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# Crystal structure of (*E*)-13-{4-[(*Z*)-2-cyano-2-(3,4,5-trimethoxyphenyl)ethenyl]phenyl}parthenolide methanol hemisolvate

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The title compound,  $C_{33}H_{35}NO_6$  [systematic name: (*Z*)-3-(4-[(*E*)-[(*E*)-1a,5-dimethyl-9-oxo-2,3,7,7a-tetrahydrooxireno[2',3':9,10]cyclodeca[1,2-*b*]furan-8(1a*H*,6*H*,9*H*,10a*H*,10b*H*)-ylidene]methyl]phenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile methanol hemisolvate],  $C_{33}H_{35}NO_6 \cdot 0.5CH_3OH$ , was prepared by the reaction of (*Z*)-3-(4-iodophenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile with parthenolide [systematic name: (*E*)-1a,5-dimethyl-8-methylene-2,3,6,7,7a,8,10a,10b-octahydrooxireno[2',3':9,10]cyclodeca[1,2-*b*]furan-9(1a*H*)-one] under Heck reaction conditions. The molecule is built up from fused ten-, five- (lactone) and three-membered (epoxide) rings with a {4-[(*Z*)-2-cyano-2-(3,4,5-trimethoxyphenyl)ethenyl]phenyl}methylidene group as a substituent. The 4-[(*Z*)-2-cyano-2-(3,4,5-trimethoxyphenyl)ethenyl]phenyl group on the parthenolide exocyclic double bond is oriented in a *trans* position to the lactone ring to form the *E* isomer. The dihedral angle between the benzene ring of the phenyl moiety and the lactone ring mean plane is 21.93 (4)°.

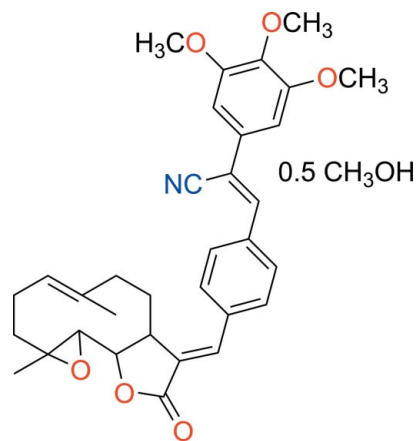
**Keywords:** crystal structure; parthenolide derivatives; Heck synthesis; biological activity.

CCDC reference: 1021449

## 1. Related literature

For the biological activity of parthenolide, see: Hall *et al.* (1979). For the biological activity of parthenolide derivatives similar to the title compound, see: Hanson *et al.* (1970); Hehner *et al.* (1998); Kupchan *et al.* (1971); Neelakantan *et al.*

(2009); Oka *et al.*, 2007); Ralstin *et al.* (2006); Sun *et al.* (2006); Penthala *et al.* (2013*b*). For the synthesis and crystal structures of similar molecules, see: Han *et al.* (2009); Penthala *et al.* (2013*a*). For details of the experimental procedure, see: Hope (1994); Parkin & Hope (1998);



## 2. Experimental

### 2.1. Crystal data

$C_{33}H_{35}NO_6 \cdot 0.5CH_4O$   
 $M_r = 557.64$   
Orthorhombic,  $P2_12_12_1$   
 $a = 9.3347$  (2) Å  
 $b = 16.2442$  (3) Å  
 $c = 19.2580$  (4) Å

$V = 2920.18$  (10) Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 0.71$  mm<sup>-1</sup>  
 $T = 90$  K  
 $0.18 \times 0.15 \times 0.10$  mm

### 2.2. Data collection

Bruker X8 Proteum diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 2008*b*)  
 $T_{min} = 0.836$ ,  $T_{max} = 0.963$

40379 measured reflections  
5349 independent reflections  
5303 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.036$

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.065$   
 $S = 1.03$   
5349 reflections  
387 parameters  
H-atom parameters constrained  
 $\Delta\rho_{max} = 0.14$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.13$  e Å<sup>-3</sup>  
Absolute structure: Flack  $x$  determined using 2283 quotients  $[(I^+)(I^-)]/[(I^-)(I^+)]$  (Parsons *et al.*, 2013)  
Absolute structure parameter: 0.02 (2)

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008*a*); molecular graphics: *XP in SHELXTL* (Sheldrick, 2008*a*); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008*a*), *CIFFIX* (Parkin, 2013), *PLATON* (Spek, 2009) and local program (Parkin, 2000).

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SJ5404).

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## supporting information

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## Crystal structure of (*E*)-13-{4-[(*Z*)-2-cyano-2-(3,4,5-trimethoxyphenyl)-ethenyl]phenyl}parthenolide methanol hemisolvate

Narsimha Reddy Penthala, Shobanbabu Bommagani, Venumadhav Janganati, Sean Parkin and Peter A. Crooks

### S1. Comment

Parthenolide (PTL) and its analogs belong to the class of sesquiterpene lactones. These compounds are currently being used in the development of anti-cancer agents for the treatment of hematological tumours (Sun *et al.*, 2006; Hehner *et al.*, 1998; Ralstin *et al.* 2006; Oka *et al.*, 2007; Kupchan *et al.*, 1971 and Hanson *et al.*, 1970). Recently, we have reported the crystal structure of (*E*)-13-(4-aminophenyl)parthenolide, a Heck reaction derivative of parthenolide (Penthala *et al.* 2013*a*), and we have also reported on *Z*-2-(3,4,5-trimethoxyphenyl)acrylonitrile analogs (Penthala *et al.* 2013*b*) as anti-cancer agents. As part of a program for the development of parthenolide analogs as anti-leukemic agents (Neelakantan *et al.* 2009), and small molecule analogs as anti-cancer agents, our research group is focusing on the synthesis of *E*-olefinic analogues of PTL which can be obtained from the reaction of parthenolide with iodoaromatic reagents utilizing Heck chemistry (Han *et al.* 2009). The title compound was obtained from the reaction of parthenolide with (*Z*)-3-(4-iodophenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile under Heck reaction conditions. To obtain detailed information on the structure of the title compound and to establish the geometry of the exocyclic C13—C14 double bond, a single-crystal X-ray structure determination has been carried out.

