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## Structure Reports

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## 3-Hydroxy-8-oxo-3-nor-methyl-chamigrane-2,7-peroxide

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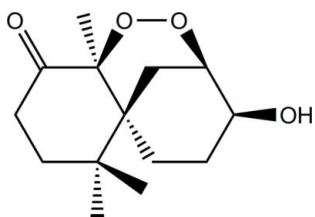
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.103; data-to-parameter ratio = 9.6.

In the title compound,  $\text{C}_{14}\text{H}_{22}\text{O}_4$  (systematic name: 9-hydroxy-1,5,5-trimethyl-1,8-epidioxyspiro[5.5]decan-2-one), which was isolated from the fermentation broth of *Steccherinum ochraceum*, the two six-membered rings adopt chair conformations and are bridged by a peroxide group. The hydroxy H atom forms a three-centre cyclic intermolecular  $\text{O}-\text{H}\cdots(\text{O},\text{O}')$  hydrogen-bonding interaction with a peroxide and a carbonyl O-atom acceptor, forming [100] chains.

## Related literature

For similar structures, see Miyashita *et al.* (1998).



## Experimental

## Crystal data

 $\text{C}_{14}\text{H}_{22}\text{O}_4$  $M_r = 254.32$ 

Orthorhombic,  $P2_12_12_1$   
 $a = 7.3138$  (4) Å  
 $b = 12.4206$  (7) Å  
 $c = 13.9408$  (8) Å  
 $V = 1266.41$  (12) Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 173$  K  
 $0.42 \times 0.38 \times 0.35$  mm

## Data collection

Bruker SMART 1000 CCD diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.961$ ,  $T_{\max} = 0.967$

6456 measured reflections  
1602 independent reflections  
1471 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.103$   
 $S = 1.08$   
1602 reflections

167 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                           | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{O2}-\text{H2A}\cdots\text{O1}^i$ | 0.84  | 2.51        | 3.112 (2)   | 130           |
| $\text{O2}-\text{H2A}\cdots\text{O4}^i$ | 0.84  | 2.17        | 2.943 (2)   | 154           |

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2003); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; 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: ZS2044).

## References

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**supplementary materials**

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### 3-Hydroxy-8-oxo-3-nor-methylchamigrane-2,7-peroxide

D. Liu

#### Comment

Mushrooms have proved to be a rich source of secondary metabolites with unusual structures as well as interesting biological activities. As a continuation of our study characterizing bioactive metabolites from higher fungi in China, a new norsesquiterpene peroxide, 3-hydroxy-8-oxo-3-nor-methylchamigrane-2,7-peroxide, C<sub>14</sub>H<sub>22</sub>O<sub>4</sub> (I) was isolated from the fermentation broth of *Steccherinum ochraceum*. The six-membered C1–C2–C3–C4–C5–C6 and C1–C2–O(1)–O(2)–C7–C6 rings both adopt chair conformations and are bridged by the peroxide group (Fig. 1). The absolute configuration was not determined for the four chiral centres in this compound and the structures of no other compounds with a similar peroxide-bridged cage system are present in the CSD. The hydroxyl H in (I) gives a three-centre cyclic intermolecular O–H···O, O' hydrogen-bonding interaction with both a peroxide and a carbonyl O acceptor, (Table 1) giving one-dimensional chains which extend down the *a* direction of the unit cell.

#### Experimental

*S. ochraceum* was collected from the Ailao Mountain of Yunnan Province, China. The strain was cultured in a liquid medium composed of potato (200 g), glucose (20 g), KH<sub>2</sub>PO<sub>4</sub> (3 g), MgSO<sub>4</sub> (1.5 g), citric acid (0.1 g), and thiamin hydrochloride (10 mg) in 1 L of deionized water. The fungus was grown in reagent bottles (500 mL; media of 300 mL). The pH was adjusted to 6.5 before autoclaving. Fermentation was carried out on a shaker at 22° C and 150 RPM for 25 days. The culture broth (20 L) was filtered to remove the mycelium. The filtrate was then successively extracted twice with ethyl acetate, and the crude extract (3.5 g) was chromatographed on a silica gel column using a CHCl<sub>3</sub>/MeOH gradient. Several fractions of increasing polarity were collected. Fraction II (850 mg) eluted with CHCl<sub>3</sub>/MeOH (100:1, v/v) was subjected to column chromatography over silica gel and Sephadex LH-20, using a petroleum ether/ethyl acetate (8:1, v/v) and CHCl<sub>3</sub>/MeOH (1:1, v/v) eluents respectively, and further purified by repeated recrystallization from MeOH to yield (I) (150 mg). Single crystals were obtained by slow evaporation of a MeOH solution.

