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(1*R**,11*R**)-Bicyclo[9.4.1]hexadecane-12,16-dione

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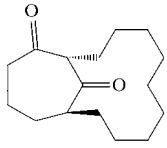
The title compound, $C_{16}H_{26}O_2$, (I), prepared by oxidation of (1*R**,11*R**)-12-hydroxybicyclo[9.4.1]hexadecan-16-one using pyridinium dichromate, has a *trans* configuration of the two fused rings and represents an interesting precursor for the synthesis of macrocyclic structures.



(I)

Experimental

Following the procedure of Fu & Cook (1992), a suspension of (1*R**,11*R**)-12-hydroxybicyclo[9.4.1]hexadecan-16-one (1.89 g, 7.5 mmol) and pyridinium dichromate (5.64 g, 15.0 mmol) in dry dichloromethane (20 ml) was stirred at room temperature for 16 h. After filtration of the salts, the solution was evaporated and filtered through a short column with silica gel using *n*-hexane/MTBE (1:1) as eluent. The crude product (1.71 g) was recrystallized from 10 ml *n*-hexane to give 1.41 g (5.6 mmol, 75%) (1*R**,11*R**)-bicyclo[9.4.1]-hexadecane-12,16-dione as colourless crystals. Analysis calculated for $C_{16}H_{26}O_2$ (250.37 g mol^{-1}): C 76.8, H 10.5%; found: C 76.8, H 10.6%. MS (EI, 70 eV): *m*/*z* (%) = 250 (M^+, 39), 232 (10), 222 (36), 179 (7), 134 (6), 123 (19), 110 (30), 95 (32), 83 (29), 67 (34), 55 (100). IR (KBr): $\tilde{\nu}$ (cm^{-1}) = 2932 (*s*), 2893 (*s*), 2859 (*s*), 1722 (*s*), 1692 (*s*), 1444 (*s*), 1336 (*s*), 1260 (*s*), 1139 (*s*). ^{1}H NMR (400 MHz, $CDCl_3$): δ (p.p.m.) = 1.00–1.43 (14H), 1.67 (*m*, 5H), 1.97 (*m*, 3H), 2.39 (*m*, 1H), 2.48 (*m*, 1H), 2.82 (*tt**, ^3J = 11.6 Hz, ^3J = 2.6 Hz, 1H), 3.13 (*dd*, ^3J = 11.9 Hz, ^3J = 3.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (p.p.m.) = 21.5 (CH_2), 21.6 (CH_2), 22.2 (CH_2), 23.6 (CH_2), 23.6 (CH_2), 23.9 (CH_2), 24.1 (CH_2), 26.7 (CH_2), 27.2 (CH_2), 31.8 (CH_2), 37.4 (CH_2), 43.2 (CH_2), 46.9 (CH), 69.3 (CH), 209.2 (Cq), 214.5 (Cq). M.p.: 380 K.

Crystal data

$C_{16}H_{26}O_2$	Z = 2
M_r = 250.37	D_x = 1.137 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 8.0114 (4) Å	Cell parameters from 8200 reflections
b = 9.8149 (7) Å	θ = 2.67–24.99°
c = 10.2505 (8) Å	μ = 0.073 mm^{-1}
α = 107.919 (2)°	T = 291 (1) K
β = 106.039 (4)°	Parallelepiped, colourless
γ = 91.831 (4)°	0.50 × 0.25 × 0.20 mm
V = 731.00 (9) Å^3	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.0230
360 frames *via* ω-rotation scans ($\Delta\omega$ = 1°) and 2 × 10 s per frame	θ_{max} = 24.99°
8200 measured reflections	$h = -8 \rightarrow 8$
2389 independent reflections	$k = -11 \rightarrow 11$
1284 reflections with $I > 2\sigma(I)$	$l = -12 \rightarrow 11$
	Intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$w = 1/[\sigma^2(F_o^2) + (0.0490P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.098	$(\Delta/\sigma)_{max} < 0.001$
S = 0.922	$\Delta\rho_{max}$ = 0.13 e Å^{-3}
2389 reflections	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
163 parameters	

Data collection: Nonius KappaCCD software; cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997), *PARST*95 (Nardelli, 1995).

References

Fu, X. & Cook, J. M. (1992). *J. Org. Chem.* **57**, 5121–5128.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Pt A, edited by C. W. Carter & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*93. University of Göttingen, Germany.