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Ethyl (*E*)-2-cyano-3-(4-methylphenyl)-acrylate: a second monoclinic polymorph

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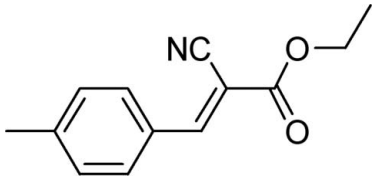
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.055; wR factor = 0.192; data-to-parameter ratio = 12.6.

The title compound, $C_{13}H_{13}NO_2$, was previously described in space group $P2_1/c$ by He *et al.* [*Acta Cryst.* (1993), C**49**, 2000–2002]. The ethyl group is disordered over two sets of sites in a 0.615 (10):0.385 (10) ratio. The C—O—C—C torsion angles containing the ethyl group are −111.6 (10) and 177.9 (7)°, while in the previously reported polymorph, the torsion angle is −167.3 (2)°. The molecules pack to form a three-dimensional structure in the *ABAB* style along the *c*-axis direction in the title compound, but parallel to the *a*-axis direction in the reported polymorph.

Related literature

For the first polymorph, see: He *et al.* (1993). For background to intramolecular charge-transfer molecules and their use in the construction of one- to three-dimesional organic nanostructures, see: Zhang *et al.* (2007); Zhang *et al.* (2008).



Experimental

Crystal data

$C_{13}H_{13}NO_2$
M_r = 215.24
Monoclinic, $P2_1/c$
a = 4.7616 (4) Å
b = 17.7989 (15) Å
c = 14.2841 (12) Å
β = 93.8021 (10)°
V = 1207.93 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan phi and omega scans
T_{min} = 0.984, T_{max} = 0.984
8359 measured reflections
2117 independent reflections
1617 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.192
S = 1.11
2117 reflections
168 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2081).

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Zhang, X. J., Zhang, X. H., Zou, K., Lee, C. S. & Lee, S. T. (2007). *J. Am. Chem. Soc.* **129**, 3527–3532.

supporting information

Acta Cryst. (2013). E**69**, o748 [https://doi.org/10.1107/S1600536813009550]

Ethyl (*E*)-2-cyano-3-(4-methylphenyl)acrylate: a second monoclinic polymorph

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S1. Comment

The title compound is a typical D–π–A (D = donor, A = acceptor) molecule. This type of compounds can be regarded as intramolecule charge transfer (ICT) molecules (Zhang *et al.*, 2007) and can be used to construct 1-dimesional to 3-dimesional organic nanostructures (Zhang *et al.*, 2008). In the title compound, the benzene cycle can be used as the electron donor unit, cyano group as the electron acceptor unit, the ester group as a flexible chain to increase the solubility of the compound. Thus, the cyano group and benzene cycle are linked by a vinyl bond to form organo-soluble D–π–A molecule.

In title compound (Fig. 1), the C═C group is almost coplanar with the attached phenyl ring, the torsion angle C9—C15—C5—C4 being 2.2 (4) °. The C═C group is also coplanar with ester group. The torsion angle C15—C9—Cl1—O2 is 177.5 (2) °. Excellent coplanarity of conjugated moieties enables the title compound to be a high delocalized electron system.

S2. Experimental

p-Toluic aldehyde (0.50 g), ethyl cyanoacetate(0.51 g), and ammonium acetate (0.32 g) were dissolved in 30 ml of ethanol and refluxed for about 4 h to give crude product as a solid. The precipitation was filtered, purified by recrystallization from acetonitrile/water (1:4) and 0.85 g of the titled compound was obtained as a white solid. Yield: 94.9%. ^{1}H NMR (400 MHz, d^{6}-DMSO): 8.35 (s, 1H), 7.98 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H), 7.32 (q, J = 7.2 Hz, 2H), 2,40 (s, 3H), 1.31 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz) 13.97, 21.33, 62.24, 101.14, 115.78, 128.68, 129.94, 130.96, 144.39, 154.94, 161.98.