#### Refinement

All H atoms were placed in calculated sites and allowed to ride with C–H = 0.98–1.00 Å and O–H = 0.84 Å and  $U_{\text{iso}} = 1.2U_{\text{eq}}$ (methylene) and O(hydroxyl) or  $1.5U_{\text{eq}}$ (methyl). The absolute configuration could not be determined for this light atom compound and Friedel pairs were averaged in the final refinement with the configuration for the four chiral centres as 2*S*,3*R*,6*S*,7*S*.

## Figures

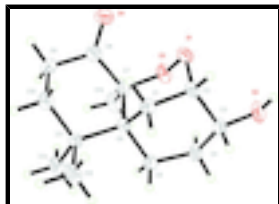


Fig. 1. Molecular configuration and atom numbering scheme for (I), with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

## 9-hydroxy-1,5,5-trimethyl-1,8-epidioxyspiro[5.5]decan-2-one

### Crystal data

|                                  |   |
|----------------------------------|---|
| $C_{14}H_{22}O_4$                | $F(000) = 552$  |
| $M_r = 254.32$                   | $D_x = 1.334 \text{ Mg m}^{-3}$                         |
| Orthorhombic, $P2_12_12_1$       | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: P 2ac 2ab           | Cell parameters from 4034 reflections                   |
| $a = 7.3138 (4) \text{ \AA}$     | $\theta = 2.8\text{--}27.0^\circ$                       |
| $b = 12.4206 (7) \text{ \AA}$    | $\mu = 0.10 \text{ mm}^{-1}$                            |
| $c = 13.9408 (8) \text{ \AA}$    | $T = 173 \text{ K}$                                     |
| $V = 1266.41 (12) \text{ \AA}^3$ | Block, colorless  |
| $Z = 4$                          | $0.42 \times 0.38 \times 0.35 \text{ mm}$               |

### Data collection

|   |  |
|---|--|
| Bruker SMART 1000 CCD diffractometer                        | 1602 independent reflections   |
| Radiation source: fine-focus sealed tube graphite           | 1471 reflections with $I > 2\sigma(I)$                                 |
| $\omega$ scans  | $R_{\text{int}} = 0.021$   |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $\theta_{\text{max}} = 27.0^\circ$ , $\theta_{\text{min}} = 2.2^\circ$ |
| $T_{\text{min}} = 0.961$ , $T_{\text{max}} = 0.967$         | $h = -5 \rightarrow 9$   |
| 6456 measured reflections                                   | $k = -15 \rightarrow 15$   |
|   | $l = -17 \rightarrow 13$   |

### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.103$               | H-atom parameters constrained                                  |
| $S = 1.08$                      | $w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.2837P]$              |
| 1602 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 167 parameters                  | $(\Delta/\sigma)_{\text{max}} < 0.001$                         |
|                                 | $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$            |

