at 100 K than as a free molecule, delocalization of the π electrons being reduced by the local electric field which each molecule experiences in the crystal. It is interesting that the ring distances in the dicarboxylic acid derivative are intermediate between the two sets for pyridazine itself.

Overall, the crystal structure provides good evidence that the accuracy of the combined analysis method matches its precision insofar as angles are concerned. For bond lengths, it is not possible to tell from this study alone how accurate the results are. The errors are certainly not more than 0·01 Å, but as the e.s.d.'s are 0·002 Å or less, further comparative studies are needed.

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Structure of 1-(3-Methyl-1-butenyl)piperidin-2-one

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Abstract. $C_{10}H_{17}NO$, $M_r = 167·25$, monoclinic, $P2_1$, $a = 6·619$ (1), $b = 7·339$ (1), $c = 11·284$ (2) Å, $\beta = 103·63$ (1)°, $V = 532·72$ (14) Å^3, $Z = 2$, $D_x = 1·043$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·06$ mm^{-1}, $F(000) = 184$, $T = 250$ K, final $R = 0·082$ for 838 observed reflections. Although disorder occurs in the six-membered lactam ring and the isopropyl group, the antiperiplanar conformation of the vinyl group relative to the amide system in the title compound is established. The crystal structure is stabilized by intermolecular hydrogen bonds between the vinylic β-H atom and the carbonyl O atom of the next molecule, with a distance of 2·5 Å.

Introduction. The pyramidality of a N atom in a conjugated system is a measure of its hybridization.

For enamines various investigations based on photo-electron spectroscopy (PES) (Brown, 1976; Domelsmith, Houk & Rouge, 1977; Müller, Previdoli & Desilvestro 1981; Lafon, Gonbeau, Pfister-Guillouzo, Lasne, Ripoll & Denis, 1986), semi-empirical calculations (Müller & Brown, 1978; Saebö & Radom, 1982) and structural studies (Brown, Damm, Dunitz, Eschenmoser, Hobi & Kratky, 1978) show a pyramidal N atom for most of the investigated compounds. The largest pyramidality was found in six-membered ring systems such as piperidine and morpholine.

Conformational analyses of the amide linkage of acyclic and cyclic systems (Kitano & Kuchitsu, 1974; Hoesterey, Neely & Worley, 1983; Norskov-Lauritsen, Bürgi, Hofmann & Schmidt, 1985; Treschanke & Rademacher, 1985; Marstokk, Möllendal, Samdal & Uggerud, 1989) have shown a

planar arrangement of the substituents around the N atom. In N-(1-alkenyl)lactams an enamine is combined with a cyclic amide (lactam). The electronic structures of unsubstituted N-ethenyllactams have been investigated by PES and semi-empirical methods (Woydt, Rademacher, Kaupp & Sauerland, 1989). While the former studies and $AM1$ calculations indicated a coplanar arrangement of the amide linkage and the vinyl moiety, $MNDO$ calculations favour a synclinal conformation of the vinyl moiety. To determine the structure, especially of mono-substituted N-alkenyllactams, an X-ray analysis of the title compound has been carried out. For N-(1-alkenyl)lactams the four stereoisomers shown in Fig. 1 are possible.

Experimental. 1-(3-Methyl-1-butenyl)piperidin-2-one was synthesized (Zezza & Smith, 1987) by condensation of 9·90 g (0·100 mol) piperidin-2-one and 10·7 ml (0·100 mol) 3-methylbutanal in the presence of 0·05 g p-toluenesulfonic acid. The reaction mixture was heated in toluene in a Dean–Stark apparatus for about 16 h. The solvent was evaporated and the crystals were purified by recrystallization from ethyl acetate. Crystal size 0·11 × 0·19 × 0·27 mm, measured at 250 K; below 160 K the crystal quality decreased and increased by heating indicating a reversible phase transition. Nicolet $R3m/V$ four-circle diffractometer, ω scan (scan width 0·5°), lattice parameters determined with angle positions of 26 centred reflections $(20 \leq 2\theta \leq 25°)$. Two standard reflections ($0\overline{2}\overline{1}$ and $\overline{3}0\overline{2}$) with no significant variation, no absorption correction applied, max. value of $(\sin\theta)/\lambda = 0·60$ Å^{-1}, hkl range $-7 \leq h \leq 8$, $-8 \leq k \leq 7$ and $0 \leq l \leq -13$, 1548 independent (unique) intensities, $R_{int} = 0·037$, 838 observed $[F_o \geq 4\sigma(F)]$. Structure solved by direct methods and full-matrix refinement on F with $SHELXTL$-$Plus$ (Sheldrick,