S3. Refinement

All hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93—0.97 Å and U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

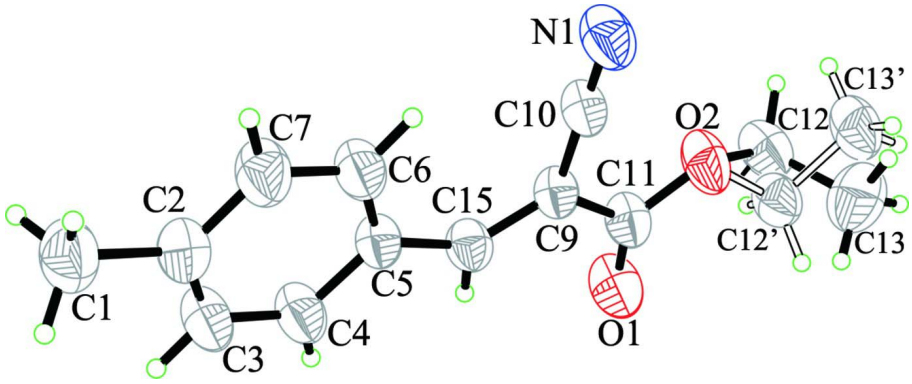

Figure 1

The molecular structure of the title molecule with 50% probability ellipsoids.

Ethyl (*E*)-2-cyano-3-(4-methylphenyl)acrylate

Crystal data

$C_{13}H_{13}NO_2$
$M_r = 215.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.7616\ (4)$ Å
$b = 17.7989\ (15)$ Å
$c = 14.2841\ (12)$ Å
$\beta = 93.8021\ (10)°$
$V = 1207.93\ (18)$ Å^3
$Z = 4$
$F(000) = 456$
$D_x = 1.184$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3950 reflections
$\theta = 2.3–25.5°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Needle, white
0.20 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan phi and omega scans
$T_{min} = 0.984$, $T_{max} = 0.984$
8359 measured reflections
2117 independent reflections
1617 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -5\rightarrow5$
$k = -21\rightarrow19$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.192$
$S = 1.11$
2117 reflections
168 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 0.1786P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.043 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2687 (8)	−0.0915 (2)	0.4400 (2)	0.1201 (10)	
H1A	0.1208	−0.1263	0.4219	0.180*	
H1B	0.2004	−0.0549	0.4823	0.180*	
H1C	0.4242	−0.1182	0.4705	0.180*	
C2	0.3650 (6)	−0.05220 (16)	0.35330 (19)	0.0954 (8)	
C3	0.2589 (7)	0.01687 (16)	0.3251 (2)	0.1062 (9)	
H3	0.1250	0.0398	0.3601	0.127*	
C4	0.3451 (6)	0.05290 (14)	0.24691 (19)	0.0924 (8)	
H4	0.2715	0.0999	0.2307	0.111*	
C5	0.5404 (5)	0.02009 (12)	0.19193 (16)	0.0740 (6)	
C6	0.6416 (6)	−0.05037 (14)	0.21981 (19)	0.0953 (8)	
H6	0.7713	−0.0744	0.1842	0.114*	
C7	0.5541 (7)	−0.08526 (15)	0.2988 (2)	0.1009 (9)	
H7	0.6256	−0.1324	0.3153	0.121*	
C9	0.6075 (5)	0.11983 (12)	0.06603 (15)	0.0713 (6)	
C10	0.4199 (5)	0.17670 (12)	0.09614 (16)	0.0777 (7)	
C11	0.7662 (5)	0.13621 (14)	−0.01789 (17)	0.0829 (7)	
C12	0.741 (3)	0.2242 (6)	−0.1547 (6)	0.109 (3)	0.385 (10)
H12A	0.5727	0.2456	−0.1863	0.131*	0.385 (10)
H12B	0.8083	0.1828	−0.1912	0.131*	0.385 (10)
C13	0.965 (3)	0.2823 (9)	−0.1321 (9)	0.136 (4)	0.385 (10)
H13A	0.8843	0.3244	−0.1017	0.204*	0.385 (10)
H13B	1.0425	0.2987	−0.1890	0.204*	0.385 (10)
H13C	1.1117	0.2608	−0.0911	0.204*	0.385 (10)
C15	0.6509 (5)	0.05304 (12)	0.10882 (16)	0.0755 (6)	
H15	0.7755	0.0220	0.0796	0.091*	
C12′	0.8890 (16)	0.2220 (4)	−0.1310 (5)	0.0961 (19)	0.615 (10)
H12C	1.0818	0.2271	−0.1053	0.115*	0.615 (10)
H12D	0.8819	0.1840	−0.1797	0.115*	0.615 (10)
C13′	0.780 (2)	0.2953 (3)	−0.1689 (5)	0.119 (2)	0.615 (10)
H13D	0.5833	0.2904	−0.1879	0.178*	0.615 (10)
H13E	0.8814	0.3094	−0.2220	0.178*	0.615 (10)
H13F	0.8040	0.3331	−0.1212	0.178*	0.615 (10)
N1	0.2704 (6)	0.22244 (13)	0.11968 (17)	0.1036 (8)	
O1	0.9398 (5)	0.09507 (11)	−0.04727 (14)	0.1101 (7)	

