

Ye-Fei Nan, Yu-Ping Wei,
Xiao-Xiao Li, Chang-Hua Hua
and Liang-Dong Sun*Department of Chemistry, College of Sciences,
Tianjin University, Tianjin 300072, People's
Republic of China

Correspondence e-mail: nyfkm@hotmail.com

Key indicatorsSingle-crystal X-ray study
 $T = 133$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.037
 wR factor = 0.094
Data-to-parameter ratio = 10.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Diethyl diprop-2-ynylmalonate**

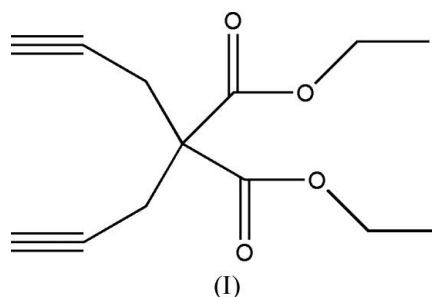
In the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_4$, the two ester groups adopt *syn*- and *anti*-conformations. The two carbonyl O atoms are acceptors of acetylene H atoms in $\text{C}-\text{H}\cdots\text{O}$ interactions which, together with van der Waals contacts, determine the crystal packing.

Received 30 May 2006

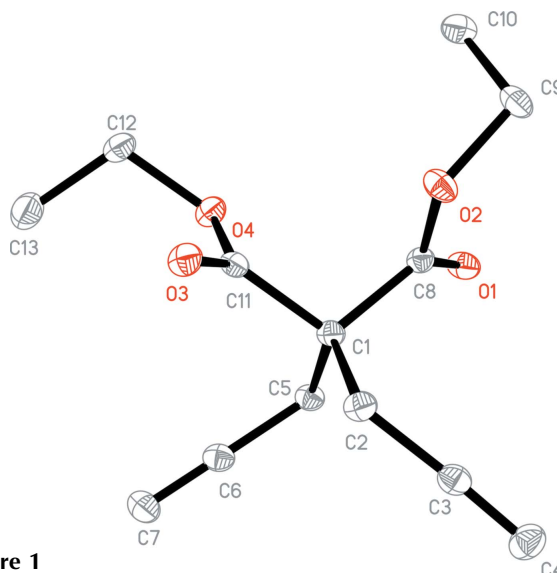
Accepted 3 July 2006

Comment

The synthesis of the title compound, (I), has already been described (Bhatarah & Smith, 1990). This compound is useful in synthesis as a precursor due to its diyne structure. The molecular structure of (I) with the atom-numbering scheme is shown in Fig. 1.



The crystal packing of (I) is determined by van der Waals contacts and $\text{C}-\text{H}\cdots\text{O}$ interactions involving acetylene H atoms (Fig. 2 and Table 1).

**Figure 1**

The molecular structure of (I), with the atom-numbering scheme, showing displacement ellipsoids at the 30% probability level. H atoms have been omitted.

Experimental

To a solution of sodium ethoxide [sodium (1.15 g, 0.05 mol) in dry ethanol (50 ml)] under nitrogen was added diethyl malonate (4.035 g, 0.025 mol) over a period of 10 min. After the solution had been stirred for 30 min at room temperature, prop-2-ynyl bromide (6 g, 0.05 mol) was added dropwise, maintaining the temperature at 299–301 K. A yellow suspension was formed and heated under reflux for 2 h. The reaction mixture was cooled to room temperature and water (50 ml) was added. The ethanol was removed and the diyne was then extracted with diethyl ether. The extracts were dried, filtered, and concentrated to give a yellow oil. Distillation gave pure (I) as a white solid (yield 84%, m.p. 315 K). Single crystals of the title compound were obtained by slow evaporation of petrol oil–diethyl ether (10:1 v/v) solution.

