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2,3,4-Tri-*O*-acetyl- β -D-xylosyl
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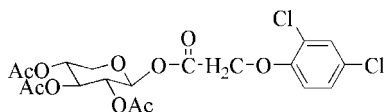
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
 R factor = 0.047; wR factor = 0.121; data-to-parameter ratio = 11.8.

In the title compound, $\text{C}_{19}\text{H}_{20}\text{Cl}_2\text{O}_{10}$, the hexopyranosyl ring adopts a chair conformation. The four substituents are in equatorial positions. The molecules are linked *via* $\text{C}-\text{H}\cdots\text{O}$ contacts along the a axis.

Related literature

For related literature, see: Hamner *et al.* (1946); Chandra-sekhar & Pattabhi (1977); Dalton (2004); Tsorteki *et al.* (2004); Yang *et al.* (2004).



Experimental

Crystal data

 $\text{C}_{19}\text{H}_{20}\text{Cl}_2\text{O}_{10}$ $M_r = 479.25$ Monoclinic, $P2_1$ $a = 5.6601$ (8) Å $b = 23.129$ (3) Å $c = 8.7456$ (13) Å $\beta = 104.281$ (2)° $V = 1109.5$ (3) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.34$ mm⁻¹ $T = 293$ (2) K $0.45 \times 0.23 \times 0.21$ mm

Data collection

Bruker SMART APEX CCD

diffractometer

Absorption correction: none

5656 measured reflections

3337 independent reflections

3044 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.089$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.121$ $S = 1.03$

3337 reflections

284 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Absolute structure: Flack (1983),

1325 Friedel pairs

Flack parameter: 0.04 (8)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{C3}-\text{H3}\cdots\text{O6}^i$ | 0.93 | 2.41 | 3.322 (6) | 168 |
| $\text{C9}-\text{H9}\cdots\text{O10}^i$ | 0.98 | 2.54 | 3.381 (4) | 144 |
| $\text{C11}-\text{H11}\cdots\text{O10}^i$ | 0.98 | 2.44 | 3.296 (4) | 146 |

Symmetry code: (i) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful to the National Natural Science Foundation of China (No. 30701041), the Postdoctoral Science Foundation of China (No. 20060400917) and Jiangsu Postdoctoral Science Foundation of China (No. 0602002B).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2071).

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supplementary materials

Acta Cryst. (2008). E64, o669 [doi:10.1107/S1600536808005837]

2,3,4-Tri-*O*-acetyl- β -D-xylosyl 2,4-dichlorophenoxyacetate

X. Wang, X. Li, Y. Yin, Y. Pang and Y. Yang

Comment

The plant growth regulator 2,4-dichlorophenoxyacetic acid cocrystallized as a guest molecule in Heptakis(2,3,6-tri-*O*-methyl)- β -cyclodextrin (Tsorteki *et al.*, 2004), and it plays an important role in graining and controlling weeds (Hamner *et al.*, 1946). However, problems such as toxic residues and environmental pollution were protruded increasingly by using amounts of herbicides during the past decades (Dalton, 2004). In order to search for a new herbicide with high efficiency and low toxicity, we obtained the title compound. All bond lengths and angles in the title molecule show normal values. The hexopyranosyl ring adopts a chair conformation (Fig. 1). The three acetyl groups are individually planar and occupy equatorial positions (Yang *et al.*, 2004). The 2,4-dichlorophenoxyacetic acid group shows a similar geometry in 2-Chlorophenoxyacetic acid (Chandrasekhar & Pattabhi, 1977), and it is twisted at the bond of O3—C8—C7—O1, with the torsion angle of 4.3°. The title molecules are linked *via* intermolecular hydrogen bonding C—H \cdots O contacts along the *a* axis by translation (Table 1).

Experimental

The title compound was prepared from α -D-1-bromo-2,3,4-tri-*O*-acetyl-xylosyl with 2,4-dichlorophenoxyacetic acid in aq NaOH at the benzyltriethylammonium chloride and 4-dimethylaminopyridine in present. Fine block colourless crystals for single-crystal X-ray diffraction were obtained by slow evaporation of an ethyl acetate at room temperature.

Refinement

The H atoms were refined by riding on their appropriate parent atoms in their as-found or calculated positions. The C—H distances for CH, CH₂ and CH₃ groups are 0.93, 0.96 and 0.97 Å, respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{Csp}^2)$ or $1.5U_{\text{eq}}(\text{Csp}^3)$. The absolute structure parameter was determined as 0.04 (8) (Flack, 1983). The number of Friedel pairs was found to be 1325 by comparison of merged intensity reflections (2012) with unmerged unique reflections of the final refinement.

