

Masood Parvez,\* Dan V. Simion  
and Ted S. SorensenDepartment of Chemistry, The University of  
Calgary, 2500 University Drive NW, Calgary,  
Alberta, Canada T2N 1N4

Correspondence e-mail: parvez@ucalgary.ca

## Key indicators

Single-crystal X-ray study  
 $T = 200\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$   
 $R$  factor = 0.040  
 $wR$  factor = 0.167  
Data-to-parameter ratio = 8.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.A novel chiral  $C_2$  axially symmetric acenaphtho-  
quinone diketal

The crystal structure of the title compound,  $(-)-(4R,5R,4''R,5''R)-4,5,4'',5''$ -tetramethyldispiro[1,3-dioxalane-2,1'(2' $H$ )-acenaphthylene-2',2''-1,3-dioxalane],  $\text{C}_{20}\text{H}_{22}\text{O}_4$ , contains molecules separated by normal van der Waals distances. The dioxacyclopentane rings adopt envelope conformations and the naphthalene moiety is essentially planar while the two C atoms bonded to it lie 0.122 (8) and 0.105 (8) Å on opposite sides from its mean plane.

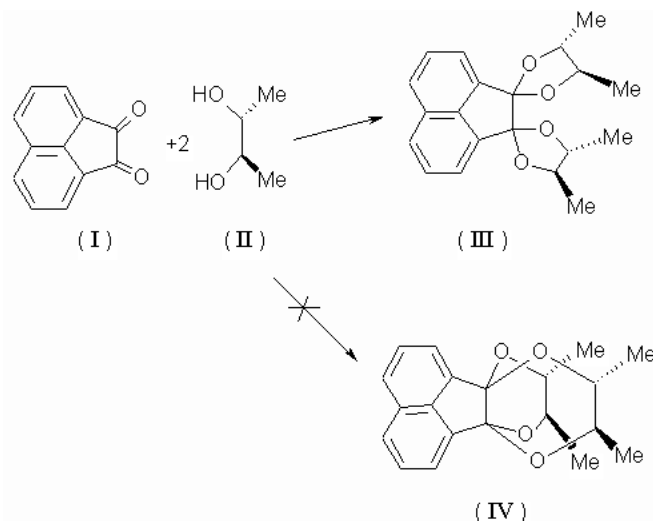
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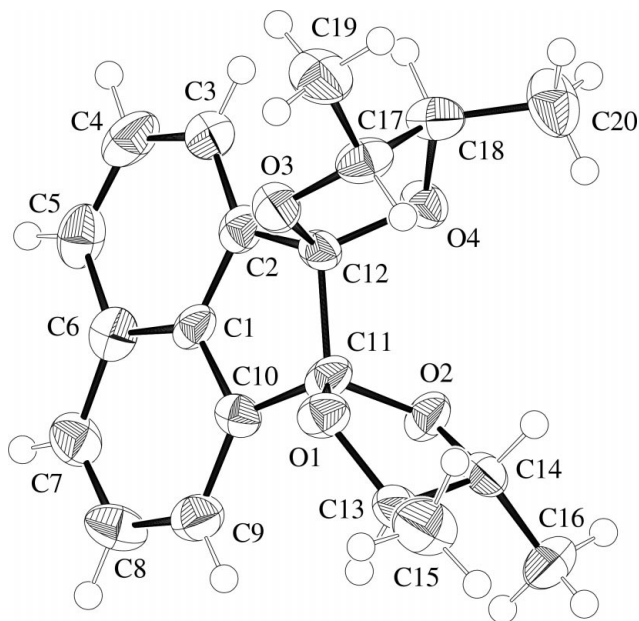
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## Comment

