C38—H38B  | 109.5     |
| C6—C8—H8A     | 109.5     | H38A—C38—H38B | 109.5     |
| C6—C8—H8B     | 109.5     | C36—C38—H38C  | 109.5     |
| H8A—C8—H8B    | 109.5     | H38A—C38—H38C | 109.5     |
| C6—C8—H8C     | 109.5     | H38B—C38—H38C | 109.5     |
| H8A—C8—H8C    | 109.5     | C40—C39—C35   | 110.0 (4) |
| H8B—C8—H8C    | 109.5     | C40—C39—H39A  | 109.7     |
| C10—C9—C5     | 109.6 (4) | C35—C39—H39A  | 109.7     |
| C10—C9—H9A    | 109.8     | C40—C39—H39B  | 109.7     |
| C5—C9—H9A     | 109.8     | C35—C39—H39B  | 109.7     |
| C10—C9—H9B    | 109.8     | H39A—C39—H39B | 108.2     |
| C5—C9—H9B     | 109.8     | C41—C40—C39   | 109.9 (4) |
| H9A—C9—H9B    | 108.2     | C41—C40—H40A  | 109.7     |
| C9—C10—C11    | 110.0 (4) | C39—C40—H40A  | 109.7     |
| C9—C10—H10A   | 109.7     | C41—C40—H40B  | 109.7     |
| C11—C10—H10A  | 109.7     | C39—C40—H40B  | 109.7     |
| C9—C10—H10B   | 109.7     | H40A—C40—H40B | 108.2     |
| C11—C10—H10B  | 109.7     | C40—C41—C48   | 114.1 (4) |
| H10A—C10—H10B | 108.2     | C40—C41—C42   | 111.7 (4) |
| C10—C11—C18   | 114.7 (4) | C48—C41—C42   | 112.6 (3) |
| C10—C11—C12   | 111.6 (4) | C40—C41—H41   | 105.9     |
| C18—C11—C12   | 112.4 (3) | C48—C41—H41   | 105.9     |
| C10—C11—H11   | 105.8     | C42—C41—H41   | 105.9     |
| C18—C11—H11   | 105.8     | C43—C42—C34   | 59.9 (3)  |
| C12—C11—H11   | 105.8     | C43—C42—C44   | 118.6 (4) |
| C13—C12—C14   | 117.8 (4) | C34—C42—C44   | 117.4 (4) |
| C13—C12—C4    | 59.1 (3)  | C43—C42—C41   | 114.3 (4) |
| C14—C12—C4    | 117.1 (4) | C34—C42—C41   | 118.0 (4) |
| C13—C12—C11   | 114.8 (4) | C44—C42—C41   | 116.6 (3) |