1988). H atoms in calculated positions and refined as rigid groups with unique isotropic displacement factors for each group (C—H 0·96 Å, H—C—H 109·5°). For the disordered atoms C(9), C(10), C(9′) and C(10′) isotropic displacement parameters were applied and site occupation factors set to 0·5. Anisotropic displacement parameters were given to all other non-H atoms. 113 parameters refined, $R = 0·082$ and $wR = 0·092$ with $w^{-1} = [\sigma^2(F_o) + 0·00200F_o^2]$, $(\Delta/\sigma)_{max} = 1·75$ for y of C(10), no convergence could be achieved owing to the inaccuracy of the determination of the disordered atoms and accordingly the R value remained unsatisfactory. Max. and min. electron densities based on final difference Fourier synthesis $(\Delta\rho)_{max} = 0·35$ and $(\Delta\rho)_{min} = -0·29$ e Å^{-3}, atomic scattering factors from $SHELXTL$-$Plus$ (Sheldrick, 1988) and Cromer & Mann (1968), $\Delta f'$, $\Delta f''$ values from Cromer & Liberman (1970).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Selected distances and angles are given in Table 2. The numbering scheme and the geometry of the model are shown in Fig. 2.

The C(9) and C(10) atoms of the isopropyl group are disordered, with the corresponding atoms denoted by C(9′) and C(10′). The anisotropic displacement parameters (ADP's) of C(3) and C(4) indicate additional disorder, which could not be resolved. The same kind of disorder was observed in different data sets from various crystals. In all data collections a low ratio of independent to observed intensities $[F_o \geq 4\sigma(F)]$ was found, as is common for disordered structures.

The molecular dimensions of the title compound can be compared with the structure of N-ethenylphthalimide (Kaupp & Matthies, 1988). The lengths of the vinylic C—C bond (1·32 Å) and the carbonyl bond (1·23 Å) are equal to the normal values of these kinds of bonds (Rademacher, 1987) considering the standard deviations. These bond lengths demonstrate that π-conjugation in N-vinylphthalimide (Kaupp & Matthies, 1988) is more distinct than in the title compound. The distances to the disordered atoms C(3), C(4), C(9), C(10), C(9′) and C(10′) are unreliable and therefore not discussed.

Because of the disorder of C(3) and C(4) a puckering of the six-membered ring can be assumed with different conformers in the crystal lattice, but a dynamic system from which an almost planar ring
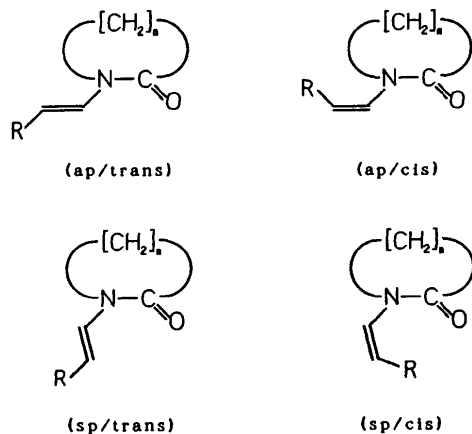

Fig. 1. The four possible stereoisomers of N-(1-alkenyl)lactams.

(ap/trans)   (ap/cis)   (sp/trans)   (sp/cis)

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$), *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}/U_{iso}
O	−2337 (6)	0	4242 (4)	88 (2)*
N	1142 (7)	52 (20)	4528 (4)	57 (2)*
C(1)	−607 (10)	−123 (22)	4945 (6)	58 (2)*
C(2)	−436 (11)	−2 (24)	6301 (6)	80 (3)*
C(3)	1734 (14)	−123 (51)	7073 (8)	159 (7)*†
C(4)	3282 (17)	−174 (39)	6611 (8)	159 (7)*†
C(5)	3268 (10)	148 (24)	5342 (6)	69 (3)*
C(6)	955 (9)	−8 (25)	3261 (5)	60 (2)*
C(7)	2503 (11)	−55 (27)	2708 (6)	77 (3)*
C(8)	2141 (12)	179 (28)	1335 (7)	98 (4)*
C(9)	2824 (46)	−1885 (34)	840 (23)	82 (6)‡
C(9')	3988 (40)	−929 (34)	946 (20)	102 (7)‡
C(10)	3195 (43)	1624 (35)	912 (22)	86 (7)‡
C(10')	2404 (39)	2271 (35)	933 (20)	95 (8)‡

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

† The ADP's of these atoms indicate disorder out of the plane of the six-membered ring.