Naphthalene- $\text{Cr}(\text{CO})_3$  can be used to transfer  $\text{Cr}(\text{CO})_3$  to monoarenes (Kündig & Timms, 1977). In cases where the monoarene is prochiral, the use of a homochiral naphthalene- $\text{Cr}(\text{CO})_3$  complex might be expected (depending on the actual mechanism of this transfer; Traylor *et al.*, 1989; Howell *et al.*, 1991) to result in some enantioselectivity in the product monoarene complex. To simplify the number of possible  $\text{Cr}(\text{CO})_3$  isomers of a chiral naphthalene and to maximize the chiral environment for subsequent  $\text{Cr}(\text{CO})_3$  transfer, it was desirable to use  $C_2$  axially symmetric chiral naphthalenes in this work. One potential candidate was the bis-acetal (III) (Simion, 1996). Acenaphthoquinone (I) was reacted with diol (II) to produce the bis-ketal (III). The X-ray crystal structure determination of (III) was undertaken for two reasons: (i) to verify that the bis-acetal (III) was actually formed; the bis-1,4-dioxane structure (IV) was also possible and NMR spectroscopy was not definitive in distinguishing (III) and (IV), and (ii) to determine the detailed conformation of (III) with regard to the steric differentiation between the two naphthalene faces.





**Figure 1**  
ORTEP (Johnson, 1976) drawing of (III). Displacement ellipsoids have been plotted at the 50% probability level.

The structure of (III) is composed of molecules (Fig. 1) separated by normal van der Waals distances. The dioxacyclopentane rings adopt C13- and C17-envelope conformations, with C13 and C17 0.543 (9) and 0.550 (10) Å, respectively, out of the planes formed by the remaining ring atoms. The naphthalene moiety in the acenaphthene ring is essentially planar, while the two C atoms bonded to it lie 0.122 (8) and 0.105 (8) Å on opposite sides from its mean plane. The molecular dimensions are as expected.

## Experimental

Acenaphthoquinone, (I), was refluxed (Dean–Stark trap) with 9 equivalents of (–)-(2*R*,3*R*)-butane-2,3-diol, (II), in toluene solvent (cat. *p*-toluenesulfonic acid) for 17 d, leading to (III), a bis-ketal product, which was purified by flash chromatography (SiO<sub>2</sub>, 1:1 benzene–ethyl acetate), 80% yield, m.p. 478–479 K (from benzene–hexane),  $[\alpha]_D^{23} = -23.8^\circ$  ( $c = 0.075$ , CHCl<sub>3</sub>). GC–MS analysis of the crude material also showed the presence of a minor isomer.

### Crystal data

C<sub>20</sub>H<sub>22</sub>O<sub>4</sub>  
 $M_r = 326.38$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 9.799$  (3) Å  
 $b = 19.331$  (5) Å  
 $c = 9.054$  (3) Å  
 $V = 1715.0$  (9) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.264$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 10.0$ – $15.0^\circ$   
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 200$  (2) K  
 Prismatic, colourless  
 $0.50 \times 0.21 \times 0.13$  mm

### Data collection

Rigaku AFC-6S diffractometer  
 $\omega/2\theta$  scans  
 1758 measured reflections  
 1758 independent reflections  
 831 reflections with  $I > 2\sigma(I)$   
 $\theta_{\max} = 25.0^\circ$

$h = 0 \rightarrow 11$   
 $k = 0 \rightarrow 23$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 every 200 reflections  
 intensity decay: <0.5%

### Refinement

Refinement on  $F^2$   
 $R(F) = 0.040$   
 $wR(F^2) = 0.167$   
 $S = 1.00$   
 1758 reflections  
 218 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0835P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL*  
 Extinction coefficient: 0.013 (3)  
 Absolute structure: (Flack, 1983)  
 Flack parameter =  $-1$  (4)

All the H atoms were located from difference maps and were included at geometrically idealized positions, with C–H = 0.95–1.00 Å, in a riding mode with isotropic displacement parameters 1.2 (non-methyl) and 1.5 (methyl) times the displacement parameters of the atoms to which they were attached. An absolute configuration was not established in this analysis since the absolute configuration of the starting material was already known.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXS97* (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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