O2	0.6971 (4)	0.20214 (10)	−0.05566 (13)	0.1045 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.1351 (17)	0.1184 (17)	0.1087 (15)	−0.0055 (14)	0.0237 (13)	0.0261 (13)
C2	0.1080 (15)	0.0895 (14)	0.0898 (13)	−0.0069 (12)	0.0141 (12)	0.0126 (11)
C3	0.123 (2)	0.094 (2)	0.108 (2)	0.0117 (16)	0.0474 (17)	0.0159 (15)
C4	0.1092 (18)	0.0723 (15)	0.0990 (17)	0.0114 (13)	0.0320 (14)	0.0128 (13)
C5	0.0863 (14)	0.0615 (12)	0.0751 (13)	−0.0038 (10)	0.0114 (11)	0.0015 (10)
C6	0.120 (2)	0.0713 (15)	0.0974 (18)	0.0148 (13)	0.0271 (15)	0.0122 (13)
C7	0.131 (2)	0.0749 (16)	0.0985 (18)	0.0101 (14)	0.0183 (16)	0.0203 (14)
C9	0.0841 (13)	0.0612 (12)	0.0695 (12)	−0.0036 (10)	0.0120 (10)	−0.0005 (9)
C10	0.0975 (15)	0.0600 (12)	0.0769 (14)	−0.0010 (11)	0.0151 (11)	0.0059 (10)
C11	0.1033 (17)	0.0688 (14)	0.0783 (14)	0.0001 (12)	0.0191 (12)	0.0043 (11)
C12	0.134 (8)	0.096 (6)	0.099 (6)	0.016 (6)	0.017 (5)	0.014 (4)
C13	0.137 (8)	0.144 (10)	0.132 (8)	−0.025 (8)	0.044 (7)	−0.004 (7)
C15	0.0883 (14)	0.0631 (13)	0.0762 (13)	0.0012 (10)	0.0137 (11)	−0.0020 (10)
C12′	0.104 (4)	0.097 (4)	0.090 (4)	0.011 (4)	0.031 (3)	0.026 (3)
C13′	0.151 (6)	0.101 (4)	0.109 (4)	0.011 (4)	0.042 (4)	0.037 (3)
N1	0.1314 (19)	0.0732 (14)	0.1099 (17)	0.0159 (12)	0.0344 (14)	0.0056 (12)
O1	0.1454 (17)	0.0902 (13)	0.1006 (13)	0.0211 (11)	0.0524 (12)	0.0065 (10)
O2	0.1456 (16)	0.0791 (12)	0.0937 (13)	0.0125 (10)	0.0435 (11)	0.0220 (9)

Geometric parameters (Å, º)

