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(*E*)-Ethyl 2-cyano-3-(2,4-dimethoxyphenyl)prop-2-enoate

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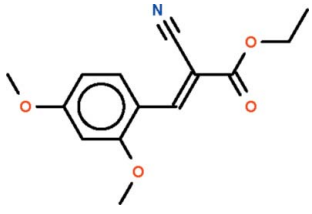
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.134; data-to-parameter ratio = 19.4.

The C=C bond in the title compound, $C_{14}H_{15}NO_4$, is in an *E* configuration. With the exception of the methyl C atoms, the non-H atoms of the molecule all lie approximately on a plane (r.m.s. deviation = 0.096 Å). π–π stacking is observed between parallel benzene rings of adjacent molecules, the centroid–centroid distance being 3.7924 (8) Å.

Related literature

For benzylidenecyanoacetate, see: Bodrikov *et al.* (1992) and for 3,4-dimethoxybenzylidenecyanoacetate, see: Nesterov *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{15}NO_4$
M_r = 261.27
Monoclinic, $P2_1/c$
a = 10.5661 (6) Å
b = 6.9715 (4) Å
c = 18.4141 (10) Å
β = 101.858 (1)°
V = 1327.47 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer
14924 measured reflections
3330 independent reflections
2382 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.134
S = 1.03
3330 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5338).

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supporting information

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S1. Comment

The synthesis of benzylidenecyanoacetate was reported by Bodrikov *et al.* in 1992; the compound was synthesized by a conventional route. In the present study, microwave radiation was used to initiate the condensation; 2,4-dimethoxy-lbenzaldehyde was used in place of the unsubstituted homolog. The carbon-carbon double-bond in $C_{14}H_{15}NO_4$ is of an *E*-configuration (Scheme I, Fig. 1). With the exception of the methyl C, the non-hydrogen atoms all lie on a plane. The features are similar to those of 3,4-dimethoxybenzylidenecyanoacetate (Bodrikov *et al.*, 1992).

S2. Experimental

2,4-Dimethoxy benzaldehyde (10 mmol), ethyl cyanoacetate (10 mmol), and 2,4-pentanedione (100 mmol, aprox. 10 ml) dissolved in ethanol (50 ml) and the solution was irradiated by microwave irradiation for 5 minutes. The mixture was cooled and the product was recrystalized from ethanol in 90% yield; m.p. 405 K.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.93 to 0.97 Å; *U*(H) 1.2 to 1.5*U*(C)] and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

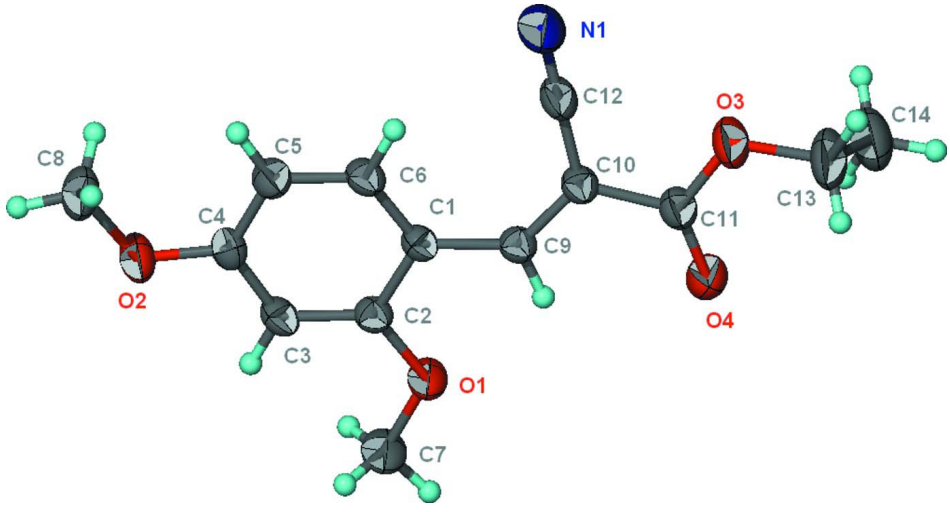

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{15}NO_4$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-Ethyl 2-cyano-3-(2,4-dimethoxyphenyl)prop-2-enoate

