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2,3-*O*-Isopropylidene-3-*C*-phenyl-erythrofuranose

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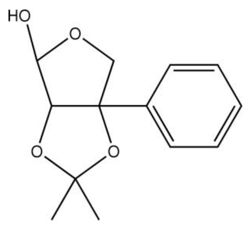
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.157; data-to-parameter ratio = 15.3.

The title compound, $C_{13}H_{16}O$, comprises two fused five-membered rings. Each ring has an envelope conformation, with the ether O atom in the furanose ring, and the CMe_2 atom in the acetonide ring as the flap atoms. In the crystal, centrosymmetrically related molecules associate *via* hydroxy–ether O—H⋯O hydrogen bonds and the resulting dimers are linked into a supramolecular chain with a flattened topology *via* C—H⋯$O_{hydroxy}$ contacts, and aligned in the *a*-axis direction.

Related literature

For the relevance and chemistry of systems related to the title compound, see: Pedersen *et al.* (2009); Robinson *et al.* (2006, 2009); Valente *et al.* (2009). For the reactions of Co(II) complexes with endoperoxides, see: Boyd *et al.* (1980); Sutbeyaz *et al.* (1988); Greatrex *et al.* (2003); Greatrex & Taylor (2005).



Experimental

Crystal data

$C_{13}H_{16}O_4$
M_r = 236.26
Triclinic, $P\bar{1}$
a = 5.716 (2) Å
b = 9.201 (4) Å
c = 11.871 (6) Å
α = 89.76 (3)°
β = 78.72 (2)°
γ = 73.70 (2)°
V = 586.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 K
0.35 × 0.35 × 0.10 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.773, T_{max} = 1.000
14572 measured reflections
2408 independent reflections
2361 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.157
S = 1.16
2408 reflections
157 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2o⋯O1^{i}	0.84	1.93	2.755 (2)	166
C5—H5a⋯O2^{ii}	0.99	2.47	3.296 (3)	140

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x-1, y, z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2687).

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supplementary materials

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2,3-*O*-Isopropylidene-3-*C*-phenylerythrofuranose

T. V. Robinson, D. K. Taylor and **E. R. T. Tiekink**

Comment

The dihydroxyation of monocyclic and bicyclic 1,2-dioxines has provided a new route for the stereoselective synthesis of a diverse range of carbohydrates and related compounds (Pedersen *et al.*, 2009; Robinson *et al.*, 2006; Robinson *et al.*, 2009; Valente *et al.*, 2009). During the course of these studies, the title compound, (I), was obtained by the Co(II)-mediated ring-opening of the precursor 1,2-dioxane, post dihydroxation. The reactions of Co(II) complexes with endoperoxides have been well documented (Boyd *et al.*, 1980; Sutbeyaz *et al.*, 1988; Greatrex *et al.*, 2003; Greatrex & Taylor, 2005).

The molecular structure of (I), Fig. 1, comprises two fused five-membered rings linked at the C3—C4 bond. Each of the five-membered rings adopts an envelope conformation, on atom O1 for the furanose (O1, C2—C5) ring, and on atom C6 for the acetonide (O3, O4, C3, C4, C6) ring. When viewed down the C3—C4 axis, the O1 atom lies above the plane through the four remaining atoms, away from the phenyl substituent and the C6 atom lies below the plane, being orientated in the same direction as the phenyl ring. In the crystal structure centrosymmetrically related pairs of molecules associate *via* O—H···O hydrogen bonds to form an eight-membered $\{\cdots OCOH\}_2$ synthon, Table 1 and Fig. 2. The dimers are linked into a supramolecular chain *via* C—H···O contacts and ten-membered $\{\cdots OH\cdots OCH\}_2$ synthons, Table 1. The resulting chain comprising alternating eight- and ten-membered synthons has a flattened topology, Fig. 2, and is aligned along the *a* axis.

