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# 4,6-*O*-Benzylidene-1-*O*-methyl-2,3-deoxy-α-D-glucopyranose-2,3-diyl 1,2-phenylene orthocarbonate

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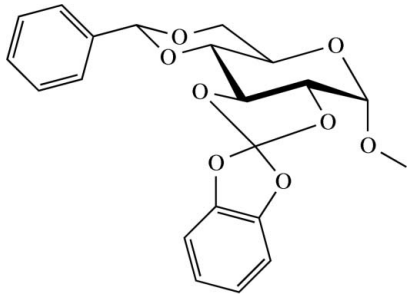
Key indicators: single-crystal X-ray study; $T$ = 200 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.043; $wR$ factor = 0.101; data-to-parameter ratio = 8.9.

The title compound, $C_{21}H_{20}O_8$, is the mixed orthocarbonic acid ester of the alcohol components benzene-1,2-diol and 4,6-*O*-benzylidene-1-*O*-methyl-α-D-glucopyranoside. The compound represents the first example of a structurally characterized carbohydrate ester of orthocarbonic acid. Both oxacyclic six-membered rings in the structure adopt a chair conformation. The puckered five-membered ring at the spiro centre is present in a twist conformation. No special interactions are observed in the molecular packing.

## Related literature

The compound was prepared according to the procedure described by Mues & Buysch (1990). A comparable spirocyclic orthocarbonate has been described recently (Betz & Klüfers, 2007).



## Experimental

### Crystal data

$C_{21}H_{20}O_8$
$M_r$ = 400.37
Monoclinic, $C2$
$a$ = 27.8221 (12) Å
$b$ = 5.1082 (1) Å
$c$ = 13.0090 (5) Å
$\beta$ = 98.710 (2)°
$V$ = 1827.53 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 200 (2) K
0.13 × 0.07 × 0.04 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.985, $T_{max}$ = 0.996
17337 measured reflections
2340 independent reflections
1770 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.056

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.101
$S$ = 1.07
2340 reflections
264 parameters
1 restraint
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Sandra Albrecht and Anna Zangl for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2136).

## References

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o4132 [ **doi:10.1107/S1600536807045266** ]

# 4,6-*O*-Benzylidene-1-*O*-methyl-2,3-deoxy-*α*-D-glucopyranose-2,3-diyl 1,2-phenylene orthocarbonate

**R. Betz and P. Klüfers**

## Comment

The spirocyclic orthocarbonate derived from benzene-1,2-diol and 4,6-*O*-benzylidene-1-*O*-methyl α-*D*-glucopyranoside has been prepared to obtain NMR data and structural details for comparison with analogous silicon compounds. In the centre of the molecular structure of (I) a carbon atom is bonded to a benzene-1,2-dioxy group and to the vicinal, *trans*-configured, 2,3-dioxy function derived from 4,6-*O*-benzylated methyl α-*D*-glucopyranoside. Both oxacyclic six-membered rings in the structure adopt a chair conformation. The puckered five-membered ring at the spiro centre is present in a twist conformation.

The molecular packing shows clustering of the aromatic groups about the twofold axes parallel to b. While two of these rings stem from the carbohydrate's protecting group, the other two are provided by phenylene residues.

## Experimental

The compound was obtained upon reaction of equimolar amounts of 2,2-dichlorobenzo[1.3]dioxole and 4,6-*O*-benzylidene-methyl-α-*D*-glucopyranoside in the presence of pyridine in dry dichloromethane. Crystals suitable for X-ray analysis were obtained after aqueous workup and subsequent recrystallization of the crude reaction product from hot ethyl acetate.

## Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms with C—H = 0.95–1.00 Å. One common isotropic displacement parameter for all H atoms was refined to $U_{iso}$(H) = 0.046 (2).

Due to the absence of significant anomalous scattering, Friedel opposites (1736 pairs) have been merged. The absolute configuration is assigned from the carbohydrate starting material.

## Figures

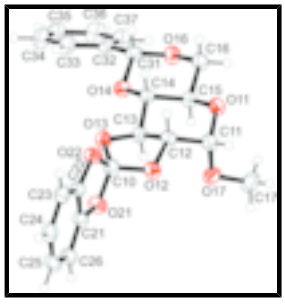

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at the 50% probability level) for non-H atoms.

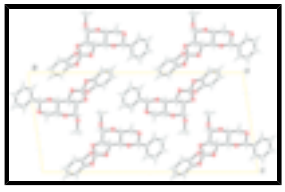

Fig. 2. The packing of (I), viewed along [010].