0 restraints

$$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$$

*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 ( $\text{\AA}^2$ )*

|      | <i>x</i>   | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|------------|--------------|--------------|----------------------------------|
| C1   | 0.4858 (3) | 0.29411 (15) | 0.68490 (13) | 0.0218 (4)                       |
| H1A  | 0.5496     | 0.2309       | 0.7124       | 0.026*                           |
| H1B  | 0.3532     | 0.2857       | 0.6971       | 0.026*                           |
| C2   | 0.5204 (3) | 0.29930 (15) | 0.57779 (13) | 0.0225 (4)                       |
| H2   | 0.4747     | 0.2304       | 0.5495       | 0.027*                           |
| C3   | 0.4112 (3) | 0.39043 (16) | 0.53239 (13) | 0.0247 (4)                       |
| H3   | 0.2786     | 0.3719       | 0.5380       | 0.030*                           |
| C4   | 0.4405 (3) | 0.49914 (16) | 0.58127 (15) | 0.0296 (5)                       |
| H4A  | 0.3429     | 0.5494       | 0.5608       | 0.036*                           |
| H4B  | 0.5591     | 0.5295       | 0.5602       | 0.036*                           |
| C5   | 0.4393 (3) | 0.49050 (15) | 0.69173 (14) | 0.0259 (4)                       |
| H5A  | 0.3114     | 0.4812       | 0.7134       | 0.031*                           |
| H5B  | 0.4846     | 0.5593       | 0.7187       | 0.031*                           |
| C6   | 0.5554 (2) | 0.39771 (14) | 0.73338 (13) | 0.0183 (4)                       |
| C7   | 0.7632 (3) | 0.40990 (16) | 0.70409 (13) | 0.0229 (4)                       |
| C8   | 0.8819 (3) | 0.32554 (18) | 0.75389 (15) | 0.0280 (4)                       |
| C9   | 0.8623 (3) | 0.3215 (2)   | 0.86128 (15) | 0.0334 (5)                       |
| H9A  | 0.9072     | 0.3896       | 0.8897       | 0.040*                           |
| H9B  | 0.9367     | 0.2617       | 0.8873       | 0.040*                           |
| C10  | 0.6617 (3) | 0.30478 (17) | 0.88798 (14) | 0.0285 (4)                       |
| H10A | 0.6507     | 0.3054       | 0.9588       | 0.034*                           |
| H10B | 0.6229     | 0.2328       | 0.8653       | 0.034*                           |
| C11  | 0.5305 (3) | 0.38957 (16) | 0.84637 (14) | 0.0221 (4)                       |
| C12  | 0.3354 (3) | 0.3526 (2)   | 0.87222 (16) | 0.0331 (5)                       |
| H12A | 0.3233     | 0.3480       | 0.9421       | 0.050*                           |
| H12B | 0.3122     | 0.2817       | 0.8438       | 0.050*                           |
| H12C | 0.2467     | 0.4046       | 0.8472       | 0.050*                           |
| C13  | 0.5579 (3) | 0.49723 (17) | 0.89893 (14) | 0.0312 (5)                       |
| H13A | 0.6821     | 0.5238       | 0.8871       | 0.047*                           |
| H13B | 0.5401     | 0.4866       | 0.9679       | 0.047*                           |
| H13C | 0.4691     | 0.5499       | 0.8752       | 0.047*                           |