Crystal data

$C_{14}H_{15}NO_4$
$M_r = 261.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.5661$ (6) Å
$b = 6.9715$ (4) Å
$c = 18.4141$ (10) Å
$\beta = 101.858$ (1)°
$V = 1327.47$ (13) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.307$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3565 reflections
$\theta = 2.3$–$27.7°$
$\mu = 0.10$ mm^{-1}
$T = 295$ K
Prism, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
14924 measured reflections
3330 independent reflections
2382 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.0°$
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.134$
$S = 1.03$
3330 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0663P)^2 + 0.2366P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.31913 (10)	0.27747 (18)	0.55366 (6)	0.0536 (3)
O2	0.29391 (10)	0.12247 (17)	0.30174 (6)	0.0537 (3)
O3	0.87131 (11)	0.3785 (2)	0.71774 (6)	0.0628 (4)
O4	0.66774 (11)	0.4110 (2)	0.73311 (6)	0.0627 (3)
N1	0.89121 (15)	0.2321 (3)	0.55058 (9)	0.0804 (6)
C1	0.51259 (12)	0.25722 (18)	0.50966 (7)	0.0351 (3)
C2	0.37586 (13)	0.2438 (2)	0.49505 (8)	0.0377 (3)
C3	0.30758 (13)	0.1990 (2)	0.42512 (8)	0.0411 (3)
H3	0.2179	0.1899	0.4163	0.049*
C4	0.37162 (14)	0.1674 (2)	0.36783 (8)	0.0401 (3)
C5	0.50565 (14)	0.1815 (2)	0.37981 (8)	0.0434 (3)
H5	0.5489	0.1616	0.3413	0.052*
C6	0.57268 (13)	0.2257 (2)	0.45012 (8)	0.0417 (3)
H6	0.6623	0.2350	0.4582	0.050*

C7	0.18101 (15)	0.2627 (3)	0.54175 (10)	0.0631 (5)
H7A	0.1535	0.2895	0.5873	0.095*
H7B	0.1548	0.1353	0.5254	0.095*
H7C	0.1422	0.3535	0.5046	0.095*
C8	0.35279 (19)	0.0981 (3)	0.23941 (9)	0.0636 (5)
H8A	0.2877	0.0668	0.1965	0.095*
H8B	0.4151	−0.0038	0.2492	0.095*
H8C	0.3953	0.2150	0.2305	0.095*
C9	0.57905 (13)	0.30444 (19)	0.58405 (7)	0.0371 (3)
H9	0.5242	0.3340	0.6160	0.045*
C10	0.70570 (13)	0.3137 (2)	0.61586 (8)	0.0383 (3)
C11	0.74281 (14)	0.3727 (2)	0.69485 (8)	0.0446 (3)
C12	0.80883 (14)	0.2688 (2)	0.57949 (8)	0.0497 (4)
C13	0.92092 (19)	0.4426 (4)	0.79351 (10)	0.0773 (6)
H13A	0.8687	0.5488	0.8047	0.093*
H13B	1.0088	0.4886	0.7977	0.093*
C14	0.9198 (2)	0.2881 (4)	0.84808 (12)	0.0922 (8)
H14A	0.9534	0.3361	0.8971	0.138*
H14B	0.9725	0.1835	0.8378	0.138*
H14C	0.8327	0.2442	0.8449	0.138*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0315 (5)	0.0867 (8)	0.0439 (6)	−0.0039 (5)	0.0112 (4)	−0.0102 (5)
O2	0.0473 (6)	0.0751 (8)	0.0352 (5)	−0.0065 (5)	0.0001 (4)	0.0012 (5)
O3	0.0433 (6)	0.0943 (10)	0.0461 (6)	−0.0115 (6)	−0.0018 (5)	−0.0126 (6)
O4	0.0539 (7)	0.0885 (9)	0.0455 (6)	0.0063 (6)	0.0098 (5)	−0.0101 (6)
N1	0.0359 (7)	0.1402 (17)	0.0662 (10)	−0.0018 (9)	0.0134 (7)	−0.0185 (10)
C1	0.0305 (6)	0.0374 (7)	0.0368 (7)	−0.0007 (5)	0.0054 (5)	0.0033 (5)
C2	0.0339 (7)	0.0416 (7)	0.0385 (7)	0.0003 (5)	0.0095 (5)	0.0027 (6)
C3	0.0296 (6)	0.0488 (8)	0.0432 (7)	−0.0020 (6)	0.0036 (6)	0.0044 (6)
C4	0.0413 (7)	0.0416 (7)	0.0348 (7)	−0.0021 (6)	0.0020 (5)	0.0049 (6)
C5	0.0408 (7)	0.0549 (9)	0.0361 (7)	0.0000 (6)	0.0117 (6)	0.0035 (6)
C6	0.0301 (7)	0.0525 (8)	0.0425 (7)	−0.0001 (6)	0.0076 (6)	0.0035 (6)
C7	0.0323 (8)	0.1026 (15)	0.0569 (10)	−0.0057 (8)	0.0149 (7)	−0.0097 (10)
C8	0.0674 (11)	0.0851 (13)	0.0361 (8)	−0.0066 (9)	0.0059 (7)	−0.0056 (8)
C9	0.0342 (7)	0.0405 (7)	0.0371 (7)	0.0004 (5)	0.0081 (5)	0.0013 (6)
C10	0.0336 (7)	0.0421 (7)	0.0387 (7)	−0.0025 (6)	0.0065 (5)	0.0004 (6)
C11	0.0398 (8)	0.0509 (9)	0.0412 (8)	−0.0030 (6)	0.0041 (6)	−0.0011 (6)
C12	0.0329 (7)	0.0704 (11)	0.0432 (8)	−0.0058 (7)	0.0017 (6)	−0.0036 (7)
C13	0.0638 (12)	0.1100 (17)	0.0506 (10)	−0.0214 (11)	−0.0059 (8)	−0.0201 (11)
C14	0.0774 (15)	0.134 (2)	0.0547 (11)	−0.0001 (14)	−0.0103 (10)	−0.0037 (13)