**4,6-*O*-Benzylidene-1-*O*-methyl-2,3-deoxy-α-*D*-glucopyranos-2,3-diyl- 1',2'-phenylene orthocarbonate**

*Crystal data*

| | |
|---|---|
| $C_{21}H_{20}O_8$ | $F_{000}$ = 840 |
| $M_r$ = 400.37 | $D_x$ = 1.455 Mg m$^{-3}$ |
| Monoclinic, *C*2 | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: C 2y | Cell parameters from 11294 reflections |
| *a* = 27.8221 (12) Å | θ = 3.1–27.5º |
| *b* = 5.1082 (1) Å | μ = 0.11 mm$^{-1}$ |
| *c* = 13.0090 (5) Å | *T* = 200 (2) K |
| β = 98.710 (2)º | Block, colourless |
| *V* = 1827.53 (11) Å$^3$ | 0.13 × 0.07 × 0.04 mm |
| *Z* = 4 | |

*Data collection*

| | |
|---|---|
| Nonius Kappa CCD diffractometer | 2340 independent reflections |
| Radiation source: fine-focus sealed tube | 1770 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.056 |
| *T* = 200(2) K | $\theta_{max}$ = 27.6º |
| φ and ω scans | $\theta_{min}$ = 3.1º |
| Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997) | *h* = −36→36 |
| $T_{min}$ = 0.985, $T_{max}$ = 0.996 | *k* = −6→6 |
| 17337 measured reflections | *l* = −16→16 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)]$ = 0.043 | Only H-atom displacement parameters refined |
| $wR(F^2)$ = 0.101 | $w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.4733P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| *S* = 1.07 | $(\Delta/\sigma)_{max} < 0.001$ |
| 2340 reflections | $\Delta\rho_{max}$ = 0.17 e Å$^{-3}$ |
| 264 parameters | $\Delta\rho_{min}$ = −0.25 e Å$^{-3}$ |
| 1 restraint | Extinction correction: none |

Primary atom site location: structure-invariant direct methods

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O11 | 0.64380 (6) | 0.9042 (4) | 0.57076 (14) | 0.0319 (4) |
| O12 | 0.74938 (6) | 0.6047 (4) | 0.73260 (13) | 0.0329 (5) |
| O13 | 0.69284 (6) | 0.4286 (4) | 0.82183 (13) | 0.0346 (5) |
| O14 | 0.58259 (6) | 0.4525 (4) | 0.73050 (13) | 0.0291 (4) |
| O16 | 0.52502 (6) | 0.6614 (4) | 0.60767 (14) | 0.0339 (5) |
| O17 | 0.69720 (7) | 0.5838 (4) | 0.52112 (14) | 0.0377 (5) |
| O21 | 0.76376 (7) | 0.2029 (4) | 0.80564 (14) | 0.0340 (5) |
| O22 | 0.76541 (7) | 0.5557 (4) | 0.91262 (14) | 0.0333 (5) |
| C10 | 0.74231 (10) | 0.4493 (6) | 0.8183 (2) | 0.0321 (6) |
| C11 | 0.69259 (10) | 0.8021 (6) | 0.5832 (2) | 0.0328 (6) |
| H11 | 0.7158 | 0.9421 | 0.5686 | 0.046 (2)* |
| C12 | 0.70311 (9) | 0.7217 (6) | 0.6967 (2) | 0.0284 (6) |
| H12 | 0.6990 | 0.8779 | 0.7409 | 0.046 (2)* |
| C13 | 0.66896 (9) | 0.5106 (6) | 0.72079 (19) | 0.0288 (6) |
| H13 | 0.6686 | 0.3638 | 0.6698 | 0.046 (2)* |
| C14 | 0.61956 (9) | 0.6315 (5) | 0.71191 (18) | 0.0260 (6) |
| H14 | 0.6202 | 0.7856 | 0.7595 | 0.046 (2)* |
| C15 | 0.60867 (9) | 0.7212 (6) | 0.5974 (2) | 0.0276 (6) |
| H15 | 0.6081 | 0.5646 | 0.5511 | 0.046 (2)* |
| C16 | 0.55935 (9) | 0.8494 (6) | 0.5802 (2) | 0.0342 (7) |
| H161 | 0.5595 | 1.0076 | 0.6242 | 0.046 (2)* |
| H162 | 0.5506 | 0.9018 | 0.5065 | 0.046 (2)* |
| C17 | 0.68845 (12) | 0.6421 (9) | 0.4118 (2) | 0.0523 (10) |
| H171 | 0.7102 | 0.7832 | 0.3967 | 0.046 (2)* |
| H172 | 0.6546 | 0.6973 | 0.3919 | 0.046 (2)* |
| H173 | 0.6946 | 0.4854 | 0.3723 | 0.046 (2)* |
| C21 | 0.80707 (10) | 0.2023 (6) | 0.8741 (2) | 0.0291 (6) |
| C22 | 0.80802 (9) | 0.4151 (6) | 0.9392 (2) | 0.0290 (6) |
| C23 | 0.84633 (10) | 0.4667 (6) | 1.0154 (2) | 0.0345 (7) |
| H23 | 0.8465 | 0.6115 | 1.0613 | 0.046 (2)* |
| C24 | 0.88525 (11) | 0.2911 (7) | 1.0211 (2) | 0.0398 (7) |

