

4-Cyano-3-fluorophenyl (2*E*)-3-(3-methoxy-4-pentyloxyphenyl)acrylateRui Ren, Xue-Mei Li, Qiang Li  
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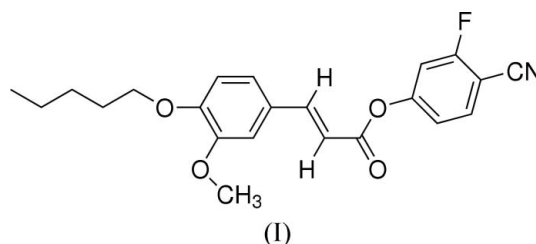
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
Disorder in main residue  
 $R$  factor = 0.058  
 $wR$  factor = 0.173  
Data-to-parameter ratio = 14.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The two molecules in the asymmetric unit of the title compound,  $\text{C}_{22}\text{H}_{22}\text{NO}_4\text{F}$ , differ in the orientation of the 4-cyano-3-fluorobenzene ring with respect to the acrylate linkage. In one of the molecules, the pentyloxy chain has an extended conformation and in the other it is folded. The molecules are linked into a three-dimensional framework by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{F}$  hydrogen bonds, and by  $\pi-\pi$  interactions.

## Comment

The nematic isotropic transition temperature of compounds containing a fluoro atom at the *ortho*- or *meta*- position with respect to the cyano group on a phenyl ring are only slightly lower than the corresponding esters with an F atom (Kelly *et al.*, 1984). However, the viscosity and driving voltage needed for the new esters are remarkably higher than those of the analogous non-fluoro-substituted phenyl esters. In order to obtain liquid-crystal materials with excellent properties (Sung *et al.*, 2004), the title compound, (I), was synthesized. We report here the crystal structure of (I).



The asymmetric unit of (I) contains two crystallographically independent molecules. The corresponding bond lengths and angles of these two molecules agree with each other (Table 1) but they differ in the orientation of the 4-cyano-3-fluorobenzene ring with respect to the acrylate linkage (Fig. 1). The dihedral angle between the  $\text{C}2-\text{C}7$  and  $\text{O}1/\text{O}2/\text{C}8-\text{C}10$  planes is  $74.93$  ( $9^\circ$ ) and that between the  $\text{C}24-\text{C}29$  and  $\text{O}5/\text{O}6/\text{C}30-\text{C}32$  planes is  $68.28$  ( $8^\circ$ ). Also, the torsion angles  $\text{O}3-\text{C}17-\text{C}18-\text{C}19$  [ $177.8$  ( $2^\circ$ )] and  $\text{O}7-\text{C}39-\text{C}40-\text{C}41$  [ $60.7$  ( $3^\circ$ )] indicate that in one of the molecules the pentyloxy chain is in an extended conformation and in the other it is folded. In both molecules, the methoxybenzene ring is nearly coplanar with the acrylate linkage. The torsion angles  $\text{C}8-\text{C}9-\text{C}10-\text{C}11$  [ $-178.5$  ( $3^\circ$ )] and  $\text{C}30-\text{C}31-\text{C}32-\text{C}33$  [ $176.9$  ( $2^\circ$ )] indicate the *trans* configuration of the double bonds.

In the crystal structure, molecules are linked into a three-dimensional framework by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{F}$

hydrogen bonds (Table 2). The packing is further stabilized by  $\pi$ - $\pi$  interactions involving the C2-C7 and C24-C29 benzene rings at  $(x, y, z)$  and  $(x - 1, y, 1 + z)$ , respectively, with a centroid-centroid distance of 3.788 (2) Å.