Recrystallization of the title compound from methanol afforded light yellow coloured crystals that were suitable for X-ray analysis. The X-ray studies revealed that the title compound was identified as the *E*-isomer (conformation about the exocyclic methylenide C=C bond; the conformation about the C=C bond in the ten-membered ring is also *E*). The molecule is built up from fused ten-, five- (lactone) and three-membered (epoxide) rings with a (*Z*)-3-(4-phenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile group as a substituent. The dihedral angle between the benzene ring of the phenyl moiety and the lactone ring mean plane is 21.93 (4) Å.

### S2. Experimental

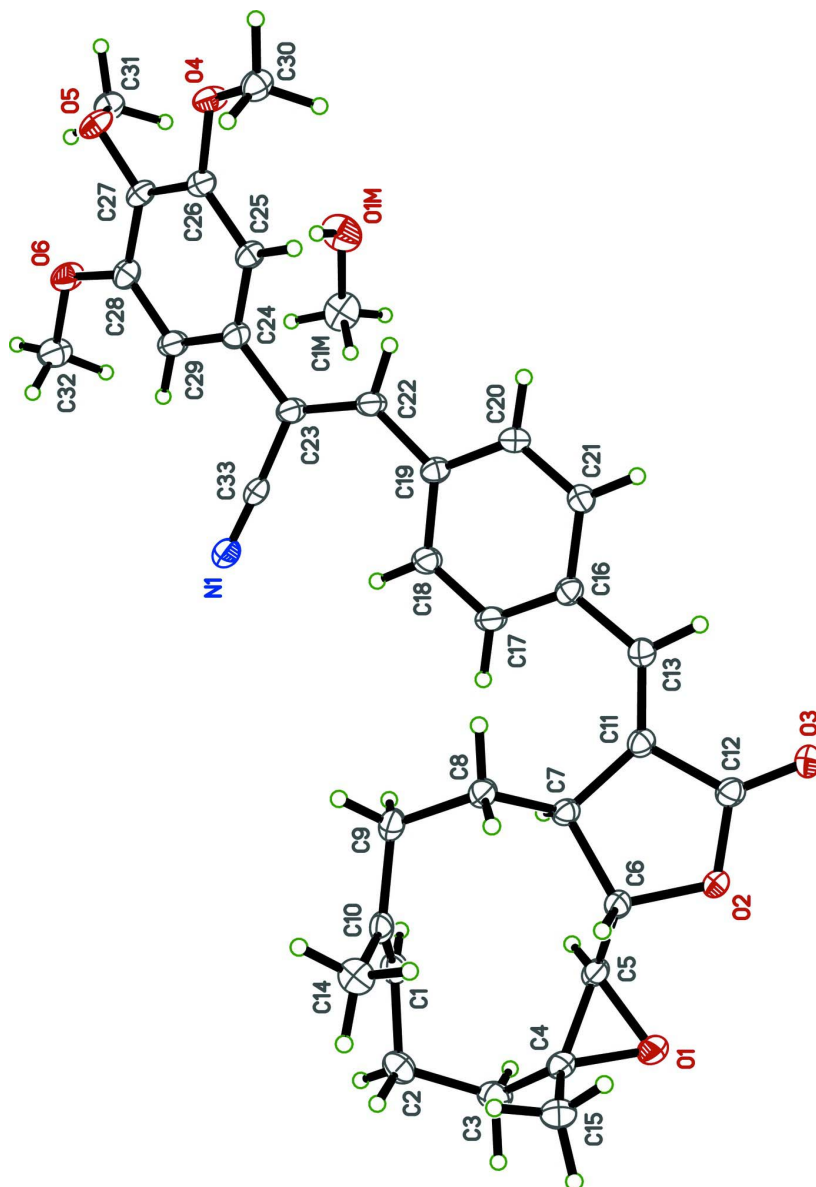
A mixture of parthenolide (1.0 mmol), diisopropylethylamine (3.0 mmol), and (*Z*)-3-(4-iodophenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile (1.1 mmol) in toluene (1 ml) was treated with palladium (II) ferrocene (0.01 mmol) and then stirred at 353 K for 24 h. The reaction mixture was cooled to room temperature, water (8 ml) was added, and the mixture was extracted with ethyl acetate (10 mlx3). The separated organics were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The obtained crude residue was purified using silica flash chromatography (7:3, hexanes/EtOAc) to afford the title compound, which was recrystallized from methanol as light yellow coloured crystals suitable for X-ray analysis (87% yield; M.P.: 478–480 K); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 3.6 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.46 (s, 1H), 6.88 (s, 2H), 5.29 (d, *J* = 11.2 Hz, 1H), 3.94 (s, 6H, 2xOCH<sub>3</sub>), 3.90 (s, 3H, OCH<sub>3</sub>), 3.3 (m, 1H), 2.85 (d, *J* = 8.4 Hz, 1H), 2.41–2.46 (m, 1H), 2.10–2.27 (m, 5H), 1.69 (s, 3H, CH<sub>3</sub>), 1.46–1.55 (m, 2H), 1.32

(s, 3H, CH<sub>3</sub>), 1.27–1.30 (m, 1H) *p.p.m.*. <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 17.60, 17.70, 24.54, 30.58, 36.33, 42.13, 47.16, 55.60, 61.27, 61.94, 66.71, 83.30, 103.75, 113.21, 118.04, 125.48, 129.53, 129.95, 130.56, 130.97, 134.88, 134.96, 135.69, 137.06, 139.77, 140.34, 153.89, 170.85 *p.p.m.*.

### S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were found in difference Fourier maps, but subsequently included in the refinement using riding models, with constrained distances set to 0.95Å (C<sub>sp2</sub>H), 0.98Å (RCH<sub>3</sub>), 0.99Å (R<sub>2</sub>CH<sub>2</sub>), 1.00Å (R<sub>3</sub>CH) and 0.84Å (OH). *U*<sub>iso</sub>(H) parameters were set to values of either 1.2*U*<sub>eq</sub> or 1.5*U*<sub>eq</sub> (RCH<sub>3</sub> and OH only) of the attached atom.

