Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 1,2,3,3′,4′,6′-Hexaacetyl-4,6-*O*-benzylidenesucrose

**Marco A. Brito-Arias,* Miguel Soto-Ortega and Efrén V. García-Báez**

Unidad Profesional Interdisciplinaria de Biotecnología, Instituto Politécnico Nacional, Avenida Acueducto s/n, Barrio La Laguna Ticomán, México DF 07340, Mexico
Correspondence e-mail: mbrito@ipn.mx

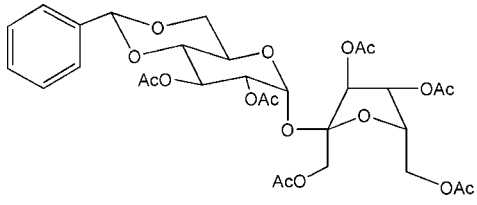
Received 17 December 2010; accepted 18 January 2011

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.036; $wR$ factor = 0.091; data-to-parameter ratio = 12.7.

In the title compound, $C_{31}H_{38}O_{17}$, the 1,3-dioxane and pyranoside rings both show $^4C_1$ chair conformations while for the D-fructofuranoside moiety an envelop 3$E$ conformation is observed. The phenyl ring is oriented almost perpendicular to the 1,3-dioxane ring [dihedral angle = 79.3 (2)°], and the acetate groups are equatorial for the pyranoside ring and axial for the furanoside ring. The analysis of potential hydrogen bonds shows both intra- and intermolecular C—H···O contacts to be present.

## Related literature

For sucrose and sucralose, see: Robyt (1998); Fairclough *et al.* (1995). For sucrose derivatives as potential pharmaceutically active substances, see: El Sayed & El Nemr (2005); Furneaux *et al.* (1993). For details of *O*-glycosidic bonds, see: Brito-Arias *et al.* (2007). For conformational analysis of five and six-membered rings, see: Cremer & Pople (1975); Evans & Boeyens (1989).




## Experimental

### *Crystal data*

$C_{31}H_{38}O_{17}$
$M_r$ = 682.61
Orthorhombic, $P2_12_12_1$
$a$ = 8.2018 (2) Å
$b$ = 18.6416 (3) Å
$c$ = 22.0994 (5) Å
$V$ = 3378.88 (12) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 293 K
0.40 × 0.30 × 0.20 mm

### *Data collection*

Oxford Diffraction CrysAlis CCD diffractometer
10881 measured reflections
5582 independent reflections
4757 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
$S$ = 1.03
5582 reflections
440 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1···O10 | 0.98 | 2.49 | 3.108 (3) | 121 |
| C1—H1···O15$^{i}$ | 0.98 | 2.56 | 3.399 (3) | 143 |
| C15—H15···O17 | 0.98 | 2.49 | 3.309 (3) | 141 |
| C25—H25$A$···O12$^{ii}$ | 0.96 | 2.51 | 3.343 (3) | 145 |
| C29—H29$C$···O12$^{iii}$ | 0.96 | 2.56 | 3.412 (4) | 148 |
| C31—H31$C$···O14$^{iv}$ | 0.96 | 2.56 | 3.504 (4) | 168 |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2009) and *WinGX2003* (Farrugia, 1999).

MBA is grateful to COFFA and SIP IPN for financial support. The authors thank Professor Hugo Jimenez, ENCB-IPN, for the generous allocation of diffractometer time.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2240).

## References

Brito-Arias, M., Cruz-Salazar, D. & Molins, E. (2007). *Acta Cryst.* E**63**, o359–o360.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
El Sayed, H. E. A. & El Nemr, A. (2005). In *Synthesis of Naturally Occurring Nitrogen Heterocycles from Carbohydrates*. Oxford: Blackwell Publishing Ltd.
Evans, D. G. & Boeyens, J. C. A. (1989). *Acta Cryst.* B**45**, 581–590.
Fairclough, P. H., Hough, L. & Richardson, A. C. (1995). *Carbohydr. Res.* **40**, 285–298.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Furneaux, R. H., Tyler, P. C. & Whitehouse, L. A. (1993). *Tetrahedron Lett.* **34**, 3613–3616.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Oxford Diffraction (2003). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abington, England.
Robyt, J. R. (1998). In *Essentials of Carbohydrate Chemistry*, Springer Advanced Texts in Chemistry. Heidelberg: Springer-Verlag GmbH.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2011). E**67**, o486 [doi:10.1107/S1600536811002546]

## 1,2,3,3′,4′,6′-Hexaacetyl-4,6-*O*-benzylidenesucrose

**Marco A. Brito-Arias, Miguel Soto-Ortega and Efrén V. García-Báez**

### S1. Comment

Sucrose is an abundant and low cost sugar mainly used as natural edulcorant (Robyt, 1998), and when substituted by chlorine at certain positions as the artificial edulcorant sucralose (Fairclough *et al.*, 1995). Despite this important usefulness sucrose has not been exploited sufficiently as synton for preparing modified derivatives containing isosteric substituents which could eventually lead us to pharmaceutical active substances (El Sayed & El Nemr, 2005; Furneaux *et al.* 1993). As a part of a strategy directed toward the preparation of modified sucrose derivatives we have prepared the title compound, a protected sucrose derivative which contains a benzylidene group at the 4 and 6 position of the gluco-pyranoside moiety and is fully acetylated at the remaining hydroxyl positions. This intermediate will allow us to functionalize the hydroxyl position at the pyranoside ring after deprotection of the benzylidene protecting group under mild conditions.

The title compound (Fig.1), shows two $^4C_1$ chair conformations belonging to the pyranoside and the 1,3-dioxane rings with puckering parameters (Cremer & Pople, 1975) Q = 0.572 (2) Å, $\theta$ = 7.7 (2)°, $\varphi$ = 304.4 (15)° and Q = 0.577 (2) Å, $\theta$ = 0.0 (2)°, $\varphi$ = 46 (7)°; both values being in agreement with a chair conformation. Also the angular disposition for the endocyclic bond C1—O5—C5 of 111.83 (15)° is in agreement with $^4C_1$ conformations having the substituents positioned at equatorial positions. The phenyl group is oriented almost perpendicular to the 1,3-dioxane and the acetate groups attached to the pyranoside ring are in equatorial positions. The $\alpha$-anomeric C1—O1 bond value of 1.412 (2)Å is more enlongated than the reference value of 1.385 (4)Å for O-glycosidic bonds (Brito-Arias *et al.*, 2007). For the furanoside ring the torsion angle values are 2.2 (2)° for C15—C14—O11—C17 revealing these elements to be almost in the plane and -28.9 (2)° for C14—C15—C16—C17 indicating an envelop *exo* E for C16, in agreement with a syn-periplanar conformation (Evans & Boeyens, 1989).

The analysis of potential hydrogen bonds shows different intramolecular and intermolecular C—H···O contacts are present (Table 1). The molecular packing is shown in Fig. 2, and some of the intramolecular O···C(acetyl) contacts, in the range 3.488 to 2.865 Å, are indicated in Fig. 3.

### S2. Experimental

The title compound was prepared by following a two step sequence starting from D-sucrose, which was treated with benzaldehyde dimethylacetal in dimethylformamide, followed by peracetylation under acetic anhydride-pyridine conditions. After purification by column chromatography the title compound was obtained as a white crystalline solid. $^1$H NMR data are available in the archived CIF.

### S3. Refinement

The absolute configuration of the structure could not determined by the X-ray analysis [Flack parameter = -0.3 (8)], but was already known from the configuration of the starting material, D-sucrose. H-atoms were placed in calculated

positions and treated as riding atoms: C—H = 0.93, 0.98, 0.97 and 0.96 Å for CH(aromatic), CH(methine), $CH_2$ and $CH_3$, respectively, with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.5 for $CH_3$ H-atoms and k = 1.2 for all other H-atoms.