### Experimental

1-Bromopentane (4.53 g, 0.03 mol) in ethanol (30 ml) was added dropwise to a solution of 3-(4-hydroxy-3-methoxyphenyl)acrylic acid (1.94 g, 0.01 mol) in 17.0% KOH solution (10 ml). The mixture was refluxed for 24 h and extra KOH solution was added to remove the additional products. The mixture was then acidified to give a white precipitate, which was filtered off and recrystallized from ethanol to obtain 3-(4-pentyloxyphenyl)acrylic acid. 3-(4-Pentyloxyphenyl)acrylic acid (0.79 g) and 1,3-dicyclohexylcarbodiimide (0.62 g) were dissolved in tetrahydrofuran (THF, 20 ml) and a solution of 2-fluoro-4-hydroxybenzotrile (0.68 g) and 4-dimethylaminopyridine (0.01 g) in THF (20 ml) was added. The mixture was stirred at room temperature for 24 h and then filtered. Colourless single crystals of (I) suitable for X-ray diffraction study were obtained by slow evaporation of a THF/ethanol (1:4 v/v) solution over a period of 2 d.

#### Crystal data

$C_{22}H_{22}FNO_4$	$Z = 4$
$M_r = 383.41$	$D_x = 1.255 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 12.193 (4) \text{ \AA}$	Cell parameters from 1724 reflections
$b = 12.506 (4) \text{ \AA}$	$\theta = 2.3\text{--}22.6^\circ$
$c = 14.449 (5) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 82.348 (7)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 68.816 (6)^\circ$	Plate, colourless
$\gamma = 83.036 (7)^\circ$	$0.41 \times 0.18 \times 0.09 \text{ mm}$
$V = 2029.7 (12) \text{ \AA}^3$	

#### Data collection

Simens SMART 1000 CCD area-detector diffractometer	7827 independent reflections
$\omega$ scans	3944 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.020$
$T_{\text{min}} = 0.963, T_{\text{max}} = 0.992$	$\theta_{\text{max}} = 26.1^\circ$
11493 measured reflections	$h = -13 \rightarrow 15$
	$k = -15 \rightarrow 14$
	$l = -9 \rightarrow 17$

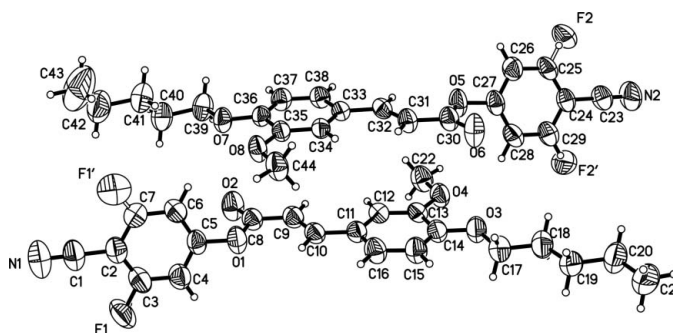
#### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.059$	$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2]$
$wR(F^2) = 0.173$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{\text{max}} = 0.001$
7827 reflections	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
524 parameters	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$

**Table 1**

Selected bond lengths (Å).

O1—C8	1.369 (3)	N2—C23	1.140 (3)
O1—C5	1.390 (3)	C1—C2	1.432 (4)
O2—C8	1.191 (3)	C8—C9	1.450 (3)
O5—C30	1.375 (3)	C9—C10	1.318 (3)
O5—C27	1.387 (3)	C23—C24	1.439 (4)
O6—C30	1.192 (3)	C30—C31	1.445 (3)
N1—C1	1.139 (4)	C31—C32	1.327 (3)



**Figure 1**

The two independent molecules of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The F atom in each molecule is disordered over two positions and both positions are shown.

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C28—H28 $\cdots$ O4	0.93	2.58	3.398 (3)	147
C31—H31 $\cdots$ O6 <sup>i</sup>	0.93	2.48	3.410 (3)	174
C37—H37 $\cdots$ O2 <sup>ii</sup>	0.93	2.39	3.246 (3)	153
C44—H44A $\cdots$ F2 <sup>iii</sup>	0.96	2.37	3.262 (4)	155

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

In both independent molecules, the F atom was found to be disordered over two positions, implying two different orientations for the 4-cyano-3-fluorobenzene ring. The occupancies of the disordered positions F1/F1' and F2/F2' were refined to 0.791 (4)/0.209 (4). The C—F distances were restrained to be equal and the  $U^{ij}$  components of the disordered atoms were approximated to isotropic behaviour. H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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