Experimental

For full synthetic procedures and characterization data see Pedersen *et al.* (2009) and Robinson *et al.* (2009). To a stirred solution of $Co(salen)_2$ (17 mg, 0.05 mmol) in THF (5 ml) at ambient temperature was added (*3aR*,*7aS*)-3a-phenyl-tetrahydro-2,2-dimethyl-[1,3]dioxolo[4,5-*d*][1,2]dioxine (501 mg, 2.12 mmol), and the reaction left to stir until complete by TLC (~16 h). All volatiles were removed *in vacuo* giving a crude mixture of regioisomers in a 40:60 ratio. The isomers were fully separated by flash chromatography giving a combined total yield of 496 mg (99%). Compound (I) was isolated as a colourless solid (198 mg), and the pure material was recrystallized from a slowly evaporating 1:1 mixture of dichloromethane/heptane to give colourless prisms, m. pt. 424–425 K. The compound was found to exist solely in its cyclic hemi-acetal form(*s*) both as a solid indicated by IR (absence of carbonyl signal), and in $CDCl_3$ solution which revealed a 90:10 ratio of anomers.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95–1.00 Å) and were included in the refinement in the riding model approximation with U_{iso}(H) set to 1.2–1.5U_{eq}(C). The O–bound H-atom was located in a difference Fourier map and was refined with an O–H restraint of 0.840±0.001 Å, and with U_{iso}(H) = 1.5U_{eq}(O).

Figures

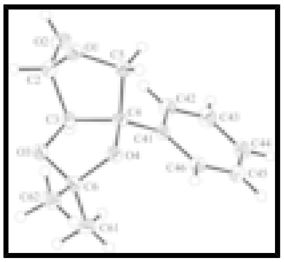

Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 35% probability level.

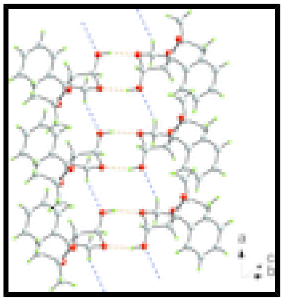

Fig. 2. Supramolecular chain formation along the *a* axis in (I) mediated by O—H···O hydrogen bonds (orange dashed lines) and C—H···O contacts (blue dashed lines).

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Crystal data

$C_{13}H_{16}O_4$
$M_r = 236.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.716$ (2) Å
$b = 9.201$ (4) Å
$c = 11.871$ (6) Å
$\alpha = 89.76$ (3)°
$\beta = 78.72$ (2)°
$\gamma = 73.70$ (2)°
$V = 586.9$ (4) Å^3

$Z = 2$
$F_{000} = 252$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 2428 reflections
$\theta = 3.5$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 173$ K
Prism, pale-yellow
0.35 × 0.35 × 0.10 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 173$ K
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
$T_{min} = 0.773$, $T_{max} = 1.000$
14572 measured reflections

2408 independent reflections
2361 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 26.5$°
$\theta_{min} = 1.8$°
$h = -7\rightarrow7$
$k = -10\rightarrow11$
$l = -14\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.157$	$w = 1/[\sigma^2(F_o^2) + (0.0929P)^2 + 0.1356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.16$	$(\Delta/\sigma)_{max} < 0.001$
2408 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
157 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}
O1	−0.11375 (18)	0.89747 (11)	0.40885 (9)	0.0294 (3)
O2	0.29860 (19)	0.86868 (13)	0.42473 (9)	0.0335 (3)
H2O	0.2683	0.9394	0.4747	0.050*
O3	0.1029 (2)	0.87393 (12)	0.15795 (9)	0.0321 (3)
O4	−0.09610 (17)	0.69527 (11)	0.20274 (8)	0.0270 (3)
C2	0.1242 (3)	0.91139 (17)	0.35425 (12)	0.0281 (3)
H2	0.1102	1.0170	0.3296	0.034*
C3	0.2058 (3)	0.79968 (16)	0.25019 (12)	0.0264 (3)
H3	0.3905	0.7548	0.2291	0.032*
C4	0.0642 (2)	0.67820 (16)	0.28391 (11)	0.0248 (3)
C5	−0.0944 (3)	0.73843 (16)	0.40319 (12)	0.0273 (3)
H5A	−0.2612	0.7226	0.4120	0.033*
H5B	−0.0130	0.6864	0.4645	0.033*
C6	−0.0013 (3)	0.77251 (17)	0.10732 (12)	0.0300 (4)
C41	0.2235 (2)	0.51612 (16)	0.28472 (12)	0.0263 (3)
C42	0.4230 (3)	0.48304 (18)	0.34152 (13)	0.0322 (4)
H42	0.4620	0.5630	0.3772	0.039*