| | | | | |
|---|---|---|---|---|
| H24 | 0.9131 | 0.3189 | 1.0720 | 0.046 (2)* |
| C25 | 0.88453 (10) | 0.0791 (7) | 0.9552 (2) | 0.0390 (7) |
| H25 | 0.9117 | −0.0353 | 0.9621 | 0.046 (2)* |
| C26 | 0.84470 (10) | 0.0285 (6) | 0.8783 (2) | 0.0355 (7) |
| H26 | 0.8439 | −0.1164 | 0.8323 | 0.046 (2)* |
| C31 | 0.53608 (9) | 0.5784 (6) | 0.71128 (19) | 0.0291 (6) |
| H31 | 0.5360 | 0.7326 | 0.7586 | 0.046 (2)* |
| C32 | 0.49829 (9) | 0.3857 (6) | 0.7332 (2) | 0.0293 (6) |
| C33 | 0.50281 (11) | 0.2628 (7) | 0.8290 (2) | 0.0398 (7) |
| H33 | 0.5299 | 0.3006 | 0.8806 | 0.046 (2)* |
| C34 | 0.46786 (11) | 0.0846 (7) | 0.8501 (2) | 0.0455 (8) |
| H34 | 0.4714 | −0.0005 | 0.9158 | 0.046 (2)* |
| C35 | 0.42799 (11) | 0.0302 (7) | 0.7761 (2) | 0.0422 (8) |
| H35 | 0.4039 | −0.0902 | 0.7909 | 0.046 (2)* |
| C36 | 0.42350 (11) | 0.1516 (7) | 0.6813 (2) | 0.0471 (8) |
| H36 | 0.3962 | 0.1144 | 0.6301 | 0.046 (2)* |
| C37 | 0.45846 (11) | 0.3288 (7) | 0.6594 (2) | 0.0405 (7) |
| H37 | 0.4550 | 0.4114 | 0.5933 | 0.046 (2)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O11 | 0.0248 (10) | 0.0316 (10) | 0.0392 (10) | −0.0017 (8) | 0.0047 (8) | 0.0066 (9) |
| O12 | 0.0215 (9) | 0.0391 (11) | 0.0371 (10) | −0.0003 (9) | 0.0013 (7) | 0.0075 (10) |
| O13 | 0.0222 (10) | 0.0473 (12) | 0.0329 (10) | 0.0002 (9) | −0.0002 (7) | 0.0123 (9) |
| O14 | 0.0204 (9) | 0.0324 (10) | 0.0342 (9) | −0.0039 (8) | 0.0033 (7) | 0.0014 (9) |
| O16 | 0.0233 (10) | 0.0419 (11) | 0.0354 (10) | −0.0035 (9) | 0.0006 (8) | 0.0060 (9) |
| O17 | 0.0357 (11) | 0.0485 (13) | 0.0297 (9) | 0.0033 (10) | 0.0076 (8) | −0.0004 (10) |
| O21 | 0.0278 (10) | 0.0324 (10) | 0.0394 (11) | −0.0006 (9) | −0.0028 (8) | −0.0020 (9) |
| O22 | 0.0273 (10) | 0.0360 (11) | 0.0349 (10) | 0.0041 (9) | −0.0010 (8) | −0.0030 (9) |
| C10 | 0.0255 (15) | 0.0357 (16) | 0.0339 (14) | 0.0003 (12) | 0.0001 (11) | 0.0034 (13) |
| C11 | 0.0225 (14) | 0.0365 (15) | 0.0390 (15) | −0.0012 (12) | 0.0028 (12) | 0.0055 (13) |
| C12 | 0.0202 (13) | 0.0315 (14) | 0.0326 (14) | 0.0009 (11) | 0.0012 (11) | −0.0005 (12) |
| C13 | 0.0232 (13) | 0.0352 (15) | 0.0275 (13) | −0.0013 (12) | 0.0018 (10) | 0.0017 (12) |
| C14 | 0.0217 (13) | 0.0284 (14) | 0.0277 (13) | −0.0020 (11) | 0.0030 (10) | 0.0001 (12) |
| C15 | 0.0245 (13) | 0.0293 (14) | 0.0285 (13) | −0.0035 (12) | 0.0023 (10) | −0.0005 (11) |
| C16 | 0.0239 (14) | 0.0399 (16) | 0.0377 (15) | 0.0004 (13) | 0.0015 (12) | 0.0081 (13) |
| C17 | 0.0449 (19) | 0.082 (3) | 0.0305 (15) | 0.011 (2) | 0.0081 (13) | 0.0072 (18) |
| C21 | 0.0252 (14) | 0.0315 (14) | 0.0300 (13) | −0.0008 (12) | 0.0019 (11) | 0.0044 (12) |
| C22 | 0.0231 (14) | 0.0337 (15) | 0.0304 (13) | 0.0014 (12) | 0.0045 (10) | 0.0057 (12) |
| C23 | 0.0346 (16) | 0.0424 (16) | 0.0254 (13) | −0.0034 (13) | 0.0009 (11) | 0.0017 (13) |
| C24 | 0.0309 (15) | 0.054 (2) | 0.0325 (15) | −0.0007 (15) | −0.0015 (12) | 0.0132 (14) |