The partial occupancy methanol molecule refined to an occupancy of about one half. For the final rounds of refinement its occupancy was fixed at exactly 0.5 for the sake of simplicity. This is reasonable because other crystals from the same batch would almost certainly have had varying amounts of solvent incorporated, due to unpredictable rates of solvent loss dependent on such things as crystal handling. The position of this half-occupancy methanol is consistent with an O—H···*π* weak hydrogen bonding interaction in which the distance between atom O1M and the centroid of the trimethoxy-phenyl ring (C24-C29) is 3.212 (3)Å.



**Figure 1**

A view of the molecule with displacement ellipsoids drawn at the 50% probability level.

**(Z)-3-(4-((E)-[(E)-1a,5-Dimethyl-9-oxo-2,3,7,7a-tetrahydrooxireno[2',3':9,10]cyclodeca[1,2-b]furan-8(1aH,6H,9H,10aH,10bH)-ylidene)methyl)phenyl)-2-(3,4,5-trimethoxyphenyl)acrylonitrile methanol hemisolvate**

*Crystal data*

$C_{33}H_{35}NO_6 \cdot 0.5CH_4O$   
 $M_r = 557.64$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 9.3347(2) \text{ \AA}$   
 $b = 16.2442(3) \text{ \AA}$   
 $c = 19.2580(4) \text{ \AA}$   
 $V = 2920.18(10) \text{ \AA}^3$

$Z = 4$   
 $F(000) = 1188$   
 $D_x = 1.268 \text{ Mg m}^{-3}$   
 Cu  $K\alpha$  radiation,  $\lambda = 1.54178 \text{ \AA}$   
 Cell parameters from 9693 reflections  
 $\theta = 3.6\text{--}68.4^\circ$   
 $\mu = 0.71 \text{ mm}^{-1}$

$T = 90$  K  $0.18 \times 0.15 \times 0.10$  mm  
 Irregular cut wedge, pale yellow

*Data collection*

|  |  |
|--|--|
| Bruker X8 Proteum<br>diffractometer<br>Radiation source: fine-focus rotating anode<br>Detector resolution: $5.6$ pixels $\text{mm}^{-1}$<br>$\varphi$ and $\omega$ scans<br>Absorption correction: multi-scan<br>(SADABS; Sheldrick, 2008b)<br>$T_{\min} = 0.836$ , $T_{\max} = 0.963$ | 40379 measured reflections<br>5349 independent reflections<br>5303 reflections with $I > 2\sigma(I)$<br>$R_{\text{int}} = 0.036$<br>$\theta_{\text{max}} = 68.4^\circ$ , $\theta_{\text{min}} = 3.6^\circ$<br>$h = -11 \rightarrow 11$<br>$k = -13 \rightarrow 19$<br>$l = -20 \rightarrow 23$ |
|--|--|

*Refinement*

|   |  |
|---|--|
| Refinement on $F^2$<br>Least-squares matrix: full<br>$R[F^2 > 2\sigma(F^2)] = 0.024$<br>$wR(F^2) = 0.065$<br>$S = 1.03$<br>5349 reflections<br>387 parameters<br>0 restraints<br>Primary atom site location: structure-invariant<br>direct methods<br>Secondary atom site location: difference Fourier<br>map<br>Hydrogen site location: difference Fourier map | H-atom parameters constrained<br>$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 0.5907P]$<br>where $P = (F_o^2 + 2F_c^2)/3$<br>$(\Delta/\sigma)_{\text{max}} < 0.001$<br>$\Delta\rho_{\text{max}} = 0.14$ e $\text{\AA}^{-3}$<br>$\Delta\rho_{\text{min}} = -0.13$ e $\text{\AA}^{-3}$<br>Extinction correction: SHELXL2014 (Sheldrick,<br>2008a), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$<br>Extinction coefficient: 0.00092 (14)<br>Absolute structure: Flack $x$ determined using<br>2283 quotients $[(I+)-(I-)]/[(I+)+(I-)]$ (Parsons <i>et</i><br><i>al.</i> , 2013)<br>Absolute structure parameter: 0.02 (2) |
|---|--|

*Special details*

**Experimental.** The crystal was mounted with polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid nitrogen based cryostat, according to published methods (Hope, 1994; Parkin & Hope, 1998). Diffraction data were collected with the crystal at 90 K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

**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 progress was checked using PLATON (Spek, 2009) and by an  $R$ -tensor (Parkin, 2000). The final model was further checked with the IUCr utility checkCIF.

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

|    | $x$          | $y$          | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|----|--------------|--------------|--------------|----------------------------------|-----------|
| O1 | 0.48351 (13) | 0.18048 (7)  | 0.54248 (6)  | 0.0242 (3)                       |           |
| O2 | 0.35142 (13) | 0.32648 (7)  | 0.48438 (5)  | 0.0210 (2)                       |           |
| O3 | 0.34451 (13) | 0.45934 (7)  | 0.45520 (6)  | 0.0237 (3)                       |           |
| O4 | 0.02191 (12) | 0.41510 (7)  | -0.22367 (6) | 0.0225 (3)                       |           |
| O5 | 0.14060 (12) | 0.30869 (7)  | -0.31036 (5) | 0.0219 (2)                       |           |
| O6 | 0.30389 (14) | 0.18270 (7)  | -0.26366 (6) | 0.0261 (3)                       |           |
| N1 | 0.35753 (19) | 0.14312 (9)  | 0.01607 (8)  | 0.0311 (4)                       |           |
| C1 | 0.35744 (17) | 0.04178 (10) | 0.37932 (8)  | 0.0203 (3)                       |           |
| H1 | 0.4318       | 0.0590       | 0.3489       | 0.024*                           |           |

