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Methyl 3-aminobut-2-enoate

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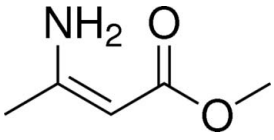
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.067; wR factor = 0.141; data-to-parameter ratio = 14.0.

The title compound, $C_5H_9NO_2$, is almost planar (r.m.s. deviation for the non-H atoms = 0.036 Å) and an intramolecular N—H···O hydrogen bond generates an S(6) ring. In the crystal, N—H···O interactions link the molecules into C(6) chains propagating along [010].

Related literature

For further synthetic details, see: Rakshit *et al.* (2010); Vanden Eynde *et al.* (1995).



Experimental

Crystal data

$C_5H_9NO_2$
M_r = 115.13
Monoclinic, $P2_1/c$
a = 8.3020 (12) Å
b = 9.7232 (14) Å
c = 7.665 (1) Å
β = 97.855 (13)°
V = 612.93 (15) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 113 K
0.18 × 0.16 × 0.10 mm

Data collection

Rigaku Saturn944 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
T_{min} = 0.868, T_{max} = 0.924
6605 measured reflections
1175 independent reflections
1019 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.141
S = 1.20
1175 reflections
84 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.84 (2)	2.05 (2)	2.8778 (16)	168.9 (19)
N1—H1B···O1	0.89 (2)	2.08 (2)	2.7168 (16)	127.7 (15)

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2009); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6788).

References

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supplementary materials

Acta Cryst. (2012). E**68**, o1930 [doi:10.1107/S160053681202288X]

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Comment

Nifedipine was found to be a highly effective calcium antagonist. Consequently,many compounds which were similar in structure to nifedipine have already been used as therapeutic agents for treatment of cerebral circulatory disorder,hypertension and so on. The title compound (I) is an intermediate for the synthesis of this family of compounds and its structure is reported here. As shown in Fig. 1, in each molecular unit, almost non-hydrogen atoms in the same plane, and the deviation is 0.036 nm. The length of the double bond is slightly longer than the normal double bond of ethylene likewise, the bond between carbon and nitrogen are shorter than normal C—N bond. A short intermolecular N—H···O interaction (Table 1) occurs [symmetry code:(i)-*x* + 1,*y* - 1/2,-*z* + 1/2], and relatively strong intramolecular N—H···O hydrogen bonds also exists.

Experimental

Impoved from the published methods by Rakshit *et al.* (2010) and Vanden Eynde *et al.* (1995) a modification of the synthetic procedure was used to prepare the title compound from methyl acetoacetate and ammonium acetate. Colorless prisms of (I) were obtained by recrystallizing from a ethyl acetate solution. mp: 355 K. Analysis, calculated for $C_5H_9NO_2$: C 52.16, H 7.88, N 12.17; found: C 52.15, H 7.87, N 12.16.

Computing details

Data collection: *CrystalClear* (Rigaku, 2009); cell refinement: *CrystalClear* (Rigaku, 2009); data reduction: *CrystalClear* (Rigaku, 2009); 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: *SHELXL97* (Sheldrick, 2008).

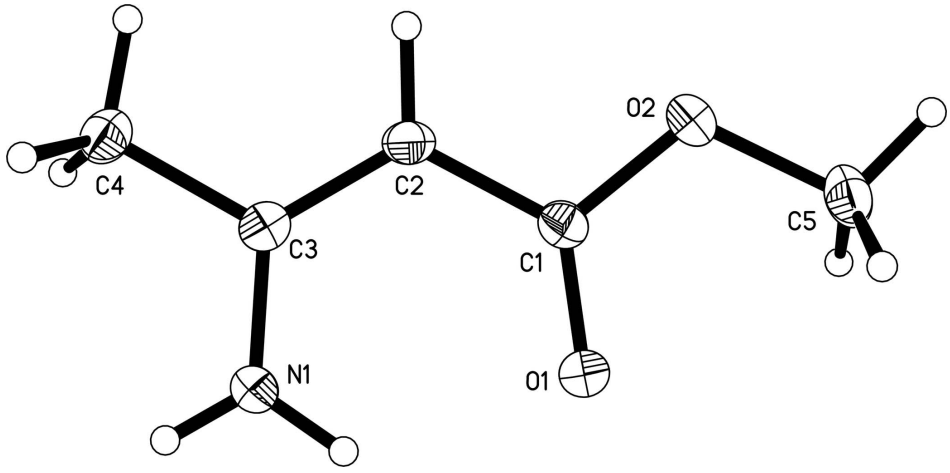

Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids.

