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Methyl (2*E*)-2-cyano-3-(dimethylamino)-prop-2-enoate

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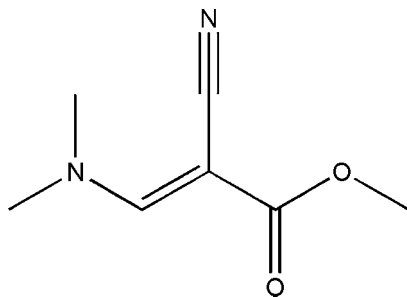
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.134; data-to-parameter ratio = 15.5.

In the title compound, $\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2$, the dimethylamino group is twisted slightly relative to the acrylate fragment, forming a dihedral angle of $11.6(1)^\circ$. In the crystal, molecules are linked *via* pairs of bifurcated $\text{C}-\text{H}/\text{H}\cdots\text{O}$ hydrogen bonds, forming inversion dimers, which are further connected by $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds into chains along the *a*-axis direction.

Related literature

For applications of enamines, see: Huang *et al.* (2007); Michael *et al.* (1999). For a related structure, see: Gupta *et al.* (2007).



Experimental

Crystal data

 $\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2$
 $M_r = 154.17$
 Triclinic, $P\bar{1}$
 $a = 7.1102(5)$ Å
 $b = 7.8170(5)$ Å

 $c = 8.2454(6)$ Å
 $\alpha = 97.270(6)^\circ$
 $\beta = 93.431(6)^\circ$
 $\gamma = 115.680(7)^\circ$
 $V = 406.31(5)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.3 \times 0.2 \times 0.2$ mm

Data collection

 Oxford Diffraction Xcalibur
 Sapphire3 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Oxford
 Diffraction, 2010)
 $T_{\min} = 0.830$, $T_{\max} = 1.000$

 6682 measured reflections
 1593 independent reflections
 1126 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.134$
 $S = 1.03$
 1593 reflections

 103 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.13$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C3—H3 \cdots O1 ⁱ	0.93	2.56	3.370 (2)	146
C4—H4A \cdots O1 ⁱ	0.96	2.58	3.415 (3)	145
C7—H7C \cdots N2 ⁱⁱ	0.96	2.58	3.535 (3)	172

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

RK acknowledges the Department of Science & Technology for access to the single-crystal X-ray diffractometer sanctioned as a National Facility under project No. SR/S2/CMP-47/2003.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2525).

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supplementary materials

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Methyl (2*E*)-2-cyano-3-(dimethylamino)prop-2-enoate

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Comment

Enamines are the multipurpose synthetic intermediates used for the synthesis of a variety of organic derivatives, bioactive natural products and their analogs (Huang *et al.*, 2007; Michael *et al.*, 1999).

In (I)(Fig.1), all bond lengths and angles are normal and correspond to those observed in the related structure (Gupta *et al.*, 2007). The dihedral angle between dimethylamino and acrylate fragment is 11.6 (1)°. In the crystal, C3—H3···O1 and C4—H4A···O1 hydrogen bonds link molecules to form dimers. Dimers are further connected by C—H···N hydrogen bonds into chains along the *a* axis (Fig. 2, Table 1.).

Experimental

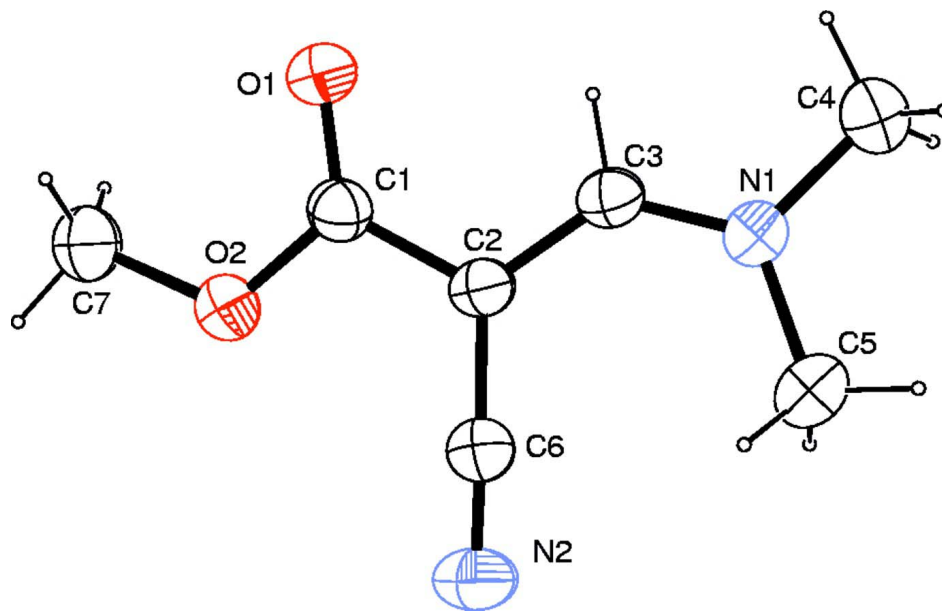
In a 50 ml round bottom flask the mixture of 5 mmole of methyl cyanoacetate and 5 mmole of dimethylformamide dimethyl acetal was stirred at room temperature for 1–1.5 h. After completion of reaction, the reaction mixture was poured on ice cold water and the separated solid was precipitated after 15 minutes and recrystallized from ethanol. Yield: 87%; m.p. 378–380 K. IR(KBr): 2205, 1695, 1621, 1433, 1373, 1287, 1216 1/cm

