

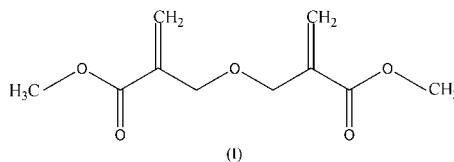
## Methyl 4-oxahepta-1,6-diene-2,6-dicarboxylate

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## Key indicators

Single-crystal X-ray study  
 $T = 153\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.039  
 $wR$  factor = 0.116  
Data-to-parameter ratio = 17.0For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The crystal packing in the title compound,  $\text{C}_{10}\text{H}_{14}\text{O}_5$ , is determined by various weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds that result in parallel sheets of molecules stacked along  $[010]$ .Received 23 October 2002  
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## Comment

The title compound, (I), is interesting due to its high functionality. It can be used as a polymerizing agent, not only on its own, but also as a crosslinker. Although (I) has been well known for several years (Drewes *et al.*, 1987), this is the first crystallographic determination.

The molecular structure of (I) shows non-crystallographic  $C_{2v}$  symmetry, with atom O3 as its centre. The molecule is almost planar, with maximum deviations of  $-0.158(1)\text{ \AA}$  for O4 and  $0.120(1)\text{ \AA}$  for O3 from the mean plane of all the atoms. The torsion angles  $\text{C4}-\text{C3}-\text{C7}-\text{C8}$  and  $\text{O2}-\text{C2}-\text{C9}-\text{O4}$  have values of  $2.6(1)$  and  $9.4(1)^\circ$ , respectively. Bond lengths and angles are in good agreement with those reported for related structures (Rohrer *et al.*, 1984; Steurer & Podlech, 1999). The crystal structure shows complex patterns of weak hydrogen bonding (Fig. 2). The  $\text{C1}-\text{H1B}\cdots\text{O4}(x, y, z+1)$  bridges of  $2.44\text{ \AA}$  (angle at H of  $160^\circ$ ) lead to infinite chains of molecules along  $[001]$ . Additional  $\text{C4}-\text{H4B}\cdots\text{O2}(x-1, y, z)$  bridges of  $2.53\text{ \AA}$  (angle at H of  $138^\circ$ ) in the  $[100]$  direction link these chains into sheets, which are then stacked parallel along  $[010]$  with  $\text{C5}-\text{H5B}\cdots\text{O2}(-x+2, -y, -z+2)$  and  $\text{C10}-\text{H10B}\cdots\text{O5}(-x, -y+1, -z+1)$  contacts of  $2.56\text{ \AA}$  (angle at H of  $152^\circ$ ) and  $2.60\text{ \AA}$  (angle at H of  $142^\circ$ ), respectively. All these geometric parameters are normalized for  $\text{C}-\text{H} = 1.08\text{ \AA}$ .

## Experimental

The compound was prepared according to a method already described in the literature (Drewes *et al.*, 1987). After evaporation of the solvent, fine crystal plate were obtained and analysed *via* X-ray analysis. Furthermore NMR spectra were recorded on a Bruker AMX 300.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ , p.p.m.):  $\delta = 3.77$  (*s*, 6H,  $2 \times \text{OCH}_3$ ), 4.55 (*m*, 4H,  $2 \times \text{CH}_2$ ), 5.90 and 6.33 (*m*, 4H,  $2 \times =\text{CH}_2$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 51.67$  (*q*,  $2 \times \text{OCH}_3$ ), 68.65 (*t*,  $2 \times \text{CH}_2$ ), 125.89 (*t*,  $2 \times \text{CH}_2$ ), 136.63 (*s*,  $2 \times =\text{C}$ ), 166.03 (*s*,  $2 \times \text{C}=\text{O}$ ).

Crystal data

$C_{10}H_{14}O_5$   
 $M_r = 214.21$   
 Triclinic,  $P\bar{1}$   
 $a = 6.8104$  (13) Å  
 $b = 7.1777$  (14) Å  
 $c = 12.458$  (3) Å  
 $\alpha = 74.713$  (3)°  
 $\beta = 78.365$  (4)°  
 $\gamma = 67.939$  (3)°  
 $V = 540.84$  (18) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.315$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 1575 reflections  
 $\theta = 3.1$ – $28.2$ °  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 153$  (2) K  
 Plate, colorless  
 $0.50 \times 0.50 \times 0.10$  mm

Data collection

SMART APEX CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.889$ ,  $T_{\max} = 0.931$   
 3269 measured reflections

