

## 2-(2-Oxoimidazolidinyl)ethyl 2-methylprop-2-enoate

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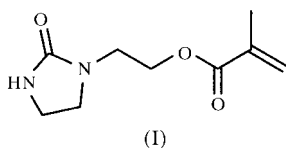
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In the crystal structure of the title compound, C<sub>9</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>, the molecules are linked by N—H···O=C bonds into chains parallel to [001]. Large crystals are readily obtained, presumably because of the hydrogen bonds and an energetically stable conformation of the molecule.

## Comment

2-(2-Oxoimidazolidinyl)ethyl 2-methylprop-2-enoate, or more simply ethylimidazolidone methacrylate, (I), is used commercially as an additive to paints, paper, textiles and leather because it favours adhesiveness on wet surfaces. It is synthesized by a transesterification reaction between 1-(2-hydroxyethyl)imidazolidin-2-one and methyl 2-methylprop-2-enoate. The synthesis requires a catalyst in order to achieve a complete conversion into the ester (I) at a temperature below 373 K and several catalysts have been patented recently (Riondel & Herbst, 1995, 1994; Herbst & Riondel, 1994). Compound (I) readily forms large crystals (up to 2 cm wide) from solution in methyl methacrylate and we wished to understand the origin of this behaviour, hence the present study. It should also be noted here that numerous reports have



been devoted to the crystallographic structures of compounds containing imidazolidin-2-one as a part of a bicyclo[3.3.0] unit, for instance in biotin. However, only three X-ray crystal structures are known so far in which an uncomplexed imidazolidin-2-one group is substituted on at least one of the N atoms and does not feature substituents directly grafted onto its ethylene moiety (Peeters *et al.*, 1984; Ueda *et al.*, 1986; Jensen, 1988).

The refined molecular structure of (I) is shown in Fig. 1, together with the atom-numbering scheme. Bond lengths and

angles in the imidazolidin-2-one ring compare favourably with the values observed in niridazole (Peeters *et al.*, 1984) and in the irindalone ion (Jensen, 1988). The imidazolidin-2-one ring is almost planar, with a mean deviation of 0.034 (2) Å from the least-squares plane through atoms N1, C2, O2, N3, C4 and C5. A similar planar arrangement was found in the case of niridazole (Peeters *et al.*, 1984), but the ring of irindalone has a significantly more twisted conformation (mean deviation 0.097 Å; Jensen, 1988).

The crystal structure of (I) is composed of chains running parallel to [001]. In neighbouring chains the imidazolidin-2-one rings are parallel to each other. The chain formation is supported by hydrogen bonding between O2 and N3<sup>i</sup>, as shown in Fig. 2 [symmetry code: (i)  $x, -\frac{1}{2} - y, z - \frac{1}{2}$ ]. The N3<sup>i</sup>···O2 distance between two hydrogen-bonded molecules is 2.915 (2) Å. These intermolecular hydrogen-bond interactions do not influence the conformation of the molecule. Indeed, an MM2 computation (Allinger, 1977) starting from the crystallographic conformation of (I) brings only negligible changes in

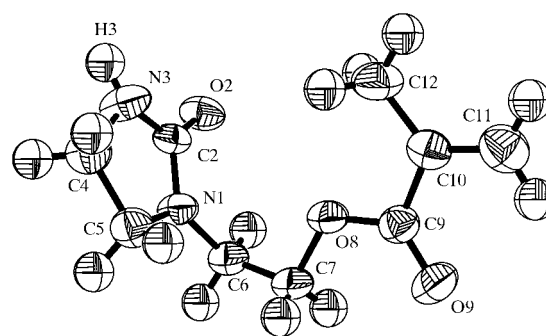


Figure 1

The molecular structure of (I), showing the atom-labelling scheme and with 50% probability displacement ellipsoids.

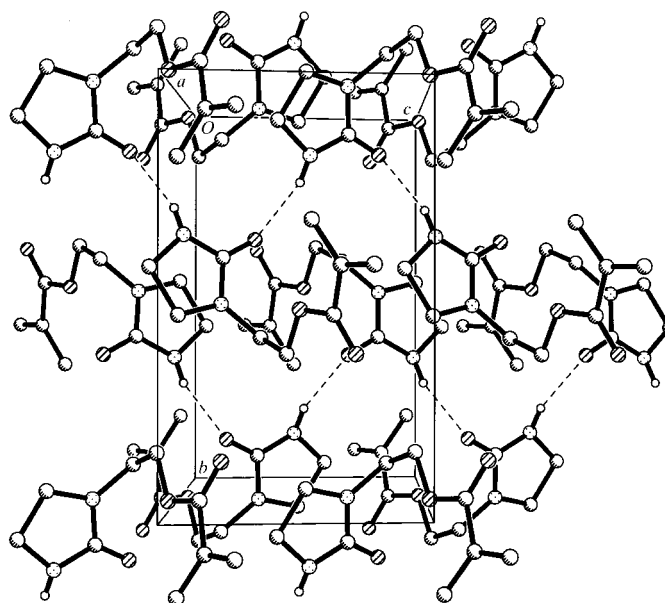


Figure 2

The crystal packing in (I). Dashed lines represent hydrogen bonds.

