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

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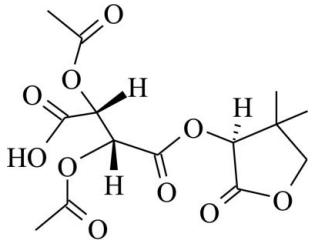
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.035; wR factor = 0.090; data-to-parameter ratio = 8.0.

The synthesis of the title compound, (1*R*,2*R*)-1-carboxy-2-[(3*S*)-4,4-dimethyl-2-oxotetrahydrofuran-3-yloxycarbonyl]-ethane-1,2-diyl diacetate, $C_{14}H_{18}O_{10}$, from diacetyltartaric acid anhydride and pantolactone gave two enantiomeric pairs and the crystal structure of the *R*,*R*,*S* enantiomer is presented here. The molecule consists of a hydrogen tartrate fragment in which the carboxyl group and the lactone ester group are in an *anti* conformation. In the crystal structure, molecules are linked into *C*(10) chains by an intermolecular O—H···O hydrogen bond and further by C—H···O interactions to form a layer structure with the second-level graph-set descriptor $R_2^2(8)[R_4^4(26)]$.

Related literature

The corresponding (*R*,*R*,*R*) diastereoisomer crystallizes as the monohydrate (Zachara *et al.*, 2007; see the following paper). The molecular geometry is close to that of the (*R*,*R*,*S*) isomer but the molecules are O—H···O linked *via* water molecules to form a layer structure. 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: Bernstein *et al.* (1995); Beutel & Tishler (1946); Etter (1990); Ghosh *et al.* (2001).



[1] Tartaric acid and its *O*-acyl derivatives. Part 3.

Experimental

Crystal data

$C_{14}H_{18}O_{10}$
M_r = 346.28
Orthorhombic, $P2_12_12_1$
a = 7.946 (2) Å
b = 13.067 (3) Å
c = 16.796 (3) Å
V = 1743.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.60 × 0.54 × 0.50 mm

Data collection

Siemens *P*3 diffractometer
Absorption correction: none
3184 measured reflections
1784 independent reflections
1420 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
2 standard reflections
every 70 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.03
1784 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.95 (5)	1.89 (4)	2.725 (3)	146 (4)
C5—H5···O2^{ii}	0.98	2.53	3.396 (4)	147
C12—H12*A*···O8^{iii}	0.96	2.54	3.476 (4)	166
C14—H14*A*···O4^{iv}	0.96	2.47	3.381 (4)	159
C14—H14*B*···O10^{v}	0.96	2.29	3.231 (5)	165

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) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iv) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (v) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

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, 1997); 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: LH2407).

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supplementary materials

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

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Comment

Four diastereomeric diacetyl hydrogentartrate esters of pantolactone representing two enantiomeric pairs have been obtained from diacetyltartaric acid anhydride and pantolactone. They may be exploited in the resolution of racemic pantolactone *via* diastereomeric ester formation (Beutel & Tishler, 1946). The esters posses unique structure, which combines two natural units: of tartaric acid and of pantolactone, what may be interesting in the synthesis of bioactive molecules from the chiral pool (Ghosh *et al.*, 2001). Although application of such compounds is unknown it seems that they may be used, *e.g.* in the polymer chemistry. The structure of the (*R*,*R*,*S*) enantiomer (I) is presented here.

The molecule of (I) (Fig. 1) consists of the hydrogentartrate fragment in which the carboxyl group and the lactone ester group are in *anti* conformation with the torsion angle C1—C2—C3—C4 equal to 172.8 (2)°. The same conformation is observed in (*S*)-tetrahydrofurfuryl -*O*,*O*'-diacetyl-(*R*,*R*)-hydrogentartrate (Mravik *et al.*, 1996) were the relevant torsion angle is 168.1 (5)°. Whereas in the second structurally characterized derivative, (*S*)-timolol-*O*,*O*'-diacetyl-(*R*,*R*)-hydrogentartrate (Kivikoski *et al.*, 1993) the *gauche* conformation is observed and the corresponding torsion angle equals to 37.0 (5)°. The ester fragment consists of (*S*)-pantalactone heterocycle showing the open envelope conformation with the C8 atom displaced by 0.626 (3) Å out of the l.s. plane defined by C5, C6, O6 and C7 atoms.