| C25 | 0.0270 (15) | 0.0445 (18) | 0.0447 (16) | 0.0086 (14) | 0.0027 (12) | 0.0160 (15) |
| C26 | 0.0353 (16) | 0.0321 (15) | 0.0393 (15) | 0.0017 (13) | 0.0063 (12) | 0.0073 (13) |
| C31 | 0.0217 (13) | 0.0352 (14) | 0.0298 (13) | 0.0016 (12) | 0.0016 (10) | −0.0019 (12) |
| C32 | 0.0227 (13) | 0.0343 (15) | 0.0316 (14) | −0.0002 (12) | 0.0062 (11) | −0.0047 (12) |
| C33 | 0.0323 (16) | 0.052 (2) | 0.0356 (15) | −0.0069 (14) | 0.0048 (12) | 0.0023 (15) |
| C34 | 0.0438 (18) | 0.054 (2) | 0.0397 (16) | −0.0089 (17) | 0.0107 (14) | 0.0083 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C35 | 0.0325 (16) | 0.0449 (18) | 0.0519 (18) | −0.0090 (15) | 0.0149 (14) | 0.0001 (15) |
| C36 | 0.0346 (17) | 0.056 (2) | 0.0487 (18) | −0.0153 (16) | −0.0012 (14) | −0.0020 (17) |
| C37 | 0.0329 (16) | 0.0477 (18) | 0.0393 (16) | −0.0100 (14) | 0.0011 (13) | 0.0036 (15) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O11—C15 | 1.432 (3) | C16—H162 | 0.990 |
| O11—C11 | 1.440 (3) | C17—H171 | 0.980 |
| O12—C10 | 1.407 (3) | C17—H172 | 0.980 |
| O12—C12 | 1.432 (3) | C17—H173 | 0.980 |
| O13—C10 | 1.388 (3) | C21—C26 | 1.367 (4) |
| O13—C13 | 1.442 (3) | C21—C22 | 1.376 (4) |
| O14—C14 | 1.424 (3) | C22—C23 | 1.367 (4) |
| O14—C31 | 1.433 (3) | C23—C24 | 1.399 (4) |
| O16—C31 | 1.402 (3) | C23—H23 | 0.950 |
| O16—C16 | 1.437 (3) | C24—C25 | 1.380 (5) |
| O17—C11 | 1.394 (4) | C24—H24 | 0.950 |
| O17—C17 | 1.437 (3) | C25—C26 | 1.400 (4) |
| O21—C21 | 1.386 (3) | C25—H25 | 0.950 |
| O21—C10 | 1.413 (4) | C26—H26 | 0.950 |
| O22—C22 | 1.385 (3) | C31—C32 | 1.499 (4) |
| O22—C10 | 1.405 (3) | C31—H31 | 1.000 |
| C11—C12 | 1.518 (4) | C32—C37 | 1.382 (4) |
| C11—H11 | 1.000 | C32—C33 | 1.384 (4) |
| C12—C13 | 1.501 (4) | C33—C34 | 1.390 (4) |
| C12—H12 | 1.000 | C33—H33 | 0.950 |
| C13—C14 | 1.495 (4) | C34—C35 | 1.382 (4) |
| C13—H13 | 1.000 | C34—H34 | 0.950 |
| C14—C15 | 1.545 (3) | C35—C36 | 1.369 (4) |
| C14—H14 | 1.000 | C35—H35 | 0.950 |
| C15—C16 | 1.506 (4) | C36—C37 | 1.389 (4) |
| C15—H15 | 1.000 | C36—H36 | 0.950 |
| C16—H161 | 0.990 | C37—H37 | 0.950 |
| C15—O11—C11 | 113.7 (2) | H161—C16—H162 | 108.5 |
| C10—O12—C12 | 105.5 (2) | O17—C17—H171 | 109.5 |
| C10—O13—C13 | 105.67 (19) | O17—C17—H172 | 109.5 |
| C14—O14—C31 | 109.77 (19) | H171—C17—H172 | 109.5 |
| C31—O16—C16 | 112.5 (2) | O17—C17—H173 | 109.5 |
| C11—O17—C17 | 113.0 (3) | H171—C17—H173 | 109.5 |
| C21—O21—C10 | 105.6 (2) | H172—C17—H173 | 109.5 |
| C22—O22—C10 | 106.2 (2) | C26—C21—C22 | 123.0 (2) |
| O13—C10—O22 | 109.0 (2) | C26—C21—O21 | 127.8 (3) |
| O13—C10—O12 | 109.1 (2) | C22—C21—O21 | 109.1 (2) |
| O22—C10—O12 | 111.9 (2) | C23—C22—C21 | 122.6 (3) |
| O13—C10—O21 | 112.0 (2) | C23—C22—O22 | 128.7 (3) |
| O22—C10—O21 | 107.5 (2) | C21—C22—O22 | 108.7 (2) |
| O12—C10—O21 | 107.3 (2) | C22—C23—C24 | 115.3 (3) |
| O17—C11—O11 | 113.1 (2) | C22—C23—H23 | 122.4 |
| O17—C11—C12 | 109.0 (2) | C24—C23—H23 | 122.4 |

