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The *O*,*O*′-diacetyl (*R*,*R*)-hydrogentartrate ester of (*R*)-pantolactone monohydrate[1]

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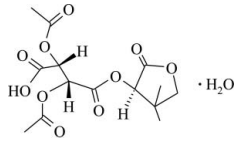
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 7.7.

In the synthesis of (1*R*,2*R*)-1-carboxy-2-[(3*R*)-4,4-dimethyl-2-oxotetrahydrofuran-3-yloxycarbonyl]ethane-1,2-diyl diacetate from diacetyltartaric acid anhydride and pantolactone, two enantiomeric pairs were obtained and the structure of the *R*,*R*,*R* enantiomer is presented here. The compound crystallizes as a monohydrate, $C_{14}H_{18}O_{10}\cdot H_2O$. The main molecule consists of a hydrogentartrate fragment in which the carboxyl group and the lactone ester group are in an *anti* conformation. In the crystal structure, molecules are linked *via* O—H···O hydrogen bonds, involving water molecules, to form a layered structure.

Related literature

The corresponding *R*,*R*,*S* diastereoisomer forms anhydrous crystals (Zachara *et al.*, 2007). There are only two other structurally characterized (*R*,*R*)-hydrogentartrate esters to date (Kivikoski *et al.*, 1993; Mravik *et al.*, 1996). For related literature, see: Allen *et al.* (1998).



Experimental

Crystal data

$C_{14}H_{18}O_{10}\cdot H_2O$
M_r = 364.30
Orthorhombic, $P2_12_12_1$
a = 8.2882 (15) Å
b = 12.905 (3) Å
c = 16.544 (4) Å
V = 1769.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
0.55 × 0.50 × 0.40 mm

Data collection

Siemens *P*3 diffractometer
Absorption correction: none
4027 measured reflections
1804 independent reflections
1602 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
2 standard reflections
every 70 reflections
intensity decay: 4.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.06
1804 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O11	0.89 (3)	1.85 (3)	2.706 (3)	163 (4)
O11—H11A···O5^{i}	0.82	2.03	2.845 (5)	171
O11—H11B···O10^{ii}	0.82	2.28	3.101 (3)	177
C5—H5···O2^{iii}	0.98	2.59	3.224 (3)	122
C10—H10B···O4^{iv}	0.96	2.59	3.522 (4)	163
C14—H14A···O10^{v}	0.96	2.52	3.436 (3)	160
C14—H14C···O4^{vi}	0.96	2.59	3.445 (3)	148

Symmetry codes: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, -y+1, z-\frac{1}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (vi) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *P3/P4-PC Software* (Siemens, 1991); cell refinement: *P3/P4-PC Software*; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2408).

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supplementary materials

Acta Cryst. (2007). E**63**, o3210 [**doi:10.1107/S1600536807027328**]

The *O*,*O*'-diacetyl (*R*,*R*)-hydrogentartrate ester of (*R*)-pantolactone monohydrate

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Comment

The syntheses of diacetyl hydrogentartrate esters of pantolactone gave two enantiomeric pairs. The structural characterization of (*R*,*R*,*S*) diastereoisomer is presented in the preceding paper (Zachara *et al.*, 2007). The title (*R*,*R*,*R*) isomer crystallizes as monohydrate.

Similarly to (*R*,*R*,*S*) the diastereoisomer (Zachara *et al.*, 2007) and (*S*)-tetrahydrofurfuryl-*O*,*O*'-diacetyl-(*R*,*R*)-hydrogentartrate (Mravik *et al.*, 1996) the molecule (Fig. 1) posseses the hydrogentartrate fragment in which the carboxyl group and the ester group are in *anti* conformation with the torsion angle C1—C2—C3—C4 equal to 167.0 (2)°. The *gauche* conformation is observed in (*S*)-timolol-*O*,*O*'-diacetyl-(*R*,*R*)-hydrogentartrate (Kivikoski *et al.*, 1993) where the corresponding torsion angle equals to 37.0 (5)°. To the best of our knowledge there are no other structurally characterized (*R*,*R*)-hydrogentartrate esters. The (*R*)-pantalactone heterocycle in (I) shows the open envelope conformation with the C8 atom displaced by 0.600 (3) Å out of the l.s. plane defined by C5, C6, O6 and C7 atoms.