Refinement

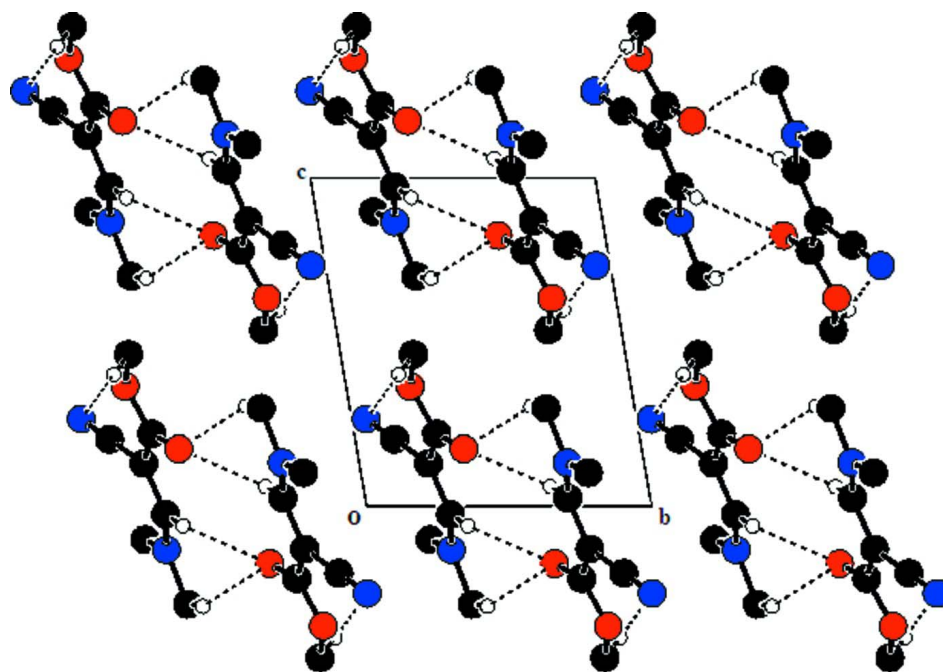
All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

**Figure 1**

ORTEP view of the molecule with the atom-labeling scheme. The displacement ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

**Figure 2**

The packing arrangement of molecules viewed down the *a* axis. The dotted lines show intermolecular C—H \cdots O and C—H \cdots N hydrogen bonds.

Methyl (2E)-2-cyano-3-(dimethylamino)prop-2-enoate

Crystal data

C₇H₁₀N₂O₂ $M_r = 154.17$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 7.1102$ (5) Å $b = 7.8170$ (5) Å $c = 8.2454$ (6) Å $\alpha = 97.270$ (6)° $\beta = 93.431$ (6)° $\gamma = 115.680$ (7)° $V = 406.31$ (5) Å³ $Z = 2$ $F(000) = 164$ $D_x = 1.260$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2720 reflections

 $\theta = 3.5$ – 29.0 ° $\mu = 0.09$ mm⁻¹ $T = 293$ K

Block, white

 $0.3 \times 0.2 \times 0.2$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire3
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 16.1049 pixels mm⁻¹ ω scan

Absorption correction: multi-scan

(CrysAlis PRO; Oxford Diffraction, 2010)

 $T_{\min} = 0.830$, $T_{\max} = 1.000$

6682 measured reflections

1593 independent reflections

1126 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.047$ $\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 3.5$ ° $h = -8 \rightarrow 8$ $k = -9 \rightarrow 9$ $l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.134$ $S = 1.03$

1593 reflections

103 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.0587P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Special details