## supplementary materials

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|      |            |              |              |            |
|------|------------|--------------|--------------|------------|
| C14  | 0.8524 (3) | 0.52104 (18) | 0.72087 (16) | 0.0341 (5) |
| H14A | 0.7730     | 0.5775       | 0.6949       | 0.051*     |
| H14B | 0.9713     | 0.5235       | 0.6885       | 0.051*     |
| H14C | 0.8695     | 0.5326       | 0.7898       | 0.051*     |
| O1   | 0.7133 (2) | 0.30323 (12) | 0.56148 (10) | 0.0295 (4) |
| O2   | 0.4515 (2) | 0.40426 (12) | 0.43304 (10) | 0.0333 (4) |
| H2A  | 0.4353     | 0.3457       | 0.4041       | 0.050*     |
| O3   | 0.7841 (2) | 0.40379 (13) | 0.60137 (10) | 0.0298 (3) |
| O4   | 0.9872 (2) | 0.26855 (15) | 0.71020 (12) | 0.0423 (5) |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$    |
|-----|-------------|-------------|-------------|--------------|-------------|-------------|
| C1  | 0.0262 (9)  | 0.0173 (8)  | 0.0219 (9)  | -0.0021 (8)  | -0.0017 (7) | -0.0004 (7) |
| C2  | 0.0273 (9)  | 0.0180 (8)  | 0.0222 (9)  | -0.0018 (8)  | -0.0007 (8) | -0.0047 (7) |
| C3  | 0.0310 (10) | 0.0245 (9)  | 0.0187 (8)  | -0.0002 (8)  | -0.0046 (8) | -0.0016 (7) |
| C4  | 0.0439 (12) | 0.0208 (9)  | 0.0242 (9)  | 0.0050 (9)   | -0.0104 (9) | -0.0004 (8) |
| C5  | 0.0338 (10) | 0.0211 (9)  | 0.0229 (9)  | 0.0069 (9)   | -0.0053 (8) | -0.0041 (8) |
| C6  | 0.0198 (8)  | 0.0179 (8)  | 0.0174 (8)  | -0.0001 (7)  | 0.0005 (7)  | -0.0018 (7) |
| C7  | 0.0234 (9)  | 0.0278 (9)  | 0.0174 (8)  | -0.0029 (8)  | -0.0002 (7) | -0.0005 (8) |
| C8  | 0.0204 (9)  | 0.0350 (10) | 0.0284 (10) | 0.0006 (9)   | -0.0018 (8) | -0.0055 (9) |
| C9  | 0.0306 (11) | 0.0433 (12) | 0.0264 (10) | 0.0114 (10)  | -0.0051 (9) | 0.0013 (10) |
| C10 | 0.0342 (11) | 0.0295 (10) | 0.0217 (9)  | 0.0024 (9)   | 0.0005 (8)  | 0.0035 (8)  |
| C11 | 0.0228 (9)  | 0.0254 (9)  | 0.0182 (8)  | 0.0007 (8)   | 0.0011 (7)  | -0.0024 (8) |
| C12 | 0.0274 (10) | 0.0467 (13) | 0.0251 (10) | -0.0053 (10) | 0.0068 (8)  | -0.0040 (9) |
| C13 | 0.0389 (12) | 0.0318 (10) | 0.0228 (9)  | 0.0023 (10)  | -0.0015 (9) | -0.0080 (8) |
| C14 | 0.0363 (11) | 0.0364 (11) | 0.0297 (10) | -0.0155 (10) | 0.0007 (9)  | 0.0004 (9)  |
| O1  | 0.0286 (7)  | 0.0345 (8)  | 0.0253 (7)  | 0.0029 (6)   | 0.0013 (6)  | -0.0095 (6) |
| O2  | 0.0511 (10) | 0.0305 (7)  | 0.0184 (6)  | -0.0015 (8)  | -0.0050 (7) | -0.0015 (6) |
| O3  | 0.0307 (7)  | 0.0378 (8)  | 0.0208 (7)  | -0.0093 (7)  | 0.0037 (6)  | -0.0027 (6) |
| O4  | 0.0329 (8)  | 0.0555 (10) | 0.0386 (9)  | 0.0160 (8)   | -0.0052 (7) | -0.0158 (8) |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|        |           |          |           |
|--------|-----------|----------|-----------|
| C1—C2  | 1.516 (3) | C8—O4    | 1.211 (3) |
| C1—C6  | 1.540 (2) | C8—C9    | 1.505 (3) |
| C1—H1A | 0.9900    | C9—C10   | 1.528 (3) |
| C1—H1B | 0.9900    | C9—H9A   | 0.9900    |
| C2—O1  | 1.430 (2) | C9—H9B   | 0.9900    |
| C2—C3  | 1.523 (3) | C10—C11  | 1.538 (3) |
| C2—H2  | 1.0000    | C10—H10A | 0.9900    |
| C3—O2  | 1.426 (2) | C10—H10B | 0.9900    |
| C3—C4  | 1.528 (3) | C11—C13  | 1.538 (3) |
| C3—H3  | 1.0000    | C11—C12  | 1.542 (3) |
| C4—C5  | 1.544 (3) | C12—H12A | 0.9800    |
| C4—H4A | 0.9900    | C12—H12B | 0.9800    |
| C4—H4B | 0.9900    | C12—H12C | 0.9800    |
| C5—C6  | 1.545 (3) | C13—H13A | 0.9800    |
| C5—H5A | 0.9900    | C13—H13B | 0.9800    |