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|---------------|-----------|---------------|-----------|
| C14—C12—C11   | 117.6 (3) | C42—C43—C34   | 60.7 (3)  |
| C4—C12—C11    | 117.4 (4) | C42—C43—H43A  | 117.7     |
| C4—C13—C12    | 61.3 (3)  | C34—C43—H43A  | 117.7     |
| C4—C13—H13A   | 117.6     | C42—C43—H43B  | 117.7     |
| C12—C13—H13A  | 117.6     | C34—C43—H43B  | 117.7     |
| C4—C13—H13B   | 117.6     | H43A—C43—H43B | 114.8     |
| C12—C13—H13B  | 117.6     | C42—C44—C45   | 117.4 (4) |
| H13A—C13—H13B | 114.7     | C42—C44—H44A  | 108.0     |
| C12—C14—C15   | 116.2 (4) | C45—C44—H44A  | 108.0     |
| C12—C14—H14A  | 108.2     | C42—C44—H44B  | 108.0     |
| C15—C14—H14A  | 108.2     | C45—C44—H44B  | 108.0     |
| C12—C14—H14B  | 108.2     | H44A—C44—H44B | 107.2     |
| C15—C14—H14B  | 108.2     | C46—C45—C44   | 113.4 (4) |
| H14A—C14—H14B | 107.4     | C46—C45—H45A  | 108.9     |
| C16—C15—C14   | 113.8 (3) | C44—C45—H45A  | 108.9     |
| C16—C15—H15A  | 108.8     | C46—C45—H45B  | 108.9     |
| C14—C15—H15A  | 108.8     | C44—C45—H45B  | 108.9     |
| C16—C15—H15B  | 108.8     | H45A—C45—H45B | 107.7     |
| C14—C15—H15B  | 108.8     | C47—C46—C45   | 108.3 (3) |
| H15A—C15—H15B | 107.7     | C47—C46—C52   | 108.3 (3) |
| C15—C16—C17   | 108.9 (3) | C45—C46—C52   | 117.7 (3) |
| C15—C16—C18   | 109.3 (3) | C47—C46—C48   | 111.8 (3) |
| C17—C16—C18   | 111.4 (3) | C45—C46—C48   | 109.2 (4) |
| C15—C16—C22   | 117.3 (3) | C52—C46—C48   | 101.6 (3) |
| C17—C16—C22   | 107.7 (3) | C46—C47—H47A  | 109.5     |
| C18—C16—C22   | 102.2 (3) | C46—C47—H47B  | 109.5     |
| C16—C17—H17A  | 109.5     | H47A—C47—H47B | 109.5     |
| C16—C17—H17B  | 109.5     | C46—C47—H47C  | 109.5     |
| H17A—C17—H17B | 109.5     | H47A—C47—H47C | 109.5     |
| C16—C17—H17C  | 109.5     | H47B—C47—H47C | 109.5     |
| H17A—C17—H17C | 109.5     | C50—C48—C49   | 107.6 (4) |
| H17B—C17—H17C | 109.5     | C50—C48—C41   | 113.2 (4) |
| C20—C18—C11   | 113.5 (3) | C49—C48—C41   | 111.4 (4) |
| C20—C18—C19   | 107.7 (3) | C50—C48—C46   | 102.8 (3) |
| C11—C18—C19   | 111.2 (4) | C49—C48—C46   | 111.6 (3) |
| C20—C18—C16   | 103.4 (3) | C41—C48—C46   | 110.0 (3) |
| C11—C18—C16   | 109.8 (3) | C48—C49—H49A  | 109.5     |
| C19—C18—C16   | 111.0 (3) | C48—C49—H49B  | 109.5     |
| C18—C19—H19A  | 109.5     | H49A—C49—H49B | 109.5     |
| C18—C19—H19B  | 109.5     | C48—C49—H49C  | 109.5     |
| H19A—C19—H19B | 109.5     | H49A—C49—H49C | 109.5     |
| C18—C19—H19C  | 109.5     | H49B—C49—H49C | 109.5     |
| H19A—C19—H19C | 109.5     | C48—C50—C51   | 105.1 (4) |
| H19B—C19—H19C | 109.5     | C48—C50—H50A  | 110.7     |
| C18—C20—C21   | 105.6 (3) | C51—C50—H50A  | 110.7     |
| C18—C20—H20A  | 110.6     | C48—C50—H50B  | 110.7     |
| C21—C20—H20A  | 110.6     | C51—C50—H50B  | 110.7     |
| C18—C20—H20B  | 110.6     | H50A—C50—H50B | 108.8     |
| C21—C20—H20B  | 110.6     | C52—C51—C50   | 107.6 (4) |