The strong intermolecular hydrogen bonds are observed between the O1—H1 donor of carboxyl group and the carbonyl $O5^{i}$ atom of pantalactone [symmetry code: (i) $3/2 - x$, $1 - y$, $1/2 + z$]. The molecules are arranged into infinite one-dimensional chain running along *c* axis with the assigned graph descriptor *C*(10) (Etter, 1990). The weak C—H···O intermolecular interactions are observed between C5—H5 chiral atom and the $O2^{ii}$ carbonyl oxygen of the carboxylic group [symmetry code: (ii) $1/2 - x$, $1 - y$, $-1/2 + z$]. This motif can be described as *C*(8) and it is also running along [001] direction. Together with the O1—H1···O5 motif they form a layer structure on (100) plane (Fig. 2) with the second level graph extended descriptor $R^2_2(8)[R^4_4(26)]$ (Bernstein *et al.*, 1995). The remaining carbonyl oxygen O4, O8 and O10 atoms act as the acceptors to the methyl groups only and weakly join the adjacent layers into three-dimensional structure.

Experimental

A (1:1 mol/mol) mixture of diacetyl-(*R*,*R*)-tartaric anhydride and (*S*)-pantolactone in toluene was heated up to boiling temperature in a nitrogen atmosphere under reflux for 18 h. The mixture was then cooled to the room temperature and filtered. The resulting white solid product was recrystallized from 2-propanol to give pure compound (I) with mp 183.8–185.5°C. $[\alpha]^{25}_{D}$ = +9.5% (*c* 2, ethyl acetate). IR (KBr): ν = 1088, 1212 cm^{-1}, (C—O), ν = 1760 cm^{-1} (C=O), ν = 2946 cm^{-1} (CH_3). Crystals suitable for single-crystal X-ray diffraction measurement were recrystallized from saturated ethyl acetate.

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 groups) × U_{eq}(C). Two methyl groups (C12, C14) were modelled as idealized disordered rotating groups with refined occupancy factors, 0.69 (5) and 0.69 (4) for major conformers, respectively. The position of the H atom attached to O atom was freely refined with U_{iso}(H) = 1.5×U_{eq}(O).

Figures

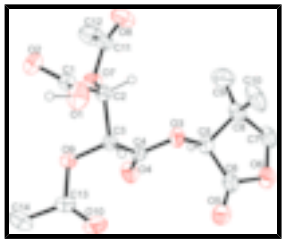

Fig. 1. *ORTEP* plot of the molecural structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms not bonded to chiral carbons or the O atom are omitted for clarity.

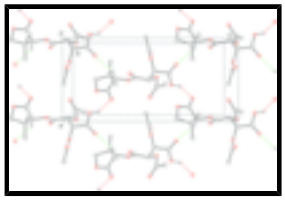

Fig. 2. An *a* axis projection showing layers of molecules linked by O—H···O (dashed lines) and C—H···O (dotted lines) H-bonds. Symmetry codes: (i) 3/2 − *x*, 1 − *y*, 1/2 + *z*; (ii) 1/2 − *x*, 1 − *y*, *z* − 1/2.