| | | | |
|---|---|---|---|
| O11—C11—C12 | 104.3 (2) | C25—C24—C23 | 122.1 (3) |
| O17—C11—H11 | 110.1 | C25—C24—H24 | 118.9 |
| O11—C11—H11 | 110.1 | C23—C24—H24 | 118.9 |
| C12—C11—H11 | 110.1 | C24—C25—C26 | 121.6 (3) |
| O12—C12—C13 | 101.4 (2) | C24—C25—H25 | 119.2 |
| O12—C12—C11 | 117.5 (2) | C26—C25—H25 | 119.2 |
| C13—C12—C11 | 111.2 (2) | C21—C26—C25 | 115.3 (3) |
| O12—C12—H12 | 108.8 | C21—C26—H26 | 122.4 |
| C13—C12—H12 | 108.8 | C25—C26—H26 | 122.4 |
| C11—C12—H12 | 108.8 | O16—C31—O14 | 111.4 (2) |
| O13—C13—C14 | 118.8 (2) | O16—C31—C32 | 108.9 (2) |
| O13—C13—C12 | 100.36 (19) | O14—C31—C32 | 108.2 (2) |
| C14—C13—C12 | 106.9 (2) | O16—C31—H31 | 109.4 |
| O13—C13—H13 | 110.1 | O14—C31—H31 | 109.4 |
| C14—C13—H13 | 110.1 | C32—C31—H31 | 109.4 |
| C12—C13—H13 | 110.1 | C37—C32—C33 | 118.9 (3) |
| O14—C14—C13 | 113.6 (2) | C37—C32—C31 | 121.2 (2) |
| O14—C14—C15 | 108.25 (19) | C33—C32—C31 | 119.9 (2) |
| C13—C14—C15 | 103.9 (2) | C32—C33—C34 | 120.3 (3) |
| O14—C14—H14 | 110.3 | C32—C33—H33 | 119.9 |
| C13—C14—H14 | 110.3 | C34—C33—H33 | 119.9 |
| C15—C14—H14 | 110.3 | C35—C34—C33 | 120.4 (3) |
| O11—C15—C16 | 108.7 (2) | C35—C34—H34 | 119.8 |
| O11—C15—C14 | 112.67 (19) | C33—C34—H34 | 119.8 |
| C16—C15—C14 | 108.2 (2) | C36—C35—C34 | 119.4 (3) |
| O11—C15—H15 | 109.1 | C36—C35—H35 | 120.3 |
| C16—C15—H15 | 109.1 | C34—C35—H35 | 120.3 |
| C14—C15—H15 | 109.1 | C35—C36—C37 | 120.6 (3) |
| O16—C16—C15 | 107.3 (2) | C35—C36—H36 | 119.7 |
| O16—C16—H161 | 110.2 | C37—C36—H36 | 119.7 |
| C15—C16—H161 | 110.2 | C32—C37—C36 | 120.5 (3) |
| O16—C16—H162 | 110.2 | C32—C37—H37 | 119.8 |
| C15—C16—H162 | 110.2 | C36—C37—H37 | 119.8 |
| C13—O13—C10—O22 | 138.5 (2) | C13—C14—C15—O11 | −59.3 (3) |
| C13—O13—C10—O12 | 16.0 (3) | O14—C14—C15—C16 | 59.4 (3) |
| C13—O13—C10—O21 | −102.6 (2) | C13—C14—C15—C16 | −179.5 (2) |
| C22—O22—C10—O13 | 138.2 (2) | C31—O16—C16—C15 | 59.3 (3) |
| C22—O22—C10—O12 | −101.0 (3) | O11—C15—C16—O16 | −179.92 (19) |
| C22—O22—C10—O21 | 16.6 (3) | C14—C15—C16—O16 | −57.3 (3) |
| C12—O12—C10—O13 | 11.6 (3) | C10—O21—C21—C26 | −167.6 (3) |
| C12—O12—C10—O22 | −109.1 (2) | C10—O21—C21—C22 | 10.7 (3) |
| C12—O12—C10—O21 | 133.1 (2) | C26—C21—C22—C23 | −1.6 (4) |
| C21—O21—C10—O13 | −136.5 (2) | O21—C21—C22—C23 | 180.0 (2) |
| C21—O21—C10—O22 | −16.8 (3) | C26—C21—C22—O22 | 177.9 (2) |
| C21—O21—C10—O12 | 103.8 (2) | O21—C21—C22—O22 | −0.5 (3) |
| C17—O17—C11—O11 | 65.2 (3) | C10—O22—C22—C23 | 169.5 (3) |
| C17—O17—C11—C12 | −179.3 (2) | C10—O22—C22—C21 | −10.1 (3) |
| C15—O11—C11—O17 | 60.5 (3) | C21—C22—C23—C24 | 1.5 (4) |
| C15—O11—C11—C12 | −57.8 (3) | O22—C22—C23—C24 | −177.9 (3) |