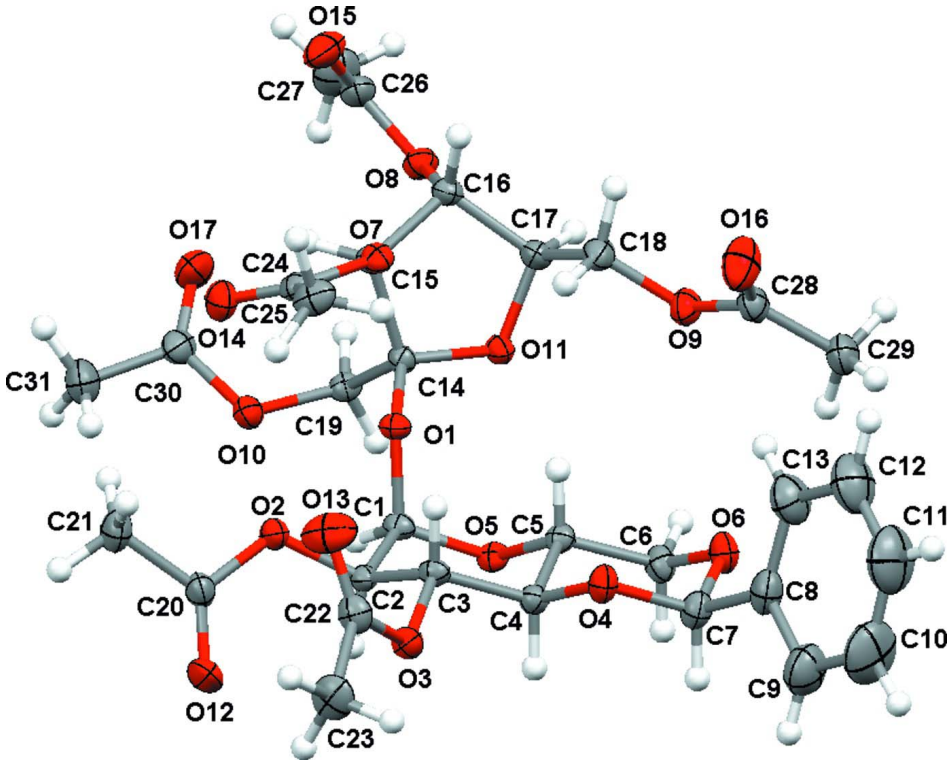



**Figure 1**

The molecular structure of the title comound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

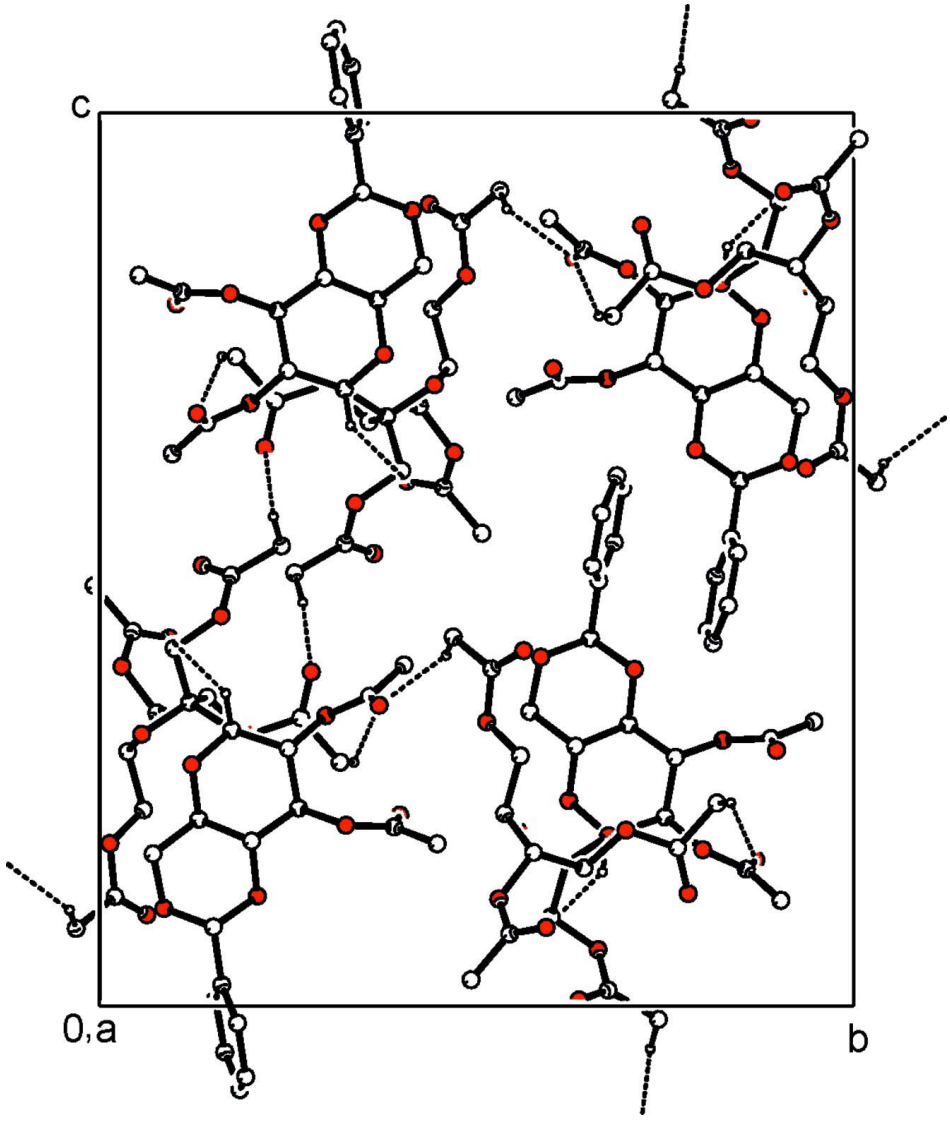



**Figure 2**

A view of the crystal packing along the a-axis of the title compound. The C-H···O interactions are shown as dotted lines [H-atoms not involved in C-H···O interactions have been omitted for clarity].

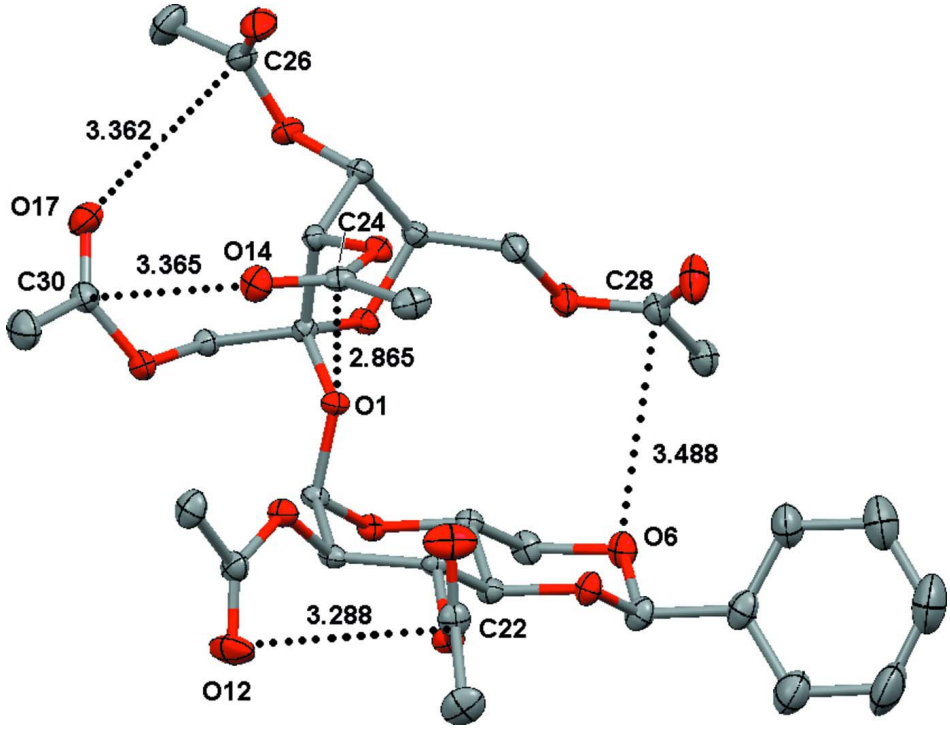



**Figure 3**

A view of the intramolecular C=O···C(acetyl) contacts in the title compound [H-atoms have been omitted for clarity].