C43	0.5645 (3)	0.33463 (19)	0.34623 (15)	0.0380 (4)
H43	0.7000	0.3135	0.3849	0.046*
C44	0.5092 (3)	0.21693 (18)	0.29478 (14)	0.0378 (4)
H44	0.6059	0.1151	0.2982	0.045*
C45	0.3115 (3)	0.24891 (18)	0.23827 (13)	0.0362 (4)
H45	0.2735	0.1686	0.2025	0.043*
C46	0.1683 (3)	0.39770 (17)	0.23361 (12)	0.0307 (4)
H46	0.0323	0.4184	0.1953	0.037*
C61	0.1984 (3)	0.6632 (2)	0.02004 (14)	0.0398 (4)
H61A	0.1252	0.5942	−0.0141	0.060*
H61B	0.3294	0.6045	0.0582	0.060*
H61C	0.2701	0.7202	−0.0405	0.060*
C62	−0.2175 (3)	0.8637 (2)	0.05769 (14)	0.0397 (4)
H62A	−0.2898	0.7948	0.0229	0.060*
H62B	−0.1591	0.9276	−0.0011	0.060*
H62C	−0.3439	0.9279	0.1191	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0253 (5)	0.0272 (6)	0.0334 (6)	−0.0055 (4)	−0.0032 (4)	−0.0028 (4)
O2	0.0280 (6)	0.0361 (6)	0.0352 (6)	−0.0045 (4)	−0.0103 (4)	−0.0069 (4)
O3	0.0409 (6)	0.0306 (6)	0.0301 (6)	−0.0151 (5)	−0.0124 (4)	0.0082 (4)
O4	0.0261 (5)	0.0309 (6)	0.0258 (5)	−0.0093 (4)	−0.0082 (4)	0.0054 (4)
C2	0.0248 (7)	0.0279 (7)	0.0316 (7)	−0.0071 (5)	−0.0062 (5)	0.0009 (6)
C3	0.0258 (7)	0.0266 (7)	0.0272 (7)	−0.0083 (5)	−0.0051 (5)	0.0028 (5)
C4	0.0236 (7)	0.0282 (8)	0.0237 (7)	−0.0080 (5)	−0.0066 (5)	0.0025 (5)
C5	0.0258 (7)	0.0279 (8)	0.0274 (7)	−0.0071 (5)	−0.0042 (5)	0.0014 (5)
C6	0.0343 (8)	0.0326 (8)	0.0258 (7)	−0.0128 (6)	−0.0079 (6)	0.0056 (6)
C41	0.0265 (7)	0.0271 (8)	0.0243 (6)	−0.0075 (6)	−0.0030 (5)	0.0025 (5)
C42	0.0308 (8)	0.0308 (8)	0.0358 (8)	−0.0077 (6)	−0.0105 (6)	0.0022 (6)
C43	0.0332 (8)	0.0367 (9)	0.0420 (9)	−0.0038 (6)	−0.0114 (7)	0.0070 (7)
C44	0.0419 (9)	0.0275 (8)	0.0365 (8)	−0.0006 (6)	−0.0037 (7)	0.0053 (6)
C45	0.0479 (9)	0.0276 (8)	0.0325 (8)	−0.0108 (7)	−0.0065 (7)	0.0011 (6)
C46	0.0347 (8)	0.0304 (8)	0.0283 (7)	−0.0102 (6)	−0.0080 (6)	0.0031 (6)
C61	0.0430 (9)	0.0468 (10)	0.0281 (8)	−0.0141 (7)	−0.0015 (6)	−0.0018 (7)
C62	0.0437 (9)	0.0442 (10)	0.0355 (8)	−0.0130 (7)	−0.0176 (7)	0.0125 (7)

Geometric parameters (Å, °)