Strong hydrogen bonds are observed between the O1—H1 donor of a carboxyl group and atom O11 of a water molecule. The water molecule acts as a double donor to $O5^{i}$ and $O10^{ii}$ carbonyl atoms [symmetry codes: (i) 3/2 − *x*, 1 − *y*, 1/2 + *z*; (ii) 1/2 − *x*, 1 − *y*, *z* + 1/2]. As a result of those interactions the molecules of (I) are linked *via* water molecules to form a layer structure on (010) plane (Fig. 2). In the layer, weaker C—H···O intermolecular H-bonds are observed between the chiral C5—H5 group of the heterocycle and $O2^{iii}$ atom [symmetry code: (iii) 3/2 − *x*, 1 − *y*, *z* − 1/2] of a carboxylic group. Additionally, the short, 3.012 (4) Å, intermolecular distance between C6 and $O2^{iii}$ indicates that non-covalent interactions between carbonyl groups cooperate with H-bonds (Allen *et al.*, 1998). Further, the adjacent layers are connected *via* weak intermolecular C—H···O bridges between carbonyl O4 and O10 atoms and the C10 and C14 methyl groups acting as donors to form a 3-D structure.

Experimental

A (1:1 mol/mol) mixture of diacetyl-(*R*,*R*)-tartaric anhydride and (*R*)-pantolactone in toluene was heated up to boiling temperature in a nitrogen atmpsphere under reflux for 18 h. The mixture was then cooled to the room temperature and filtered. The resulting white solid product was recrystallized from saturated 2-propanol solution to give pure title compound with mp. 461–462 K and crystals suitable for X-ray diffraction measurement. $[\alpha]^{20}_{D}$ = +1.0% (*c* 2, ethyl acetate). IR (KBr): ν = 1088, 1208 cm^{-1}, (C—O), ν = 1752 cm^{-1} (C=O), ν = 2824 cm^{-1} (CH_3), 2880, 2946, 2972 cm^{-1} (CH_3), ν = 3440 (OH).

Refinement

Due to the absence of significant anomalous scattering effects, the measured Friedel pairs have been merged. The absolute structure was assigned on the basis of the known configuration of the starting materials. H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 (1.5 for methyl

carbons)×U_{eq}(C). The methyl group (C12) was modelled as idealized disordered rotating groups with refined occupancy factor 0.67 (3) for major conformer. The position of the H atom attached to O1 atom was freely refined with U_{iso}(H) = 1.5×U_{eq}(O). The water molecule was refined as a rigid group with O—H = 0.82 Å and U_{iso}(H) = 1.5×U_{eq}(O).

Figures

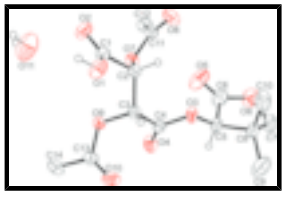

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms not bonded to chiral carbons or O atoms are omitted for clarity

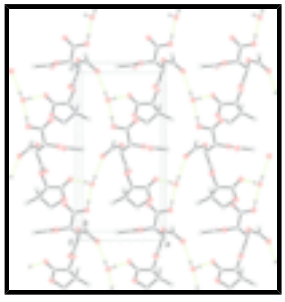

Fig. 2. A *b* axis projection showing layers of molecules linked by O—H···O bonds (dashed lines). Symmetry codes: (i) 3/2 − *x*, 1 − *y*, 1/2 + *z*; (ii) 1/2 − *x*, 1 − *y*, *z* + 1/2.