Figures

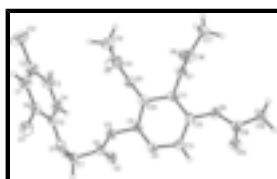


Fig. 1. The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,3,4-Tri-*O*-acetyl- β -D-xylosyl 2,4-dichlorophenoxyacetate

Crystal data

| | |
|--------------------------------|---|
| $C_{19}H_{20}Cl_2O_{10}$ | $F_{000} = 496$ |
| $M_r = 479.25$ | $D_x = 1.435 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation |
| Hall symbol: P 2yb | $\lambda = 0.71073 \text{ \AA}$ |
| $a = 5.6601 (8) \text{ \AA}$ | Cell parameters from 90 reflections |
| $b = 23.129 (3) \text{ \AA}$ | $\theta = 2.4\text{--}25.0^\circ$ |
| $c = 8.7456 (13) \text{ \AA}$ | $\mu = 0.35 \text{ mm}^{-1}$ |
| $\beta = 104.281 (2)^\circ$ | $T = 293 (2) \text{ K}$ |
| $V = 1109.5 (3) \text{ \AA}^3$ | Block, colourless |
| $Z = 2$ | $0.45 \times 0.23 \times 0.21 \text{ mm}$ |

Data collection

| | |
|---|--|
| Bruker SMART APEX CCD diffractometer | 3337 independent reflections |
| Radiation source: fine-focus sealed tube | 3044 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.089$ |
| Detector resolution: 9.00cm pixels mm^{-1} | $\theta_{\text{max}} = 25.0^\circ$ |
| $T = 293(2) \text{ K}$ | $\theta_{\text{min}} = 2.4^\circ$ |
| ω and φ scans | $h = -6 \rightarrow 5$ |
| Absorption correction: none | $k = -27 \rightarrow 15$ |
| 5656 measured reflections | $l = -9 \rightarrow 10$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | $w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 0.2029P]$ |
| $wR(F^2) = 0.121$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.04$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 3337 reflections | $\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$ |
| 284 parameters | $\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 1325 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack parameter: 0.04 (8) |

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C11 | 0.39611 (18) | 0.41178 (5) | 0.40852 (13) | 0.0708 (3) |
| C12 | -0.4306 (3) | 0.37317 (8) | -0.0324 (2) | 0.1303 (7) |
| O1 | 0.2663 (5) | 0.31057 (11) | 0.5567 (3) | 0.0593 (6) |
| O2 | 0.1992 (6) | 0.16524 (12) | 0.6787 (3) | 0.0662 (7) |
| O3 | 0.3421 (4) | 0.20235 (10) | 0.4807 (3) | 0.0492 (5) |
| O4 | 0.6349 (4) | 0.13402 (10) | 0.5269 (3) | 0.0501 (6) |
| O5 | 0.1431 (4) | 0.15293 (10) | 0.1744 (3) | 0.0452 (5) |
| O6 | 0.2612 (5) | 0.22405 (14) | 0.0364 (4) | 0.0748 (9) |
| O7 | 0.4821 (4) | 0.08031 (10) | 0.0669 (2) | 0.0478 (5) |
| O8 | 0.1649 (6) | 0.01975 (16) | 0.0038 (4) | 0.0854 (10) |
| O9 | 0.7485 (4) | 0.00745 (10) | 0.2985 (2) | 0.0451 (5) |
| O10 | 1.1379 (4) | 0.01306 (11) | 0.4309 (3) | 0.0552 (6) |
| C1 | -0.0205 (8) | 0.38597 (19) | 0.1966 (5) | 0.0648 (10) |
| H1 | 0.0105 | 0.4181 | 0.1406 | 0.078* |
| C2 | -0.2255 (8) | 0.3534 (2) | 0.1430 (5) | 0.0733 (11) |
| C3 | -0.2759 (8) | 0.30616 (19) | 0.2235 (6) | 0.0724 (11) |
| H3 | -0.4165 | 0.2846 | 0.1841 | 0.087* |
| C4 | -0.1159 (7) | 0.29073 (17) | 0.3640 (5) | 0.0601 (9) |
| H4 | -0.1510 | 0.2592 | 0.4207 | 0.072* |
| C5 | 0.0968 (6) | 0.32194 (15) | 0.4211 (4) | 0.0502 (8) |
| C6 | 0.1387 (6) | 0.36984 (16) | 0.3357 (4) | 0.0520 (8) |
| C7 | 0.2153 (9) | 0.26725 (17) | 0.6570 (4) | 0.0632 (10) |
| H7A | 0.0481 | 0.2720 | 0.6641 | 0.076* |
| H7B | 0.3196 | 0.2734 | 0.7617 | 0.076* |
| C8 | 0.2479 (6) | 0.20569 (15) | 0.6091 (4) | 0.0470 (7) |
| C9 | 0.3960 (5) | 0.14523 (14) | 0.4394 (3) | 0.0406 (6) |
| H9 | 0.2811 | 0.1173 | 0.4647 | 0.049* |
| C10 | 0.3903 (5) | 0.14345 (14) | 0.2641 (3) | 0.0396 (6) |
| H10 | 0.5002 | 0.1726 | 0.2382 | 0.048* |
| C11 | 0.4660 (5) | 0.08292 (14) | 0.2273 (3) | 0.0383 (6) |
| H11 | 0.3442 | 0.0550 | 0.2435 | 0.046* |
| C12 | 0.7106 (5) | 0.06757 (13) | 0.3323 (4) | 0.0407 (6) |
| H12 | 0.8380 | 0.0917 | 0.3066 | 0.049* |