### 1,2,3,3′,4′,6′-Hexaacetyl-4,6-*O*-benzylidenesucrose

*Crystal data*

$C_{31}H_{38}O_{17}$
$M_r = 682.61$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.2018$ (2) Å
$b = 18.6416$ (3) Å
$c = 22.0994$ (5) Å
$V = 3378.88$ (12) Å$^3$
$Z = 4$
$F(000) = 1440$
$D_x = 1.342$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 600 reflections
$\theta = 20$–25°
$\mu = 0.11$ mm$^{-1}$
$T = 293$ K
Block, colourless
0.40 × 0.30 × 0.20 mm

*Data collection*

Oxford Diffraction CrysAlis CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
10881 measured reflections
5582 independent reflections
4757 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$h = -9\rightarrow9$
$k = -22\rightarrow18$
$l = -22\rightarrow26$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.091$
$S = 1.03$
5582 reflections
440 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.4056P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.15$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (5)
Absolute structure: Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Absolute structure parameter: −0.3 (8)

*Special details*

**Experimental**. Spectroscopic $^1$H NMR analysis in $CDCl_3$, 300 MHz, $\delta$, p.p.m. shows: 2.0–2.1(6 s, 18H acetates), 3.6–3.8 (m, 2*H*-16,17), 4.1–4.4 (m, 7*H*-5,6,18,19), 4.8 (dd, 1*H*-2), 5.3(t, 1*H*-4), 5.4(d, 1*H*-15), 5.5(s, 1*H*-benzyl), 5.6(t, 1*H*-3), 5.7(d, 1*H*-1), 7.3–7.4(m, 5H aromatics).

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.73111 (16) | 0.17519 (7) | 0.30944 (6) | 0.0333 (4) |
| O2 | 0.86704 (18) | 0.30014 (7) | 0.32503 (7) | 0.0406 (5) |
| O3 | 1.00795 (18) | 0.32682 (7) | 0.20241 (7) | 0.0401 (5) |
| O4 | 0.9344 (2) | 0.20942 (7) | 0.12304 (7) | 0.0443 (5) |
| O5 | 0.96831 (16) | 0.12242 (7) | 0.27017 (6) | 0.0353 (5) |
| O6 | 0.9303 (2) | 0.08528 (8) | 0.10929 (7) | 0.0504 (5) |
| O7 | 0.42743 (17) | 0.19824 (7) | 0.30318 (6) | 0.0382 (5) |
| O8 | 0.36078 (19) | 0.02962 (8) | 0.38025 (7) | 0.0444 (5) |
| O9 | 0.5580 (2) | 0.01360 (9) | 0.18192 (7) | 0.0515 (6) |
| O10 | 0.74965 (19) | 0.16117 (8) | 0.43694 (7) | 0.0441 (5) |
| O11 | 0.64870 (17) | 0.05572 (7) | 0.30428 (7) | 0.0377 (4) |
| O12 | 1.0842 (3) | 0.37072 (12) | 0.33670 (11) | 0.0865 (9) |
| O13 | 0.7900 (3) | 0.39817 (10) | 0.21369 (12) | 0.0833 (9) |
| O14 | 0.4859 (2) | 0.28084 (8) | 0.37415 (8) | 0.0552 (6) |
| O15 | 0.1459 (2) | 0.09224 (12) | 0.41209 (9) | 0.0705 (7) |
| O16 | 0.4388 (3) | 0.06265 (12) | 0.10214 (9) | 0.0840 (8) |
| O17 | 0.5304 (3) | 0.13599 (12) | 0.49345 (9) | 0.0731 (8) |
| C1 | 0.9033 (2) | 0.17528 (10) | 0.30896 (10) | 0.0344 (6) |
| C2 | 0.9547 (2) | 0.25001 (10) | 0.28831 (9) | 0.0342 (6) |
| C3 | 0.9194 (3) | 0.26387 (10) | 0.22161 (10) | 0.0340 (6) |
| C4 | 0.9826 (3) | 0.20166 (10) | 0.18466 (9) | 0.0361 (6) |
| C5 | 0.9142 (3) | 0.13166 (10) | 0.20908 (9) | 0.0347 (6) |
| C6 | 0.9739 (3) | 0.07081 (11) | 0.17039 (11) | 0.0458 (8) |
| C7 | 0.9946 (3) | 0.15103 (12) | 0.08835 (11) | 0.0508 (8) |
| C8 | 0.9397 (4) | 0.16212 (14) | 0.02379 (12) | 0.0613 (10) |