(1*R*,2*R*)-1-carboxy-2-[(3*R*)-4,4-dimethyl-2-oxotetrahydrofuran-3- yloxycarbonyl]ethane-1,2-diyl diacetate monohydrate

Crystal data

$C_{14}H_{18}O_{10}·H_2O$	D_x = 1.367 Mg m^{-3}
M_r = 364.30	Melting point: 188.0 K
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 32 reflections
a = 8.2882 (15) Å	θ = 14–33º
b = 12.905 (3) Å	μ = 0.12 mm^{-1}
c = 16.544 (4) Å	*T* = 293 (2) K
V = 1769.5 (7) Å^3	Prism, white
Z = 4	0.55 × 0.50 × 0.40 mm
F_{000} = 768	

Data collection

Siemens P3 diffractometer	R_{int} = 0.020
Radiation source: fine-focus sealed tube	θ_{max} = 25.1º
Monochromator: graphite	θ_{min} = 2.5º
T = 293(2) K	*h* = −9→9
profile data from ω–2θ scans	*k* = −15→15
Absorption correction: none	*l* = −19→19

4027 measured reflections
1804 independent reflections
1602 reflections with $I > 2\sigma(I)$
2 standard reflections
every 70 reflections
intensity decay: 4.6%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.085$
$S = 1.06$
1804 reflections
233 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.1825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}
Extinction correction: none

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 $I > 2\sigma(I)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.4135 (2)	0.40210 (19)	0.64862 (11)	0.0635 (5)	
H1	0.392 (5)	0.401 (3)	0.701 (2)	0.095*	
O2	0.6718 (2)	0.41764 (18)	0.68431 (11)	0.0653 (6)	
O3	0.5667 (2)	0.41482 (12)	0.37263 (9)	0.0460 (4)	
O4	0.5964 (3)	0.58696 (13)	0.38505 (10)	0.0582 (5)	
O5	0.9050 (3)	0.4119 (3)	0.33816 (14)	0.0983 (9)	
O6	0.8368 (3)	0.3414 (2)	0.22103 (13)	0.0846 (7)	
O7	0.76978 (18)	0.44152 (11)	0.53189 (9)	0.0399 (4)	
O8	0.8423 (2)	0.27519 (13)	0.52033 (13)	0.0653 (6)	
O9	0.50341 (18)	0.58436 (12)	0.53907 (9)	0.0404 (4)	
O10	0.2776 (2)	0.62970 (15)	0.47394 (13)	0.0634 (5)	
O11	0.3488 (4)	0.4401 (3)	0.80616 (14)	0.1023 (9)	
H11A	0.4256	0.4772	0.8179	0.153*	