supplementary materials

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|------|-------------|---------------|-------------|-------------|
| C13 | 0.7069 (6) | 0.07587 (15) | 0.5044 (4) | 0.0462 (7) |
| H13A | 0.5931 | 0.0489 | 0.5323 | 0.055* |
| H13B | 0.8676 | 0.0684 | 0.5719 | 0.055* |
| C14 | 0.1046 (6) | 0.19246 (16) | 0.0576 (4) | 0.0479 (8) |
| C15 | -0.1479 (7) | 0.1900 (2) | -0.0418 (5) | 0.0628 (10) |
| H15A | -0.2249 | 0.2269 | -0.0404 | 0.094* |
| H15B | -0.2378 | 0.1610 | -0.0013 | 0.094* |
| H15C | -0.1450 | 0.1804 | -0.1481 | 0.094* |
| C16 | 0.3333 (7) | 0.04317 (17) | -0.0295 (4) | 0.0543 (9) |
| C17 | 0.4131 (10) | 0.0352 (2) | -0.1786 (5) | 0.0802 (14) |
| H17A | 0.5523 | 0.0101 | -0.1593 | 0.120* |
| H17B | 0.4557 | 0.0721 | -0.2148 | 0.120* |
| H17C | 0.2827 | 0.0184 | -0.2575 | 0.120* |
| C18 | 0.9736 (5) | -0.01395 (15) | 0.3484 (4) | 0.0439 (7) |
| C19 | 0.9926 (7) | -0.07338 (17) | 0.2889 (5) | 0.0620 (9) |
| H19A | 1.0063 | -0.0716 | 0.1818 | 0.093* |
| H19B | 0.8497 | -0.0950 | 0.2934 | 0.093* |
| H19C | 1.1343 | -0.0920 | 0.3531 | 0.093* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Cl1 | 0.0728 (6) | 0.0576 (6) | 0.0782 (6) | -0.0171 (5) | 0.0114 (5) | -0.0004 (5) |
| Cl2 | 0.1225 (12) | 0.1201 (14) | 0.1079 (10) | -0.0217 (10) | -0.0481 (9) | 0.0335 (10) |
| O1 | 0.0780 (16) | 0.0361 (13) | 0.0569 (14) | 0.0026 (12) | 0.0038 (12) | -0.0004 (11) |
| O2 | 0.100 (2) | 0.0457 (16) | 0.0624 (16) | 0.0022 (14) | 0.0379 (14) | 0.0033 (12) |
| O3 | 0.0682 (14) | 0.0353 (13) | 0.0468 (12) | 0.0000 (10) | 0.0191 (10) | -0.0025 (10) |
| O4 | 0.0557 (13) | 0.0461 (15) | 0.0444 (12) | 0.0009 (10) | 0.0044 (10) | -0.0115 (10) |
| O5 | 0.0425 (11) | 0.0481 (14) | 0.0462 (12) | -0.0016 (9) | 0.0130 (9) | 0.0082 (10) |
| O6 | 0.0692 (16) | 0.076 (2) | 0.0702 (17) | -0.0201 (15) | -0.0009 (13) | 0.0305 (15) |
| O7 | 0.0600 (13) | 0.0497 (14) | 0.0362 (10) | -0.0045 (11) | 0.0170 (9) | -0.0007 (10) |
| O8 | 0.090 (2) | 0.091 (3) | 0.0718 (19) | -0.0339 (19) | 0.0142 (16) | -0.0312 (17) |