| | | | | |
|---|---|---|---|---|
| C9 | 1.0530 (5) | 0.1828 (2) | −0.01927 (16) | 0.0947 (16) |
| C10 | 1.0007 (8) | 0.1938 (3) | −0.07948 (19) | 0.130 (3) |
| C11 | 0.8399 (9) | 0.1862 (3) | −0.0937 (2) | 0.129 (2) |
| C12 | 0.7291 (7) | 0.1660 (2) | −0.05162 (19) | 0.1057 (19) |
| C13 | 0.7789 (5) | 0.15374 (19) | 0.00733 (14) | 0.0813 (14) |
| C14 | 0.6485 (3) | 0.11941 (10) | 0.33997 (9) | 0.0314 (6) |
| C15 | 0.4675 (2) | 0.14265 (10) | 0.34567 (9) | 0.0334 (6) |
| C16 | 0.3740 (3) | 0.07573 (11) | 0.32814 (10) | 0.0379 (7) |
| C17 | 0.4873 (3) | 0.03726 (12) | 0.28463 (10) | 0.0391 (7) |
| C18 | 0.4578 (3) | 0.05827 (14) | 0.21980 (11) | 0.0509 (8) |
| C19 | 0.7226 (3) | 0.09899 (11) | 0.40039 (10) | 0.0366 (7) |
| C20 | 0.9452 (3) | 0.35802 (12) | 0.34689 (11) | 0.0468 (8) |
| C21 | 0.8321 (4) | 0.40377 (13) | 0.38178 (12) | 0.0580 (9) |
| C22 | 0.9294 (3) | 0.39056 (11) | 0.20049 (11) | 0.0487 (8) |
| C23 | 1.0434 (4) | 0.44780 (12) | 0.18165 (15) | 0.0703 (10) |
| C24 | 0.4529 (3) | 0.26677 (11) | 0.32286 (11) | 0.0402 (7) |
| C25 | 0.4346 (3) | 0.31905 (13) | 0.27285 (12) | 0.0559 (8) |
| C26 | 0.2401 (3) | 0.04389 (14) | 0.41925 (11) | 0.0509 (8) |
| C27 | 0.2438 (5) | −0.00724 (19) | 0.47071 (14) | 0.0857 (14) |
| C28 | 0.5332 (3) | 0.02021 (14) | 0.12248 (11) | 0.0491 (8) |
| C29 | 0.6319 (4) | −0.03097 (15) | 0.08675 (12) | 0.0603 (9) |
| C30 | 0.6439 (3) | 0.17443 (15) | 0.48229 (12) | 0.0510 (8) |
| C31 | 0.6844 (4) | 0.24131 (19) | 0.51508 (14) | 0.0777 (12) |
| H1 | 0.94339 | 0.16705 | 0.35016 | 0.0413* |
| H2 | 1.07174 | 0.25597 | 0.29557 | 0.0410* |
| H3 | 0.80215 | 0.27032 | 0.21503 | 0.0408* |
| H4 | 1.10184 | 0.20050 | 0.18705 | 0.0434* |
| H5 | 0.79477 | 0.13321 | 0.20806 | 0.0416* |
| H6A | 1.09139 | 0.06637 | 0.17394 | 0.0549* |
| H6B | 0.92478 | 0.02612 | 0.18353 | 0.0549* |
| H7 | 1.11397 | 0.14992 | 0.09022 | 0.0610* |
| H9 | 1.16182 | 0.18930 | −0.00874 | 0.1136* |
| H10 | 1.07559 | 0.20612 | −0.10930 | 0.1562* |
| H11 | 0.80557 | 0.19516 | −0.13305 | 0.1542* |
| H12 | 0.62016 | 0.16029 | −0.06227 | 0.1268* |
| H13 | 0.70281 | 0.13969 | 0.03619 | 0.0977* |
| H15 | 0.44170 | 0.15735 | 0.38716 | 0.0400* |
| H16 | 0.26775 | 0.08664 | 0.31002 | 0.0454* |
| H17 | 0.47181 | −0.01463 | 0.28886 | 0.0469* |
| H18A | 0.34372 | 0.05174 | 0.20961 | 0.0611* |
| H18B | 0.48561 | 0.10834 | 0.21379 | 0.0611* |
| H19A | 0.65003 | 0.06634 | 0.42141 | 0.0439* |
| H19B | 0.82535 | 0.07445 | 0.39372 | 0.0439* |
| H21A | 0.89344 | 0.43589 | 0.40696 | 0.0869* |
| H21B | 0.76373 | 0.37413 | 0.40665 | 0.0869* |
| H21C | 0.76555 | 0.43100 | 0.35440 | 0.0869* |
| H23A | 1.07607 | 0.43993 | 0.14047 | 0.1054* |
| H23B | 1.13776 | 0.44709 | 0.20733 | 0.1054* |