| | | | |
|---|---|---|---|
| C10—O12—C12—C13 | −33.0 (2) | C22—C23—C24—C25 | −0.9 (4) |
| C10—O12—C12—C11 | −154.4 (2) | C23—C24—C25—C26 | 0.3 (4) |
| O17—C11—C12—O12 | 57.3 (3) | C22—C21—C26—C25 | 0.9 (4) |
| O11—C11—C12—O12 | 178.4 (2) | O21—C21—C26—C25 | 179.0 (3) |
| O17—C11—C12—C13 | −58.9 (3) | C24—C25—C26—C21 | −0.3 (4) |
| O11—C11—C12—C13 | 62.2 (3) | C16—O16—C31—O14 | −61.3 (3) |
| C10—O13—C13—C14 | −151.3 (2) | C16—O16—C31—C32 | 179.4 (2) |
| C10—O13—C13—C12 | −35.4 (3) | C14—O14—C31—O16 | 61.2 (3) |
| O12—C12—C13—O13 | 41.6 (2) | C14—O14—C31—C32 | −179.14 (18) |
| C11—C12—C13—O13 | 167.3 (2) | O16—C31—C32—C37 | −4.4 (4) |
| O12—C12—C13—C14 | 166.13 (19) | O14—C31—C32—C37 | −125.6 (3) |
| C11—C12—C13—C14 | −68.2 (3) | O16—C31—C32—C33 | 176.1 (3) |
| C31—O14—C14—C13 | −174.84 (19) | O14—C31—C32—C33 | 54.9 (3) |
| C31—O14—C14—C15 | −60.0 (2) | C37—C32—C33—C34 | 0.3 (4) |
| O13—C13—C14—O14 | −68.9 (3) | C31—C32—C33—C34 | 179.8 (3) |
| C12—C13—C14—O14 | 178.70 (19) | C32—C33—C34—C35 | −0.7 (5) |
| O13—C13—C14—C15 | 173.7 (2) | C33—C34—C35—C36 | 0.7 (5) |
| C12—C13—C14—C15 | 61.3 (3) | C34—C35—C36—C37 | −0.3 (5) |
| C11—O11—C15—C16 | −179.5 (2) | C33—C32—C37—C36 | 0.2 (5) |
| C11—O11—C15—C14 | 60.6 (3) | C31—C32—C37—C36 | −179.4 (3) |
| O14—C14—C15—O11 | 179.6 (2) | C35—C36—C37—C32 | −0.2 (5) |

