Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,9-Diphenyl-2,4,8,10-tetraoxaspiro[5.5]undecane

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.108
Data-to-parameter ratio = 8.8

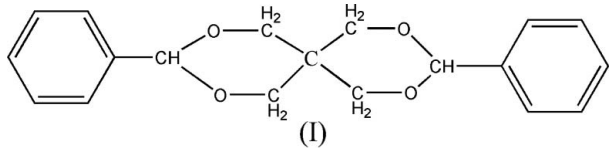
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 June 2006
Accepted 22 June 2006

In the molecule of the title compound, $C_{19}H_{20}O_4$, the 1,3-dioxane rings have chair conformations. Intermolecular C—H···O hydrogen bonds link the molecules and may be effective in the stabilization of the crystal structure.

Comment

The title compound, (I), is an important intermediate in the synthesis of pesticides (Jermy & Pandurangan, 2005). The crystal structure determination of (I) has been carried out in order to elucidate the molecular conformation. We report here the synthesis and crystal structure of (I).



(I)

In the molecule of compound (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings *B* (O1/O2/C7–C10) and *C* (O3/O4/C19–C13), with total puckering amplitudes Q_T of 1.424 (3) and 1.425 (4) Å, respectively, have chair conformations [φ = −144.95 (15)° and θ = 1.70 (3)°, and φ = 35.27 (9)° and θ = 178.46 (3)°, respectively] (Cremer & Pople, 1975). Rings *A* (C1–C6) and *D* (C14–C19) are, of course, planar, and the dihedral angle between them is 82.05 (4)°.

As can be seen from the packing diagram (Fig. 2), intermolecular C—H···O hydrogen bonds [H2*A*···O1^{i} = 2.55 Å, C2···O1^{i} = 3.214 (4) Å and C2—H2*A*···O1^{i} = 129°; symmetry code: (i) $x-\frac{1}{2}, \frac{3}{2}-y, -z$] link the molecules and these may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

The title compound was prepared from a mixture of 2,2-bis(hydroxymethyl)propane-1,3-diol (0.68 g, 5 mmol), benzaldehyde (10 mmol), freshly activated catalyst TiO_2/SO_4^{2-} (0.3 g, 0.16 mmol) and benzene (20 ml), heated with stirring at refluxing temperature for 2 h, using a Dean-Stark apparatus in a nitrogen atmosphere (Sun *et al.*, 2001). The progress of the reaction was monitored by thin-layer chromatography. After cooling to room temperature, the catalyst was filtered off, the crude product was isolated by distillation and the solid was recrystallized from ethanol. Crystals of (I) were obtained by dissolving the title compound (1.0 g) in toluene (15 ml) and evaporating the solvent slowly at room temperature for about 3 d.

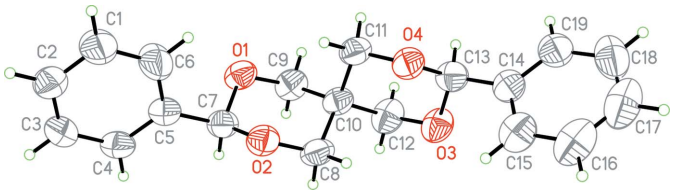


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Crystal data

$C_{19}H_{20}O_4$
M_r = 312.35
Orthorhombic, $P2_12_12_1$
a = 9.1890 (18) Å
b = 11.483 (2) Å
c = 15.449 (3) Å
V = 1630.1 (5) Å^3
Z = 4
D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.965, T_{max} = 0.974
3499 measured reflections
1839 independent reflections
1296 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.0°
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 0.99
1839 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.05P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. H atoms were positioned geometrically, with C—H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

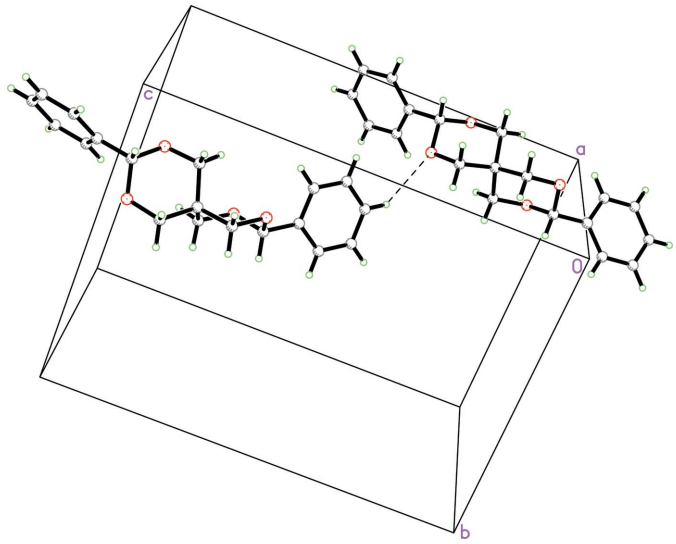


Figure 2
A partial packing diagram for (I). Hydrogen bonds are shown as dashed lines.

The authors thank the Centre of Testing and Analysis, Nanjing University, for support.

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