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

Crystal data

$C_{14}H_{18}O_{10}$	D_x = 1.319 Mg m^{-3}
M_r = 346.28	Melting point: 183.8 K
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 28 reflections
a = 7.946 (2) Å	θ = 15–24º
b = 13.067 (3) Å	μ = 0.11 mm^{-1}
c = 16.796 (3) Å	*T* = 293 (2) K
V = 1743.9 (7) Å^3	Prism, white
Z = 4	0.60 × 0.54 × 0.50 mm
F_{000} = 728	

Data collection

Siemens P3 diffractometer	R_{int} = 0.017
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º

Monochromator: graphite	θ_{min} = 2.4º
T = 293(2) K	h = −5→9
profile data from ω/2θ scans	k = −15→15
Absorption correction: none	l = −20→20
3184 measured reflections	2 standard reflections
1784 independent reflections	every 70 reflections
1420 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.1521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.090	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.11 e Å^{-3}
1784 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
223 parameters	Extinction coefficient: 0.0133 (17)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 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)
O2	0.3053 (3)	0.56229 (18)	0.66759 (13)	0.0712 (7)	
O1	0.5720 (3)	0.6007 (2)	0.63717 (13)	0.0694 (7)	
H1	0.591 (5)	0.586 (3)	0.692 (3)	0.104*	
O3	0.5095 (3)	0.55520 (13)	0.37040 (10)	0.0507 (5)	
O4	0.4240 (3)	0.39211 (15)	0.38236 (12)	0.0646 (7)	
O5	0.7548 (4)	0.4649 (2)	0.26751 (13)	0.0772 (7)	
O6	0.6492 (4)	0.56521 (18)	0.17255 (12)	0.0783 (8)	
O7	0.2241 (2)	0.54728 (13)	0.51039 (12)	0.0485 (5)	

O8	0.1568 (3)	0.71252 (15)	0.50821 (17)	0.0749 (7)	
O9	0.4740 (2)	0.39796 (13)	0.54300 (10)	0.0434 (5)	
O10	0.7233 (3)	0.34030 (19)	0.50182 (17)	0.0838 (8)	
C1	0.4147 (4)	0.5780 (2)	0.62065 (17)	0.0469 (7)	
C2	0.3928 (3)	0.5724 (2)	0.53104 (15)	0.0370 (6)	
H2	0.4236	0.6382	0.5070	0.044*	
C3	0.5048 (3)	0.48876 (18)	0.49835 (14)	0.0386 (6)	
H3	0.6226	0.5089	0.5057	0.046*	
C4	0.4737 (3)	0.4697 (2)	0.41073 (15)	0.0420 (7)	
C5	0.4859 (4)	0.5518 (2)	0.28598 (15)	0.0493 (7)	
H5	0.3951	0.5040	0.2728	0.059*	
C6	0.6447 (5)	0.5206 (2)	0.24441 (18)	0.0603 (9)	
C7	0.5031 (5)	0.6327 (2)	0.16463 (18)	0.0686 (10)	
H7A	0.4133	0.5989	0.1357	0.082*	
H7B	0.5332	0.6952	0.1368	0.082*	
C8	0.4488 (5)	0.6560 (2)	0.25043 (18)	0.0600 (9)	
C9	0.2646 (6)	0.6854 (3)	0.2554 (2)	0.0922 (13)	
H9A	0.2487	0.7517	0.2321	0.138*	
H9B	0.2303	0.6871	0.3102	0.138*	
H9C	0.1982	0.6360	0.2272	0.138*	
C10	0.5602 (7)	0.7381 (2)	0.2871 (2)	0.0944 (15)	
H10A	0.5345	0.8031	0.2634	0.142*	
H10B	0.6762	0.7216	0.2775	0.142*	
H10C	0.5402	0.7413	0.3434	0.142*	
C11	0.1167 (4)	0.6263 (2)	0.50047 (19)	0.0500 (7)	
C12	−0.0534 (4)	0.5889 (3)	0.4791 (3)	0.0923 (14)	
H12A	−0.1281	0.6460	0.4731	0.138*	0.69 (5)
H12B	−0.0944	0.5447	0.5205	0.138*	0.69 (5)
H12C	−0.0477	0.5516	0.4300	0.138*	0.69 (5)
H12D	−0.0520	0.5155	0.4759	0.138*	0.31 (5)
H12E	−0.0858	0.6168	0.4285	0.138*	0.31 (5)
H12F	−0.1325	0.6100	0.5191	0.138*	0.31 (5)
C13	0.5991 (4)	0.3268 (2)	0.53940 (19)	0.0537 (8)	
C14	0.5592 (5)	0.2359 (2)	0.5890 (2)	0.0724 (10)	
H14A	0.6507	0.1881	0.5865	0.109*	0.69 (4)
H14B	0.4585	0.2040	0.5695	0.109*	0.69 (4)
H14C	0.5424	0.2569	0.6432	0.109*	0.69 (4)
H14D	0.4504	0.2446	0.6130	0.109*	0.31 (4)
H14E	0.6426	0.2287	0.6300	0.109*	0.31 (4)
H14F	0.5587	0.1758	0.5562	0.109*	0.31 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0817 (17)	0.0784 (15)	0.0533 (12)	0.0062 (14)	0.0256 (13)	0.0025 (12)
O1	0.0672 (16)	0.0949 (17)	0.0462 (11)	−0.0177 (14)	−0.0139 (11)	−0.0016 (12)
O3	0.0744 (14)	0.0423 (10)	0.0352 (9)	−0.0148 (12)	0.0038 (10)	−0.0028 (8)
O4	0.0955 (18)	0.0464 (11)	0.0520 (11)	−0.0215 (13)	−0.0023 (12)	−0.0031 (10)