## supplementary materials

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|---------------|-----------|---------------|-----------|
| H20A—C20—H20B | 108.7     | C52—C51—H51A  | 110.2     |
| C20—C21—C22   | 107.2 (3) | C50—C51—H51A  | 110.2     |
| C20—C21—H21A  | 110.3     | C52—C51—H51B  | 110.2     |
| C22—C21—H21A  | 110.3     | C50—C51—H51B  | 110.2     |
| C20—C21—H21B  | 110.3     | H51A—C51—H51B | 108.5     |
| C22—C21—H21B  | 110.3     | C53—C52—C51   | 113.2 (4) |
| H21A—C21—H21B | 108.5     | C53—C52—C46   | 119.4 (4) |
| C23—C22—C16   | 119.4 (4) | C51—C52—C46   | 102.9 (3) |
| C23—C22—C21   | 112.5 (4) | C53—C52—H52   | 106.9     |
| C16—C22—C21   | 103.0 (3) | C51—C52—H52   | 106.9     |
| C23—C22—H22   | 107.1     | C46—C52—H52   | 106.9     |
| C16—C22—H22   | 107.1     | C54—C53—C55   | 110.5 (4) |
| C21—C22—H22   | 107.1     | C54—C53—C52   | 112.9 (4) |
| C22—C23—C25   | 110.9 (3) | C55—C53—C52   | 110.1 (4) |
| C22—C23—C24   | 112.3 (4) | C54—C53—H53   | 107.7     |
| C25—C23—C24   | 110.9 (4) | C55—C53—H53   | 107.7     |
| C22—C23—H23   | 107.5     | C52—C53—H53   | 107.7     |
| C25—C23—H23   | 107.5     | C53—C54—H54A  | 109.5     |
| C24—C23—H23   | 107.5     | C53—C54—H54B  | 109.5     |
| C23—C24—H24A  | 109.5     | H54A—C54—H54B | 109.5     |
| C23—C24—H24B  | 109.5     | C53—C54—H54C  | 109.5     |
| H24A—C24—H24B | 109.5     | H54A—C54—H54C | 109.5     |
| C23—C24—H24C  | 109.5     | H54B—C54—H54C | 109.5     |
| H24A—C24—H24C | 109.5     | C56—C55—C53   | 114.8 (4) |
| H24B—C24—H24C | 109.5     | C56—C55—H55A  | 108.6     |
| C26—C25—C23   | 115.1 (4) | C53—C55—H55A  | 108.6     |
| C26—C25—H25A  | 108.5     | C56—C55—H55B  | 108.6     |
| C23—C25—H25A  | 108.5     | C53—C55—H55B  | 108.6     |
| C26—C25—H25B  | 108.5     | H55A—C55—H55B | 107.5     |
| C23—C25—H25B  | 108.5     | C57—C56—C55   | 111.6 (4) |
| H25A—C25—H25B | 107.5     | C57—C56—H56A  | 109.3     |
| C27—C26—C25   | 112.8 (4) | C55—C56—H56A  | 109.3     |
| C27—C26—H26A  | 109.0     | C57—C56—H56B  | 109.3     |
| C25—C26—H26A  | 109.0     | C55—C56—H56B  | 109.3     |
| C27—C26—H26B  | 109.0     | H56A—C56—H56B | 108.0     |
| C25—C26—H26B  | 109.0     | C58—C57—C56   | 128.7 (5) |
| H26A—C26—H26B | 107.8     | C58—C57—H57   | 115.7     |
| C28—C27—C26   | 128.2 (4) | C56—C57—H57   | 115.7     |
| C28—C27—H27   | 115.9     | C57—C58—C59   | 125.3 (5) |
| C26—C27—H27   | 115.9     | C57—C58—C60   | 119.2 (5) |
| C27—C28—C29   | 124.5 (4) | C59—C58—C60   | 115.5 (4) |
| C27—C28—C30   | 119.9 (5) | C58—C59—H59A  | 109.5     |
| C29—C28—C30   | 115.6 (4) | C58—C59—H59B  | 109.5     |
| C28—C29—H29A  | 109.5     | H59A—C59—H59B | 109.5     |
| C28—C29—H29B  | 109.5     | C58—C59—H59C  | 109.5     |
| H29A—C29—H29B | 109.5     | H59A—C59—H59C | 109.5     |
| C28—C29—H29C  | 109.5     | H59B—C59—H59C | 109.5     |
| H29A—C29—H29C | 109.5     | O4—C60—C58    | 112.1 (4) |
| H29B—C29—H29C | 109.5     | O4—C60—H60A   | 109.2     |

