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## Key indicators

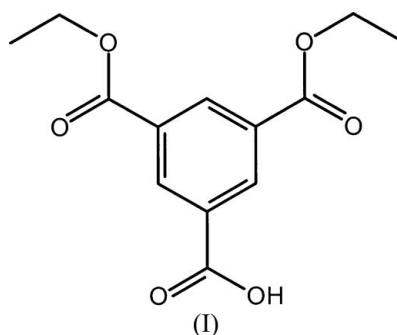
Single-crystal X-ray study  
 $T = 110$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.055  
 $wR$  factor = 0.168  
Data-to-parameter ratio = 16.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Supramolecular assembly of diethyl  
5-carboxybenzene-1,3-dicarboxylate

In the crystal structure of the title compound,  $\text{C}_{13}\text{H}_{14}\text{O}_6$ , hydrogen-bonded dimers are held together by the cyclic  $(\text{COOH})_2$  synthon. The planar dimers are arranged in layers *via* additional  $\text{C}-\text{H}\cdots\text{O}$  interactions. Tight offset-stacking of such layers characterizes the crystal packing.

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## Comment

The aldehyde analogue of the title compound, (I), can be utilized in the preparation of functionalized porphyrin derivatives suitable for supramolecular self-assembly (Goldberg, 2005, and references therein), such as, for example, tetrakis(*meso*-3,5-dicarboxyphenyl)porphyrin. As (I) had not been analysed before by X-ray diffraction, we report here its structure determined precisely at *ca* 110 K with relatively high resolution ( $d = 0.70$  Å), focusing also on its interesting supramolecular aggregation. This precise analysis also allowed the refinement of the H-atom parameters. The molecular structure of (I) is shown in Fig. 1.



The self-assembly of the molecular species into dimeric entities by the cyclic  $(\text{COOH})_2$  hydrogen-bonding synthon

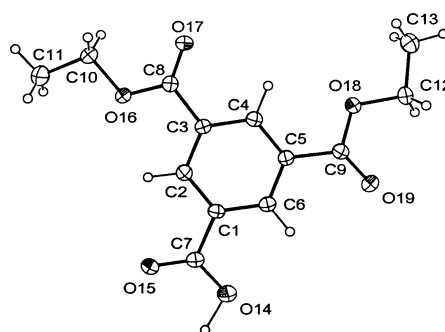


Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Ellipsoids represent displacement parameters at the 50% probability level at *ca* 110 K.

around the inversion centre at  $(\frac{1}{2}, 1, \frac{1}{2})$ , which is abundant in the crystal structures of carboxylic acids (CSD, Version 5.27, August 2006 update; Allen, 2002), is depicted in Fig. 2. This Figure shows also that the planar dimers assemble further into layers by weak  $C-H \cdots O=C$  hydrogen bonds between molecules related by the inversion centre at  $(0, \frac{1}{2}, \frac{1}{2})$  (Table 1). Both types of hydrogen bonds [graph-set representations  $R_2^2(8)$  and  $R_2^2(10)$ , respectively] are characterized by a nearly linear geometry. The so-formed supramolecular layers are aligned nearly parallel to the  $(1\bar{1}3)$  plane of the crystal lattice. They are stacked in the crystal structure along the vertical direction in an offset manner (Fig. 3). The tight organization should be attributed to strong dispersive interactions between the polarized surfaces of adjacent layers, and specific contacts between negatively charged and electron-deficient sites (Fig. 3).

## Experimental

Compound (I) is commercially available from Aldrich. Single crystals of (I) were obtained by slow evaporation of a dichloromethane solution.

### Crystal data

$C_{13}H_{14}O_6$	$V = 633.81 (5) \text{ \AA}^3$
$M_r = 266.24$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.395 \text{ Mg m}^{-3}$
$a = 4.5368 (2) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.7315 (4) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 14.5803 (6) \text{ \AA}$	$T = 110 (2) \text{ K}$
$\alpha = 89.3231 (13)^\circ$	Block, colourless
$\beta = 84.7769 (14)^\circ$	$0.45 \times 0.30 \times 0.15 \text{ mm}$
$\gamma = 81.383 (2)^\circ$	

