

9,9-Bis(ethoxycarbonyl)ethyl)-9H-fluorene

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Key indicators

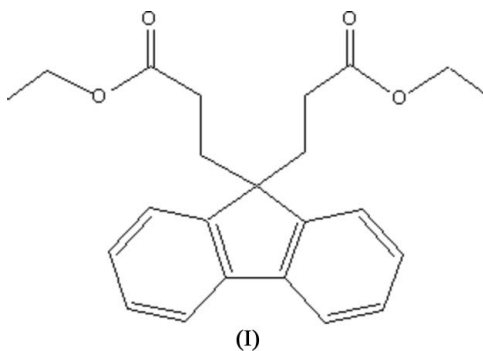
Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.060
 wR factor = 0.172
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound [systematic name: diethyl 3,3'-(9H-fluorene-9,9-diyl)dipropionate], $\text{C}_{23}\text{H}_{26}\text{O}_4$, the fluorene ring system is planar. The ethoxycarbonyl ethyl groups display extended conformations and are nearly perpendicular to the fluorene plane. A weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interaction occurs in the crystal structure.

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Comment

Fluorene and its polymeric derivatives have been used as laser-generating (Bazyl, 1986) or photo-active fluorescent materials (Johansson *et al.*, 2001). However, insolubility is a common problem for fluorene polymers. Appropriate substitution of the fluorene ring system may modify the solubility of fluorene polymers (Redecker *et al.*, 1999). We present here the crystal structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1. The fluorene ring system is planar. The ethoxycarbonyl ethyl groups display extended conformations with a normal geometry (Table 1), and are nearly perpendicular to the fluorene plane. Weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions occurs between neighboring molecules (Table 2 and Fig. 2).

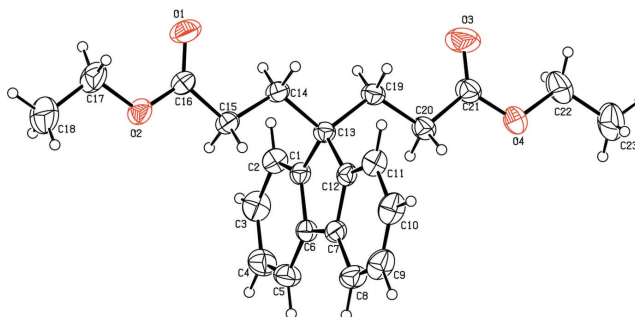


Figure 1

The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Experimental

A 50 ml three-necked round-bottomed flask was charged with fluorene (4 g) and ethyl acrylate (25 ml), and then heated to 308 K. A methanol solution (1 ml) of benzyltrimethylammonium methoxide (40% in methanol) was added rapidly dropwise to the stirred solution, raising the temperature to 362 K. Excess ethyl acrylate was removed by distillation to a temperature of 363 K at a pressure of 8 mm Hg, then cooling to 263 K, and the residue was removed by filtration, and air-dried, yielding a white solid. Recrystallization from methanol gave (I) in 53% yield. Single crystals of (I) were obtained by slow evaporation of methanol solution at room temperature (m.p. 377–378 K). IR (KBr, ν cm^{-1}): 3034 (Ar–H), 2950 (C–H), 1736 (C=O), 1453 (C=C), 1187, 1114 (C–O–C); ^1H NMR (CDCl_3): δ 7.71–7.29 (m, 8H), 3.94–3.88 (q, 4H), 2.43–2.39 (t, 4H), 1.57–1.52 (t, 4H), 1.11–1.08 (t, 6H). Elemental analysis calculated for $\text{C}_{23}\text{H}_{26}\text{O}_4$: C 75.38, H 7.15%; found: C 75.51, H 7.32%.

Crystal data

$\text{C}_{23}\text{H}_{26}\text{O}_4$ Mo $K\alpha$ radiation
 $M_r = 366.44$ Cell parameters from 6837 reflections
 Orthorhombic, *Pbca* $\theta = 2.4$ – 20.7°
 $a = 10.8464$ (7) \AA $\mu = 0.08$ mm^{-1}
 $b = 16.9377$ (11) \AA $T = 292$ (2) K
 $c = 22.2437$ (14) \AA Block, colorless
 $V = 4086.5$ (5) \AA^3 $0.30 \times 0.20 \times 0.10$ mm
 $Z = 8$
 $D_x = 1.191$ Mg m^{-3}

Data collection

Bruker SMART CCD area-detector 2931 reflections with $I > 2\sigma(I)$
 diffractometer $R_{\text{int}} = 0.072$
 φ and ω scans $\theta_{\text{max}} = 26.0^\circ$
 Absorption correction: none $h = -13 \rightarrow 13$
 40279 measured reflections $k = -20 \rightarrow 20$
 4005 independent reflections $l = -27 \rightarrow 27$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0954P)^2 + 0.5495P]$
 $R[F^2 > 2\sigma(F^2)] = 0.060$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.172$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 $S = 1.02$ $\Delta\rho_{\text{max}} = 0.32$ e \AA^{-3}
 4005 reflections $\Delta\rho_{\text{min}} = -0.21$ e \AA^{-3}
 246 parameters
 H-atom parameters constrained

Table 1

Selected geometric parameters (\AA , $^\circ$).

C1–C13	1.519 (3)	C17–O2	1.451 (2)
C12–C13	1.526 (3)	C21–O3	1.194 (3)
C16–O1	1.194 (2)	C21–O4	1.324 (3)
C16–O2	1.324 (2)	C22–O4	1.456 (3)
C13–C14–C15–C16	179.02 (16)	C19–C20–C21–O4	172.54 (18)
C14–C15–C16–O2	179.46 (17)	C18–C17–O2–C16	165.8 (2)
C13–C19–C20–C21	175.83 (18)	C23–C22–O4–C21	171.1 (2)

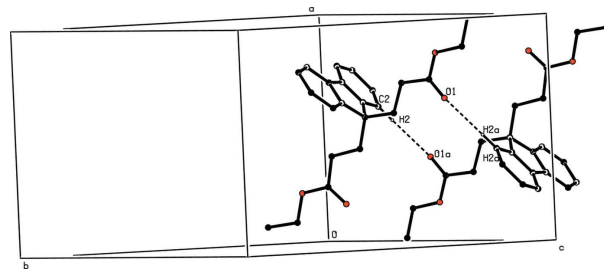


Figure 2

Partial packing diagram of (I) showing the weak C–H...O hydrogen bonds (dashed lines) [symmetry code: (a) $1 - x, -y, 1 - z$].

Table 2

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

D–H...A	D–H	H...A	D...A	D–H...A
C2–H2...O1 ⁱ	0.93	2.57	3.485 (3)	169

Symmetry code: (i) $-x + 1, -y, -z + 1$.

Methyl H atoms were placed in calculated positions, with C–H = 0.96 \AA , and refined to fit the electron density [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$]. Other H atoms were placed in calculated positions, with C–H = 0.93 (aromatic) or 0.97 \AA (methylene), and refined in riding mode [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2001); software used to prepare material for publication: SHELXTL.

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