|                 |            |                 |            |
|-----------------|------------|-----------------|------------|
| O2—C30—C28      | 110.5 (4)  | C58—C60—H60A    | 109.2      |
| O2—C30—H30A     | 109.6      | O4—C60—H60B     | 109.2      |
| C28—C30—H30A    | 109.6      | C58—C60—H60B    | 109.2      |
| O2—C30—H30B     | 109.6      | H60A—C60—H60B   | 107.9      |
| O1—C1—C2—C3     | -179.9 (4) | O3—C31—C32—C33  | 179.7 (4)  |
| C6—C1—C2—C3     | 53.4 (5)   | C36—C31—C32—C33 | 55.1 (6)   |
| C1—C2—C3—C4     | -53.2 (5)  | C31—C32—C33—C34 | -56.6 (6)  |
| C2—C3—C4—C13    | -87.5 (5)  | C32—C33—C34—C35 | 58.7 (5)   |
| C2—C3—C4—C5     | 57.1 (5)   | C32—C33—C34—C43 | -86.3 (5)  |
| C2—C3—C4—C12    | -156.4 (4) | C32—C33—C34—C42 | -154.2 (4) |
| C13—C4—C5—C9    | -51.6 (5)  | C33—C34—C35—C39 | 166.6 (4)  |
| C3—C4—C5—C9     | 166.2 (4)  | C43—C34—C35—C39 | -51.5 (6)  |
| C12—C4—C5—C9    | 19.9 (6)   | C42—C34—C35—C39 | 19.6 (6)   |
| C13—C4—C5—C6    | 79.5 (5)   | C33—C34—C35—C36 | -60.6 (5)  |
| C3—C4—C5—C6     | -62.7 (5)  | C43—C34—C35—C36 | 81.2 (5)   |
| C12—C4—C5—C6    | 151.0 (4)  | C42—C34—C35—C36 | 152.4 (4)  |
| O1—C1—C6—C8     | -56.3 (5)  | O3—C31—C36—C38  | -53.7 (5)  |
| C2—C1—C6—C8     | 67.9 (5)   | C32—C31—C36—C38 | 71.1 (5)   |
| O1—C1—C6—C7     | 63.5 (5)   | O3—C31—C36—C37  | 65.3 (5)   |
| C2—C1—C6—C7     | -172.3 (4) | C32—C31—C36—C37 | -169.9 (5) |
| O1—C1—C6—C5     | -179.1 (4) | O3—C31—C36—C35  | -176.1 (4) |
| C2—C1—C6—C5     | -54.9 (5)  | C32—C31—C36—C35 | -51.3 (6)  |
| C4—C5—C6—C8     | -61.4 (5)  | C34—C35—C36—C38 | -67.2 (5)  |
| C9—C5—C6—C8     | 68.4 (6)   | C39—C35—C36—C38 | 65.4 (6)   |
| C4—C5—C6—C7     | 177.1 (4)  | C34—C35—C36—C37 | 170.8 (4)  |
| C9—C5—C6—C7     | -53.1 (6)  | C39—C35—C36—C37 | -56.7 (6)  |
| C4—C5—C6—C1     | 59.8 (5)   | C34—C35—C36—C31 | 54.2 (6)   |
| C9—C5—C6—C1     | -170.3 (4) | C39—C35—C36—C31 | -173.3 (5) |
| C4—C5—C9—C10    | -49.5 (5)  | C34—C35—C39—C40 | -47.9 (6)  |
| C6—C5—C9—C10    | -178.9 (4) | C36—C35—C39—C40 | 179.6 (4)  |
| C5—C9—C10—C11   | 70.3 (5)   | C35—C39—C40—C41 | 68.2 (5)   |
| C9—C10—C11—C18  | 173.8 (4)  | C39—C40—C41—C48 | 174.4 (4)  |
| C9—C10—C11—C12  | -56.8 (5)  | C39—C40—C41—C42 | -56.5 (5)  |
| C3—C4—C12—C13   | 105.3 (5)  | C35—C34—C42—C43 | -112.7 (5) |
| C5—C4—C12—C13   | -112.1 (5) | C33—C34—C42—C43 | 103.4 (5)  |
| C13—C4—C12—C14  | -107.6 (4) | C35—C34—C42—C44 | 138.6 (4)  |
| C3—C4—C12—C14   | -2.3 (6)   | C33—C34—C42—C44 | -5.3 (7)   |
| C5—C4—C12—C14   | 140.2 (4)  | C43—C34—C42—C44 | -108.8 (5) |
| C13—C4—C12—C11  | 103.9 (5)  | C35—C34—C42—C41 | -9.3 (6)   |
| C3—C4—C12—C11   | -150.8 (4) | C33—C34—C42—C41 | -153.2 (4) |
| C5—C4—C12—C11   | -8.2 (6)   | C43—C34—C42—C41 | 103.3 (5)  |
| C10—C11—C12—C13 | 92.5 (5)   | C40—C41—C42—C43 | 94.8 (5)   |
| C18—C11—C12—C13 | -137.0 (4) | C48—C41—C42—C43 | -135.2 (4) |
| C10—C11—C12—C14 | -122.3 (4) | C40—C41—C42—C34 | 27.4 (6)   |
| C18—C11—C12—C14 | 8.2 (6)    | C48—C41—C42—C34 | 157.3 (4)  |
| C10—C11—C12—C4  | 26.0 (6)   | C40—C41—C42—C44 | -120.8 (4) |
| C18—C11—C12—C4  | 156.5 (4)  | C48—C41—C42—C44 | 9.2 (6)    |
| C3—C4—C13—C12   | -111.2 (4) | C44—C42—C43—C34 | 106.9 (5)  |
| C5—C4—C13—C12   | 109.4 (4)  | C41—C42—C43—C34 | -109.5 (4) |