Crystal data

$C_{13}H_{16}O_4$ $Z = 4$
 $M_r = 236.26$ $D_x = 1.205 \text{ Mg m}^{-3}$
 Orthorhombic, $Pna2_1$ Mo $K\alpha$ radiation
 $a = 17.695 (4) \text{ \AA}$ $\mu = 0.09 \text{ mm}^{-1}$
 $b = 8.3610 (9) \text{ \AA}$ $T = 133 (2) \text{ K}$
 $c = 8.8050 (12) \text{ \AA}$ Block, colourless
 $V = 1302.7 (4) \text{ \AA}^3$ $0.24 \times 0.16 \times 0.14 \text{ mm}$

Data collection

Rigaku Saturn diffractometer 5303 measured reflections
 ω scans 1572 independent reflections
 Absorption correction: multi-scan 1448 reflections with $I > 2\sigma(I)$
 (Jacobson, 1998) $R_{\text{int}} = 0.055$
 $T_{\text{min}} = 0.979, T_{\text{max}} = 0.988$ $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2 H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.037$ $w = 1/[\sigma^2(F_o^2) + (0.0632P)^2]$
 $wR(F^2) = 0.094$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.01$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 1572 reflections $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 155 parameters $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C4-H4 \cdots O3^i$	0.95	2.46	3.184 (3)	133
$C7-H7 \cdots O1^{ii}$	0.95	2.29	3.214 (2)	163
$C9-H9B \cdots O3^{iii}$	0.99	2.60	3.509 (3)	153
$C12-H12A \cdots O1^{iv}$	0.99	2.58	3.480 (2)	151

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

All H atoms were initially located in a difference Fourier map, and were then constrained to an ideal geometry, with $C-H = 0.95-0.99 \text{ \AA}$, and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

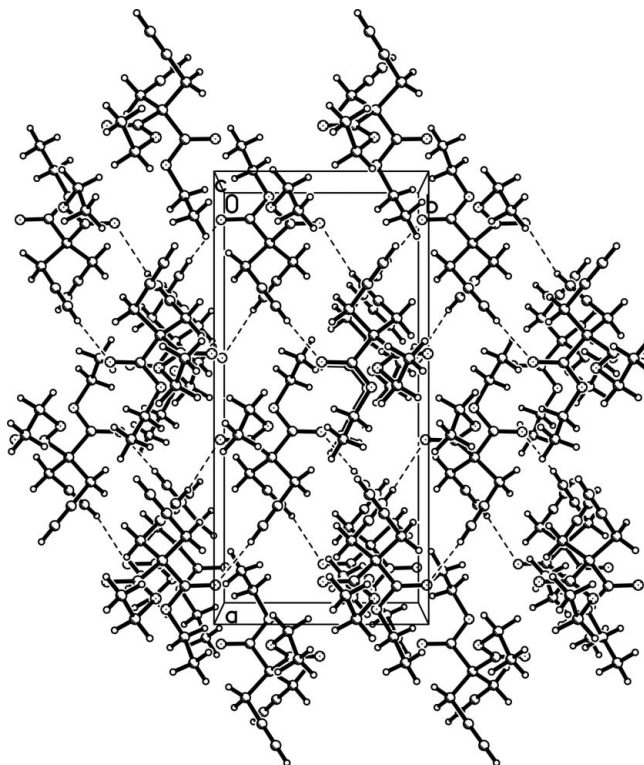


Figure 2 The molecular packing of (I), viewed along the c axis. Dashed lines indicate the intermolecular $C-H \cdots O$ contacts involving the acetylene H atom.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MS, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure*.

References

Bhatarah, P. & Smith, E. H. (1990). *J. Chem. Soc. Perkin Trans. 1*, pp. 2603–2606.
 Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Jacobson, R. (1998). *REQAB*. Private communication to Rigaku Corporation, Tokyo, Japan.
 Molecular Structure Corporation & Rigaku (1999). *CrystalClear*. Version 1.3.6. MSC, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2005). *CrystalStructure*. Version 3.7.0. Rigaku/MS, The Woodlands, Texas, USA.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.