| | | | | |
|---|---|---|---|---|
| H23C | 0.99033 | 0.49354 | 0.18490 | 0.1054* |
| H25A | 0.32536 | 0.33750 | 0.27267 | 0.0838* |
| H25B | 0.45654 | 0.29572 | 0.23499 | 0.0838* |
| H25C | 0.51018 | 0.35782 | 0.27848 | 0.0838* |
| H27A | 0.35180 | −0.00885 | 0.48739 | 0.1286* |
| H27B | 0.21358 | −0.05416 | 0.45674 | 0.1286* |
| H27C | 0.16841 | 0.00820 | 0.50128 | 0.1286* |
| H29A | 0.65424 | −0.01087 | 0.04762 | 0.0904* |
| H29B | 0.57280 | −0.07507 | 0.08206 | 0.0904* |
| H29C | 0.73279 | −0.04019 | 0.10737 | 0.0904* |
| H31A | 0.58619 | 0.26275 | 0.53026 | 0.1167* |
| H31B | 0.73752 | 0.27412 | 0.48800 | 0.1167* |
| H31C | 0.75590 | 0.23046 | 0.54824 | 0.1167* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0262 (7) | 0.0325 (7) | 0.0412 (8) | −0.0004 (6) | 0.0000 (6) | 0.0034 (6) |
| O2 | 0.0395 (8) | 0.0333 (7) | 0.0489 (9) | −0.0014 (7) | 0.0031 (7) | −0.0078 (7) |
| O3 | 0.0446 (8) | 0.0261 (7) | 0.0497 (9) | −0.0036 (6) | 0.0056 (7) | 0.0012 (6) |
| O4 | 0.0603 (9) | 0.0355 (7) | 0.0370 (9) | 0.0018 (8) | 0.0028 (8) | −0.0014 (7) |
| O5 | 0.0338 (8) | 0.0310 (7) | 0.0412 (9) | 0.0052 (6) | −0.0009 (7) | 0.0037 (6) |
| O6 | 0.0671 (10) | 0.0372 (8) | 0.0469 (10) | 0.0008 (8) | −0.0025 (9) | −0.0078 (7) |
| O7 | 0.0347 (7) | 0.0380 (8) | 0.0419 (9) | 0.0044 (7) | −0.0049 (7) | 0.0020 (7) |
| O8 | 0.0434 (8) | 0.0408 (8) | 0.0490 (10) | −0.0032 (7) | 0.0079 (8) | 0.0029 (7) |
| O9 | 0.0530 (10) | 0.0620 (10) | 0.0395 (10) | 0.0060 (9) | −0.0050 (9) | −0.0087 (8) |
| O10 | 0.0430 (9) | 0.0517 (9) | 0.0376 (9) | −0.0053 (8) | −0.0030 (7) | −0.0051 (7) |
| O11 | 0.0332 (7) | 0.0336 (7) | 0.0462 (9) | 0.0005 (6) | −0.0023 (7) | −0.0068 (7) |
| O12 | 0.0566 (12) | 0.0907 (15) | 0.1121 (18) | −0.0310 (12) | 0.0178 (12) | −0.0535 (14) |
| O13 | 0.0627 (13) | 0.0481 (10) | 0.139 (2) | 0.0146 (10) | 0.0125 (14) | 0.0245 (12) |
| O14 | 0.0667 (11) | 0.0432 (9) | 0.0556 (11) | 0.0024 (8) | 0.0027 (10) | −0.0081 (8) |
| O15 | 0.0507 (10) | 0.0970 (15) | 0.0637 (13) | 0.0153 (12) | 0.0120 (10) | −0.0033 (11) |
| O16 | 0.1039 (16) | 0.0990 (15) | 0.0491 (11) | 0.0343 (15) | −0.0173 (12) | −0.0057 (11) |
| O17 | 0.0702 (13) | 0.0932 (14) | 0.0558 (12) | −0.0139 (13) | 0.0200 (10) | −0.0090 (10) |
| C1 | 0.0293 (10) | 0.0339 (10) | 0.0401 (12) | 0.0002 (9) | −0.0027 (9) | 0.0029 (9) |
| C2 | 0.0263 (10) | 0.0335 (10) | 0.0427 (12) | −0.0002 (9) | 0.0012 (10) | −0.0043 (9) |
| C3 | 0.0313 (10) | 0.0271 (9) | 0.0436 (12) | −0.0032 (9) | 0.0021 (10) | 0.0032 (9) |
| C4 | 0.0341 (10) | 0.0348 (10) | 0.0395 (12) | −0.0015 (9) | 0.0018 (10) | −0.0013 (9) |
| C5 | 0.0331 (10) | 0.0309 (10) | 0.0400 (13) | 0.0025 (9) | 0.0004 (10) | 0.0004 (9) |
| C6 | 0.0532 (14) | 0.0334 (11) | 0.0507 (14) | 0.0066 (10) | −0.0044 (12) | −0.0049 (10) |
| C7 | 0.0613 (15) | 0.0443 (13) | 0.0469 (15) | 0.0029 (12) | 0.0069 (13) | −0.0062 (11) |
| C8 | 0.087 (2) | 0.0494 (14) | 0.0475 (15) | 0.0023 (15) | 0.0097 (17) | −0.0070 (12) |
| C9 | 0.113 (3) | 0.107 (3) | 0.064 (2) | −0.006 (3) | 0.017 (2) | −0.006 (2) |
| C10 | 0.172 (5) | 0.166 (5) | 0.053 (3) | −0.009 (4) | 0.024 (3) | 0.016 (3) |
| C11 | 0.182 (5) | 0.147 (4) | 0.057 (3) | 0.005 (4) | −0.011 (3) | 0.000 (3) |
| C12 | 0.133 (4) | 0.113 (3) | 0.071 (3) | 0.008 (3) | −0.031 (3) | −0.009 (2) |
| C13 | 0.099 (3) | 0.090 (2) | 0.055 (2) | −0.004 (2) | −0.0155 (18) | −0.0001 (17) |
| C14 | 0.0335 (10) | 0.0283 (9) | 0.0324 (11) | −0.0015 (9) | 0.0014 (10) | 0.0007 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0317 (10) | 0.0347 (10) | 0.0337 (11) | 0.0000 (9) | −0.0017 (9) | 0.0038 (9) |
| C16 | 0.0309 (10) | 0.0426 (11) | 0.0401 (13) | −0.0047 (9) | −0.0023 (10) | 0.0011 (10) |
| C17 | 0.0351 (11) | 0.0399 (11) | 0.0422 (13) | −0.0064 (10) | −0.0005 (10) | −0.0037 (10) |
| C18 | 0.0417 (13) | 0.0657 (15) | 0.0454 (14) | 0.0054 (12) | −0.0051 (12) | −0.0104 (12) |
| C19 | 0.0345 (11) | 0.0369 (11) | 0.0385 (12) | 0.0026 (10) | −0.0001 (10) | 0.0046 (9) |
| C20 | 0.0546 (15) | 0.0436 (12) | 0.0423 (13) | −0.0086 (12) | 0.0026 (12) | −0.0088 (10) |
| C21 | 0.0783 (18) | 0.0436 (13) | 0.0520 (16) | 0.0016 (13) | 0.0075 (15) | −0.0094 (12) |
| C22 | 0.0619 (16) | 0.0325 (12) | 0.0516 (15) | 0.0000 (11) | −0.0031 (14) | 0.0048 (10) |
| C23 | 0.090 (2) | 0.0320 (12) | 0.089 (2) | −0.0106 (14) | 0.0151 (19) | 0.0066 (13) |
| C24 | 0.0321 (11) | 0.0362 (11) | 0.0523 (15) | 0.0044 (10) | 0.0084 (12) | −0.0007 (11) |
| C25 | 0.0529 (14) | 0.0431 (12) | 0.0717 (17) | 0.0081 (12) | 0.0089 (14) | 0.0133 (12) |
| C26 | 0.0443 (14) | 0.0585 (15) | 0.0499 (15) | −0.0088 (14) | 0.0085 (13) | −0.0049 (12) |
| C27 | 0.104 (3) | 0.092 (2) | 0.061 (2) | −0.011 (2) | 0.0239 (19) | 0.0173 (17) |
| C28 | 0.0531 (14) | 0.0553 (14) | 0.0390 (14) | −0.0040 (13) | −0.0067 (13) | −0.0040 (11) |
| C29 | 0.0673 (17) | 0.0643 (16) | 0.0492 (15) | −0.0045 (15) | −0.0032 (13) | −0.0125 (13) |
| C30 | 0.0457 (14) | 0.0653 (16) | 0.0419 (14) | 0.0026 (14) | −0.0047 (12) | −0.0006 (13) |
| C31 | 0.080 (2) | 0.088 (2) | 0.065 (2) | −0.0055 (18) | −0.0028 (18) | −0.0296 (18) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.412 (2) | C20—C21 | 1.477 (4) |
| O1—C14 | 1.413 (2) | C22—C23 | 1.479 (4) |
| O2—C2 | 1.431 (2) | C24—C25 | 1.481 (3) |
| O2—C20 | 1.345 (3) | C26—C27 | 1.484 (4) |
| O3—C3 | 1.444 (2) | C28—C29 | 1.480 (4) |
| O3—C22 | 1.352 (3) | C30—C31 | 1.480 (4) |
| O4—C4 | 1.425 (3) | C1—H1 | 0.9800 |
| O4—C7 | 1.420 (3) | C2—H2 | 0.9800 |
| O5—C1 | 1.411 (2) | C3—H3 | 0.9800 |
| O5—C5 | 1.432 (2) | C4—H4 | 0.9800 |
| O6—C6 | 1.423 (3) | C5—H5 | 0.9800 |
| O6—C7 | 1.412 (3) | C6—H6A | 0.9700 |
| O7—C15 | 1.437 (2) | C6—H6B | 0.9700 |
| O7—C24 | 1.366 (2) | C7—H7 | 0.9800 |
| O8—C16 | 1.441 (3) | C9—H9 | 0.9300 |
| O8—C26 | 1.339 (3) | C10—H10 | 0.9300 |
| O9—C18 | 1.439 (3) | C11—H11 | 0.9300 |
| O9—C28 | 1.335 (3) | C12—H12 | 0.9300 |
| O10—C19 | 1.430 (3) | C13—H13 | 0.9300 |
| O10—C30 | 1.348 (3) | C15—H15 | 0.9800 |
| O11—C14 | 1.425 (2) | C16—H16 | 0.9800 |
| O11—C17 | 1.435 (3) | C17—H17 | 0.9800 |
| O12—C20 | 1.186 (3) | C18—H18A | 0.9700 |
| O13—C22 | 1.189 (3) | C18—H18B | 0.9700 |
| O14—C24 | 1.195 (3) | C19—H19A | 0.9700 |
| O15—C26 | 1.198 (3) | C19—H19B | 0.9700 |
| O16—C28 | 1.195 (3) | C21—H21A | 0.9600 |
| O17—C30 | 1.200 (4) | C21—H21B | 0.9600 |

| | | | |
|---|---|---|---|
| C1—C2 | 1.525 (3) | C21—H21C | 0.9600 |
| C2—C3 | 1.524 (3) | C23—H23A | 0.9600 |
| C3—C4 | 1.510 (3) | C23—H23B | 0.9600 |
| C4—C5 | 1.520 (3) | C23—H23C | 0.9600 |
| C5—C6 | 1.503 (3) | C25—H25A | 0.9600 |
| C7—C8 | 1.510 (4) | C25—H25B | 0.9600 |
| C8—C9 | 1.385 (5) | C25—H25C | 0.9600 |
| C8—C13 | 1.377 (5) | C27—H27A | 0.9600 |
| C9—C10 | 1.413 (6) | C27—H27B | 0.9600 |
| C10—C11 | 1.363 (10) | C27—H27C | 0.9600 |
| C11—C12 | 1.354 (8) | C29—H29A | 0.9600 |
| C12—C13 | 1.384 (5) | C29—H29B | 0.9600 |
| C14—C15 | 1.552 (3) | C29—H29C | 0.9600 |
| C14—C19 | 1.516 (3) | C31—H31A | 0.9600 |
| C15—C16 | 1.515 (3) | C31—H31B | 0.9600 |
| C16—C17 | 1.517 (3) | C31—H31C | 0.9600 |
| C17—C18 | 1.505 (3) | | |