the molecular conformation. The relative orientation of the ester and imidazolidin-2-one groups remains essentially unaltered and the ethyl groups keep the same fully staggered conformation found in the solid state. The only significant difference between the crystallographic and *MM2* structures of (I) is a reduction of the C2–N1–C6–C7 torsion angle from  $-110.9$  to  $-90.2^\circ$ . The two protons on C6 are then more staggered with respect to the imidazolidone.

The large size of the crystals of (I) is tentatively ascribed to the energetically favourable arrangement of the molecule and to the hydrogen-bond network, although the latter is not oriented along the principal growing direction.

## Experimental

Compound (I) was obtained by reacting 1-(2-hydroxyethyl)-imidazolidin-2-one with a large excess of methyl 2-methylprop-2-enoate, as reported previously by Riondel & Herbst (1995). Slow recrystallization from a solution of (I) in methyl methacrylate at room temperature afforded colourless tabular crystals suitable for X-ray analysis.

### Crystal data

$C_9H_{14}N_2O_3$	$D_x = 1.319 \text{ Mg m}^{-3}$
$M_r = 198.22$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 39 reflections
$a = 12.6304 (8) \text{ \AA}$	$\theta = 5\text{--}25^\circ$
$b = 11.3329 (10) \text{ \AA}$	$\mu = 0.100 \text{ mm}^{-1}$
$c = 7.0306 (6) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 97.197 (6)^\circ$	Tabular, colourless
$V = 998.42 (14) \text{ \AA}^3$	$0.4 \times 0.3 \times 0.2 \text{ mm}$
$Z = 4$	

### Data collection

Siemens R3m/V4 diffractometer	$\theta_{\text{max}} = 25^\circ$
$\omega/2\theta$ scans	$h = -14 \rightarrow 14$
Absorption correction: $\psi$ -scan (North <i>et al.</i> , 1968)	$k = 0 \rightarrow 13$
$T_{\text{min}} = 0.933$ , $T_{\text{max}} = 0.980$	$l = 0 \rightarrow 8$
1866 measured reflections	3 standard reflections
1739 independent reflections	every 97 reflections
1287 reflections with $I > 2\sigma(I)$	intensity decay: none

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.950$	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
1737 reflections	$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$
184 parameters	Extinction correction: <i>SHELXL97</i>
All H-atom parameters refined	(Sheldrick, 1997)
	Extinction coefficient: 0.024 (8)

All H atoms were located from difference Fourier maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

N1–C2	1.3523 (18)	C4–C5	1.519 (2)
N1–C5	1.438 (2)	C6–C7	1.502 (2)
N1–C6	1.4451 (19)	C7–O8	1.4450 (19)
C2–O2	1.2318 (16)	O8–C9	1.3357 (18)
C2–N3	1.3374 (19)	O9–C9	1.1956 (18)
O2...N3 <sup>i</sup>	2.9151 (18)	C9–C10	1.491 (2)
N3–C4	1.433 (2)	C10–C11	1.330 (3)
N3–H3	0.85 (2)	C10–C12	1.468 (3)
C2–N1–C5	112.60 (12)	N1–C6–C7	114.15 (14)
C2–N1–C6	122.83 (13)	O8–C7–C6	107.61 (12)
C5–N1–C6	124.16 (12)	C9–O8–C7	117.19 (12)
O2–C2–N3	126.69 (14)	O9–C9–O8	122.76 (15)
O2–C2–N1	125.58 (14)	O9–C9–C10	125.86 (15)
N3–C2–N1	107.73 (13)	O8–C9–C10	111.38 (13)
C2–N3–C4	113.00 (14)	C11–C10–C12	123.79 (17)
N3–C4–C5	103.11 (13)	C11–C10–C9	117.28 (16)
N1–C5–C4	102.89 (12)	C12–C10–C9	118.91 (15)
N1–C6–C7–O8	–50.8 (2)		

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

structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXTL-Plus*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1113). Services for accessing these data are described at the back of the journal.

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