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|      |              |               |              |            |
|------|--------------|---------------|--------------|------------|
| C2   | 0.40090 (19) | -0.01340 (10) | 0.43865 (9)  | 0.0231 (3) |
| H2A  | 0.4617       | -0.0586       | 0.4208       | 0.028*     |
| H2B  | 0.3143       | -0.0381       | 0.4598       | 0.028*     |
| C3   | 0.48435 (19) | 0.03537 (10)  | 0.49458 (9)  | 0.0249 (4) |
| H3A  | 0.5000       | 0.0001        | 0.5358       | 0.030*     |
| H3B  | 0.5792       | 0.0516        | 0.4761       | 0.030*     |
| C4   | 0.40199 (18) | 0.11124 (10)  | 0.51545 (8)  | 0.0213 (3) |
| C5   | 0.42653 (17) | 0.18504 (10)  | 0.47296 (8)  | 0.0187 (3) |
| H5   | 0.5005       | 0.1772        | 0.4360       | 0.022*     |
| C6   | 0.31309 (17) | 0.24713 (9)   | 0.45417 (8)  | 0.0180 (3) |
| H6   | 0.2182       | 0.2288        | 0.4727       | 0.022*     |
| C7   | 0.30239 (17) | 0.26039 (9)   | 0.37424 (8)  | 0.0170 (3) |
| H7   | 0.3909       | 0.2376        | 0.3522       | 0.020*     |
| C8   | 0.17085 (17) | 0.21982 (10)  | 0.33974 (8)  | 0.0197 (3) |
| H8A  | 0.0932       | 0.2152        | 0.3745       | 0.024*     |
| H8B  | 0.1362       | 0.2559        | 0.3019       | 0.024*     |
| C9   | 0.20236 (18) | 0.13380 (10)  | 0.30982 (8)  | 0.0211 (3) |
| H9A  | 0.1211       | 0.1168        | 0.2801       | 0.025*     |
| H9B  | 0.2887       | 0.1370        | 0.2801       | 0.025*     |
| C10  | 0.22602 (17) | 0.06913 (9)   | 0.36481 (8)  | 0.0189 (3) |
| C11  | 0.30617 (16) | 0.35309 (9)   | 0.36819 (8)  | 0.0175 (3) |
| C12  | 0.33264 (17) | 0.38809 (9)   | 0.43821 (8)  | 0.0191 (3) |
| C13  | 0.29827 (17) | 0.40414 (9)   | 0.31380 (8)  | 0.0188 (3) |
| H13  | 0.2959       | 0.4610        | 0.3254       | 0.023*     |
| C14  | 0.09048 (18) | 0.04115 (11)  | 0.39995 (9)  | 0.0253 (4) |
| H14A | 0.1121       | -0.0048       | 0.4313       | 0.038*     |
| H14B | 0.0213       | 0.0231        | 0.3648       | 0.038*     |
| H14C | 0.0498       | 0.0869        | 0.4267       | 0.038*     |
| C15  | 0.2653 (2)   | 0.09726 (11)  | 0.55515 (9)  | 0.0265 (4) |
| H15A | 0.2874       | 0.0715        | 0.5999       | 0.040*     |
| H15B | 0.2021       | 0.0610        | 0.5283       | 0.040*     |
| H15C | 0.2175       | 0.1501        | 0.5631       | 0.040*     |
| C16  | 0.29273 (16) | 0.38648 (9)   | 0.23919 (8)  | 0.0175 (3) |
| C17  | 0.33360 (19) | 0.31137 (10)  | 0.20977 (8)  | 0.0217 (3) |
| H17  | 0.3699       | 0.2690        | 0.2389       | 0.026*     |
| C18  | 0.32232 (19) | 0.29741 (10)  | 0.13921 (8)  | 0.0226 (3) |
| H18  | 0.3514       | 0.2459        | 0.1207       | 0.027*     |
| C19  | 0.26861 (17) | 0.35823 (10)  | 0.09458 (8)  | 0.0182 (3) |
| C20  | 0.23537 (17) | 0.43490 (10)  | 0.12358 (8)  | 0.0189 (3) |
| H20  | 0.2037       | 0.4782        | 0.0942       | 0.023*     |
| C21  | 0.24764 (17) | 0.44897 (9)   | 0.19415 (8)  | 0.0191 (3) |
| H21  | 0.2252       | 0.5018        | 0.2123       | 0.023*     |
| C22  | 0.24427 (18) | 0.34952 (10)  | 0.01995 (8)  | 0.0202 (3) |
| H22  | 0.2131       | 0.3986        | -0.0022      | 0.024*     |
| C23  | 0.25803 (18) | 0.28451 (10)  | -0.02315 (8) | 0.0201 (3) |
| C24  | 0.22173 (17) | 0.28790 (10)  | -0.09850 (8) | 0.0201 (3) |
| C25  | 0.13268 (17) | 0.35015 (10)  | -0.12349 (8) | 0.0194 (3) |
| H25  | 0.0903       | 0.3882        | -0.0922      | 0.023*     |