H11B	0.3130	0.4199	0.8496	0.153*	
C1	0.5697 (3)	0.41106 (19)	0.63381 (14)	0.0446 (5)	
C2	0.6042 (3)	0.41135 (17)	0.54372 (13)	0.0364 (5)	
H2	0.5877	0.3415	0.5220	0.044*	
C3	0.4996 (3)	0.48740 (17)	0.49778 (12)	0.0364 (5)	
H3	0.3884	0.4616	0.4960	0.044*	
C4	0.5606 (3)	0.50471 (17)	0.41201 (12)	0.0384 (5)	
C5	0.6269 (3)	0.4182 (2)	0.29114 (13)	0.0476 (6)	
H5	0.6096	0.4874	0.2683	0.057*	
C6	0.8043 (4)	0.3918 (3)	0.28876 (17)	0.0673 (8)	
C7	0.6883 (4)	0.3302 (3)	0.17520 (16)	0.0718 (9)	
H7A	0.6856	0.2638	0.1478	0.086*	
H7B	0.6790	0.3848	0.1352	0.086*	
C8	0.5516 (4)	0.3378 (2)	0.23704 (14)	0.0548 (7)	
C9	0.3956 (6)	0.3711 (4)	0.1981 (2)	0.1072 (15)	
H9A	0.3100	0.3677	0.2371	0.161*	
H9B	0.3714	0.3258	0.1536	0.161*	
H9C	0.4059	0.4409	0.1787	0.161*	
C10	0.5331 (5)	0.2352 (2)	0.28152 (18)	0.0862 (11)	
H10A	0.6341	0.2166	0.3059	0.129*	
H10B	0.5013	0.1823	0.2440	0.129*	
H10C	0.4523	0.2421	0.3227	0.129*	
C11	0.8788 (3)	0.36404 (18)	0.52200 (14)	0.0419 (5)	
C12	1.0441 (3)	0.4065 (2)	0.51447 (17)	0.0555 (7)	
H12A	1.0946	0.4077	0.5667	0.083*	0.67 (3)
H12B	1.0389	0.4758	0.4934	0.083*	0.67 (3)
H12C	1.1060	0.3638	0.4785	0.083*	0.67 (3)
H12D	1.0650	0.4238	0.4590	0.083*	0.33 (3)
H12E	1.1208	0.3557	0.5323	0.083*	0.33 (3)
H12F	1.0537	0.4677	0.5472	0.083*	0.33 (3)
C13	0.3856 (3)	0.65216 (17)	0.51929 (14)	0.0427 (5)	
C14	0.4109 (3)	0.75371 (19)	0.55937 (18)	0.0589 (7)	
H14A	0.5034	0.7873	0.5363	0.088*	
H14B	0.4282	0.7432	0.6162	0.088*	
H14C	0.3173	0.7964	0.5516	0.088*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0497 (11)	0.0973 (15)	0.0434 (9)	−0.0147 (11)	0.0049 (9)	0.0030 (10)
O2	0.0589 (12)	0.0909 (15)	0.0461 (9)	0.0041 (12)	−0.0129 (9)	0.0006 (10)
O3	0.0657 (11)	0.0397 (8)	0.0327 (7)	−0.0110 (9)	0.0054 (8)	−0.0023 (6)
O4	0.0827 (14)	0.0424 (9)	0.0493 (9)	−0.0128 (10)	0.0104 (10)	−0.0008 (8)
O5	0.0701 (15)	0.159 (3)	0.0659 (13)	−0.0302 (18)	−0.0102 (13)	0.0110 (16)
O6	0.0761 (15)	0.1177 (19)	0.0598 (12)	0.0158 (15)	0.0146 (12)	−0.0072 (13)
O7	0.0303 (8)	0.0379 (8)	0.0514 (9)	−0.0011 (6)	−0.0028 (7)	−0.0017 (7)
O8	0.0502 (11)	0.0432 (11)	0.1025 (16)	0.0062 (9)	−0.0019 (11)	−0.0060 (10)
O9	0.0376 (8)	0.0413 (8)	0.0424 (7)	0.0072 (7)	−0.0048 (7)	−0.0069 (7)

O10	0.0451 (10)	0.0586 (11)	0.0865 (13)	0.0046 (9)	−0.0213 (11)	0.0036 (10)
O11	0.117 (2)	0.127 (2)	0.0637 (12)	−0.0471 (19)	0.0178 (15)	0.0035 (15)
C1	0.0461 (14)	0.0449 (12)	0.0428 (12)	0.0013 (12)	−0.0038 (11)	0.0018 (11)
C2	0.0310 (11)	0.0367 (11)	0.0417 (11)	−0.0029 (10)	−0.0015 (9)	−0.0021 (9)
C3	0.0309 (10)	0.0401 (11)	0.0383 (10)	−0.0035 (9)	−0.0018 (9)	−0.0042 (9)
C4	0.0355 (11)	0.0399 (11)	0.0398 (11)	−0.0046 (10)	−0.0034 (10)	−0.0025 (10)
C5	0.0653 (16)	0.0445 (12)	0.0329 (10)	−0.0063 (13)	0.0026 (11)	−0.0021 (10)
C6	0.0679 (19)	0.086 (2)	0.0478 (14)	−0.0114 (17)	0.0068 (16)	0.0052 (15)
C7	0.108 (3)	0.0647 (17)	0.0428 (13)	−0.006 (2)	0.0108 (17)	−0.0094 (13)
C8	0.0709 (18)	0.0555 (15)	0.0378 (12)	−0.0095 (15)	−0.0063 (12)	−0.0072 (11)
C9	0.091 (3)	0.153 (4)	0.077 (2)	0.004 (3)	−0.029 (2)	−0.021 (3)
C10	0.137 (3)	0.0586 (17)	0.0628 (16)	−0.031 (2)	0.010 (2)	−0.0091 (14)
C11	0.0377 (12)	0.0428 (13)	0.0453 (12)	0.0049 (10)	−0.0046 (11)	−0.0021 (10)
C12	0.0350 (12)	0.0644 (16)	0.0670 (16)	0.0032 (12)	−0.0037 (12)	−0.0018 (14)
C13	0.0333 (11)	0.0451 (12)	0.0496 (12)	0.0016 (10)	0.0043 (11)	0.0047 (10)
C14	0.0467 (14)	0.0466 (13)	0.0835 (18)	0.0071 (12)	0.0075 (14)	−0.0053 (13)