O5	0.0898 (18)	0.0856 (16)	0.0564 (13)	0.0162 (18)	0.0171 (13)	0.0121 (13)
O6	0.104 (2)	0.0833 (16)	0.0478 (12)	0.0046 (16)	0.0161 (13)	0.0164 (12)
O7	0.0340 (10)	0.0401 (10)	0.0713 (13)	0.0010 (9)	−0.0013 (10)	−0.0011 (9)
O8	0.0598 (14)	0.0443 (12)	0.121 (2)	0.0062 (12)	−0.0055 (15)	0.0025 (13)
O9	0.0411 (11)	0.0433 (10)	0.0459 (10)	0.0077 (9)	0.0020 (9)	0.0041 (8)
O10	0.0643 (16)	0.0783 (16)	0.109 (2)	0.0252 (14)	0.0259 (16)	0.0027 (15)
C1	0.056 (2)	0.0394 (14)	0.0456 (15)	0.0036 (14)	0.0067 (16)	0.0000 (13)
C2	0.0304 (14)	0.0380 (13)	0.0424 (13)	−0.0031 (12)	0.0012 (12)	−0.0006 (11)
C3	0.0341 (14)	0.0414 (13)	0.0404 (13)	−0.0030 (12)	0.0020 (13)	0.0023 (11)
C4	0.0430 (17)	0.0410 (14)	0.0420 (14)	−0.0033 (14)	0.0061 (13)	−0.0014 (12)
C5	0.069 (2)	0.0420 (14)	0.0366 (14)	−0.0108 (17)	−0.0009 (15)	−0.0061 (11)
C6	0.085 (3)	0.0526 (17)	0.0435 (17)	−0.0069 (19)	0.0091 (19)	0.0018 (15)
C7	0.102 (3)	0.0585 (18)	0.0455 (16)	−0.009 (2)	−0.008 (2)	0.0058 (15)
C8	0.090 (3)	0.0427 (15)	0.0477 (16)	−0.0034 (18)	−0.0056 (19)	−0.0014 (13)
C9	0.126 (4)	0.077 (2)	0.074 (3)	0.027 (3)	0.000 (3)	−0.004 (2)
C10	0.167 (4)	0.0466 (18)	0.069 (2)	−0.035 (3)	−0.021 (3)	0.0054 (16)
C11	0.0372 (16)	0.0461 (17)	0.0669 (18)	0.0045 (14)	0.0022 (15)	0.0040 (15)
C12	0.0417 (19)	0.073 (2)	0.162 (4)	0.0073 (18)	−0.015 (2)	0.001 (3)
C13	0.0474 (19)	0.0510 (17)	0.0627 (18)	0.0127 (16)	−0.0046 (17)	−0.0060 (15)
C14	0.067 (2)	0.0540 (18)	0.096 (3)	0.0116 (18)	−0.021 (2)	0.0115 (18)