Geometric parameters (Å, º)

O1—C2	1.3584 (16)	C6—H6	0.9300
O1—C7	1.4342 (18)	C7—H7A	0.9600

O2—C4	1.3573 (17)	C7—H7B	0.9600
O2—C8	1.4238 (19)	C7—H7C	0.9600
O3—C11	1.3370 (18)	C8—H8A	0.9600
O3—C13	1.456 (2)	C8—H8B	0.9600
O4—C11	1.1945 (18)	C8—H8C	0.9600
N1—C12	1.139 (2)	C9—C10	1.3475 (19)
C1—C6	1.3925 (19)	C9—H9	0.9300
C1—C2	1.4172 (18)	C10—C12	1.426 (2)
C1—C9	1.4431 (19)	C10—C11	1.485 (2)
C2—C3	1.376 (2)	C13—C14	1.475 (3)
C3—C4	1.383 (2)	C13—H13A	0.9700
C3—H3	0.9300	C13—H13B	0.9700
C4—C5	1.391 (2)	C14—H14A	0.9600
C5—C6	1.377 (2)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600

C2—O1—C7	117.86 (12)	O2—C8—H8B	109.5
C4—O2—C8	117.79 (12)	H8A—C8—H8B	109.5
C11—O3—C13	116.96 (13)	O2—C8—H8C	109.5
C6—C1—C2	116.87 (12)	H8A—C8—H8C	109.5
C6—C1—C9	124.86 (12)	H8B—C8—H8C	109.5
C2—C1—C9	118.26 (12)	C10—C9—C1	132.04 (13)
O1—C2—C3	123.40 (12)	C10—C9—H9	114.0
O1—C2—C1	115.90 (12)	C1—C9—H9	114.0
C3—C2—C1	120.70 (12)	C9—C10—C12	124.87 (13)
C2—C3—C4	120.35 (12)	C9—C10—C11	118.55 (12)
C2—C3—H3	119.8	C12—C10—C11	116.57 (12)
C4—C3—H3	119.8	O4—C11—O3	124.19 (14)
O2—C4—C3	114.83 (12)	O4—C11—C10	124.48 (14)
O2—C4—C5	124.54 (13)	O3—C11—C10	111.33 (12)
C3—C4—C5	120.63 (13)	N1—C12—C10	179.7 (2)
C6—C5—C4	118.36 (13)	O3—C13—C14	112.15 (18)
C6—C5—H5	120.8	O3—C13—H13A	109.2
C4—C5—H5	120.8	C14—C13—H13A	109.2
C5—C6—C1	123.09 (13)	O3—C13—H13B	109.2
C5—C6—H6	118.5	C14—C13—H13B	109.2
C1—C6—H6	118.5	H13A—C13—H13B	107.9
O1—C7—H7A	109.5	C13—C14—H14A	109.5
O1—C7—H7B	109.5	C13—C14—H14B	109.5
H7A—C7—H7B	109.5	H14A—C14—H14B	109.5
O1—C7—H7C	109.5	C13—C14—H14C	109.5
H7A—C7—H7C	109.5	H14A—C14—H14C	109.5
H7B—C7—H7C	109.5	H14B—C14—H14C	109.5
O2—C8—H8A	109.5		

C7—O1—C2—C3	0.9 (2)	C4—C5—C6—C1	−0.1 (2)
C7—O1—C2—C1	−179.11 (14)	C2—C1—C6—C5	−0.6 (2)
C6—C1—C2—O1	−179.18 (12)	C9—C1—C6—C5	−179.78 (13)

C9—C1—C2—O1	0.05 (19)	C6—C1—C9—C10	−6.4 (2)
C6—C1—C2—C3	0.8 (2)	C2—C1—C9—C10	174.47 (15)
C9—C1—C2—C3	−179.93 (13)	C1—C9—C10—C12	−2.2 (3)
O1—C2—C3—C4	179.66 (13)	C1—C9—C10—C11	178.48 (14)
C1—C2—C3—C4	−0.4 (2)	C13—O3—C11—O4	−2.3 (3)
C8—O2—C4—C3	176.73 (14)	C13—O3—C11—C10	177.39 (15)
C8—O2—C4—C5	−3.5 (2)	C9—C10—C11—O4	0.9 (2)
C2—C3—C4—O2	179.36 (13)	C12—C10—C11—O4	−178.46 (16)
C2—C3—C4—C5	−0.4 (2)	C9—C10—C11—O3	−178.79 (14)
O2—C4—C5—C6	−179.10 (14)	C12—C10—C11—O3	1.84 (19)
C3—C4—C5—C6	0.6 (2)	C11—O3—C13—C14	81.1 (2)