C1—C2	1.519 (4)	C10—N1	1.147 (3)
C1—H1A	0.9600	C11—O1	1.201 (3)
C1—H1B	0.9600	C11—O2	1.324 (3)
C1—H1C	0.9600	C12—O2	1.496 (8)
C2—C7	1.363 (4)	C12—C13	1.507 (6)
C2—C3	1.379 (4)	C12—H12A	0.9700
C3—C4	1.374 (4)	C12—H12B	0.9700
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.385 (3)	C13—H13B	0.9600
C4—H4	0.9300	C13—H13C	0.9600
C5—C6	1.392 (3)	C15—H15	0.9300
C5—C15	1.453 (3)	C12′—C13′	1.492 (5)
C6—C7	1.376 (4)	C12′—O2	1.500 (5)
C6—H6	0.9300	C12′—H12C	0.9700
C7—H7	0.9300	C12′—H12D	0.9700
C9—C15	1.346 (3)	C13′—H13D	0.9600
C9—C10	1.435 (3)	C13′—H13E	0.9600
C9—C11	1.488 (3)	C13′—H13F	0.9600
C2—C1—H1A	109.5	N1—C10—C9	179.5 (3)
C2—C1—H1B	109.5	O1—C11—O2	123.8 (2)
H1A—C1—H1B	109.5	O1—C11—C9	124.1 (2)

C2—C1—H1C	109.5	O2—C11—C9	112.1 (2)
H1A—C1—H1C	109.5	O2—C12—C13	96.8 (8)
H1B—C1—H1C	109.5	O2—C12—H12A	112.4
C7—C2—C3	117.4 (2)	C13—C12—H12A	112.4
C7—C2—C1	120.9 (3)	O2—C12—H12B	112.4
C3—C2—C1	121.6 (3)	C13—C12—H12B	112.4
C4—C3—C2	122.0 (3)	H12A—C12—H12B	110.0
C4—C3—H3	119.0	C9—C15—C5	132.4 (2)
C2—C3—H3	119.0	C9—C15—H15	113.8
C3—C4—C5	120.8 (2)	C5—C15—H15	113.8
C3—C4—H4	119.6	C13′—C12′—O2	104.6 (4)
C5—C4—H4	119.6	C13′—C12′—H12C	110.8
C4—C5—C6	116.7 (2)	O2—C12′—H12C	110.8
C4—C5—C15	125.9 (2)	C13′—C12′—H12D	110.8
C6—C5—C15	117.4 (2)	O2—C12′—H12D	110.8
C7—C6—C5	121.5 (2)	H12C—C12′—H12D	108.9
C7—C6—H6	119.2	C12′—C13′—H13D	109.5
C5—C6—H6	119.2	C12′—C13′—H13E	109.5
C2—C7—C6	121.4 (3)	H13D—C13′—H13E	109.5
C2—C7—H7	119.3	C12′—C13′—H13F	109.5
C6—C7—H7	119.3	H13D—C13′—H13F	109.5
C15—C9—C10	124.5 (2)	H13E—C13′—H13F	109.5
C15—C9—C11	117.9 (2)	C11—O2—C12	124.9 (6)
C10—C9—C11	117.57 (19)	C11—O2—C12′	110.8 (3)

C7—C2—C3—C4	−2.1 (5)	C10—C9—C11—O2	−2.9 (3)
C1—C2—C3—C4	179.9 (3)	C10—C9—C15—C5	−1.5 (4)
C2—C3—C4—C5	1.2 (5)	C11—C9—C15—C5	178.0 (2)
C3—C4—C5—C6	0.2 (4)	C4—C5—C15—C9	2.2 (4)
C3—C4—C5—C15	−178.9 (3)	C6—C5—C15—C9	−177.0 (2)
C4—C5—C6—C7	−0.7 (4)	O1—C11—O2—C12	22.4 (7)
C15—C5—C6—C7	178.5 (2)	C9—C11—O2—C12	−158.7 (6)
C3—C2—C7—C6	1.6 (5)	O1—C11—O2—C12′	−7.2 (5)
C1—C2—C7—C6	179.6 (3)	C9—C11—O2—C12′	171.8 (4)
C5—C6—C7—C2	−0.2 (5)	C13—C12—O2—C11	−111.6 (10)
C15—C9—C11—O1	−3.5 (4)	C13—C12—O2—C12′	−42.2 (9)
C10—C9—C11—O1	176.0 (2)	C13′—C12′—O2—C11	177.9 (7)
C15—C9—C11—O2	177.5 (2)	C13′—C12′—O2—C12	53.2 (10)