| | | | |
|---|---|---|---|
| O1···O2 | 2.6052 (19) | C22···O2 | 3.268 (3) |
| O1···O7 | 2.5313 (19) | C22···O12 | 3.288 (3) |
| O1···O10 | 2.834 (2) | C23···O11$^{iii}$ | 3.244 (3) |
| O1···O14 | 3.157 (2) | C24···O1 | 2.865 (3) |
| O1···C24 | 2.865 (3) | C25···O12$^{iv}$ | 3.343 (3) |
| O2···O3 | 2.988 (2) | C26···O17 | 3.362 (3) |
| O2···O13 | 3.129 (3) | C29···O6 | 3.307 (3) |
| O2···O1 | 2.6052 (19) | C29···O12$^{i}$ | 3.412 (4) |
| O2···C22 | 3.268 (3) | C30···O14 | 3.365 (3) |
| O3···O4 | 2.869 (2) | C30···C15 | 3.400 (3) |
| O3···O12 | 3.141 (3) | C1···H19B | 2.7300 |
| O3···O2 | 2.988 (2) | C2···H5 | 3.1000 |
| O3···C20 | 3.286 (3) | C12···H9$^{ix}$ | 3.0600 |
| O4···O3 | 2.869 (2) | C12···H23A$^{ix}$ | 3.0500 |
| O5···O11 | 2.9977 (19) | C13···H9$^{ix}$ | 3.0800 |
| O6···C29 | 3.307 (3) | C14···H18B | 3.1000 |
| O7···C18 | 3.204 (3) | C15···H18B | 2.9900 |
| O7···O11 | 3.2176 (19) | C16···H19A | 3.0700 |
| O7···O1 | 2.5313 (19) | C19···H1 | 2.4700 |
| O8···O11 | 2.938 (2) | C23···H19B$^{iii}$ | 3.0800 |
| O8···C19 | 3.268 (3) | C26···H15 | 2.7800 |
| O9···O11 | 2.912 (2) | C28···H21C$^{xi}$ | 3.0100 |
| O10···C1 | 3.108 (3) | C30···H15 | 2.7000 |
| O10···O1 | 2.834 (2) | H1···O10 | 2.4900 |
| O11···O8 | 2.938 (2) | H1···O15$^{ii}$ | 2.5600 |
| O11···C23$^{i}$ | 3.244 (3) | H1···C19 | 2.4700 |
| O11···C5 | 3.343 (3) | H1···H19B | 2.2000 |
| O11···O7 | 3.2176 (19) | H2···O12 | 2.3300 |
| O11···O5 | 2.9977 (19) | H3···O1 | 2.8000 |
| O11···O9 | 2.912 (2) | H3···O13 | 2.3900 |

| | | | |
|---|---|---|---|
| O12···C25$^{ii}$ | 3.343 (3) | H3···H5 | 2.5600 |
| O12···C22 | 3.288 (3) | H4···H6A | 2.5200 |
| O12···O3 | 3.141 (3) | H4···H7 | 2.3400 |
| O12···C29$^{iii}$ | 3.412 (4) | H5···O1 | 2.4300 |
| O13···O2 | 3.129 (3) | H5···O11 | 2.8400 |
| O13···C20 | 3.293 (4) | H5···C2 | 3.1000 |
| O14···C30 | 3.365 (3) | H5···H3 | 2.5600 |
| O14···O1 | 3.157 (2) | H5···H18B | 2.5800 |
| O14···C14 | 3.377 (3) | H6A···H4 | 2.5200 |
| O15···C15 | 3.162 (2) | H6A···H7 | 2.4300 |
| O15···C1$^{iv}$ | 3.399 (3) | H6A···H18A$^{ii}$ | 2.2300 |
| O17···C26 | 3.362 (3) | H7···H4 | 2.3400 |
| O17···C21$^{v}$ | 3.286 (4) | H7···H6A | 2.4300 |
| O17···C15 | 3.309 (3) | H7···H9 | 2.3400 |
| O1···H5 | 2.4300 | H9···H7 | 2.3400 |
| O1···H3 | 2.8000 | H9···C12$^{vi}$ | 3.0600 |
| O3···H11$^{vi}$ | 2.9100 | H9···C13$^{vi}$ | 3.0800 |
| O5···H16$^{ii}$ | 2.6900 | H11···O3$^{ix}$ | 2.9100 |
| O5···H23C$^{i}$ | 2.6200 | H11···H25B$^{vi}$ | 2.5800 |
| O6···H13 | 2.6700 | H12···H23A$^{ix}$ | 2.5700 |
| O6···H29C | 2.8500 | H13···O6 | 2.6700 |
| O7···H18B | 2.6300 | H15···O10 | 2.7600 |
| O8···H19A | 2.6300 | H15···O14 | 2.3500 |
| O10···H15 | 2.7600 | H15···O15 | 2.7700 |
| O10···H1 | 2.4900 | H15···O17 | 2.4900 |
| O11···H23B$^{i}$ | 2.6900 | H15···C26 | 2.7800 |
| O11···H5 | 2.8400 | H15···C30 | 2.7000 |
| O12···H25A$^{ii}$ | 2.5100 | H15···H19A | 2.5200 |
| O12···H2 | 2.3300 | H16···O5$^{iv}$ | 2.6900 |
| O12···H29C$^{iii}$ | 2.5600 | H16···O15 | 2.4700 |
| O13···H25C | 2.8100 | H16···H18A | 2.3900 |
| O13···H17$^{vii}$ | 2.6900 | H17···O13$^{xi}$ | 2.6900 |
| O13···H3 | 2.3900 | H18A···O16 | 2.5100 |
| O14···H29B$^{vii}$ | 2.9000 | H18A···H6A$^{iv}$ | 2.2300 |
| O14···H15 | 2.3500 | H18A···H16 | 2.3900 |
| O14···H31C$^{v}$ | 2.5600 | H18B···O7 | 2.6300 |
| O15···H16 | 2.4700 | H18B···O16 | 2.6400 |
| O15···H19B$^{iv}$ | 2.6800 | H18B···C14 | 3.1000 |
| O15···H15 | 2.7700 | H18B···C15 | 2.9900 |
| O15···H1$^{iv}$ | 2.5600 | H18B···H5 | 2.5800 |
| O16···H27C$^{viii}$ | 2.7400 | H19A···O8 | 2.6300 |
| O16···H18B | 2.6400 | H19A···O17 | 2.2800 |
| O16···H18A | 2.5100 | H19A···C16 | 3.0700 |
| O17···H15 | 2.4900 | H19A···H15 | 2.5200 |
| O17···H19A | 2.2800 | H19B···O15$^{ii}$ | 2.6800 |
| O17···H21A$^{v}$ | 2.8100 | H19B···C1 | 2.7300 |
| C1···O10 | 3.108 (3) | H19B···H1 | 2.2000 |
| C1···O15$^{ii}$ | 3.399 (3) | H19B···C23$^{i}$ | 3.0800 |

| | | | |
|---|---|---|---|
| C5···O11 | 3.343 (3) | H21A···O17$^{x}$ | 2.8100 |
| C9···C12$^{vi}$ | 3.533 (6) | H21B···H31B | 2.6000 |
| C9···C13$^{vi}$ | 3.576 (5) | H21C···C28$^{vii}$ | 3.0100 |
| C12···C9$^{ix}$ | 3.533 (6) | H23A···C12$^{vi}$ | 3.0500 |
| C13···C9$^{ix}$ | 3.576 (5) | H23A···H12$^{vi}$ | 2.5700 |
| C14···O14 | 3.377 (3) | H23B···O11$^{iii}$ | 2.6900 |
| C15···C30 | 3.400 (3) | H23C···O5$^{iii}$ | 2.6200 |
| C15···O17 | 3.309 (3) | H25A···O12$^{iv}$ | 2.5100 |
| C15···O15 | 3.162 (2) | H25B···H11$^{ix}$ | 2.5800 |
| C18···O7 | 3.204 (3) | H25C···O13 | 2.8100 |
| C19···O8 | 3.268 (3) | H27C···O16$^{xii}$ | 2.7400 |
| C20···C22 | 3.294 (3) | H29B···O14$^{xi}$ | 2.9000 |
| C20···O13 | 3.293 (4) | H29C···O6 | 2.8500 |
| C20···O3 | 3.286 (3) | H29C···O12$^{i}$ | 2.5600 |
| C21···O17$^{x}$ | 3.286 (4) | H31B···H21B | 2.6000 |
| C22···C20 | 3.294 (3) | H31C···O14$^{x}$ | 2.5600 |