| O9 | 0.0458 (11) | 0.0385 (13) | 0.0508 (12) | -0.0028 (9) | 0.0114 (9) | -0.0053 (10) |
| O10 | 0.0480 (12) | 0.0565 (16) | 0.0585 (14) | -0.0034 (11) | 0.0084 (10) | -0.0008 (12) |
| C1 | 0.075 (2) | 0.057 (2) | 0.058 (2) | -0.0028 (19) | 0.0095 (17) | 0.0090 (18) |
| C2 | 0.070 (2) | 0.067 (3) | 0.071 (2) | 0.001 (2) | -0.0058 (19) | 0.006 (2) |
| C3 | 0.065 (2) | 0.050 (2) | 0.095 (3) | -0.0128 (18) | 0.003 (2) | -0.003 (2) |
| C4 | 0.068 (2) | 0.037 (2) | 0.074 (2) | -0.0035 (16) | 0.0142 (18) | 0.0021 (17) |
| C5 | 0.0619 (19) | 0.0340 (18) | 0.0543 (18) | 0.0064 (15) | 0.0134 (15) | -0.0033 (14) |
| C6 | 0.0598 (18) | 0.0414 (19) | 0.0548 (18) | 0.0017 (15) | 0.0142 (15) | -0.0048 (15) |
| C7 | 0.097 (3) | 0.041 (2) | 0.052 (2) | 0.0103 (19) | 0.0190 (19) | -0.0014 (17) |
| C8 | 0.0599 (18) | 0.0381 (19) | 0.0414 (15) | 0.0056 (14) | 0.0095 (14) | 0.0016 (14) |
| C9 | 0.0506 (16) | 0.0316 (16) | 0.0395 (15) | -0.0021 (13) | 0.0112 (12) | -0.0031 (12) |
| C10 | 0.0399 (14) | 0.0401 (17) | 0.0389 (14) | -0.0072 (12) | 0.0099 (11) | 0.0027 (13) |
| C11 | 0.0443 (14) | 0.0404 (17) | 0.0331 (13) | -0.0098 (12) | 0.0150 (11) | -0.0017 (12) |
| C12 | 0.0418 (15) | 0.0334 (17) | 0.0477 (16) | -0.0036 (13) | 0.0125 (12) | -0.0020 (13) |
| C13 | 0.0502 (16) | 0.0423 (19) | 0.0442 (16) | 0.0044 (14) | 0.0081 (13) | -0.0024 (14) |
| C14 | 0.0517 (17) | 0.051 (2) | 0.0423 (16) | -0.0055 (15) | 0.0146 (13) | -0.0017 (14) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C15 | 0.0564 (19) | 0.073 (3) | 0.058 (2) | 0.0036 (18) | 0.0110 (16) | 0.0126 (19) |
| C16 | 0.069 (2) | 0.046 (2) | 0.0420 (17) | 0.0046 (17) | 0.0039 (16) | -0.0052 (15) |
| C17 | 0.132 (4) | 0.065 (3) | 0.044 (2) | 0.019 (3) | 0.023 (2) | -0.0076 (19) |
| C18 | 0.0437 (17) | 0.0473 (19) | 0.0421 (16) | -0.0012 (14) | 0.0136 (13) | 0.0055 (13) |
| C19 | 0.063 (2) | 0.051 (2) | 0.071 (2) | 0.0030 (17) | 0.0147 (17) | -0.0049 (18) |

Geometric parameters (Å, °)