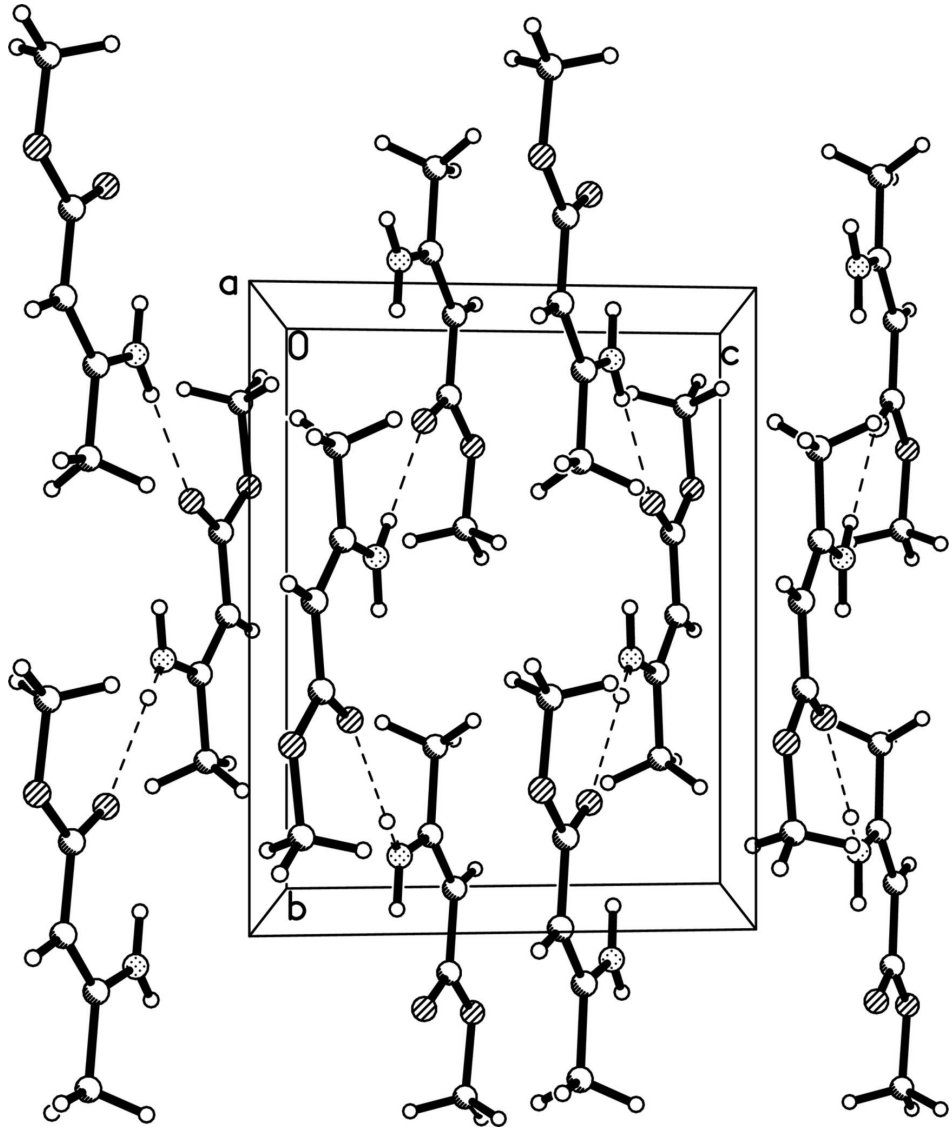

Figure 2

Packing diagram for (I).

Methyl 3-aminobut-2-enoate

Crystal data

$C_5H_9NO_2$
$M_r = 115.13$
Monoclinic, $P2_1/c$
$a = 8.3020$ (12) Å
$b = 9.7232$ (14) Å
$c = 7.665$ (1) Å
$\beta = 97.855$ (13)°
$V = 612.93$ (15) Å^3
$Z = 4$
$F(000) = 248$
$D_x = 1.248$ Mg m^{-3}
Melting point: 355 K
Cu $K\alpha$ radiation, $\lambda = 1.54187$ Å
Cell parameters from 670 reflections
$\theta = 27.9$–$71.6°$
$\mu = 0.81$ mm^{-1}
$T = 113$ K
Prism, colorless
0.18 × 0.16 × 0.10 mm