Geometric parameters (Å, °)

O2—C1	1.191 (3)	C7—H7A	0.9700
O1—C1	1.314 (4)	C7—H7B	0.9700
O1—H1	0.95 (4)	C8—C9	1.516 (6)
O3—C4	1.337 (3)	C8—C10	1.521 (5)
O3—C5	1.431 (3)	C9—H9A	0.9600
O4—C4	1.188 (3)	C9—H9B	0.9600
O5—C6	1.202 (4)	C9—H9C	0.9600
O6—C6	1.341 (4)	C10—H10A	0.9600
O6—C7	1.464 (5)	C10—H10B	0.9600
O7—C11	1.350 (3)	C10—H10C	0.9600
O7—C2	1.424 (3)	C11—C12	1.481 (5)
O8—C11	1.178 (3)	C12—H12A	0.9600
O9—C13	1.363 (3)	C12—H12B	0.9600
O9—C3	1.425 (3)	C12—H12C	0.9600
O10—C13	1.184 (4)	C12—H12D	0.9600
C1—C2	1.517 (4)	C12—H12E	0.9600
C2—C3	1.513 (4)	C12—H12F	0.9600
C2—H2	0.9800	C13—C14	1.486 (4)
C3—C4	1.513 (3)	C14—H14A	0.9600
C3—H3	0.9800	C14—H14B	0.9600
C5—C6	1.499 (5)	C14—H14C	0.9600
C5—C8	1.515 (4)	C14—H14D	0.9600
C5—H5	0.9800	C14—H14E	0.9600
C7—C8	1.535 (5)	C14—H14F	0.9600
C1—O1—H1	108 (3)	H10A—C10—H10B	109.5
C4—O3—C5	116.7 (2)	C8—C10—H10C	109.5

