

Lin-Hai Jing,<sup>a\*</sup> Da-Bin Qin,<sup>a</sup>  
Shao-Jin Gu,<sup>a</sup> Huan-Xia Zhang<sup>a</sup>  
and Zhi-Hua Mao<sup>b</sup><sup>a</sup>Department of Chemistry, China West Normal University, Nanchong 637002, People's Republic of China, and <sup>b</sup>Centre for Testing and Analysis, Sichuan University, Chengdu 610064, People's Republic of China

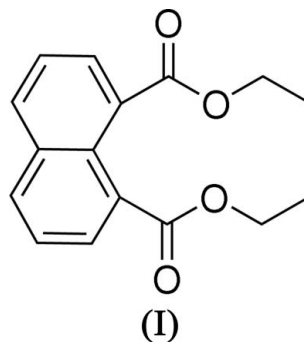
Correspondence e-mail: jlhxg@yahoo.com.cn

**Key indicators**Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
Disorder in main residue  
 $R$  factor = 0.041  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 13.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**Diethyl naphthalene-1,8-dicarboxylate**

The title compound,  $\text{C}_{16}\text{H}_{16}\text{O}_4$ , crystallizes with two molecules in the asymmetric unit. The two molecules differ in the orientations of the ethyl groups. The crystal packing is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

Received 3 April 2006  
Accepted 17 April 2006**Comment**

1,8-Naphthalenedicarboxylic acid derivatives are a class of intermediates important for applications as monomers in the preparation of polymers (Nakayama *et al.*, 2001; Isoda & Yamada, 1995, 1996) or as sludge inhibitors (Raymond *et al.*, 1968). Previously, we have reported the crystal structure of dimethyl naphthalene-1,8-dicarboxylate (Jing *et al.*, 2005). We now report the crystal structure of the title compound, (I).



The bond lengths and angles in (I) are normal. Compound (I) crystallizes in the monoclinic space group  $P2_1/c$  with  $Z' = 2$ , and the two molecules in the asymmetric unit differ in the orientations of the ethyl groups (Fig. 1). In both independent molecules, the naphthalene ring system is slightly distorted from planarity. The dihedral angle between the  $\text{C}1-\text{C}4/\text{C}9/\text{C}10$  and  $\text{C}5-\text{C}10$  planes is  $5.5(1)^\circ$  and that between the  $\text{C}17-\text{C}20/\text{C}25/\text{C}26$  and  $\text{C}21-\text{C}26$  planes is  $5.2(1)^\circ$ . As a result of steric effects, the substituent groups are twisted away from the planes of the naphthalene ring systems (Fig. 1). The dihedral angles formed between various planes are as follows:  $\text{C}1-\text{C}4/\text{C}9/\text{C}10$  and  $\text{O}1/\text{O}2/\text{C}11/\text{C}12$   $46.1(4)^\circ$ ,  $\text{C}5-\text{C}10$  and  $\text{O}3/\text{O}4/\text{C}14/\text{C}15$   $39.9(1)^\circ$ ,  $\text{C}17-\text{C}20/\text{C}25/\text{C}26$  and  $\text{O}5/\text{O}6/\text{C}27/\text{C}28'$   $38.5(2)^\circ$ , and  $\text{C}21-\text{C}26$  and  $\text{O}7/\text{O}8/\text{C}30/\text{C}31$   $45.7(1)^\circ$ . The crystal packing is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds (Table 1).

**Experimental**

Compound (I) was prepared according to the reported procedure of Errera (1911). Colourless single crystals suitable for X-ray diffraction were obtained by recrystallization from ethanol.