Geometric parameters (Å, °)

O1—C1	1.323 (3)	C5—H5	0.9800
O1—H1	0.88 (4)	C7—C8	1.530 (4)
O2—C1	1.192 (3)	C7—H7A	0.9700
O3—C4	1.332 (3)	C7—H7B	0.9700
O3—C5	1.438 (3)	C8—C9	1.507 (5)
O4—C4	1.189 (3)	C8—C10	1.522 (4)
O5—C6	1.197 (4)	C9—H9A	0.9600
O6—C6	1.323 (4)	C9—H9B	0.9600
O6—C7	1.453 (4)	C9—H9C	0.9600
O7—C11	1.357 (3)	C10—H10A	0.9600
O7—C2	1.440 (3)	C10—H10B	0.9600
O8—C11	1.186 (3)	C10—H10C	0.9600
O9—C13	1.351 (3)	C11—C12	1.481 (4)
O9—C3	1.426 (3)	C12—H12A	0.9600
O10—C13	1.204 (3)	C12—H12B	0.9600
O11—H11A	0.8200	C12—H12C	0.9600
O11—H11B	0.8200	C12—H12D	0.9600
C1—C2	1.518 (3)	C12—H12E	0.9600
C2—C3	1.514 (3)	C12—H12F	0.9600
C2—H2	0.9800	C13—C14	1.484 (3)
C3—C4	1.523 (3)	C14—H14A	0.9600
C3—H3	0.9800	C14—H14B	0.9600
C5—C8	1.505 (3)	C14—H14C	0.9600
C5—C6	1.510 (4)		
C1—O1—H1	112 (3)	C8—C9—H9A	109.5
C4—O3—C5	116.45 (18)	C8—C9—H9B	109.5
C6—O6—C7	108.5 (2)	H9A—C9—H9B	109.5
C11—O7—C2	116.83 (17)	C8—C9—H9C	109.5
C13—O9—C3	115.85 (17)	H9A—C9—H9C	109.5
H11A—O11—H11B	105.0	H9B—C9—H9C	109.5