O1—C2	1.4278 (18)	C41—C46	1.388 (2)
O1—C5	1.4370 (19)	C41—C42	1.397 (2)
O2—C2	1.3972 (17)	C42—C43	1.385 (2)
O2—H2O	0.8400	C42—H42	0.9500
O3—C6	1.4295 (18)	C43—C44	1.385 (3)
O3—C3	1.4254 (17)	C43—H43	0.9500
O4—C6	1.4323 (18)	C44—C45	1.386 (2)
O4—C4	1.4339 (16)	C44—H44	0.9500
C2—C3	1.522 (2)	C45—C46	1.391 (2)

C2—H2	1.0000	C45—H45	0.9500
C3—C4	1.563 (2)	C46—H46	0.9500
C3—H3	1.0000	C61—H61A	0.9800
C4—C41	1.515 (2)	C61—H61B	0.9800
C4—C5	1.535 (2)	C61—H61C	0.9800
C5—H5A	0.9900	C62—H62A	0.9800
C5—H5B	0.9900	C62—H62B	0.9800
C6—C62	1.509 (2)	C62—H62C	0.9800
C6—C61	1.513 (2)		

C2—O1—C5	106.30 (11)	O4—C6—C61	111.43 (13)
C2—O2—H2O	107.7	C62—C6—C61	113.45 (14)
C6—O3—C3	107.42 (11)	C46—C41—C42	118.91 (14)
C6—O4—C4	108.29 (10)	C46—C41—C4	120.93 (13)
O2—C2—O1	111.99 (12)	C42—C41—C4	120.10 (13)
O2—C2—C3	108.35 (12)	C43—C42—C41	120.58 (15)
O1—C2—C3	104.47 (11)	C43—C42—H42	119.7
O2—C2—H2	110.6	C41—C42—H42	119.7
O1—C2—H2	110.6	C44—C43—C42	120.26 (15)
C3—C2—H2	110.6	C44—C43—H43	119.9
O3—C3—C2	108.22 (12)	C42—C43—H43	119.9
O3—C3—C4	104.64 (11)	C43—C44—C45	119.46 (15)
C2—C3—C4	104.60 (11)	C43—C44—H44	120.3
O3—C3—H3	112.9	C45—C44—H44	120.3
C2—C3—H3	112.9	C46—C45—C44	120.51 (15)
C4—C3—H3	112.9	C46—C45—H45	119.7
O4—C4—C41	112.22 (11)	C44—C45—H45	119.7
O4—C4—C5	108.90 (11)	C45—C46—C41	120.27 (14)
C41—C4—C5	112.12 (12)	C45—C46—H46	119.9
O4—C4—C3	103.36 (10)	C41—C46—H46	119.9
C41—C4—C3	116.44 (11)	C6—C61—H61A	109.5
C5—C4—C3	102.99 (11)	C6—C61—H61B	109.5
O1—C5—C4	105.33 (11)	H61A—C61—H61B	109.5
O1—C5—H5A	110.7	C6—C61—H61C	109.5
C4—C5—H5A	110.7	H61A—C61—H61C	109.5
O1—C5—H5B	110.7	H61B—C61—H61C	109.5
C4—C5—H5B	110.7	C6—C62—H62A	109.5
H5A—C5—H5B	108.8	C6—C62—H62B	109.5
O3—C6—O4	104.00 (11)	H62A—C62—H62B	109.5
O3—C6—C62	109.07 (13)	C6—C62—H62C	109.5
O4—C6—C62	108.34 (12)	H62A—C62—H62C	109.5
O3—C6—C61	110.10 (13)	H62B—C62—H62C	109.5

C5—O1—C2—O2	−76.45 (14)	C3—O3—C6—O4	34.96 (14)
C5—O1—C2—C3	40.60 (13)	C3—O3—C6—C62	150.39 (12)
C6—O3—C3—C2	−133.84 (12)	C3—O3—C6—C61	−84.52 (14)
C6—O3—C3—C4	−22.73 (13)	C4—O4—C6—O3	−33.70 (14)
O2—C2—C3—O3	−154.79 (11)	C4—O4—C6—C62	−149.65 (13)
O1—C2—C3—O3	85.67 (13)	C4—O4—C6—C61	84.87 (14)
O2—C2—C3—C4	94.07 (13)	O4—C4—C41—C46	−14.92 (18)