| | | | |
|------------|-----------|--------------|-----------|
| C11—C6 | 1.735 (4) | C5—C6 | 1.389 (5) |
| C12—C2 | 1.740 (4) | C7—C8 | 1.508 (5) |
| O1—C5 | 1.355 (4) | C7—H7A | 0.9700 |
| O1—C7 | 1.407 (5) | C7—H7B | 0.9700 |
| O2—C8 | 1.185 (4) | C9—C10 | 1.526 (4) |
| O3—C8 | 1.359 (4) | C9—H9 | 0.9800 |
| O3—C9 | 1.422 (4) | C10—C11 | 1.521 (4) |
| O4—C9 | 1.404 (4) | C10—H10 | 0.9800 |
| O4—C13 | 1.433 (4) | C11—C12 | 1.503 (4) |
| O5—C14 | 1.347 (4) | C11—H11 | 0.9800 |
| O5—C10 | 1.442 (4) | C12—C13 | 1.523 (4) |
| O6—C14 | 1.198 (4) | C12—H12 | 0.9800 |
| O7—C16 | 1.344 (4) | C13—H13A | 0.9700 |
| O7—C11 | 1.429 (3) | C13—H13B | 0.9700 |
| O8—C16 | 1.193 (5) | C14—C15 | 1.479 (5) |
| O9—C18 | 1.336 (4) | C15—H15A | 0.9600 |
| O9—C12 | 1.448 (4) | C15—H15B | 0.9600 |
| O10—C18 | 1.202 (4) | C15—H15C | 0.9600 |
| C1—C2 | 1.366 (6) | C16—C17 | 1.493 (5) |
| C1—C6 | 1.375 (5) | C17—H17A | 0.9600 |
| C1—H1 | 0.9300 | C17—H17B | 0.9600 |
| C2—C3 | 1.367 (6) | C17—H17C | 0.9600 |
| C3—C4 | 1.381 (6) | C18—C19 | 1.483 (5) |
| C3—H3 | 0.9300 | C19—H19A | 0.9600 |
| C4—C5 | 1.387 (5) | C19—H19B | 0.9600 |
| C4—H4 | 0.9300 | C19—H19C | 0.9600 |
| C5—O1—C7 | 118.3 (3) | O7—C11—C12 | 108.5 (2) |
| C8—O3—C9 | 114.5 (2) | O7—C11—C10 | 109.6 (2) |
| C9—O4—C13 | 111.5 (2) | C12—C11—C10 | 110.7 (2) |
| C14—O5—C10 | 118.0 (2) | O7—C11—H11 | 109.3 |
| C16—O7—C11 | 117.4 (3) | C12—C11—H11 | 109.3 |
| C18—O9—C12 | 117.8 (2) | C10—C11—H11 | 109.3 |
| C2—C1—C6 | 118.0 (4) | O9—C12—C11 | 105.2 (2) |
| C2—C1—H1 | 121.0 | O9—C12—C13 | 111.2 (2) |
| C6—C1—H1 | 121.0 | C11—C12—C13 | 109.8 (2) |
| C1—C2—C3 | 122.1 (4) | O9—C12—H12 | 110.2 |
| C1—C2—C12 | 118.9 (4) | C11—C12—H12 | 110.2 |
| C3—C2—C12 | 119.1 (3) | C13—C12—H12 | 110.2 |
| C2—C3—C4 | 119.4 (4) | O4—C13—C12 | 109.1 (3) |
| C2—C3—H3 | 120.3 | O4—C13—H13A | 109.9 |
| C4—C3—H3 | 120.3 | C12—C13—H13A | 109.9 |

