Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

(1*R**,2*R**)-1,2-(2,3-Dimethoxybutane-2,3-dioxy)cyclohexane

Hans-Georg Weinig, Ulrich Koert and Burkhard Ziemer*

Institut für Chemie, Humboldt-Universität zu Berlin, Hessische Strasse 1-2, 10115 Berlin, Germany
Correspondence e-mail: burkhard=ziemer@chemie.hu-berlin.de

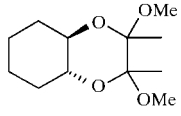
Received 24 March 2000
Accepted 10 April 2000

Data validation number: IUC0000100

The title compound, 2,3-dimethoxy-2,3-dimethyl-5,6,7,8-tetrahydro-4-oxachroman, $C_{12}H_{22}O_4$, was synthesized as a model compound for substituted diequatorial fixed vicinal *trans*-cyclohexanediols.

Comment

The title compound, (I), was synthesized as a model compound for substituted diequatorial fixed vicinal *trans*-cyclohexanediols (Berninger *et al.*, 1999). Following the method of Ley *et al.* (1994) and Montchamp *et al.* (1996), the reaction of *trans*-cyclohexane-1,2-diol with 2,2,3,3-tetramethoxybutane, trimethyl orthoformate and catalytic camphorsulfonic acid in methanol at 313 K for 3 h led to the title compound, (I), in 86% yield.



(I)

Experimental

trans-Cyclohexane-1,2-diol (500 mg, 4.30 mmol) was dissolved in MeOH (20 ml). 2,2,3,3-Tetramethoxybutane (1.53 g, 8.61 mmol), trimethyl orthoformate (1.9 ml, 17.2 mmol) and a catalytic amount of camphorsulfonic acid were added. The mixture was stirred at 313 K for 3 h, after which time solid $NaHCO_3$ was added. The solvent was removed *in vacuo* and the residue purified by flash chromatography (petrol ether/ethyl acetate, 10:1, as eluent) to give 850 mg of the title compound (3.69 mmol, 86%) as a colourless solid, which was recrystallized from MeOH [m.p.: 350 K (MeOH)].

Crystal data

$C_{12}H_{22}O_4$	$Z = 2$
$M_r = 230.30$	$D_x = 1.225$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.2971$ (11) Å	Cell parameters from 55 reflections
$b = 9.4297$ (14) Å	$\theta = 15.1$–$17.6°$
$c = 9.6480$ (13) Å	$\mu = 0.090$ mm^{-1}
$\alpha = 68.794$ (12)°	$T = 180$ (2) K
$\beta = 67.620$ (9)°	Block, colorless
$\gamma = 67.565$ (10)°	0.82 × 0.78 × 0.50 mm
$V = 624.14$ (15) Å^3	

Data collection

Stoe Stadi-4 diffractometer	$\theta_{max} = 26.04°$
$2\theta/\omega$ scans [ratio = 0.5, width (ω) = 1.7–1.82°]	$h = -9 \rightarrow 10$
2961 measured reflections	$k = -10 \rightarrow 11$
2467 independent reflections	$l = -9 \rightarrow 11$
2176 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{int} = 0.015$	frequency: 120 min
	intensity decay: 3.0%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.2295P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} = 0.006$
$wR(F^2) = 0.112$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
$S = 1.070$	$\Delta\rho_{min} = -0.18$ e Å^{-3}
2467 reflections	Extinction correction: *SHELXL*97
234 parameters	Extinction coefficient: 0.022 (5)
All H-atom parameters refined	

Refined C—H distances are in the range 0.94 (2)–1.02 (2) Å.

Data collection: *STADI*4-1.06 (Stoe & Cie, 1997); cell refinement: *STADI*4-1.06; data reduction: *XRED*-1.07 (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XSTEP*-2.18 (Stoe, 1997); software used to prepare material for publication: *SHELXL*97.

References

Berninger, J., Krauss, R., Weinig, H.-G., Koert, U., Ziemer, B. & Harms, K. (1999). *Eur. J. Org. Chem.* pp. 875–884.
Ley, S. V., Priepke, H. W. M. & Warriner, S. L. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 2290–2292.
Montchamp, J.-L., Tian, F., Hart, M. E. & Frost, J. W. (1996). *J. Org. Chem.* **61**, 3897–3899.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Stoe & Cie (1997). *STADI*4-1.06, *XRED*-1.07 and *XSTEP*-2.18. Stoe & Cie, Darmstadt, Germany.