O1—C2—C3—C4	−25.47 (13)	C5—C4—C41—C46	108.02 (15)
C6—O4—C4—C41	−107.03 (13)	C3—C4—C41—C46	−133.74 (14)
C6—O4—C4—C5	128.23 (12)	O4—C4—C41—C42	167.76 (12)
C6—O4—C4—C3	19.23 (13)	C5—C4—C41—C42	−69.29 (16)
O3—C3—C4—O4	2.14 (13)	C3—C4—C41—C42	48.95 (18)
C2—C3—C4—O4	115.84 (12)	C46—C41—C42—C43	0.4 (2)
O3—C3—C4—C41	125.66 (12)	C4—C41—C42—C43	177.73 (13)
C2—C3—C4—C41	−120.64 (13)	C41—C42—C43—C44	−0.2 (2)
O3—C3—C4—C5	−111.22 (12)	C42—C43—C44—C45	0.2 (2)
C2—C3—C4—C5	2.48 (13)	C43—C44—C45—C46	−0.4 (2)
C2—O1—C5—C4	−39.18 (13)	C44—C45—C46—C41	0.6 (2)
O4—C4—C5—O1	−88.03 (13)	C42—C41—C46—C45	−0.6 (2)
C41—C4—C5—O1	147.17 (11)	C4—C41—C46—C45	−177.92 (13)
C3—C4—C5—O1	21.22 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2o···O1^{i}	0.84	1.93	2.755 (2)	166
C5—H5a···O2^{ii}	0.99	2.47	3.296 (3)	140

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

Fig. 1

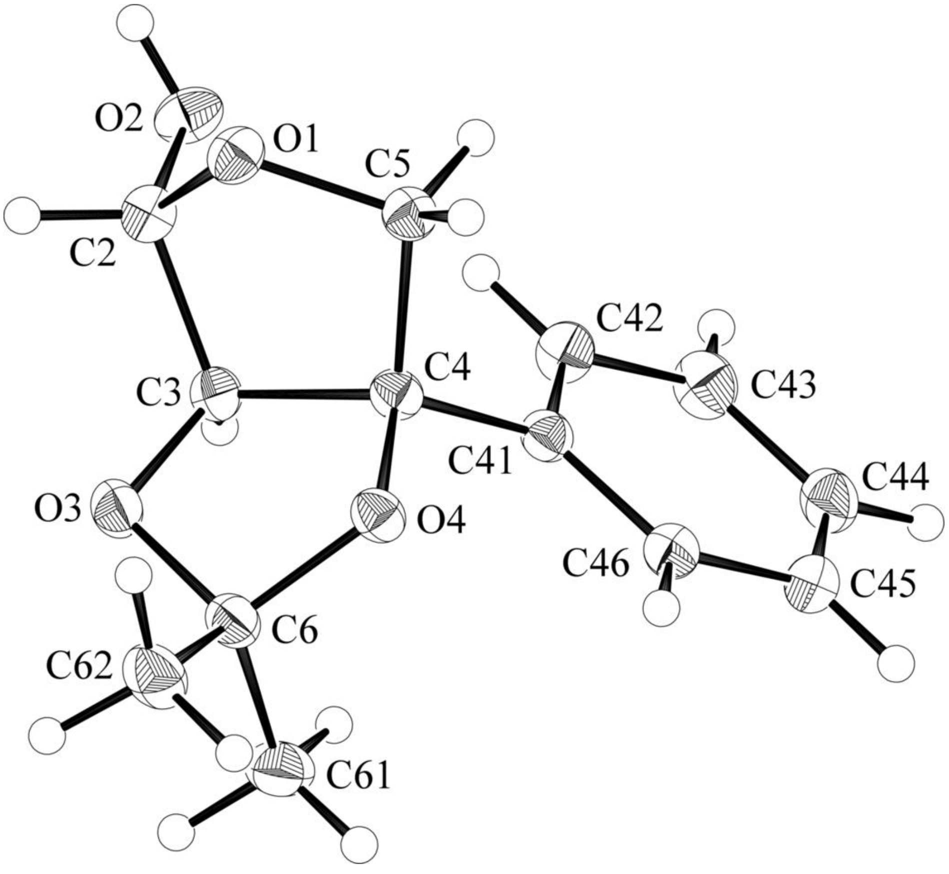

Fig. 2