## supplementary materials

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| C14—C12—C13—C4  | 106.6 (4)  | C35—C34—C43—C42 | 108.4 (5)  |
| C11—C12—C13—C4  | -108.3 (4) | C33—C34—C43—C42 | -111.4 (4) |
| C13—C12—C14—C15 | 106.9 (4)  | C43—C42—C44—C45 | 103.8 (5)  |
| C4—C12—C14—C15  | 174.4 (4)  | C34—C42—C44—C45 | 172.7 (4)  |
| C11—C12—C14—C15 | -37.2 (6)  | C41—C42—C44—C45 | -39.0 (6)  |
| C12—C14—C15—C16 | 10.9 (5)   | C42—C44—C45—C46 | 12.5 (6)   |
| C14—C15—C16—C17 | -81.3 (4)  | C44—C45—C46—C47 | -82.4 (4)  |
| C14—C15—C16—C18 | 40.5 (5)   | C44—C45—C46—C52 | 154.6 (4)  |
| C14—C15—C16—C22 | 156.2 (4)  | C44—C45—C46—C48 | 39.6 (5)   |
| C10—C11—C18—C20 | -71.9 (5)  | C40—C41—C48—C50 | -73.8 (5)  |
| C12—C11—C18—C20 | 159.2 (4)  | C42—C41—C48—C50 | 157.5 (4)  |
| C10—C11—C18—C19 | 49.8 (5)   | C40—C41—C48—C49 | 47.7 (5)   |
| C12—C11—C18—C19 | -79.2 (5)  | C42—C41—C48—C49 | -81.0 (5)  |
| C10—C11—C18—C16 | 173.0 (4)  | C40—C41—C48—C46 | 171.9 (4)  |
| C12—C11—C18—C16 | 44.1 (5)   | C42—C41—C48—C46 | 43.3 (5)   |
| C15—C16—C18—C20 | 168.1 (3)  | C47—C46—C48—C50 | -70.8 (4)  |
| C17—C16—C18—C20 | -71.6 (4)  | C45—C46—C48—C50 | 169.4 (3)  |
| C22—C16—C18—C20 | 43.1 (4)   | C52—C46—C48—C50 | 44.4 (4)   |
| C15—C16—C18—C11 | -70.5 (4)  | C47—C46—C48—C49 | 174.1 (3)  |
| C17—C16—C18—C11 | 49.8 (5)   | C45—C46—C48—C49 | 54.3 (4)   |
| C22—C16—C18—C11 | 164.5 (3)  | C52—C46—C48—C49 | -70.6 (4)  |
| C15—C16—C18—C19 | 52.9 (4)   | C47—C46—C48—C41 | 49.9 (5)   |
| C17—C16—C18—C19 | 173.2 (3)  | C45—C46—C48—C41 | -69.8 (4)  |
| C22—C16—C18—C19 | -72.1 (4)  | C52—C46—C48—C41 | 165.2 (4)  |
| C11—C18—C20—C21 | -149.6 (4) | C49—C48—C50—C51 | 86.1 (4)   |
| C19—C18—C20—C21 | 86.8 (4)   | C41—C48—C50—C51 | -150.4 (4) |
| C16—C18—C20—C21 | -30.8 (4)  | C46—C48—C50—C51 | -31.8 (4)  |
| C18—C20—C21—C22 | 6.7 (4)    | C48—C50—C51—C52 | 7.1 (5)    |
| C15—C16—C22—C23 | 76.7 (5)   | C50—C51—C52—C53 | 150.6 (4)  |
| C17—C16—C22—C23 | -46.5 (5)  | C50—C51—C52—C46 | 20.4 (5)   |
| C18—C16—C22—C23 | -163.8 (4) | C47—C46—C52—C53 | -48.1 (5)  |
| C15—C16—C22—C21 | -157.7 (4) | C45—C46—C52—C53 | 75.0 (5)   |
| C17—C16—C22—C21 | 79.1 (4)   | C48—C46—C52—C53 | -165.9 (4) |
| C18—C16—C22—C21 | -38.3 (4)  | C47—C46—C52—C51 | 78.3 (4)   |
| C20—C21—C22—C23 | 149.7 (3)  | C45—C46—C52—C51 | -158.6 (4) |
| C20—C21—C22—C16 | 19.8 (4)   | C48—C46—C52—C51 | -39.5 (4)  |
| C16—C22—C23—C25 | 176.3 (4)  | C51—C52—C53—C54 | 178.0 (4)  |
| C21—C22—C23—C25 | 55.4 (5)   | C46—C52—C53—C54 | -60.7 (5)  |
| C16—C22—C23—C24 | -59.0 (5)  | C51—C52—C53—C55 | 53.9 (5)   |
| C21—C22—C23—C24 | -179.8 (3) | C46—C52—C53—C55 | 175.2 (4)  |
| C22—C23—C25—C26 | -175.7 (4) | C54—C53—C55—C56 | 59.1 (5)   |
| C24—C23—C25—C26 | 58.8 (5)   | C52—C53—C55—C56 | -175.5 (4) |
| C23—C25—C26—C27 | 164.9 (4)  | C53—C55—C56—C57 | 159.8 (4)  |
| C25—C26—C27—C28 | 152.7 (5)  | C55—C56—C57—C58 | 142.3 (5)  |
| C26—C27—C28—C29 | -2.5 (8)   | C56—C57—C58—C59 | 0.3 (9)    |
| C26—C27—C28—C30 | 178.1 (5)  | C56—C57—C58—C60 | 178.1 (5)  |
| C27—C28—C30—O2  | -119.1 (5) | C57—C58—C60—O4  | -119.2 (5) |
| C29—C28—C30—O2  | 61.5 (6)   | C59—C58—C60—O4  | 58.8 (6)   |



Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i>   | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| O1—H1···O2 <sup>i</sup>   | 0.84        | 2.11          | 2.807 (5)             | 140                     |
| O2—H2···O3 <sup>ii</sup>  | 0.84        | 1.97          | 2.784 (5)             | 162                     |
| O3—H3···O4 <sup>iii</sup> | 0.84        | 2.00          | 2.747 (5)             | 148                     |
| O4—H4···O1                | 0.84        | 1.89          | 2.722 (4)             | 173                     |

Symmetry codes: (i)  $-x+1, y+1/2, -z+1$ ; (ii)  $x-2, y-1, z-1$ ; (iii)  $-x+3, y+1/2, -z+2$ .

Fig. 1

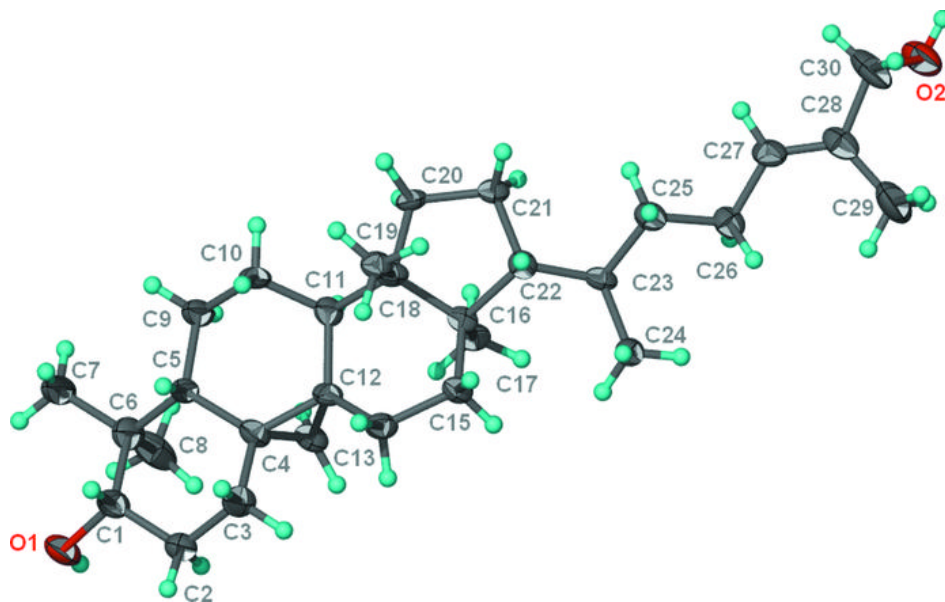


Fig. 2

