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Key indicators

Single-crystal X-ray study

T = 100 K

Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$

R factor = 0.070

wR factor = 0.165

Data-to-parameter ratio = 10.2

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

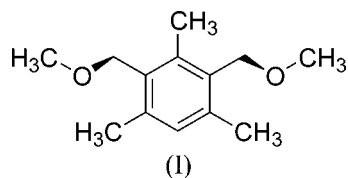
2,4-Bis(methoxymethyl)-1,3,5-trimethylbenzene

The title compound, $\text{C}_{13}\text{H}_{20}\text{O}_2$, displays polar ordering owing to intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds that link the molecules to form a three-dimensional network. A twofold rotation axis bisects the ring, passing through the unsubstituted C atom, the *para*-C atom and C of the attached methyl group.

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Comment

In our studies of complexes generated from ditopic ligands (Dobrzańska, 2005; Dobrzańska, Lloyd *et al.*, 2005; Dobrzańska, Raubenheimer *et al.*, 2005; Dobrzańska *et al.*, 2006; Dobrzańska & Lloyd, 2006), we have unexpectedly obtained single crystals of the title compound, (I).



The asymmetric unit of (I) (Fig. 1) consists of half a molecule. The two methoxy groups adopt the *trans* configuration with respect to the plane of the benzene ring, allowing the molecule to straddle a twofold rotation axis along the *c* axis. The twofold axis passes through C4, C1 and C5. As a result, the methyl group on the twofold axis is disordered. The C1—C2—C7—O8 torsion angle, which defines the rotation of the $\text{H}_2\text{C}-\text{O}$ bonds out of the plane of the central benzene ring, is $-74.5(4)^\circ$. 2,4-Bis(methoxymethyl)-1,3,5-trimethylbenzene molecules pack parallel to the *bc* plane and display polar ordering (Fig. 2), most likely due to the formation of intermolecular $\text{C7}-\text{H7A}\cdots\text{O8}^i$ hydrogen bonds [symmetry code: (i) $x - \frac{1}{4}, \frac{1}{4} - y, z - \frac{1}{4}$] (Fig. 3 and Table 1). A similar influence of hydrogen bonding on ordering of molecules was observed for 2,6-lutidine, where the presence of linear $\text{C}-\text{H}\cdots\text{N}$ interactions results in macroscopically polar crystals (Bond *et al.*, 2001). Intramolecular $\text{C5}-\text{H5B}\cdots\text{O8}$ hydrogen bonds are also formed (Table 1). The aromatic rings are at an angle of 17.43° relative to the *bc* plane. The molecules are stacked in a herringbone-like pattern as a result of forming a three-dimensional hydrogen-bonded network. $\pi-\pi$ Interactions between the benzene rings in adjacent layers is not possible because the aromatic groups are tilted at an angle of 34.86° (centroid-to-centroid) relative to one another. Since the centroid-to-centroid distance between alternate layers is 8.186 \AA and the offset is complete, $\pi-\pi$ stacking is implausible in this instance as well.

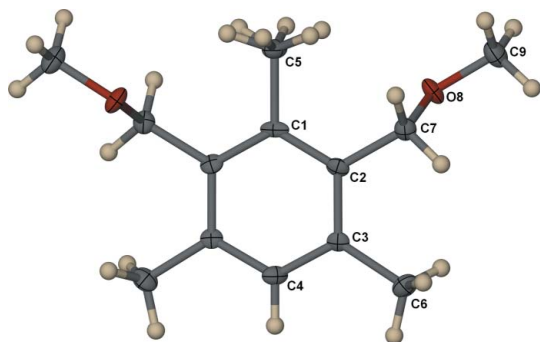


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabeled atoms are related to labeled atoms by the symmetry operation $-x, -y, z$. Both methyl group disorder components are shown.

Experimental

Colorless crystals suitable for single-crystal X-ray diffraction were obtained by slow evaporation of the solution after an $\text{S}_{\text{N}}2$ reaction of 2,4-bis-(chloromethyl)-1,3,5-trimethylbenzol (0.7238 g) with 4,5-dichloroimidazole in methanol (molar ratio: 0.0033:0.0233).

Crystal data

$\text{C}_{13}\text{H}_{20}\text{O}_2$	$Z = 8$
$M_r = 208.29$	$D_x = 1.166 \text{ Mg m}^{-3}$
Orthorhombic, $Fdd2$	Mo $K\alpha$ radiation
$a = 14.253 (16) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$b = 20.66 (2) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 8.057 (9) \text{ \AA}$	Block, colorless
$V = 2373 (4) \text{ \AA}^3$	$0.50 \times 0.38 \times 0.26 \text{ mm}$

Data collection

Bruker APEX CCD area-detector diffractometer	747 independent reflections
ω scans	623 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.084$
3575 measured reflections	$\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 6.8504P]$
$R[F^2 > 2\sigma(F^2)] = 0.070$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.165$	$(\Delta/\sigma)_{\text{max}} = 0.008$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
747 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
73 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5B}\cdots\text{O8}$	0.98	2.45	3.172 (5)	131
$\text{C7}-\text{H7A}\cdots\text{O8}^i$	0.98	2.57	3.542 (7)	166

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

H atoms were positioned geometrically, with $\text{C}-\text{H} = 0.95, 0.99$ and 0.98 \AA for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms. The

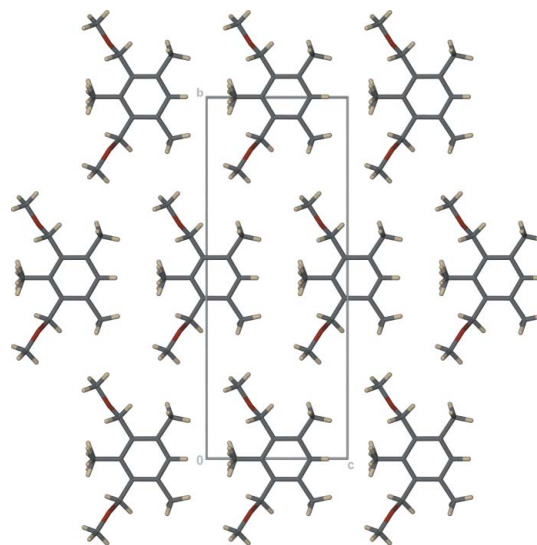


Figure 2

Polar ordering of molecules in the bc plane of the unit cell.

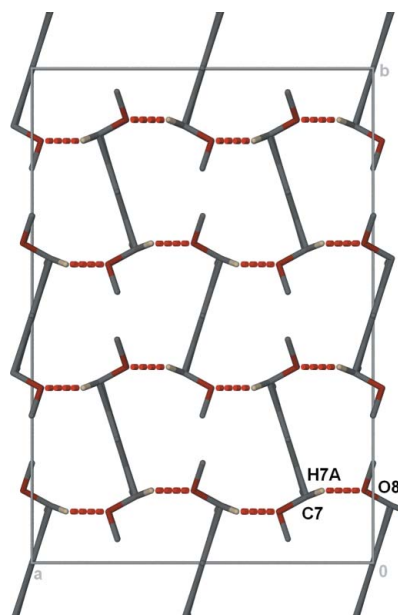


Figure 3

The hydrogen bond pattern (dashed red lines), as viewed along the c axis.

methyl group C5 has its H atoms disordered over two sets of sites, with 0.5 occupancy. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour 2001); software used to prepare material for publication: *SHELXL97*.

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