Experimental. CrysAlis PRO, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171. NET) (compiled Aug 27 2010, 11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0160 (2)	0.6248 (2)	0.82511 (17)	0.0648 (5)
O2	0.20619 (19)	0.77535 (19)	0.63425 (16)	0.0535 (4)
C1	0.1860 (3)	0.7109 (2)	0.7792 (2)	0.0438 (4)
C2	0.3885 (3)	0.7570 (2)	0.8695 (2)	0.0405 (4)
N1	0.5414 (2)	0.7293 (2)	1.13299 (19)	0.0475 (4)
N2	0.7150 (3)	0.9493 (3)	0.7343 (2)	0.0733 (6)
C3	0.3876 (3)	0.7106 (2)	1.0252 (2)	0.0423 (4)
H3	0.2552	0.6567	1.0599	0.051*
C4	0.5009 (3)	0.6883 (3)	1.2987 (3)	0.0666 (6)
H4A	0.3525	0.6365	1.3051	0.100*
H4B	0.5737	0.8051	1.3773	0.100*
H4C	0.5498	0.5964	1.3228	0.100*
C5	0.7594 (3)	0.7984 (3)	1.1018 (3)	0.0616 (6)
H5A	0.7642	0.7414	0.9929	0.092*
H5B	0.8346	0.7633	1.1813	0.092*
H5C	0.8230	0.9360	1.1107	0.092*
C6	0.5718 (3)	0.8620 (3)	0.7958 (2)	0.0478 (5)
C7	0.0158 (3)	0.7445 (3)	0.5374 (3)	0.0611 (6)
H7A	-0.0765	0.6090	0.5118	0.092*
H7B	0.0491	0.7950	0.4371	0.092*
H7C	-0.0524	0.8090	0.5988	0.092*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0394 (8)	0.0845 (10)	0.0603 (9)	0.0136 (7)	0.0073 (7)	0.0287 (8)
O2	0.0476 (8)	0.0646 (9)	0.0472 (8)	0.0215 (6)	0.0059 (6)	0.0186 (6)
C1	0.0443 (11)	0.0417 (9)	0.0417 (10)	0.0157 (8)	0.0064 (8)	0.0071 (7)
C2	0.0356 (9)	0.0396 (9)	0.0435 (10)	0.0140 (7)	0.0079 (8)	0.0061 (7)
N1	0.0395 (8)	0.0516 (9)	0.0457 (9)	0.0151 (7)	0.0027 (7)	0.0095 (7)
N2	0.0502 (11)	0.0953 (15)	0.0766 (13)	0.0263 (10)	0.0241 (10)	0.0380 (11)
C3	0.0369 (10)	0.0385 (9)	0.0467 (10)	0.0122 (8)	0.0069 (8)	0.0073 (7)
C4	0.0583 (13)	0.0804 (15)	0.0516 (12)	0.0205 (11)	0.0027 (10)	0.0200 (11)
C5	0.0422 (11)	0.0738 (14)	0.0650 (14)	0.0223 (10)	0.0028 (10)	0.0140 (11)
C6	0.0430 (10)	0.0534 (11)	0.0475 (11)	0.0210 (9)	0.0086 (9)	0.0114 (9)
C7	0.0567 (13)	0.0726 (14)	0.0569 (13)	0.0298 (11)	0.0024 (10)	0.0200 (11)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.213 (2)	C3—H3	0.9300
O2—C1	1.346 (2)	C4—H4A	0.9600
O2—C7	1.438 (2)	C4—H4B	0.9600
C1—C2	1.454 (2)	C4—H4C	0.9600
C2—C3	1.377 (2)	C5—H5A	0.9600
C2—C6	1.427 (2)	C5—H5B	0.9600
N1—C3	1.311 (2)	C5—H5C	0.9600
N1—C5	1.455 (2)	C7—H7A	0.9600

N1—C4	1.460 (2)	C7—H7B	0.9600
N2—C6	1.143 (2)	C7—H7C	0.9600
C1—O2—C7	116.82 (15)	N1—C4—H4C	109.5
O1—C1—O2	122.40 (17)	H4A—C4—H4C	109.5
O1—C1—C2	125.53 (17)	H4B—C4—H4C	109.5
O2—C1—C2	112.07 (15)	N1—C5—H5A	109.5
C3—C2—C6	125.51 (17)	N1—C5—H5B	109.5
C3—C2—C1	117.00 (15)	H5A—C5—H5B	109.5
C6—C2—C1	117.21 (16)	N1—C5—H5C	109.5
C3—N1—C5	124.10 (16)	H5A—C5—H5C	109.5
C3—N1—C4	120.07 (15)	H5B—C5—H5C	109.5
C5—N1—C4	115.77 (16)	N2—C6—C2	177.6 (2)
N1—C3—C2	131.07 (16)	O2—C7—H7A	109.5
N1—C3—H3	114.5	O2—C7—H7B	109.5
C2—C3—H3	114.5	H7A—C7—H7B	109.5
N1—C4—H4A	109.5	O2—C7—H7C	109.5
N1—C4—H4B	109.5	H7A—C7—H7C	109.5
H4A—C4—H4B	109.5	H7B—C7—H7C	109.5
C7—O2—C1—O1	2.5 (3)	O2—C1—C2—C6	0.9 (2)
C7—O2—C1—C2	-177.56 (15)	C5—N1—C3—C2	-4.1 (3)
O1—C1—C2—C3	-4.8 (3)	C4—N1—C3—C2	173.00 (19)
O2—C1—C2—C3	175.27 (15)	C6—C2—C3—N1	-8.1 (3)
O1—C1—C2—C6	-179.14 (17)	C1—C2—C3—N1	178.06 (17)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...O1 ⁱ	0.93	2.56	3.370 (2)	146
C4—H4A...O1 ⁱ	0.96	2.58	3.415 (3)	145
C7—H7C...N2 ⁱⁱ	0.96	2.58	3.535 (3)	172

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