supplementary materials

| | | | |
|--------------|------------|-----------------|------------|
| C3—C4—C5 | 120.4 (4) | O4—C13—H13B | 109.9 |
| C3—C4—H4 | 119.8 | C12—C13—H13B | 109.9 |
| C5—C4—H4 | 119.8 | H13A—C13—H13B | 108.3 |
| O1—C5—C4 | 125.3 (3) | O6—C14—O5 | 122.9 (3) |
| O1—C5—C6 | 116.6 (3) | O6—C14—C15 | 125.2 (3) |
| C4—C5—C6 | 118.0 (3) | O5—C14—C15 | 111.8 (3) |
| C1—C6—C5 | 122.1 (3) | C14—C15—H15A | 109.5 |
| C1—C6—C11 | 118.7 (3) | C14—C15—H15B | 109.5 |
| C5—C6—C11 | 119.2 (3) | H15A—C15—H15B | 109.5 |
| O1—C7—C8 | 116.2 (3) | C14—C15—H15C | 109.5 |
| O1—C7—H7A | 108.2 | H15A—C15—H15C | 109.5 |
| C8—C7—H7A | 108.2 | H15B—C15—H15C | 109.5 |
| O1—C7—H7B | 108.2 | O8—C16—O7 | 123.5 (3) |
| C8—C7—H7B | 108.2 | O8—C16—C17 | 126.1 (4) |
| H7A—C7—H7B | 107.4 | O7—C16—C17 | 110.4 (4) |
| O2—C8—O3 | 124.6 (3) | C16—C17—H17A | 109.5 |
| O2—C8—C7 | 122.9 (3) | C16—C17—H17B | 109.5 |
| O3—C8—C7 | 112.5 (3) | H17A—C17—H17B | 109.5 |
| O4—C9—O3 | 105.7 (2) | C16—C17—H17C | 109.5 |
| O4—C9—C10 | 108.8 (2) | H17A—C17—H17C | 109.5 |
| O3—C9—C10 | 109.1 (2) | H17B—C17—H17C | 109.5 |
| O4—C9—H9 | 111.0 | O10—C18—O9 | 122.5 (3) |
| O3—C9—H9 | 111.0 | O10—C18—C19 | 125.5 (3) |
| C10—C9—H9 | 111.0 | O9—C18—C19 | 112.0 (3) |
| O5—C10—C11 | 108.1 (2) | C18—C19—H19A | 109.5 |
| O5—C10—C9 | 108.6 (2) | C18—C19—H19B | 109.5 |
| C11—C10—C9 | 107.4 (2) | H19A—C19—H19B | 109.5 |
| O5—C10—H10 | 110.8 | C18—C19—H19C | 109.5 |
| C11—C10—H10 | 110.8 | H19A—C19—H19C | 109.5 |
| C9—C10—H10 | 110.8 | H19B—C19—H19C | 109.5 |
| C6—C1—C2—C3 | 0.4 (7) | O4—C9—C10—O5 | 178.0 (2) |
| C6—C1—C2—C12 | 179.7 (3) | O3—C9—C10—O5 | -67.1 (3) |
| C1—C2—C3—C4 | 0.3 (7) | O4—C9—C10—C11 | 61.2 (3) |
| C12—C2—C3—C4 | -179.0 (4) | O3—C9—C10—C11 | 176.1 (2) |
| C2—C3—C4—C5 | -1.6 (7) | C16—O7—C11—C12 | 120.4 (3) |
| C7—O1—C5—C4 | -6.4 (5) | C16—O7—C11—C10 | -118.6 (3) |
| C7—O1—C5—C6 | 171.7 (3) | O5—C10—C11—O7 | 67.4 (3) |
| C3—C4—C5—O1 | -179.8 (4) | C9—C10—C11—O7 | -175.5 (2) |
| C3—C4—C5—C6 | 2.1 (5) | O5—C10—C11—C12 | -172.9 (2) |
| C2—C1—C6—C5 | 0.2 (6) | C9—C10—C11—C12 | -55.9 (3) |
| C2—C1—C6—C11 | -178.3 (3) | C18—O9—C12—C11 | 165.2 (2) |
| O1—C5—C6—C1 | -179.7 (3) | C18—O9—C12—C13 | -76.0 (3) |
| C4—C5—C6—C1 | -1.4 (5) | O7—C11—C12—O9 | -66.0 (3) |
| O1—C5—C6—C11 | -1.2 (4) | C10—C11—C12—O9 | 173.7 (2) |
| C4—C5—C6—C11 | 177.0 (3) | O7—C11—C12—C13 | 174.3 (2) |
| C5—O1—C7—C8 | 78.3 (4) | C10—C11—C12—C13 | 54.0 (3) |
| C9—O3—C8—O2 | -4.1 (5) | C9—O4—C13—C12 | 63.7 (3) |
| C9—O3—C8—C7 | 174.8 (3) | O9—C12—C13—O4 | -171.9 (2) |
| O1—C7—C8—O2 | -176.0 (4) | C11—C12—C13—O4 | -55.8 (3) |

| | | | |
|----------------|------------|----------------|------------|
| O1—C7—C8—O3 | 5.0 (5) | C10—O5—C14—O6 | -8.1 (5) |
| C13—O4—C9—O3 | 176.0 (2) | C10—O5—C14—C15 | 169.6 (3) |
| C13—O4—C9—C10 | -66.9 (3) | C11—O7—C16—O8 | 12.6 (5) |
| C8—O3—C9—O4 | -87.9 (3) | C11—O7—C16—C17 | -166.3 (3) |
| C8—O3—C9—C10 | 155.2 (3) | C12—O9—C18—O10 | 6.7 (4) |
| C14—O5—C10—C11 | -112.4 (3) | C12—O9—C18—C19 | -172.9 (3) |
| C14—O5—C10—C9 | 131.3 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C3—H3 \cdots O6 ⁱ | 0.93 | 2.41 | 3.322 (6) | 168 |
| C9—H9 \cdots O10 ⁱ | 0.98 | 2.54 | 3.381 (4) | 144 |
| C11—H11 \cdots O10 ⁱ | 0.98 | 2.44 | 3.296 (4) | 146 |

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

Fig. 1