C6—O6—C7	108.8 (3)	H10A—C10—H10C	109.5
C11—O7—C2	116.7 (2)	H10B—C10—H10C	109.5
C13—O9—C3	114.8 (2)	O8—C11—O7	123.1 (3)
O2—C1—O1	126.4 (3)	O8—C11—C12	126.1 (3)
O2—C1—C2	124.4 (3)	O7—C11—C12	110.7 (3)
O1—C1—C2	109.2 (3)	C11—C12—H12A	109.5
O7—C2—C3	107.4 (2)	C11—C12—H12B	109.5
O7—C2—C1	111.1 (2)	H12A—C12—H12B	109.5
C3—C2—C1	109.1 (2)	C11—C12—H12C	109.5
O7—C2—H2	109.7	H12A—C12—H12C	109.5
C3—C2—H2	109.7	H12B—C12—H12C	109.5
C1—C2—H2	109.7	C11—C12—H12D	109.5
O9—C3—C2	108.03 (19)	H12A—C12—H12D	141.1
O9—C3—C4	110.3 (2)	H12B—C12—H12D	56.3
C2—C3—C4	112.1 (2)	H12C—C12—H12D	56.3
O9—C3—H3	108.8	C11—C12—H12E	109.5
C2—C3—H3	108.8	H12A—C12—H12E	56.3
C4—C3—H3	108.8	H12B—C12—H12E	141.1
O4—C4—O3	125.5 (2)	H12C—C12—H12E	56.3
O4—C4—C3	125.8 (2)	H12D—C12—H12E	109.5
O3—C4—C3	108.7 (2)	C11—C12—H12F	109.5
O3—C5—C6	111.1 (3)	H12A—C12—H12F	56.3
O3—C5—C8	112.9 (2)	H12B—C12—H12F	56.3
C6—C5—C8	103.0 (3)	H12C—C12—H12F	141.1
O3—C5—H5	109.9	H12D—C12—H12F	109.5
C6—C5—H5	109.9	H12E—C12—H12F	109.5
C8—C5—H5	109.9	O10—C13—O9	122.0 (3)
O5—C6—O6	122.3 (3)	O10—C13—C14	126.6 (3)
O5—C6—C5	128.8 (3)	O9—C13—C14	111.4 (3)
O6—C6—C5	108.8 (3)	C13—C14—H14A	109.5
O6—C7—C8	104.9 (3)	C13—C14—H14B	109.5
O6—C7—H7A	110.8	H14A—C14—H14B	109.5
C8—C7—H7A	110.8	C13—C14—H14C	109.5
O6—C7—H7B	110.8	H14A—C14—H14C	109.5
C8—C7—H7B	110.8	H14B—C14—H14C	109.5
H7A—C7—H7B	108.8	C13—C14—H14D	109.5
C5—C8—C9	113.2 (3)	H14A—C14—H14D	141.1
C5—C8—C10	111.2 (3)	H14B—C14—H14D	56.3
C9—C8—C10	111.1 (3)	H14C—C14—H14D	56.3
C5—C8—C7	97.9 (2)	C13—C14—H14E	109.5
C9—C8—C7	111.9 (3)	H14A—C14—H14E	56.3
C10—C8—C7	110.9 (3)	H14B—C14—H14E	141.1
C8—C9—H9A	109.5	H14C—C14—H14E	56.3
C8—C9—H9B	109.5	H14D—C14—H14E	109.5
H9A—C9—H9B	109.5	C13—C14—H14F	109.5
C8—C9—H9C	109.5	H14A—C14—H14F	56.3
H9A—C9—H9C	109.5	H14B—C14—H14F	56.3
H9B—C9—H9C	109.5	H14C—C14—H14F	141.1
C8—C10—H10A	109.5	H14D—C14—H14F	109.5

C8—C10—H10B	109.5	H14E—C14—H14F	109.5
C11—O7—C2—C3	−151.0 (2)	C7—O6—C6—O5	−176.5 (3)
C11—O7—C2—C1	89.7 (3)	C7—O6—C6—C5	3.9 (3)
O2—C1—C2—O7	0.8 (4)	O3—C5—C6—O5	31.1 (5)
O1—C1—C2—O7	179.6 (2)	C8—C5—C6—O5	152.2 (4)
O2—C1—C2—C3	−117.5 (3)	O3—C5—C6—O6	−149.3 (2)
O1—C1—C2—C3	61.4 (3)	C8—C5—C6—O6	−28.2 (3)
C13—O9—C3—C2	−159.5 (2)	C6—O6—C7—C8	21.8 (3)
C13—O9—C3—C4	77.7 (3)	O3—C5—C8—C9	−84.0 (3)
O7—C2—C3—O9	−69.4 (3)	C6—C5—C8—C9	156.2 (3)
C1—C2—C3—O9	51.1 (3)	O3—C5—C8—C10	42.0 (4)
O7—C2—C3—C4	52.3 (3)	C6—C5—C8—C10	−77.9 (3)
C1—C2—C3—C4	172.8 (2)	O3—C5—C8—C7	158.0 (3)
C5—O3—C4—O4	−0.2 (4)	C6—C5—C8—C7	38.2 (3)
C5—O3—C4—C3	−179.5 (2)	O6—C7—C8—C5	−36.8 (3)
O9—C3—C4—O4	2.9 (4)	O6—C7—C8—C9	−155.8 (3)
C2—C3—C4—O4	−117.5 (3)	O6—C7—C8—C10	79.5 (3)
O9—C3—C4—O3	−177.8 (2)	C2—O7—C11—O8	−0.1 (5)
C2—C3—C4—O3	61.8 (3)	C2—O7—C11—C12	179.9 (3)
C4—O3—C5—C6	−91.8 (3)	C3—O9—C13—O10	−0.1 (4)
C4—O3—C5—C8	153.1 (3)	C3—O9—C13—C14	178.6 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O5^{i}	0.95 (5)	1.89 (4)	2.725 (3)	146 (4)
C5—H5···O2^{ii}	0.98	2.53	3.396 (4)	147
C12—H12A···O8^{iii}	0.96	2.54	3.476 (4)	166
C14—H14A···O4^{iv}	0.96	2.47	3.381 (4)	159
C14—H14B···O10^{v}	0.96	2.29	3.231 (5)	165