|      |               |              |               |            |     |
|------|---------------|--------------|---------------|------------|-----|
| C26  | 0.10621 (17)  | 0.35628 (10) | -0.19439 (8)  | 0.0188 (3) |     |
| C27  | 0.16914 (17)  | 0.30063 (10) | -0.24085 (8)  | 0.0189 (3) |     |
| C28  | 0.25283 (18)  | 0.23611 (10) | -0.21509 (8)  | 0.0213 (3) |     |
| C29  | 0.27962 (18)  | 0.22993 (10) | -0.14397 (8)  | 0.0229 (3) |     |
| H29  | 0.3372        | 0.1863       | -0.1266       | 0.027*     |     |
| C30  | -0.07334 (18) | 0.45919 (11) | -0.17877 (8)  | 0.0228 (3) |     |
| H30A | -0.1350       | 0.4201       | -0.1541       | 0.034*     |     |
| H30B | -0.1327       | 0.4966       | -0.2064       | 0.034*     |     |
| H30C | -0.0175       | 0.4910       | -0.1451       | 0.034*     |     |
| C31  | 0.26452 (18)  | 0.32355 (10) | -0.35293 (8)  | 0.0219 (3) |     |
| H31A | 0.3352        | 0.3552       | -0.3265       | 0.033*     |     |
| H31B | 0.2362        | 0.3547       | -0.3943       | 0.033*     |     |
| H31C | 0.3065        | 0.2709       | -0.3671       | 0.033*     |     |
| C32  | 0.3951 (2)    | 0.11810 (11) | -0.24020 (9)  | 0.0294 (4) |     |
| H32A | 0.4758        | 0.1416       | -0.2143       | 0.044*     |     |
| H32B | 0.4314        | 0.0873       | -0.2802       | 0.044*     |     |
| H32C | 0.3409        | 0.0810       | -0.2099       | 0.044*     |     |
| C33  | 0.31313 (19)  | 0.20649 (10) | 0.00030 (8)   | 0.0219 (3) |     |
| O1M  | 0.4544 (3)    | 0.42249 (17) | -0.16592 (16) | 0.0371 (6) | 0.5 |
| H1M  | 0.3705        | 0.4054       | -0.1596       | 0.056*     | 0.5 |
| C1M  | 0.5516 (4)    | 0.3704 (3)   | -0.1313 (2)   | 0.0338 (8) | 0.5 |
| H1M1 | 0.6414        | 0.4001       | -0.1230       | 0.051*     | 0.5 |
| H1M2 | 0.5705        | 0.3218       | -0.1600       | 0.051*     | 0.5 |
| H1M3 | 0.5104        | 0.3532       | -0.0868       | 0.051*     | 0.5 |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|-------------|------------|------------|-------------|-------------|-------------|
| O1  | 0.0286 (6)  | 0.0255 (6) | 0.0187 (5) | -0.0042 (5) | -0.0075 (5) | 0.0020 (5)  |
| O2  | 0.0323 (6)  | 0.0182 (5) | 0.0125 (5) | -0.0012 (5) | -0.0003 (5) | -0.0012 (4) |
| O3  | 0.0345 (7)  | 0.0187 (5) | 0.0181 (5) | -0.0004 (5) | -0.0002 (5) | -0.0042 (4) |
| O4  | 0.0247 (6)  | 0.0275 (6) | 0.0153 (5) | 0.0082 (5)  | -0.0013 (5) | 0.0017 (5)  |
| O5  | 0.0190 (6)  | 0.0349 (6) | 0.0118 (5) | 0.0016 (5)  | -0.0008 (4) | 0.0001 (5)  |
| O6  | 0.0331 (7)  | 0.0291 (6) | 0.0161 (5) | 0.0110 (5)  | -0.0011 (5) | -0.0044 (5) |
| N1  | 0.0477 (10) | 0.0227 (7) | 0.0228 (7) | 0.0045 (7)  | -0.0131 (7) | -0.0036 (6) |
| C1  | 0.0219 (8)  | 0.0184 (7) | 0.0205 (8) | -0.0015 (6) | 0.0034 (6)  | -0.0030 (6) |
| C2  | 0.0208 (8)  | 0.0193 (7) | 0.0293 (9) | 0.0014 (6)  | 0.0023 (7)  | 0.0014 (7)  |
| C3  | 0.0251 (8)  | 0.0239 (8) | 0.0258 (8) | 0.0009 (7)  | -0.0036 (7) | 0.0060 (7)  |
| C4  | 0.0245 (8)  | 0.0228 (8) | 0.0165 (7) | -0.0033 (7) | -0.0049 (7) | 0.0017 (6)  |
| C5  | 0.0200 (8)  | 0.0223 (8) | 0.0137 (7) | -0.0030 (6) | -0.0017 (6) | -0.0007 (6) |
| C6  | 0.0217 (8)  | 0.0178 (7) | 0.0145 (7) | -0.0029 (6) | 0.0008 (6)  | -0.0011 (6) |
| C7  | 0.0189 (7)  | 0.0183 (7) | 0.0137 (7) | 0.0018 (6)  | 0.0008 (6)  | -0.0001 (6) |
| C8  | 0.0226 (8)  | 0.0192 (7) | 0.0172 (7) | 0.0022 (6)  | -0.0032 (6) | -0.0011 (6) |
| C9  | 0.0252 (8)  | 0.0208 (8) | 0.0174 (7) | -0.0003 (6) | -0.0019 (6) | -0.0049 (6) |
| C10 | 0.0232 (8)  | 0.0165 (7) | 0.0169 (7) | -0.0015 (6) | 0.0018 (6)  | -0.0057 (6) |
| C11 | 0.0179 (7)  | 0.0192 (7) | 0.0154 (7) | 0.0020 (6)  | 0.0005 (6)  | -0.0017 (6) |
| C12 | 0.0209 (8)  | 0.0205 (8) | 0.0158 (7) | 0.0009 (6)  | 0.0022 (6)  | 0.0000 (6)  |
| C13 | 0.0206 (7)  | 0.0175 (7) | 0.0183 (7) | 0.0008 (6)  | -0.0012 (6) | -0.0012 (6) |