|            |             |               |             |
|------------|-------------|---------------|-------------|
| C5—H5B     | 0.9900      | C13—H13C      | 0.9800      |
| C6—C7      | 1.581 (3)   | C14—H14A      | 0.9800      |
| C6—C11     | 1.589 (2)   | C14—H14B      | 0.9800      |
| C7—O3      | 1.442 (2)   | C14—H14C      | 0.9800      |
| C7—C8      | 1.527 (3)   | O1—O3         | 1.462 (2)   |
| C7—C14     | 1.545 (3)   | O2—H2A        | 0.8400      |
| C2—C1—C6   | 109.98 (15) | O4—C8—C9      | 122.8 (2)   |
| C2—C1—H1A  | 109.7       | O4—C8—C7      | 122.27 (19) |
| C6—C1—H1A  | 109.7       | C9—C8—C7      | 114.91 (18) |
| C2—C1—H1B  | 109.7       | C8—C9—C10     | 109.78 (18) |
| C6—C1—H1B  | 109.7       | C8—C9—H9A     | 109.7       |
| H1A—C1—H1B | 108.2       | C10—C9—H9A    | 109.7       |
| O1—C2—C1   | 108.82 (16) | C8—C9—H9B     | 109.7       |
| O1—C2—C3   | 115.22 (17) | C10—C9—H9B    | 109.7       |
| C1—C2—C3   | 110.70 (16) | H9A—C9—H9B    | 108.2       |
| O1—C2—H2   | 107.3       | C9—C10—C11    | 114.46 (17) |
| C1—C2—H2   | 107.3       | C9—C10—H10A   | 108.6       |
| C3—C2—H2   | 107.3       | C11—C10—H10A  | 108.6       |
| O2—C3—C2   | 112.61 (17) | C9—C10—H10B   | 108.6       |
| O2—C3—C4   | 107.32 (16) | C11—C10—H10B  | 108.6       |
| C2—C3—C4   | 113.44 (16) | H10A—C10—H10B | 107.6       |
| O2—C3—H3   | 107.7       | C13—C11—C10   | 109.54 (16) |
| C2—C3—H3   | 107.7       | C13—C11—C12   | 105.55 (17) |
| C4—C3—H3   | 107.7       | C10—C11—C12   | 106.57 (17) |
| C3—C4—C5   | 112.51 (16) | C13—C11—C6    | 113.71 (16) |
| C3—C4—H4A  | 109.1       | C10—C11—C6    | 110.25 (15) |
| C5—C4—H4A  | 109.1       | C12—C11—C6    | 110.88 (16) |
| C3—C4—H4B  | 109.1       | C11—C12—H12A  | 109.5       |
| C5—C4—H4B  | 109.1       | C11—C12—H12B  | 109.5       |
| H4A—C4—H4B | 107.8       | H12A—C12—H12B | 109.5       |
| C4—C5—C6   | 115.08 (16) | C11—C12—H12C  | 109.5       |
| C4—C5—H5A  | 108.5       | H12A—C12—H12C | 109.5       |
| C6—C5—H5A  | 108.5       | H12B—C12—H12C | 109.5       |
| C4—C5—H5B  | 108.5       | C11—C13—H13A  | 109.5       |
| C6—C5—H5B  | 108.5       | C11—C13—H13B  | 109.5       |
| H5A—C5—H5B | 107.5       | H13A—C13—H13B | 109.5       |
| C1—C6—C5   | 106.09 (15) | C11—C13—H13C  | 109.5       |
| C1—C6—C7   | 106.51 (15) | H13A—C13—H13C | 109.5       |
| C5—C6—C7   | 111.08 (16) | H13B—C13—H13C | 109.5       |
| C1—C6—C11  | 110.13 (15) | C7—C14—H14A   | 109.5       |
| C5—C6—C11  | 110.94 (15) | C7—C14—H14B   | 109.5       |
| C7—C6—C11  | 111.84 (15) | H14A—C14—H14B | 109.5       |
| O3—C7—C8   | 110.81 (16) | C7—C14—H14C   | 109.5       |
| O3—C7—C14  | 98.79 (15)  | H14A—C14—H14C | 109.5       |
| C8—C7—C14  | 107.71 (16) | H14B—C14—H14C | 109.5       |
| O3—C7—C6   | 110.67 (15) | C2—O1—O3      | 108.54 (14) |
| C8—C7—C6   | 111.29 (16) | C3—O2—H2A     | 109.5       |
| C14—C7—C6  | 116.91 (17) | C7—O3—O1      | 112.66 (14) |