### Data collection

Nonius KappaCCD diffractometer	3794 independent reflections
$\varphi$ and $\omega$ scans	2600 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.029$
7439 measured reflections	$\theta_{\text{max}} = 30.5^\circ$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2 + 0.0936P]$
$R[F^2 > 2\sigma(F^2)] = 0.055$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.168$	$(\Delta/\sigma)_{\text{max}} = 0.011$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.46 \text{ e \AA}^{-3}$
3794 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
228 parameters	
All H-atom parameters refined	

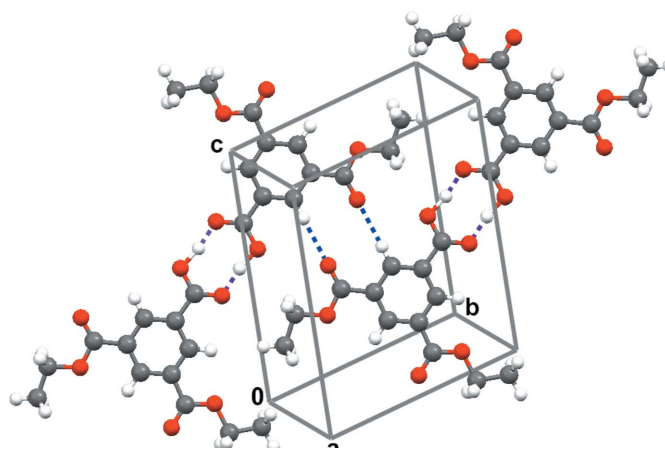
**Table 1**

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O14-H14 \cdots O15^i$	0.98 (1)	1.67 (1)	2.637 (2)	171 (3)
$C6-H6 \cdots O19^{ii}$	0.99 (2)	2.40 (2)	3.378 (2)	169 (2)

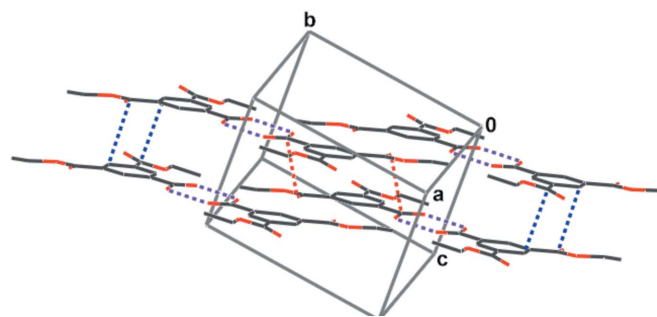
Symmetry codes: (i)  $-x + 1, -y + 2, -z + 1$ ; (ii)  $-x, -y + 1, -z + 1$ .

Initially, the H atoms bound to carbon were placed in calculated positions and constrained to ride on their parent atoms. The H atom of the carboxyl group was located in a difference Fourier map. In the final stages of the least-squares refinement, the coordinates and the



**Figure 2**

Illustration of the hydrogen-bonding interactions within the layered arrangement of (I). The  $\text{COOH} \cdots \text{COOH}$  bonds are indicated by purple dotted lines; the  $\text{C-H} \cdots \text{O}=\text{C}$  bonds are denoted by blue dotted lines (Table 1).



**Figure 3**

The crystal packing of (I), showing two adjacent molecular layers. The  $(\text{COOH})_2$  hydrogen bonding within the layers is indicated by dotted purple lines. The blue and red dotted lines depict selected dispersive interactions between the layers. The blue lines relate to the  $\text{C}4 \cdots \text{C}8(x-1, y, z)$  non-bonding contact at  $3.371 (2) \text{ \AA}$  and its symmetry equivalents. The red lines represent characteristic  $\text{O}(\delta^-) \cdots \text{C}(\delta^+) = \text{O}(\delta^-)$  interactions (Goldberg *et al.*, 1982; Allen, 2002) at  $\text{O}19 \cdots \text{C}7(1-x, 1-y, 1-z) = 3.143 (2) \text{ \AA}$  indicated by dotted blue lines. H atoms have been omitted for clarity.

displacement parameters of all H atoms were refined freely, with a single restraint that the  $\text{O}14-H14$  bond length to a target value of  $0.95 (1) \text{ \AA}$ .

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *MERCURY* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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