| | | | |
|---|---|---|---|
| C1—O1—C14 | 118.95 (15) | C4—C3—H3 | 111.00 |
| C2—O2—C20 | 119.21 (16) | O4—C4—H4 | 109.00 |
| C3—O3—C22 | 118.93 (17) | C3—C4—H4 | 109.00 |
| C4—O4—C7 | 109.97 (16) | C5—C4—H4 | 109.00 |
| C1—O5—C5 | 111.83 (15) | O5—C5—H5 | 110.00 |
| C6—O6—C7 | 112.44 (17) | C4—C5—H5 | 110.00 |
| C15—O7—C24 | 115.57 (16) | C6—C5—H5 | 110.00 |
| C16—O8—C26 | 116.84 (18) | O6—C6—H6A | 110.00 |
| C18—O9—C28 | 115.60 (19) | O6—C6—H6B | 110.00 |
| C19—O10—C30 | 117.95 (18) | C5—C6—H6A | 110.00 |
| C14—O11—C17 | 111.48 (16) | C5—C6—H6B | 110.00 |
| O1—C1—O5 | 112.44 (15) | H6A—C6—H6B | 108.00 |
| O1—C1—C2 | 106.24 (14) | O4—C7—H7 | 110.00 |
| O5—C1—C2 | 110.59 (16) | O6—C7—H7 | 110.00 |
| O2—C2—C1 | 106.73 (15) | C8—C7—H7 | 110.00 |
| O2—C2—C3 | 110.01 (15) | C8—C9—H9 | 121.00 |
| C1—C2—C3 | 113.06 (16) | C10—C9—H9 | 121.00 |
| O3—C3—C2 | 109.05 (17) | C9—C10—H10 | 120.00 |
| O3—C3—C4 | 107.01 (18) | C11—C10—H10 | 120.00 |
| C2—C3—C4 | 109.12 (16) | C10—C11—H11 | 119.00 |
| O4—C4—C3 | 110.09 (17) | C12—C11—H11 | 119.00 |
| O4—C4—C5 | 108.91 (16) | C11—C12—H12 | 120.00 |
| C3—C4—C5 | 109.92 (18) | C13—C12—H12 | 120.00 |
| O5—C5—C4 | 108.90 (16) | C8—C13—H13 | 120.00 |
| O5—C5—C6 | 110.17 (17) | C12—C13—H13 | 120.00 |
| C4—C5—C6 | 109.02 (18) | O7—C15—H15 | 111.00 |
| O6—C6—C5 | 108.36 (17) | C14—C15—H15 | 111.00 |
| O4—C7—O6 | 111.01 (18) | C16—C15—H15 | 111.00 |
| O4—C7—C8 | 107.54 (19) | O8—C16—H16 | 113.00 |
| O6—C7—C8 | 108.5 (2) | C15—C16—H16 | 113.00 |
| C7—C8—C9 | 119.2 (3) | C17—C16—H16 | 113.00 |

| | | | |
|---|---|---|---|
| C7—C8—C13 | 121.3 (3) | O11—C17—H17 | 109.00 |
| C9—C8—C13 | 119.5 (3) | C16—C17—H17 | 109.00 |
| C8—C9—C10 | 118.9 (4) | C18—C17—H17 | 109.00 |
| C9—C10—C11 | 119.7 (5) | O9—C18—H18A | 110.00 |
| C10—C11—C12 | 121.3 (5) | O9—C18—H18B | 110.00 |
| C11—C12—C13 | 119.6 (5) | C17—C18—H18A | 110.00 |
| C8—C13—C12 | 120.8 (4) | C17—C18—H18B | 110.00 |
| O1—C14—O11 | 110.38 (16) | H18A—C18—H18B | 108.00 |
| O1—C14—C15 | 106.99 (15) | O10—C19—H19A | 109.00 |
| O1—C14—C19 | 114.41 (18) | O10—C19—H19B | 109.00 |
| O11—C14—C15 | 106.18 (17) | C14—C19—H19A | 109.00 |
| O11—C14—C19 | 106.13 (16) | C14—C19—H19B | 109.00 |
| C15—C14—C19 | 112.47 (18) | H19A—C19—H19B | 108.00 |
| O7—C15—C14 | 111.55 (15) | C20—C21—H21A | 109.00 |
| O7—C15—C16 | 108.13 (15) | C20—C21—H21B | 109.00 |
| C14—C15—C16 | 103.51 (16) | C20—C21—H21C | 109.00 |
| O8—C16—C15 | 108.96 (17) | H21A—C21—H21B | 109.00 |
| O8—C16—C17 | 105.70 (17) | H21A—C21—H21C | 109.00 |
| C15—C16—C17 | 103.96 (18) | H21B—C21—H21C | 109.00 |
| O11—C17—C16 | 105.06 (17) | C22—C23—H23A | 109.00 |
| O11—C17—C18 | 111.96 (19) | C22—C23—H23B | 109.00 |
| C16—C17—C18 | 112.5 (2) | C22—C23—H23C | 109.00 |
| O9—C18—C17 | 108.15 (19) | H23A—C23—H23B | 109.00 |
| O10—C19—C14 | 110.87 (16) | H23A—C23—H23C | 109.00 |
| O2—C20—O12 | 123.4 (2) | H23B—C23—H23C | 109.00 |
| O2—C20—C21 | 110.6 (2) | C24—C25—H25A | 109.00 |
| O12—C20—C21 | 126.0 (2) | C24—C25—H25B | 109.00 |
| O3—C22—O13 | 123.8 (2) | C24—C25—H25C | 109.00 |
| O3—C22—C23 | 110.0 (2) | H25A—C25—H25B | 109.00 |
| O13—C22—C23 | 126.3 (2) | H25A—C25—H25C | 109.00 |
| O7—C24—O14 | 122.8 (2) | H25B—C25—H25C | 109.00 |
| O7—C24—C25 | 111.3 (2) | C26—C27—H27A | 109.00 |
| O14—C24—C25 | 125.9 (2) | C26—C27—H27B | 109.00 |
| O8—C26—O15 | 122.8 (2) | C26—C27—H27C | 109.00 |
| O8—C26—C27 | 110.5 (2) | H27A—C27—H27B | 109.00 |
| O15—C26—C27 | 126.7 (3) | H27A—C27—H27C | 109.00 |
| O9—C28—O16 | 122.0 (2) | H27B—C27—H27C | 109.00 |
| O9—C28—C29 | 112.5 (2) | C28—C29—H29A | 109.00 |
| O16—C28—C29 | 125.5 (2) | C28—C29—H29B | 109.00 |
| O10—C30—O17 | 122.8 (2) | C28—C29—H29C | 109.00 |
| O10—C30—C31 | 112.0 (2) | H29A—C29—H29B | 109.00 |
| O17—C30—C31 | 125.2 (3) | H29A—C29—H29C | 109.00 |
| O1—C1—H1 | 109.00 | H29B—C29—H29C | 109.00 |
| O5—C1—H1 | 109.00 | C30—C31—H31A | 109.00 |
| C2—C1—H1 | 109.00 | C30—C31—H31B | 109.00 |
| O2—C2—H2 | 109.00 | C30—C31—H31C | 109.00 |
| C1—C2—H2 | 109.00 | H31A—C31—H31B | 109.00 |
| C3—C2—H2 | 109.00 | H31A—C31—H31C | 109.00 |