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

Fig. 1

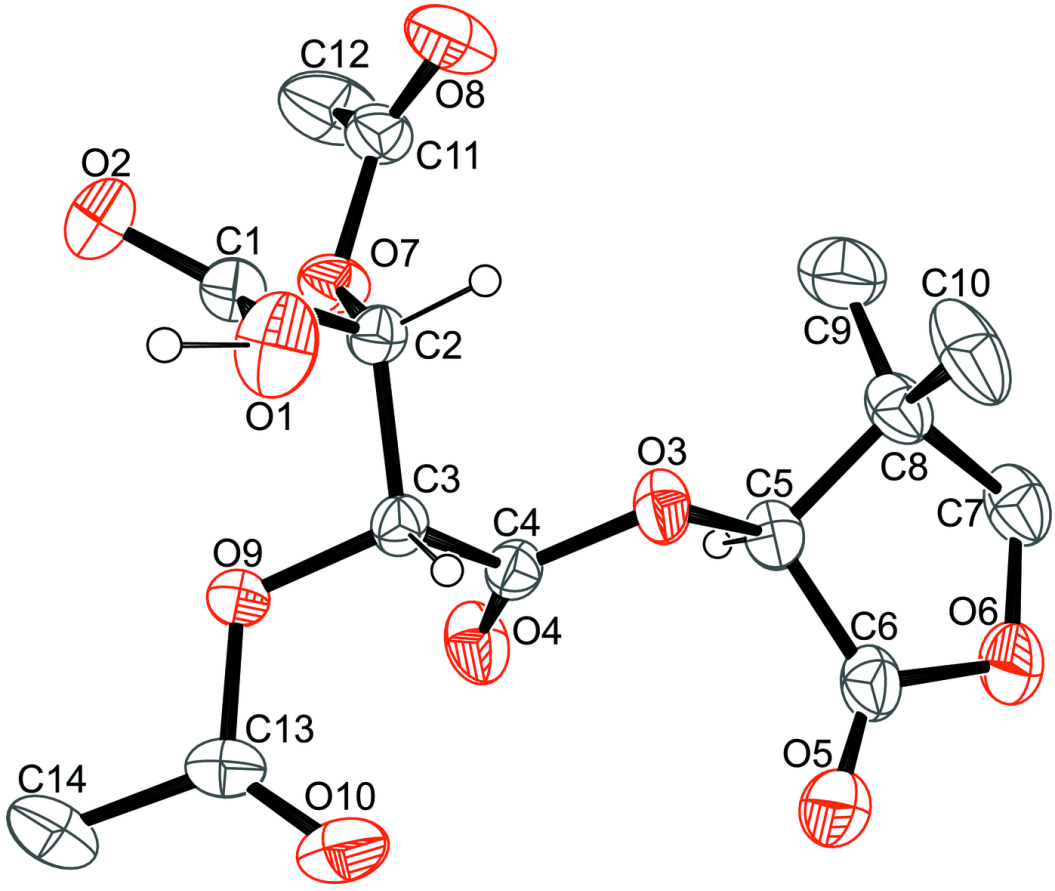
C12
O8
C11
O2
C1
O7
C2
O1
C3
O3
C5
C10
C9
C8
C7
C4
O9
O4
C6
O6
C13
O5
C14
O10

Fig. 2