## supplementary materials

|               |              |                |              |
|---------------|--------------|----------------|--------------|
| C6—C1—C2—O1   | 63.6 (2)     | C6—C7—C8—O4    | 128.6 (2)    |
| C6—C1—C2—C3   | -63.9 (2)    | O3—C7—C8—C9    | -176.93 (18) |
| O1—C2—C3—O2   | 50.9 (2)     | C14—C7—C8—C9   | 76.0 (2)     |
| C1—C2—C3—O2   | 174.94 (16)  | C6—C7—C8—C9    | -53.3 (2)    |
| O1—C2—C3—C4   | -71.2 (2)    | O4—C8—C9—C10   | -126.6 (2)   |
| C1—C2—C3—C4   | 52.8 (2)     | C7—C8—C9—C10   | 55.4 (3)     |
| O2—C3—C4—C5   | -168.47 (18) | C8—C9—C10—C11  | -56.0 (3)    |
| C2—C3—C4—C5   | -43.4 (3)    | C9—C10—C11—C13 | -72.0 (2)    |
| C3—C4—C5—C6   | 46.4 (3)     | C9—C10—C11—C12 | 174.30 (18)  |
| C2—C1—C6—C5   | 63.35 (19)   | C9—C10—C11—C6  | 53.9 (2)     |
| C2—C1—C6—C7   | -55.08 (19)  | C1—C6—C11—C13  | -167.83 (16) |
| C2—C1—C6—C11  | -176.53 (15) | C5—C6—C11—C13  | -50.7 (2)    |
| C4—C5—C6—C1   | -55.3 (2)    | C7—C6—C11—C13  | 73.9 (2)     |
| C4—C5—C6—C7   | 60.0 (2)     | C1—C6—C11—C10  | 68.7 (2)     |
| C4—C5—C6—C11  | -174.90 (17) | C5—C6—C11—C10  | -174.15 (16) |
| C1—C6—C7—O3   | 52.6 (2)     | C7—C6—C11—C10  | -49.5 (2)    |
| C5—C6—C7—O3   | -62.5 (2)    | C1—C6—C11—C12  | -49.1 (2)    |
| C11—C6—C7—O3  | 172.94 (15)  | C5—C6—C11—C12  | 68.1 (2)     |
| C1—C6—C7—C8   | -71.10 (18)  | C7—C6—C11—C12  | -167.31 (16) |
| C5—C6—C7—C8   | 173.81 (15)  | C1—C2—O1—O3    | -64.81 (18)  |
| C11—C6—C7—C8  | 49.3 (2)     | C3—C2—O1—O3    | 60.17 (19)   |
| C1—C6—C7—C14  | 164.57 (16)  | C8—C7—O3—O1    | 66.13 (19)   |
| C5—C6—C7—C14  | 49.5 (2)     | C14—C7—O3—O1   | 178.97 (15)  |
| C11—C6—C7—C14 | -75.1 (2)    | C6—C7—O3—O1    | -57.8 (2)    |
| O3—C7—C8—O4   | 5.0 (3)      | C2—O1—O3—C7    | 64.05 (18)   |
| C14—C7—C8—O4  | -102.0 (2)   |                |              |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                   | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|-------|-------------|-------------|---------------|
| O2—H2A $\cdots$ O1 <sup>i</sup> | 0.84  | 2.51        | 3.112 (2)   | 130           |
| O2—H2A $\cdots$ O4 <sup>i</sup> | 0.84  | 2.17        | 2.943 (2)   | 154           |
| C2—H2 $\cdots$ O1 <sup>i</sup>  | 1.00  | 2.49        | 3.231 (2)   | 130           |

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



Fig. 1