| | | | |
|---|---|---|---|
| O3—C3—H3 | 111.00 | H31B—C31—H31C | 109.00 |
| C2—C3—H3 | 111.00 | | |
| | | | |
| C14—O1—C1—O5 | 73.7 (2) | O5—C1—C2—C3 | 51.6 (2) |
| C14—O1—C1—C2 | −165.23 (16) | O2—C2—C3—O3 | 75.5 (2) |
| C1—O1—C14—O11 | −77.8 (2) | O2—C2—C3—C4 | −167.97 (17) |
| C1—O1—C14—C15 | 167.07 (16) | C1—C2—C3—O3 | −165.34 (15) |
| C1—O1—C14—C19 | 41.8 (2) | C1—C2—C3—C4 | −48.8 (2) |
| C20—O2—C2—C1 | 137.53 (18) | O3—C3—C4—O4 | −68.8 (2) |
| C20—O2—C2—C3 | −99.5 (2) | O3—C3—C4—C5 | 171.19 (17) |
| C2—O2—C20—O12 | 1.1 (3) | C2—C3—C4—O4 | 173.30 (17) |
| C2—O2—C20—C21 | 178.19 (18) | C2—C3—C4—C5 | 53.3 (2) |
| C22—O3—C3—C2 | −97.8 (2) | O4—C4—C5—O5 | 177.60 (16) |
| C22—O3—C3—C4 | 144.32 (19) | O4—C4—C5—C6 | 57.4 (2) |
| C3—O3—C22—O13 | −0.1 (4) | C3—C4—C5—O5 | −61.7 (2) |
| C3—O3—C22—C23 | 178.2 (2) | C3—C4—C5—C6 | 178.05 (19) |
| C7—O4—C4—C3 | 179.74 (18) | O5—C5—C6—O6 | −174.82 (17) |
| C7—O4—C4—C5 | −59.7 (2) | C4—C5—C6—O6 | −55.4 (2) |
| C4—O4—C7—O6 | 61.4 (2) | O4—C7—C8—C9 | 107.1 (3) |
| C4—O4—C7—C8 | 179.9 (2) | O4—C7—C8—C13 | −71.4 (3) |
| C5—O5—C1—O1 | 58.8 (2) | O6—C7—C8—C9 | −132.8 (3) |
| C5—O5—C1—C2 | −59.80 (19) | O6—C7—C8—C13 | 48.8 (3) |
| C1—O5—C5—C4 | 65.5 (2) | C7—C8—C9—C10 | −179.4 (3) |
| C1—O5—C5—C6 | −175.03 (16) | C13—C8—C9—C10 | −0.9 (5) |
| C7—O6—C6—C5 | 57.8 (2) | C7—C8—C13—C12 | 178.2 (3) |
| C6—O6—C7—O4 | −61.1 (2) | C9—C8—C13—C12 | −0.3 (5) |
| C6—O6—C7—C8 | −179.1 (2) | C8—C9—C10—C11 | 2.1 (7) |
| C24—O7—C15—C14 | −88.8 (2) | C9—C10—C11—C12 | −2.1 (8) |
| C24—O7—C15—C16 | 158.04 (18) | C10—C11—C12—C13 | 1.0 (7) |
| C15—O7—C24—O14 | −9.4 (3) | C11—C12—C13—C8 | 0.3 (6) |
| C15—O7—C24—C25 | 170.49 (17) | O1—C14—C15—O7 | 19.1 (2) |
| C26—O8—C16—C15 | 83.2 (2) | O1—C14—C15—C16 | 135.10 (16) |
| C26—O8—C16—C17 | −165.66 (19) | O11—C14—C15—O7 | −98.83 (17) |
| C16—O8—C26—O15 | 0.2 (3) | O11—C14—C15—C16 | 17.2 (2) |
| C16—O8—C26—C27 | −179.2 (2) | C19—C14—C15—O7 | 145.51 (16) |
| C28—O9—C18—C17 | 173.1 (2) | C19—C14—C15—C16 | −98.46 (19) |
| C18—O9—C28—O16 | 3.0 (4) | O1—C14—C19—O10 | 49.9 (2) |
| C18—O9—C28—C29 | −175.9 (2) | O11—C14—C19—O10 | 171.83 (17) |
| C30—O10—C19—C14 | 102.9 (2) | C15—C14—C19—O10 | −72.5 (2) |
| C19—O10—C30—O17 | 0.9 (4) | O7—C15—C16—O8 | −158.16 (16) |
| C19—O10—C30—C31 | −178.5 (2) | O7—C15—C16—C17 | 89.49 (18) |
| C17—O11—C14—O1 | −113.43 (19) | C14—C15—C16—O8 | 83.41 (19) |
| C17—O11—C14—C15 | 2.2 (2) | C14—C15—C16—C17 | −28.9 (2) |
| C17—O11—C14—C19 | 122.06 (19) | O8—C16—C17—O11 | −83.85 (19) |
| C14—O11—C17—C16 | −20.8 (2) | O8—C16—C17—C18 | 154.14 (19) |
| C14—O11—C17—C18 | 101.6 (2) | C15—C16—C17—O11 | 30.8 (2) |
| O1—C1—C2—O2 | 50.38 (19) | C15—C16—C17—C18 | −91.2 (2) |

| | | | |
|---|---|---|---|
| O1—C1—C2—C3 | −70.7 (2) | O11—C17—C18—O9 | 68.1 (2) |
| O5—C1—C2—O2 | 172.67 (14) | C16—C17—C18—O9 | −173.87 (18) |

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+1/2; (ii) *x*+1, *y*, *z*; (iii) −*x*+2, *y*+1/2, −*z*+1/2; (iv) *x*−1, *y*, *z*; (v) *x*−1/2, −*y*+1/2, −*z*+1; (vi) *x*+1/2, −*y*+1/2, −*z*; (vii) −*x*+1, *y*+1/2, −*z*+1/2; (viii) −*x*+1/2, −*y*, *z*−1/2; (ix) *x*−1/2, −*y*+1/2, −*z*; (x) *x*+1/2, −*y*+1/2, −*z*+1; (xi) −*x*+1, *y*−1/2, −*z*+1/2; (xii) −*x*+1/2, −*y*, *z*+1/2.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C1—H1···O10 | 0.98 | 2.49 | 3.108 (3) | 121 |
| C1—H1···O15$^{ii}$ | 0.98 | 2.56 | 3.399 (3) | 143 |
| C2—H2···O12 | 0.98 | 2.33 | 2.708 (3) | 102 |
| C3—H3···O13 | 0.98 | 2.39 | 2.725 (3) | 100 |
| C5—H5···O1 | 0.98 | 2.43 | 2.799 (3) | 102 |
| C15—H15···O17 | 0.98 | 2.49 | 3.309 (3) | 141 |
| C19—H19*A*···O17 | 0.97 | 2.28 | 2.682 (3) | 104 |
| C25—H25*A*···O12$^{iv}$ | 0.96 | 2.51 | 3.343 (3) | 145 |
| C29—H29*C*···O12$^{i}$ | 0.96 | 2.56 | 3.412 (4) | 148 |
| C31—H31*C*···O14$^{x}$ | 0.96 | 2.56 | 3.504 (4) | 168 |

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