‡ Atoms with site occupation factors of 0·5.

Table 2. *Distances* (Å), *angles* (°) *and torsion angles* (°) *for* 1-(3-methyl-1-butenyl)piperidin-2-one

O—C(1)	1·233 (7)	C(4)—C(5)	1·450 (13)
N—C(1)	1·355 (9)	C(6)—C(7)	1·320 (10)
N—C(5)	1·490 (7)	C(7)—C(8)	1·521 (10)
N—C(6)	1·406 (8)	C(8)—C(9)	1·712 (32)
C(1)—C(2)	1·510 (10)	C(8)—C(9')	1·613 (31)
C(2)—C(3)	1·497 (11)	C(8)—C(10)	1·412 (32)
C(3)—C(4)	1·255 (16)	C(8)—C(10')	1·622 (32)

C(1)—N—C(5)	123·4 (5)	N—C(5)—C(4)	112·8 (7)
C(1)—N—C(6)	118·2 (5)	N—C(6)—C(7)	126·1 (5)
C(5)—N—C(6)	118·2 (5)	C(6)—C(7)—C(8)	121·7 (6)
O—C(1)—N	120·7 (6)	C(7)—C(8)—C(9)	104·0 (15)
O—C(1)—C(2)	118·9 (7)	C(7)—C(8)—C(9')	105·8 (11)
N—C(1)—C(2)	118·7 (6)	C(7)—C(8)—C(10)	117·2 (14)
C(1)—C(2)—C(3)	114·8 (7)	C(9)—C(8)—C(10)	111·4 (16)
C(2)—C(3)—C(4)	121·7 (8)	C(7)—C(8)—C(10')	113·0 (14)
C(3)—C(4)—C(5)	126·1 (11)	C(9')—C(8)—C(10')	105·3 (15)

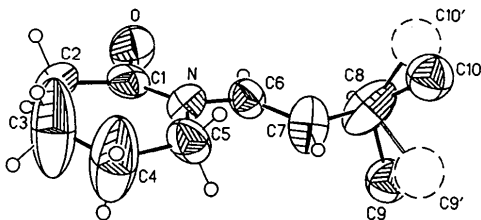|			
C(5)—N—C(1)—O	172 (9)	C(1)—N—C(6)—C(7)	−172 (1)
C(5)—N—C(1)—C(2)	7 (2)	C(5)—N—C(6)—C(7)	3 (2)
C(6)—N—C(1)—O	−13 (2)	N—C(6)—C(7)—C(8)	−170 (1)
O—C(1)—C(2)—C(3)	−179 (1)	C(6)—C(7)—C(8)—C(9)	−115 (2)
C(1)—C(2)—C(3)—C(4)	5 (3)	C(6)—C(7)—C(8)—C(9')	−152 (1)
C(2)—C(3)—C(4)—C(5)	10 (3)	C(6)—C(7)—C(8)—C(10)	122 (2)
C(1)—N—C(5)—C(4)	7 (2)	C(6)—C(7)—C(8)—C(10')	94 (2)
C(6)—N—C(5)—C(4)	−168 (1)		

Fig. 2. Perspective view of the model showing the atomic labelling. The disordered atoms C(9') and C(10') are drawn as circles with broken lines, C(9) and C(10) as full ellipsoids. Except for H atoms, ellipsoids are drawn at 50% probability.
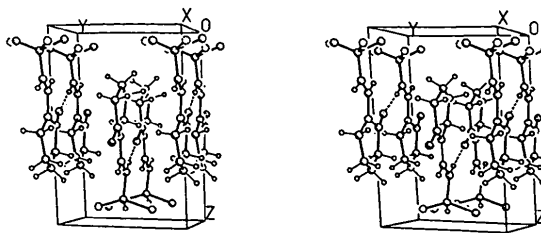

Fig. 3. Stereoview showing the crystal packing along the *x* axis. The broken lines represent H···O interactions, C(9') and C(10') are omitted.

results cannot totally be excluded; the mean deviation from planarity in this model was found to be 0·045 Å. The vinyl group is antiperiplanar to the amide linkage. The N atom has a planar surrounding (sum of angles 359·8°) and is therefore assumed to be sp^2 hybridized.

In Fig. 3 the crystal packing of the title compound is shown. The distance between the vinylic β-H atom and the carbonyl O atom of 2·5 Å indicates an intermolecular H···O interaction.

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