**Fig. 1**

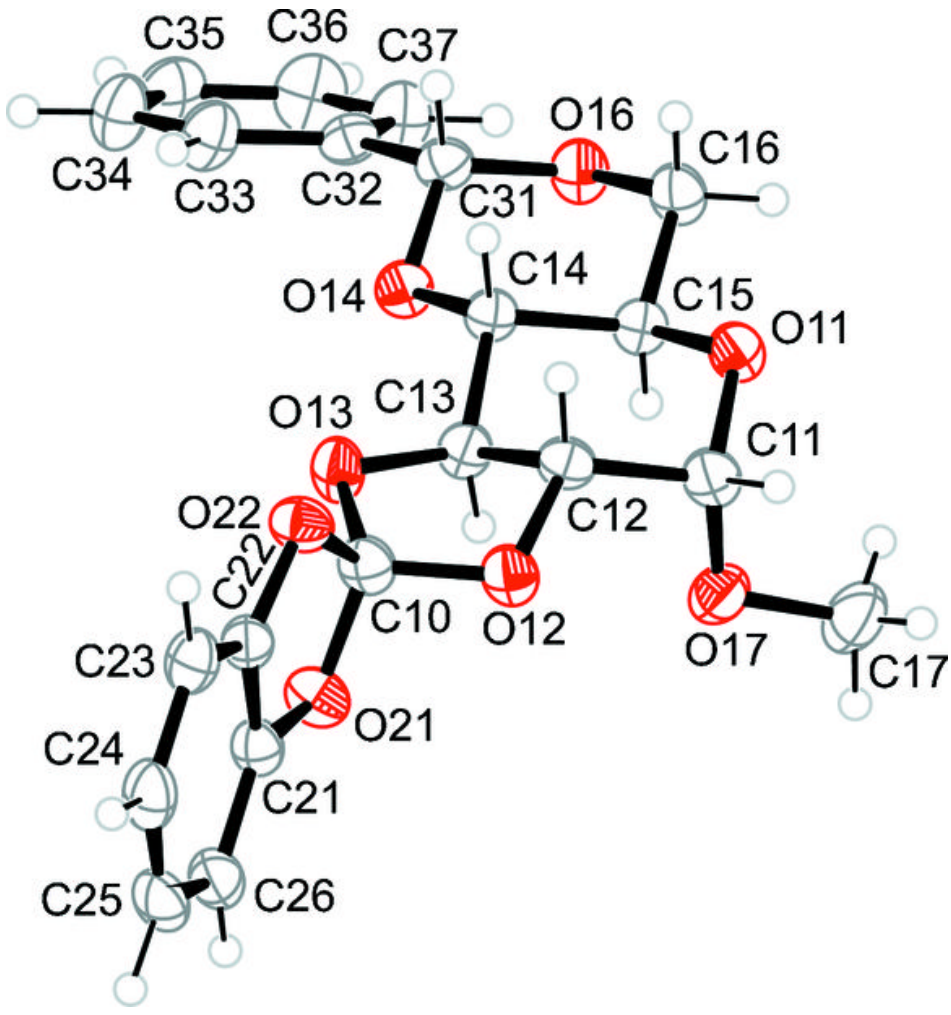
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**Fig. 2**