2342 independent reflections  
 1880 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$   
 $\theta_{\text{max}} = 28.3$ °  
 $h = -7 \rightarrow 8$   
 $k = -9 \rightarrow 9$   
 $l = -14 \rightarrow 15$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.116$   
 $S = 1.05$   
 2342 reflections  
 138 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.24$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

O1—C2	1.3447 (13)	O5—C10	1.4549 (14)
O1—C1	1.4524 (13)	C2—C3	1.4911 (14)
O2—C2	1.2151 (13)	C3—C4	1.3293 (15)
O3—C6	1.4212 (13)	C3—C5	1.5016 (15)
O3—C5	1.4230 (13)	C6—C7	1.5023 (15)
O4—C9	1.2093 (14)	C7—C8	1.3308 (16)
O5—C9	1.3416 (13)	C7—C9	1.4949 (15)
C2—O1—C1	115.15 (9)	O3—C5—C3	110.04 (8)
C6—O3—C5	110.40 (8)	O3—C6—C7	109.70 (8)
C9—O5—C10	115.18 (9)	C8—C7—C9	122.51 (10)
O2—C2—O1	123.04 (10)	C8—C7—C6	124.25 (10)
O2—C2—C3	123.25 (10)	C9—C7—C6	113.22 (9)
O1—C2—C3	113.71 (9)	O4—C9—O5	123.20 (10)
C4—C3—C2	122.70 (10)	O4—C9—C7	123.12 (10)
C4—C3—C5	124.47 (10)	O5—C9—C7	113.68 (9)
C2—C3—C5	112.82 (9)		

All H atoms were included in calculated positions, with C—H distances of 0.95 (for  $sp^2$  H atoms), 0.99 (for  $sp^3$  H atoms) and 0.98 Å (for methyl  $sp^3$  H atoms). The H atoms were then included in the

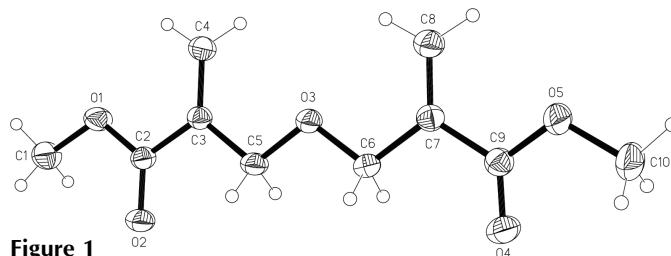


Figure 1 The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

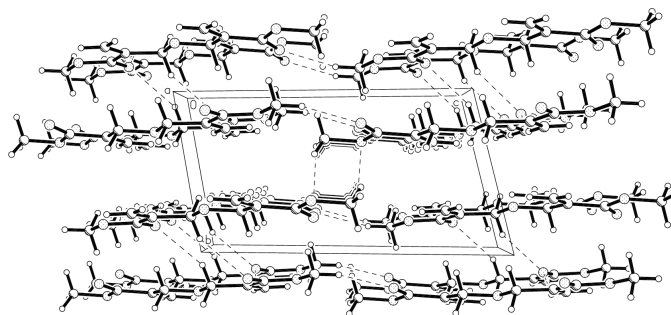


Figure 2 Packing diagram, viewed along [100]. Hydrogen bonding is indicated by dashed lines.

refinement, riding on their parent atoms, with  $U_{\text{iso}} = 1.2U_{\text{eq}}$  (or  $1.5U_{\text{eq}}$  for methyl H atoms).

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 2000); program(s) used to solve structure: SHELXTL (Bruker, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

References

- Bruker (1997). SMART. Version 5.625. Bruker AXS GmbH, Karlsruhe, Germany.  
 Bruker (1998). SHELXTL. Version 5.1. Bruker AXS GmbH, Karlsruhe, Germany.  
 Bruker (2000). SAINT. Version 6.02a. Bruker AXS GmbH, Karlsruhe, Germany.  
 Drewes, S., Loizou, G. & Roos, G. (1987). Synth. Commun. **17**, 291–298.  
 Rohrer, D. C., Kihara, M., Detto, T., Rathore, H., Ahmed, K., From, A. H. L. & Fullerton, D. S. (1984). J. Am. Chem. Soc. **106**, 8269–8276.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Steurer, S. & Podlech, J. (1999). Eur. J. Org. Chem. pp. 1551–1560.