## Crystal data

$C_{16}H_{16}O_4$   
 $M_r = 272.29$   
 Monoclinic,  $P2_1/c$   
 $a = 10.210 (2) \text{ \AA}$   
 $b = 9.672 (1) \text{ \AA}$   
 $c = 29.163 (4) \text{ \AA}$   
 $\beta = 90.15 (1)^\circ$   
 $V = 2879.9 (8) \text{ \AA}^3$

$Z = 8$   
 $D_x = 1.256 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09 \text{ mm}^{-1}$   
 $T = 295 (2) \text{ K}$   
 Block, colourless  
 $0.58 \times 0.46 \times 0.46 \text{ mm}$

## Data collection

Bruker P4 diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 5684 measured reflections  
 5365 independent reflections  
 2323 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.010$   
 $\theta_{\text{max}} = 25.5^\circ$   
 3 standard reflections  
 every 97 reflections  
 intensity decay: 6.2%

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.106$   
 $S = 0.80$   
 5365 reflections  
 406 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 (Sheldrick, 1997)  
 Extinction coefficient: 0.0087 (6)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C15-H15A\cdots O4$	0.97	2.32	2.679 (3)	101
$C18-H18\cdots O4^i$	0.93	2.44	3.307 (3)	155
$C20-H20\cdots O4^{ii}$	0.93	2.58	3.440 (3)	154
$C23-H23\cdots O8^{iii}$	0.93	2.52	3.312 (3)	142

Symmetry codes: (i)  $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (ii)  $x - 1, y, z$ ; (iii)  $-x, -y + 2, -z$ .

The ethyl groups on atoms O1 and O5 are each disordered over two different orientations, with site-occupancy factors of 0.598 (14) and 0.637 (16), respectively, for the major conformations. The disorder was modelled with O—C and C—C distance restraints of 1.47 (1) and 1.53 (1)  $\text{\AA}$ , respectively. The  $U_{ij}$  components of atom C29' were approximated to isotropic behaviour. The H atoms were placed in calculated positions, with C—H = 0.93–0.97  $\text{\AA}$ , and included in the final cycles of refinement using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

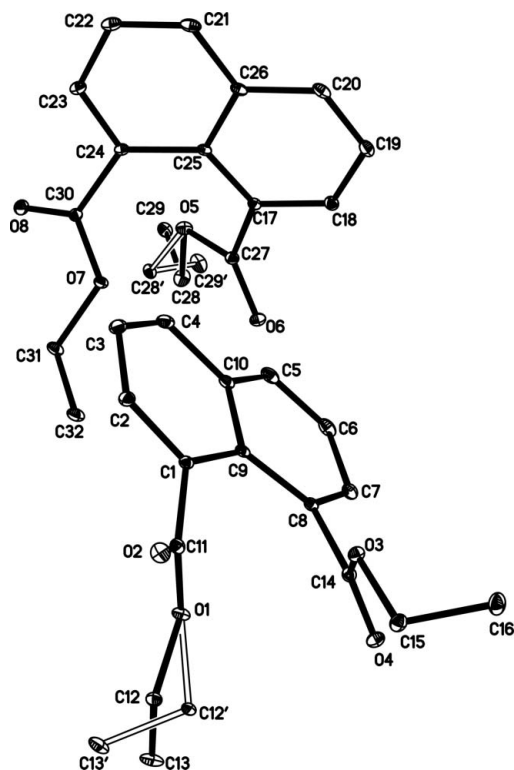


Figure 1

The structure of the asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atomic numbering. H atoms have been omitted for clarity. Both the major and minor components of the disordered ethyl groups are shown.

The authors thank the Centre for Testing and Analysis, Sichuan University, for financial support.

## References

- Errera, G. (1911). *Gazz. Chim. Ital.* **41**, 190–197.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Isoda, H. & Yamada, Y. (1995). Jpn Patent JP 07 197 366.  
 Isoda, H. & Yamada, Y. (1996). Jpn Patent JP 08 024 068.  
 Jing, L.-H., Qin, D.-B., He, L., Gu, S.-J., Zhang, H.-X. & Lei, G. (2005). *Acta Cryst. E* **61**, o3595–o3596.  
 Nakayama, S., Ide, N., Gyobu, S. & Kuwata, M. (2001). Jpn. Patent JP 2001 172 411.  
 Raymond, R. L., Douros, J. D. & Melchiorre, J. J. (1968). US Patent No. 3 361 545.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
 Siemens (1996). *XSCANS*. Version 2.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.