Data collection

Rigaku Saturn944 CCD diffractometer
Radiation source: fine-focus sealed tube
Multilayer monochromator
Detector resolution: 14.629 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
T_{min} = 0.868, T_{max} = 0.924
6605 measured reflections
1175 independent reflections
1019 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 71.9°, θ_{min} = 5.4°
$h = -10\rightarrow10$
$k = -11\rightarrow9$
$l = -9\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.141
S = 1.20
1175 reflections
84 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0954P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.32 (2)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.36825 (11)	0.18110 (10)	0.32340 (12)	0.0327 (4)
O2	0.12958 (12)	0.21372 (10)	0.42596 (13)	0.0357 (4)
N1	0.45092 (13)	−0.08490 (14)	0.27315 (14)	0.0320 (4)
C1	0.24663 (14)	0.13160 (14)	0.37507 (15)	0.0279 (4)
C2	0.21064 (14)	−0.01182 (15)	0.38684 (15)	0.0294 (4)
H2	0.1142	−0.0387	0.4317	0.035*
C3	0.31094 (14)	−0.11148 (14)	0.33542 (15)	0.0285 (4)
C4	0.26670 (18)	−0.26132 (15)	0.34523 (18)	0.0348 (4)
H4A	0.2644	−0.3035	0.2288	0.042*
H4B	0.1592	−0.2697	0.3836	0.042*
H4C	0.3477	−0.3083	0.4297	0.042*
C5	0.15619 (19)	0.35898 (16)	0.4094 (2)	0.0400 (5)
H5A	0.2563	0.3854	0.4849	0.048*
H5B	0.0641	0.4096	0.4455	0.048*
H5C	0.1663	0.3810	0.2866	0.048*

H1B	0.487 (2)	0.002 (2)	0.269 (3)	0.048 (5)*
H1A	0.515 (2)	−0.147 (2)	0.249 (2)	0.046 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0298 (6)	0.0295 (6)	0.0403 (6)	−0.0019 (4)	0.0102 (4)	−0.0002 (4)
O2	0.0328 (6)	0.0327 (7)	0.0437 (6)	0.0049 (4)	0.0128 (4)	−0.0031 (4)
N1	0.0287 (6)	0.0280 (8)	0.0409 (7)	0.0012 (5)	0.0109 (5)	−0.0021 (5)
C1	0.0263 (6)	0.0312 (9)	0.0262 (6)	0.0009 (5)	0.0040 (5)	−0.0015 (5)
C2	0.0259 (6)	0.0325 (9)	0.0311 (7)	−0.0039 (5)	0.0080 (5)	−0.0006 (5)
C3	0.0299 (7)	0.0301 (8)	0.0252 (6)	−0.0030 (5)	0.0024 (5)	−0.0002 (5)
C4	0.0423 (8)	0.0294 (8)	0.0335 (7)	−0.0043 (6)	0.0078 (6)	−0.0007 (5)
C5	0.0457 (8)	0.0316 (9)	0.0436 (8)	0.0095 (6)	0.0097 (6)	−0.0031 (6)

Geometric parameters (Å, º)

O1—C1	1.2317 (16)	C2—H2	0.9500
O2—C1	1.3557 (15)	C3—C4	1.5069 (18)
O2—C5	1.4379 (18)	C4—H4A	0.9800
N1—C3	1.3404 (17)	C4—H4B	0.9800
N1—H1B	0.89 (2)	C4—H4C	0.9800
N1—H1A	0.84 (2)	C5—H5A	0.9800
C1—C2	1.432 (2)	C5—H5B	0.9800
C2—C3	1.3702 (19)	C5—H5C	0.9800
C1—O2—C5	115.36 (11)	C3—C4—H4A	109.5
C3—N1—H1B	120.4 (12)	C3—C4—H4B	109.5
C3—N1—H1A	123.3 (14)	H4A—C4—H4B	109.5
H1B—N1—H1A	115.8 (18)	C3—C4—H4C	109.5
O1—C1—O2	120.91 (13)	H4A—C4—H4C	109.5
O1—C1—C2	126.03 (12)	H4B—C4—H4C	109.5
O2—C1—C2	113.05 (11)	O2—C5—H5A	109.5
C3—C2—C1	122.04 (12)	O2—C5—H5B	109.5
C3—C2—H2	119.0	H5A—C5—H5B	109.5
C1—C2—H2	119.0	O2—C5—H5C	109.5
N1—C3—C2	123.82 (13)	H5A—C5—H5C	109.5
N1—C3—C4	115.67 (12)	H5B—C5—H5C	109.5
C2—C3—C4	120.50 (12)		
C5—O2—C1—O1	1.92 (16)	O2—C1—C2—C3	177.61 (10)
C5—O2—C1—C2	−177.39 (11)	C1—C2—C3—N1	1.13 (19)
O1—C1—C2—C3	−1.7 (2)	C1—C2—C3—C4	−178.35 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.84 (2)	2.05 (2)	2.8778 (16)	168.9 (19)

N1—H1*B*···O1	0.89 (2)	2.08 (2)	2.7168 (16)	127.7 (15)

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+1/2.