|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C14 | 0.0209 (8)  | 0.0256 (8)  | 0.0295 (9)  | 0.0005 (7)   | 0.0010 (7)  | 0.0009 (7)   |
| C15 | 0.0329 (9)  | 0.0265 (8)  | 0.0201 (8)  | -0.0039 (7)  | 0.0032 (7)  | 0.0039 (7)   |
| C16 | 0.0160 (7)  | 0.0200 (7)  | 0.0165 (7)  | -0.0013 (6)  | -0.0008 (6) | 0.0002 (6)   |
| C17 | 0.0293 (9)  | 0.0201 (7)  | 0.0158 (7)  | 0.0052 (7)   | -0.0011 (6) | 0.0029 (6)   |
| C18 | 0.0316 (9)  | 0.0192 (8)  | 0.0172 (7)  | 0.0065 (7)   | -0.0005 (7) | -0.0003 (6)  |
| C19 | 0.0194 (7)  | 0.0205 (7)  | 0.0147 (7)  | 0.0005 (6)   | 0.0011 (6)  | 0.0015 (6)   |
| C20 | 0.0196 (7)  | 0.0192 (7)  | 0.0178 (7)  | 0.0018 (6)   | -0.0006 (6) | 0.0040 (6)   |
| C21 | 0.0226 (8)  | 0.0164 (7)  | 0.0183 (7)  | 0.0002 (6)   | 0.0002 (6)  | -0.0005 (6)  |
| C22 | 0.0245 (8)  | 0.0202 (7)  | 0.0159 (7)  | 0.0027 (7)   | -0.0007 (6) | 0.0042 (6)   |
| C23 | 0.0226 (8)  | 0.0219 (8)  | 0.0158 (7)  | 0.0015 (7)   | -0.0013 (6) | 0.0023 (6)   |
| C24 | 0.0237 (8)  | 0.0215 (7)  | 0.0151 (7)  | -0.0016 (6)  | -0.0008 (6) | 0.0014 (6)   |
| C25 | 0.0215 (8)  | 0.0227 (7)  | 0.0140 (7)  | 0.0003 (6)   | 0.0002 (6)  | -0.0006 (6)  |
| C26 | 0.0177 (7)  | 0.0217 (7)  | 0.0170 (7)  | -0.0006 (6)  | -0.0011 (6) | 0.0024 (6)   |
| C27 | 0.0174 (7)  | 0.0263 (8)  | 0.0130 (7)  | -0.0016 (6)  | -0.0006 (6) | 0.0010 (6)   |
| C28 | 0.0225 (8)  | 0.0245 (8)  | 0.0168 (7)  | 0.0003 (7)   | 0.0003 (6)  | -0.0029 (6)  |
| C29 | 0.0277 (8)  | 0.0229 (8)  | 0.0181 (8)  | 0.0047 (7)   | -0.0028 (6) | 0.0007 (6)   |
| C30 | 0.0222 (8)  | 0.0269 (8)  | 0.0192 (8)  | 0.0048 (7)   | -0.0010 (6) | -0.0031 (7)  |
| C31 | 0.0230 (8)  | 0.0264 (8)  | 0.0165 (7)  | 0.0005 (7)   | 0.0029 (6)  | 0.0007 (6)   |
| C32 | 0.0359 (10) | 0.0292 (9)  | 0.0231 (8)  | 0.0125 (8)   | -0.0005 (8) | -0.0024 (7)  |
| C33 | 0.0301 (8)  | 0.0225 (8)  | 0.0130 (7)  | -0.0001 (7)  | -0.0046 (6) | -0.0031 (6)  |
| O1M | 0.0289 (13) | 0.0360 (14) | 0.0465 (16) | -0.0027 (12) | 0.0055 (12) | 0.0064 (13)  |
| C1M | 0.0213 (17) | 0.044 (2)   | 0.0359 (19) | -0.0039 (15) | 0.0058 (16) | -0.0010 (18) |

*Geometric parameters (Å, °)*

|        |             |          |           |
|--------|-------------|----------|-----------|
| O1—C5  | 1.4426 (18) | C14—H14B | 0.9800    |
| O1—C4  | 1.454 (2)   | C14—H14C | 0.9800    |
| O2—C12 | 1.3500 (19) | C15—H15A | 0.9800    |
| O2—C6  | 1.4587 (18) | C15—H15B | 0.9800    |
| O3—C12 | 1.208 (2)   | C15—H15C | 0.9800    |
| O4—C26 | 1.3602 (19) | C16—C17  | 1.398 (2) |
| O4—C30 | 1.4321 (19) | C16—C21  | 1.400 (2) |
| O5—C27 | 1.3713 (18) | C17—C18  | 1.382 (2) |
| O5—C31 | 1.4381 (19) | C17—H17  | 0.9500    |
| O6—C28 | 1.3619 (19) | C18—C19  | 1.402 (2) |
| O6—C32 | 1.425 (2)   | C18—H18  | 0.9500    |
| N1—C33 | 1.151 (2)   | C19—C20  | 1.400 (2) |
| C1—C10 | 1.334 (2)   | C19—C22  | 1.462 (2) |
| C1—C2  | 1.508 (2)   | C20—C21  | 1.383 (2) |
| C1—H1  | 0.9500      | C20—H20  | 0.9500    |
| C2—C3  | 1.547 (2)   | C21—H21  | 0.9500    |
| C2—H2A | 0.9900      | C22—C23  | 1.349 (2) |
| C2—H2B | 0.9900      | C22—H22  | 0.9500    |
| C3—C4  | 1.507 (2)   | C23—C33  | 1.440 (2) |
| C3—H3A | 0.9900      | C23—C24  | 1.491 (2) |
| C3—H3B | 0.9900      | C24—C29  | 1.395 (2) |
| C4—C5  | 1.470 (2)   | C24—C25  | 1.395 (2) |
| C4—C15 | 1.505 (2)   | C25—C26  | 1.391 (2) |

