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## Key indicators

Single-crystal X-ray study

T = 150 K

Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$ 

R factor = 0.049

wR factor = 0.113

Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(2*S*)-1-(4-Fluorophenyl)-2-hydroxy-2-[(2*S*,5*R*)-5-(1-hydroxy-1-methylethyl)-tetrahydrofuran-2-yl]ethanone**

The asymmetric unit of the title compound,  $\text{C}_{15}\text{H}_{19}\text{FO}_4$ , contains two independent molecules that hydrogen bond to form dimers.

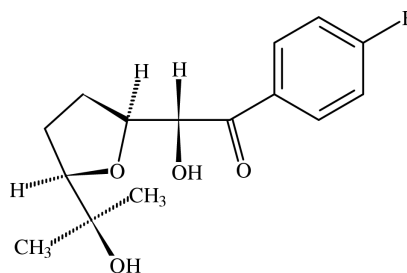
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## Comment

During the development of chiral phase-transfer catalysed permanganate oxidations of 1,5-dienes, (2*S*)-1-(4-fluorophenyl)-2-hydroxy-2-[(2*S*,5*R*)-5-(1-hydroxy-1-methylethyl)-tetrahydrofuran-2-yl]ethanone, (I), was prepared (Brown & Keily, 2001).



(I)

The two independent molecules in the asymmetric unit form dimers *via* four intermolecular hydrogen bonds (Table 1).

Molecules of opposite chirality are related by a pseudo-centre of symmetry at  $x = 0.50$  (1),  $y = 0.26$  (3),  $z = 0.50$  (1), whilst molecules of the same chirality are related by a pseudo-twofold axis at  $y = 0.01$  (3),  $z = 0.75$  (2) parallel to  $x$  with a translation of 0.49 (2).

## Experimental

(*E*)-1-(4-Fluorophenyl)-7-methylocta-2,6-dien-1-one was oxidized by potassium permanganate under solid-liquid phase-transfer conditions using a chiral quaternary ammonium salt. Following a standard

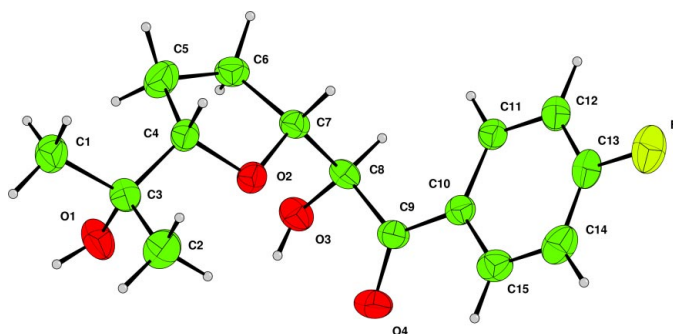
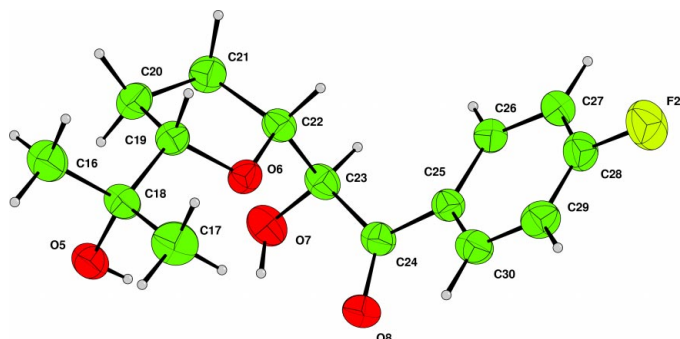
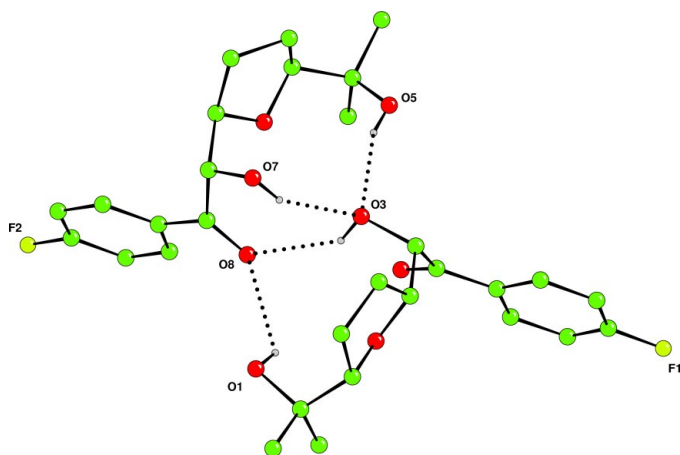


Figure 1

A view of the title compound showing the atomic numbering scheme of molecule 1. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
A view of the title compound showing the atomic numbering scheme of molecule 2. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 3**  
A view showing the hydrogen-bonded dimers.

work-up and column chromatography, the product was recrystallized from ethanol/hexane to give colourless crystals of the title compound.

#### Crystal data

$C_{15}H_{19}FO_4$   
 $M_r = 282.30$   
 Orthorhombic,  $Pna2_1$   
 $a = 10.973(2) \text{ \AA}$   
 $b = 12.764(3) \text{ \AA}$   
 $c = 20.721(4) \text{ \AA}$   
 $V = 2902.2(10) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.292 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 Cell parameters from 23180 reflections  
 $\theta = 3.1\text{--}25.0^\circ$   
 $\mu = 0.10 \text{ mm}^{-1}$   
 $T = 150(2) \text{ K}$   
 Block, colourless  
 $0.10 \times 0.10 \times 0.10 \text{ mm}$

#### Data collection

Nonius Kappa CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans to fill the Ewald sphere  
 Absorption correction: none  
 23180 measured reflections  
 5012 independent reflections

2859 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.096$   
 $\theta_{\text{max}} = 25.0^\circ$   
 $h = -11 \rightarrow 13$   
 $k = -13 \rightarrow 15$   
 $l = -22 \rightarrow 24$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.113$   
 $S = 1.05$   
 5012 reflections  
 378 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0381P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.048$   
 $\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O5—H05 $\cdots$ O3	0.83 (4)	2.09 (4)	2.854 (5)	153 (3)
O7—H07 $\cdots$ O3	0.91 (5)	2.11 (4)	2.943 (4)	150 (4)
O1—H01 $\cdots$ O8	0.95 (10)	2.25 (11)	2.946 (5)	130 (7)
O3—H03 $\cdots$ O8	0.93 (8)	2.17 (9)	2.831 (5)	127 (6)

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* (Otwinowski & Minor, 1997), *COLLECT* and *MAXUS* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WINGX* (Farrugia, 1998).

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#### References

- Brown, R. C. D. & Keily, J. F. (2001). *Angew. Chem. Int. Ed.* In the press.  
 Farrugia, L. J. (1998). *WINGX*. University of Glasgow, Scotland.  
 Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.  
 Mackay, S., Gilmore, C. J., Edwards, C., Tremayne, M., Stuart, N. & Shankland, K. (1998). *MAXUS*. University of Glasgow, Scotland, Nonius BV, Delft, The Netherlands, and MacScience Co. Ltd, Yokohama, Japan.  
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.  
 Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.