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Key indicators

Single-crystal X-ray study T = 213 K Mean σ (C–C) = 0.003 Å R factor = 0.058 wR factor = 0.165 Data-to-parameter ratio = 11.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e. The title compound, $C_{28}H_{30}O_2$, was obtained by an unprecedented osmium tetraoxide-mediated dimerization of (2'-methyl-3'-oxocyclohex-1'-enyl)phenylethane. The *trans* stereochemistry was revealed by X-ray crystallography. The molecule exhibits a crystallographic center of inversion.

enyl)-1,2-diphenylethane

 (\pm) -(1RS,2SR)-1,2-Bis(2'-methyl-3'-oxocyclohex-1'-

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Comment

The osmium tetraoxide-mediated bishydroxylation of alkenes is a well established reaction in organic synthesis. In particular, catalytic versions of this reaction using stoichiometric cooxidants are routinely used for the conversion of terminal, diand trisubstituted alkenes to the corresponding diols (Schröder, 1980; Kolb *et al.*, 1994). Tetrasubstituted alkenes and in particular hindered substrates, however, are less suitable and usually require harsh reaction conditions or the use of a stoichiometric amount of the osmium tetraoxide (Ogino *et al.*, 1994).



The reaction of enone (1) with a catalytic amount of osmium tetraoxide for a period of four days following the conditions described by Ray & Matteson (1980) did not result in the formation of the desired ketodiol. Instead two new compounds were isolated.

The ¹H and ¹³C NMR spectra of these compounds revealed that the alkene moiety remained intact, suggesting a dimerization of the starting material in the course of this reaction. Examining the mass spectra showed indeed molecular ion peaks at 398 corresponding to the diastereomeric pair (2*a*) and (2*b*).

An X-ray analysis of one of the compounds confirmed the dimeric structure and revealed a *trans* relative stereochemistry that corresponds to (2*a*).

Experimental

Crystals of (2*a*) were obtained from dichloromethane/pentane. TLC: $R_{\rm F} = 0.34$ (pentane/EtOAc = 70:30); m.p. 503 K. HRMS (EI): analysis calculated for C₂₈H₃₀O₂: 398.2246, found: 398.2252. IR (Film): $\nu =$

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organic papers

3028 cm⁻¹ (w, C_{ar}-H), 2959 (m, C–H), 1656 (vs, C=O), 1616 (m, C_{ar}- C_{ar}), 1449 (m), 1349 (m), 763 (s, C_{ar}-H), 705 (s, C_{ar}-H). ¹H NMR (300 MHz, CDCl₃): δ 1.58–1.73 (m, 4H, CH₂CH₂CH₂), 1.95 (t, ⁵J = 1.4 Hz, 6H, CH₃), 2.14–2.38 (m, 8H, COCH₂CH₂CH₂CBn), 4.80 (s, 2H, PhCHCHPh), 7.20–7.32 (m, 10H, aromatic H). ¹³C NMR (75.5 MHz, CDCl₃): δ 11.5 (CH₃), 22.4 (CH₂CH₂CH₂), 26.5 (CH₂CCPh), 37.7 (CH₂CO), 49.7 (PhCH), 127.4 (C_{ar}H), 127.7 (2C, C_{ar}H), 128.8 (2C, C_{ar}H), 132.6 (PhCHCCCO), 139.4 (C_{ar}), 157.3 (PhCHCCCO), 199.2 (CO). MS (EI), m/z (%): 398 (1) [M⁺], 200 (50) [C₁₄H₁₆O⁺], 199 (100) [M⁺ - C₁₄H₁₅O], 184 (25) [199 - CH₃⁺], 129 (29) [184 - C₃H₃O⁺], 91 (30) [C₇H₇⁺].

 $D_x = 1.192 \text{ Mg m}^{-3}$

Cell parameters from 25

Cu K α radiation

reflections

 $\begin{array}{l} \theta = 40.3 \text{--} 46.2^{\circ} \\ \mu = 0.57 \ \text{mm}^{-1} \end{array}$

T = 213 (2) K

 $R_{\rm int}=0.039$

 $\theta_{\rm max} = 69.9^{\circ}$ $h = 0 \rightarrow 13$

 $k=-11\to 0$

 $l = -14 \rightarrow 13$

2 standard reflections

frequency: 120 min

intensity decay: 3%

 $w = 1/[\sigma^2(F_o^2) + (0.0912P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: SHELXL97

Extinction coefficient: 0.0092 (16)

+ 0.2417P]

 $(\Delta/\sigma)_{\rm max} < 0.001$

 $\Delta \rho_{\rm max} = 0.22 \text{ e } \text{\AA}^{-3}$

 $\Delta \rho_{\rm min} = -0.18 \text{ e } \text{\AA}^{-3}$

Prism, colourless $0.50 \times 0.50 \times 0.20 \text{ mm}$

Crystal data

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\begin{array}{l} C_{28}H_{30}O_2 \\ M_r = 398.52 \\ \text{Monoclinic, } P2_1/n \\ a = 10.8658 \ (7) \ \text{\AA} \\ b = 9.4435 \ (5) \ \text{\AA} \\ c = 11.7323 \ (8) \ \text{\AA} \\ \beta = 112.740 \ (6)^\circ \\ V = 1110.29 \ (12) \ \text{\AA}^3 \\ Z = 2 \end{array}
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Data collection

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Enraf-Nonius CAD-4
diffractometer
\omega scans
Absorption correction: \psi scan
(SHELXTL; Sheldrick, 1996)
T_{min} = 0.765, T_{max} = 0.895
2229 measured reflections
2116 independent reflections
1794 reflections with I > 2\sigma(I)
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Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.058$ $wR(F^2) = 0.165$ S = 1.072116 reflections 187 parameters H atoms treated by a mixture of independent and constrained refinement

Table 1

Selected geometric parameters (Å, °).

C1-C1′	1.525 (3)	C2'-C3'	1.484 (3)
C1-C1 ⁱ	1.549 (4)	C3′-C4′	1.496 (3)
C1' - C2'	1.350 (3)	C4′-C5′	1.511 (3)
C1' - C6'	1.511 (3)	C5′-C6′	1.516 (3)
$C1' - C1 - C1^{i}$	111.61 (19)	C2' - C3' - C4'	118.82 (17)
$C1" - C1 - C1^{i}$	113.01 (19)	C3'-C4'-C5'	111.72 (19)
C2' - C1' - C6'	121.83 (17)	C4' - C5' - C6'	109.34 (18)
C1' - C2' - C3'	119.84 (17)	C1' - C6' - C5'	112.56 (17)

Symmetry code: (i) -x, 1 - y, 2 - z.



SHELXTL (Sheldrick, 1996) drawing of (2a). Displacement ellipsoids are drawn at the 50% probability level.

The H atoms were located using difference Fourier synthesis maps and allowed to refine isotropically (C–H distances 0.96 to 1.01 Å). In the final stage, constraints and a common isotropic displacement parameter for the H atoms were included for the disordered methyl group.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXTL*.

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