|            |             |               |             |
|------------|-------------|---------------|-------------|
| C5—C6      | 1.506 (2)   | C25—H25       | 0.9500      |
| C5—H5      | 1.0000      | C26—C27       | 1.401 (2)   |
| C6—C7      | 1.558 (2)   | C27—C28       | 1.398 (2)   |
| C6—H6      | 1.0000      | C28—C29       | 1.396 (2)   |
| C7—C11     | 1.511 (2)   | C29—H29       | 0.9500      |
| C7—C8      | 1.544 (2)   | C30—H30A      | 0.9800      |
| C7—H7      | 1.0000      | C30—H30B      | 0.9800      |
| C8—C9      | 1.540 (2)   | C30—H30C      | 0.9800      |
| C8—H8A     | 0.9900      | C31—H31A      | 0.9800      |
| C8—H8B     | 0.9900      | C31—H31B      | 0.9800      |
| C9—C10     | 1.508 (2)   | C31—H31C      | 0.9800      |
| C9—H9A     | 0.9900      | C32—H32A      | 0.9800      |
| C9—H9B     | 0.9900      | C32—H32B      | 0.9800      |
| C10—C14    | 1.505 (2)   | C32—H32C      | 0.9800      |
| C11—C13    | 1.338 (2)   | O1M—C1M       | 1.408 (5)   |
| C11—C12    | 1.484 (2)   | O1M—H1M       | 0.8400      |
| C13—C16    | 1.466 (2)   | C1M—H1M1      | 0.9800      |
| C13—H13    | 0.9500      | C1M—H1M2      | 0.9800      |
| C14—H14A   | 0.9800      | C1M—H1M3      | 0.9800      |
|            |             |               |             |
| C5—O1—C4   | 60.96 (10)  | C4—C15—H15A   | 109.5       |
| C12—O2—C6  | 111.14 (11) | C4—C15—H15B   | 109.5       |
| C26—O4—C30 | 117.40 (12) | H15A—C15—H15B | 109.5       |
| C27—O5—C31 | 114.60 (12) | C4—C15—H15C   | 109.5       |
| C28—O6—C32 | 117.42 (12) | H15A—C15—H15C | 109.5       |
| C10—C1—C2  | 127.16 (15) | H15B—C15—H15C | 109.5       |
| C10—C1—H1  | 116.4       | C17—C16—C21   | 117.62 (14) |
| C2—C1—H1   | 116.4       | C17—C16—C13   | 123.93 (14) |
| C1—C2—C3   | 111.01 (13) | C21—C16—C13   | 118.42 (14) |
| C1—C2—H2A  | 109.4       | C18—C17—C16   | 121.39 (15) |
| C3—C2—H2A  | 109.4       | C18—C17—H17   | 119.3       |
| C1—C2—H2B  | 109.4       | C16—C17—H17   | 119.3       |
| C3—C2—H2B  | 109.4       | C17—C18—C19   | 120.96 (14) |
| H2A—C2—H2B | 108.0       | C17—C18—H18   | 119.5       |
| C4—C3—C2   | 110.34 (14) | C19—C18—H18   | 119.5       |
| C4—C3—H3A  | 109.6       | C20—C19—C18   | 117.49 (14) |
| C2—C3—H3A  | 109.6       | C20—C19—C22   | 116.35 (14) |
| C4—C3—H3B  | 109.6       | C18—C19—C22   | 126.16 (15) |
| C2—C3—H3B  | 109.6       | C21—C20—C19   | 121.39 (14) |
| H3A—C3—H3B | 108.1       | C21—C20—H20   | 119.3       |
| O1—C4—C5   | 59.12 (10)  | C19—C20—H20   | 119.3       |
| O1—C4—C15  | 112.24 (13) | C20—C21—C16   | 120.93 (14) |
| C5—C4—C15  | 122.56 (15) | C20—C21—H21   | 119.5       |
| O1—C4—C3   | 117.45 (14) | C16—C21—H21   | 119.5       |
| C5—C4—C3   | 116.05 (14) | C23—C22—C19   | 131.73 (15) |
| C15—C4—C3  | 116.40 (14) | C23—C22—H22   | 114.1       |
| O1—C5—C4   | 59.92 (10)  | C19—C22—H22   | 114.1       |
| O1—C5—C6   | 121.09 (13) | C22—C23—C33   | 122.00 (14) |

|               |              |                 |             |
|---------------|--------------|-----------------|-------------|
| C4—C5—C6      | 124.78 (14)  | C22—C23—C24     | 123.24 (15) |
| O1—C5—H5      | 113.6        | C33—C23—C24     | 114.74 (14) |
| C4—C5—H5      | 113.6        | C29—C24—C25     | 120.25 (14) |
| C6—C5—H5      | 113.6        | C29—C24—C23     | 119.87 (14) |
| O2—C6—C5      | 108.87 (12)  | C25—C24—C23     | 119.86 (14) |
| O2—C6—C7      | 106.71 (12)  | C26—C25—C24     | 119.76 (15) |
| C5—C6—C7      | 112.02 (12)  | C26—C25—H25     | 120.1       |
| O2—C6—H6      | 109.7        | C24—C25—H25     | 120.1       |
| C5—C6—H6      | 109.7        | O4—C26—C25      | 124.05 (14) |
| C7—C6—H6      | 109.7        | O4—C26—C27      | 115.54 (13) |
| C11—C7—C8     | 114.26 (13)  | C25—C26—C27     | 120.41 (15) |
| C11—C7—C6     | 102.28 (12)  | O5—C27—C28      | 121.77 (14) |
| C8—C7—C6      | 114.66 (13)  | O5—C27—C26      | 118.71 (14) |
| C11—C7—H7     | 108.4        | C28—C27—C26     | 119.43 (14) |
| C8—C7—H7      | 108.4        | O6—C28—C29      | 124.43 (15) |
| C6—C7—H7      | 108.4        | O6—C28—C27      | 115.43 (13) |
| C9—C8—C7      | 113.36 (13)  | C29—C28—C27     | 120.14 (14) |
| C9—C8—H8A     | 108.9        | C24—C29—C28     | 119.87 (15) |
| C7—C8—H8A     | 108.9        | C24—C29—H29     | 120.1       |
| C9—C8—H8B     | 108.9        | C28—C29—H29     | 120.1       |
| C7—C8—H8B     | 108.9        | O4—C30—H30A     | 109.5       |
| H8A—C8—H8B    | 107.7        | O4—C30—H30B     | 109.5       |
| C10—C9—C8     | 113.42 (12)  | H30A—C30—H30B   | 109.5       |
| C10—C9—H9A    | 108.9        | O4—C30—H30C     | 109.5       |
| C8—C9—H9A     | 108.9        | H30A—C30—H30C   | 109.5       |
| C10—C9—H9B    | 108.9        | H30B—C30—H30C   | 109.5       |
| C8—C9—H9B     | 108.9        | O5—C31—H31A     | 109.5       |
| H9A—C9—H9B    | 107.7        | O5—C31—H31B     | 109.5       |
| C1—C10—C14    | 125.32 (15)  | H31A—C31—H31B   | 109.5       |
| C1—C10—C9     | 120.92 (15)  | O5—C31—H31C     | 109.5       |
| C14—C10—C9    | 113.76 (14)  | H31A—C31—H31C   | 109.5       |
| C13—C11—C12   | 118.88 (14)  | H31B—C31—H31C   | 109.5       |
| C13—C11—C7    | 132.60 (14)  | O6—C32—H32A     | 109.5       |
| C12—C11—C7    | 108.40 (13)  | O6—C32—H32B     | 109.5       |
| O3—C12—O2     | 121.34 (14)  | H32A—C32—H32B   | 109.5       |
| O3—C12—C11    | 128.93 (15)  | O6—C32—H32C     | 109.5       |
| O2—C12—C11    | 109.63 (13)  | H32A—C32—H32C   | 109.5       |
| C11—C13—C16   | 130.38 (15)  | H32B—C32—H32C   | 109.5       |
| C11—C13—H13   | 114.8        | N1—C33—C23      | 177.04 (16) |
| C16—C13—H13   | 114.8        | C1M—O1M—H1M     | 109.5       |
| C10—C14—H14A  | 109.5        | O1M—C1M—H1M1    | 109.5       |
| C10—C14—H14B  | 109.5        | O1M—C1M—H1M2    | 109.5       |
| H14A—C14—H14B | 109.5        | H1M1—C1M—H1M2   | 109.5       |
| C10—C14—H14C  | 109.5        | O1M—C1M—H1M3    | 109.5       |
| H14A—C14—H14C | 109.5        | H1M1—C1M—H1M3   | 109.5       |
| H14B—C14—H14C | 109.5        | H1M2—C1M—H1M3   | 109.5       |
| C10—C1—C2—C3  | -107.33 (19) | C11—C13—C16—C17 | -18.2 (3)   |