O2—C1—O1	124.8 (2)	C8—C10—H10A	109.5
O2—C1—C2	123.7 (2)	C8—C10—H10B	109.5
O1—C1—C2	111.5 (2)	H10A—C10—H10B	109.5
O7—C2—C3	107.61 (17)	C8—C10—H10C	109.5
O7—C2—C1	108.28 (18)	H10A—C10—H10C	109.5
C3—C2—C1	112.75 (19)	H10B—C10—H10C	109.5
O7—C2—H2	109.4	O8—C11—O7	123.1 (2)
C3—C2—H2	109.4	O8—C11—C12	126.3 (2)
C1—C2—H2	109.4	O7—C11—C12	110.7 (2)
O9—C3—C2	108.38 (16)	C11—C12—H12A	109.5
O9—C3—C4	108.06 (18)	C11—C12—H12B	109.5
C2—C3—C4	111.86 (18)	H12A—C12—H12B	109.5
O9—C3—H3	109.5	C11—C12—H12C	109.5
C2—C3—H3	109.5	H12A—C12—H12C	109.5
C4—C3—H3	109.5	H12B—C12—H12C	109.5
O4—C4—O3	125.79 (19)	C11—C12—H12D	109.5
O4—C4—C3	124.3 (2)	H12A—C12—H12D	141.1
O3—C4—C3	109.91 (19)	H12B—C12—H12D	56.3
O3—C5—C8	113.1 (2)	H12C—C12—H12D	56.3
O3—C5—C6	110.8 (2)	C11—C12—H12E	109.5
C8—C5—C6	103.4 (2)	H12A—C12—H12E	56.3
O3—C5—H5	109.8	H12B—C12—H12E	141.1
C8—C5—H5	109.8	H12C—C12—H12E	56.3
C6—C5—H5	109.8	H12D—C12—H12E	109.5
O5—C6—O6	122.9 (3)	C11—C12—H12F	109.5
O5—C6—C5	127.8 (3)	H12A—C12—H12F	56.3
O6—C6—C5	109.4 (3)	H12B—C12—H12F	56.3
O6—C7—C8	105.8 (2)	H12C—C12—H12F	141.1
O6—C7—H7A	110.6	H12D—C12—H12F	109.5
C8—C7—H7A	110.6	H12E—C12—H12F	109.5
O6—C7—H7B	110.6	O10—C13—O9	122.2 (2)
C8—C7—H7B	110.6	O10—C13—C14	126.6 (2)
H7A—C7—H7B	108.7	O9—C13—C14	111.2 (2)
C5—C8—C9	114.4 (3)	C13—C14—H14A	109.5
C5—C8—C10	110.7 (2)	C13—C14—H14B	109.5
C9—C8—C10	111.6 (3)	H14A—C14—H14B	109.5
C5—C8—C7	97.7 (2)	C13—C14—H14C	109.5
C9—C8—C7	111.6 (3)	H14A—C14—H14C	109.5
C10—C8—C7	110.0 (3)	H14B—C14—H14C	109.5
C11—O7—C2—C3	−141.06 (18)	C7—O6—C6—O5	−180.0 (3)
C11—O7—C2—C1	96.8 (2)	C7—O6—C6—C5	−0.4 (3)
O2—C1—C2—O7	−11.5 (3)	O3—C5—C6—O5	−34.4 (5)
O1—C1—C2—O7	168.8 (2)	C8—C5—C6—O5	−155.9 (3)
O2—C1—C2—C3	−130.4 (3)	O3—C5—C6—O6	146.1 (2)
O1—C1—C2—C3	49.9 (3)	C8—C5—C6—O6	24.6 (3)
C13—O9—C3—C2	−163.21 (17)	C6—O6—C7—C8	−23.7 (3)
C13—O9—C3—C4	75.4 (2)	O3—C5—C8—C9	86.5 (3)
O7—C2—C3—O9	−71.4 (2)	C6—C5—C8—C9	−153.5 (3)
C1—C2—C3—O9	48.0 (2)	O3—C5—C8—C10	−40.6 (4)