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|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C1—C2—C3—C4     | 51.42 (18)   | C11—C13—C16—C21 | 163.60 (17)  |
| C5—O1—C4—C15    | -115.66 (16) | C21—C16—C17—C18 | -3.6 (2)     |
| C5—O1—C4—C3     | 105.42 (16)  | C13—C16—C17—C18 | 178.17 (16)  |
| C2—C3—C4—O1     | -154.06 (13) | C16—C17—C18—C19 | -0.3 (3)     |
| C2—C3—C4—C5     | -87.00 (17)  | C17—C18—C19—C20 | 3.8 (2)      |
| C2—C3—C4—C15    | 68.71 (18)   | C17—C18—C19—C22 | -176.49 (17) |
| C4—O1—C5—C6     | 114.86 (17)  | C18—C19—C20—C21 | -3.4 (2)     |
| C15—C4—C5—O1    | 98.13 (16)   | C22—C19—C20—C21 | 176.89 (15)  |
| C3—C4—C5—O1     | -107.79 (15) | C19—C20—C21—C16 | -0.6 (2)     |
| O1—C4—C5—C6     | -108.93 (16) | C17—C16—C21—C20 | 4.1 (2)      |
| C15—C4—C5—C6    | -10.8 (2)    | C13—C16—C21—C20 | -177.64 (15) |
| C3—C4—C5—C6     | 143.28 (15)  | C20—C19—C22—C23 | -176.47 (17) |
| C12—O2—C6—C5    | 135.46 (13)  | C18—C19—C22—C23 | 3.8 (3)      |
| C12—O2—C6—C7    | 14.37 (17)   | C19—C22—C23—C33 | -4.2 (3)     |
| O1—C5—C6—O2     | 44.08 (18)   | C19—C22—C23—C24 | 177.52 (16)  |
| C4—C5—C6—O2     | 116.99 (15)  | C22—C23—C24—C29 | 159.30 (17)  |
| O1—C5—C6—C7     | 161.85 (13)  | C33—C23—C24—C29 | -19.1 (2)    |
| C4—C5—C6—C7     | -125.24 (16) | C22—C23—C24—C25 | -19.2 (3)    |
| O2—C6—C7—C11    | -11.56 (16)  | C33—C23—C24—C25 | 162.36 (15)  |
| C5—C6—C7—C11    | -130.62 (13) | C29—C24—C25—C26 | -2.5 (2)     |
| O2—C6—C7—C8     | -135.79 (13) | C23—C24—C25—C26 | 175.96 (15)  |
| C5—C6—C7—C8     | 105.15 (15)  | C30—O4—C26—C25  | -16.8 (2)    |
| C11—C7—C8—C9    | 147.81 (13)  | C30—O4—C26—C27  | 164.02 (14)  |
| C6—C7—C8—C9     | -94.58 (15)  | C24—C25—C26—O4  | -179.51 (15) |
| C7—C8—C9—C10    | 70.67 (17)   | C24—C25—C26—C27 | -0.4 (2)     |
| C2—C1—C10—C14   | -8.4 (3)     | C31—O5—C27—C28  | -63.9 (2)    |
| C2—C1—C10—C9    | 171.04 (14)  | C31—O5—C27—C26  | 119.53 (16)  |
| C8—C9—C10—C1    | -104.35 (17) | O4—C26—C27—O5   | -0.7 (2)     |
| C8—C9—C10—C14   | 75.17 (18)   | C25—C26—C27—O5  | -179.93 (14) |
| C8—C7—C11—C13   | -54.0 (2)    | O4—C26—C27—C28  | -177.44 (14) |
| C6—C7—C11—C13   | -178.52 (17) | C25—C26—C27—C28 | 3.4 (2)      |
| C8—C7—C11—C12   | 130.10 (14)  | C32—O6—C28—C29  | -2.5 (2)     |
| C6—C7—C11—C12   | 5.60 (16)    | C32—O6—C28—C27  | 177.21 (15)  |
| C6—O2—C12—O3    | 172.42 (15)  | O5—C27—C28—O6   | 0.3 (2)      |
| C6—O2—C12—C11   | -10.86 (17)  | C26—C27—C28—O6  | 176.89 (14)  |
| C13—C11—C12—O3  | 2.6 (3)      | O5—C27—C28—C29  | 179.97 (15)  |
| C7—C11—C12—O3   | 179.18 (17)  | C26—C27—C28—C29 | -3.4 (2)     |
| C13—C11—C12—O2  | -173.76 (14) | C25—C24—C29—C28 | 2.5 (2)      |
| C7—C11—C12—O2   | 2.78 (18)    | C23—C24—C29—C28 | -176.03 (15) |
| C12—C11—C13—C16 | 170.76 (16)  | O6—C28—C29—C24  | -179.82 (16) |
| C7—C11—C13—C16  | -4.8 (3)     | C27—C28—C29—C24 | 0.5 (2)      |

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