O7—C2—C3—C4	47.7 (2)	C6—C5—C8—C10	79.4 (3)
C1—C2—C3—C4	167.00 (19)	O3—C5—C8—C7	−155.5 (2)
C5—O3—C4—O4	2.1 (4)	C6—C5—C8—C7	−35.5 (3)
C5—O3—C4—C3	−178.22 (19)	O6—C7—C8—C5	36.5 (3)
O9—C3—C4—O4	−3.5 (3)	O6—C7—C8—C9	156.7 (3)
C2—C3—C4—O4	−122.7 (3)	O6—C7—C8—C10	−78.9 (3)
O9—C3—C4—O3	176.85 (18)	C2—O7—C11—O8	2.4 (3)
C2—C3—C4—O3	57.6 (2)	C2—O7—C11—C12	−177.3 (2)
C4—O3—C5—C8	−148.3 (2)	C3—O9—C13—O10	5.0 (3)
C4—O3—C5—C6	96.0 (3)	C3—O9—C13—C14	−174.20 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O11	0.89 (3)	1.85 (3)	2.706 (3)	163 (4)
O11—H11A···O5^{i}	0.82	2.03	2.845 (5)	171
O11—H11B···O10^{ii}	0.82	2.28	3.101 (3)	177
C2—H2···O8	0.98	2.28	2.671 (3)	103
C5—H5···O4	0.98	2.32	2.687 (3)	101
C5—H5···O2^{iii}	0.98	2.59	3.224 (3)	122
C10—H10B···O4^{iv}	0.96	2.59	3.522 (4)	163
C14—H14A···O10^{v}	0.96	2.52	3.436 (3)	160
C14—H14C···O4^{vi}	0.96	2.59	3.445 (3)	148

Symmetry codes: (i) −*x*+3/2, −*y*+1, *z*+1/2; (ii) −*x*+1/2, −*y*+1, *z*+1/2; (iii) −*x*+3/2, −*y*+1, *z*−1/2; (iv) −*x*+1, *y*−1/2, −*z*+1/2; (v) *x*+1/2, −*y*+3/2, −*z*+1; (vi) *x*−1/2, −*y*+3/2, −*z*+1.

Fig. 1

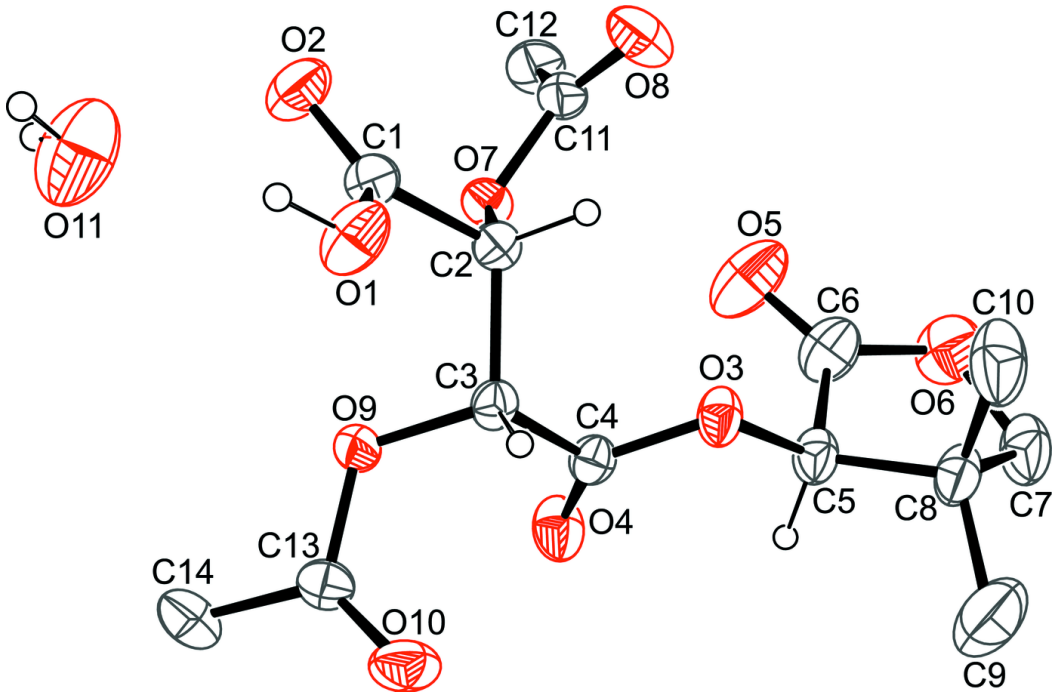
O2
C12
O8
C1
O7
C11
O11
O1
C2
O5
C6
C10
C3
O3
O9
C4
O6
C5
C8
C7
O4
C13
C14
O10
C9

Fig. 2