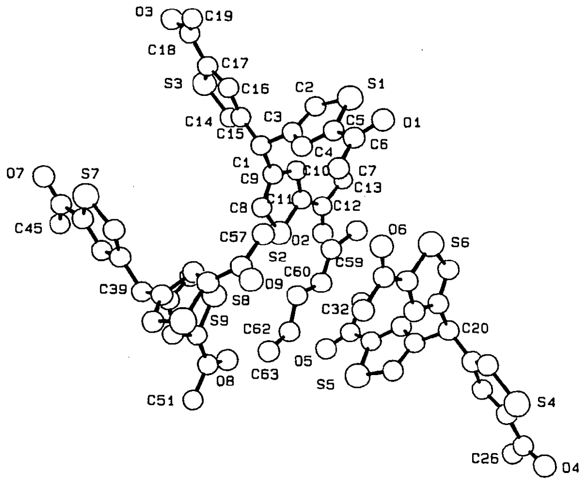

Fig. 1. Perspective drawing showing the numbering scheme.

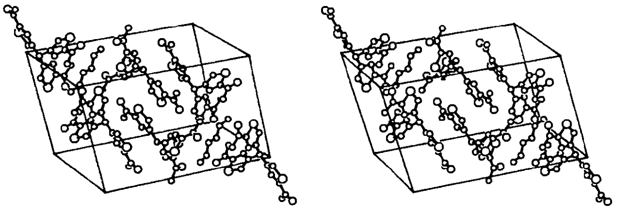

Fig. 2. Stereopair of the unit cell showing the packing.

Related literature. Related compounds are clathrates of TATM with ethyl acetate (van Rooyen & Roos, 1991*a*), benzene (van Rooyen & Roos, 1991*b*) and ethanol (Dillen & Roos, 1992). In contrast to the title compound, they crystallize in a ratio of 2/1.

The authors wish to thank Dr Meth-Cohn for supplying the title compound.

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Acta Cryst. (1992). C**48**, 1884–1886

N,*N*-Diethyl-6-(hydroxyimino)-α-methyl-1-cyclohexene-1-acetamide

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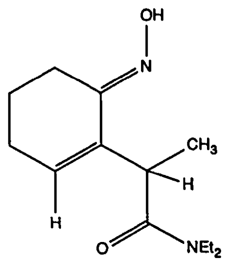
Abstract. $C_{13}H_{22}N_2O_2$, $M_r = 238.3$, monoclinic, $P2_1/c$, $a = 6.228$ (3), $b = 20.315$ (8), $c = 11.104$ (6) Å, $\beta = 103.87$ (5)°, $V = 1364$ (2) Å^3, $Z = 4$, $D_x = 1.16$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.73$ cm^{-1}, $F(000) = 520$, $T = 293$ K, final $R = 0.059$ for 1524 observed reflections. In the crystal structure disorder was found for one of the cyclohexene C atoms and in one of the ethyl groups. Molecules are connected by O—H···O hydrogen bonds.

Experimental. The title compound (I) was prepared by the reaction of the cyclic nitrone *N*,*N*-diethyl-8-methyl-7-azabicyclo[4.2.0]oct-6-ene-8-carboxamide 7-oxide with potassium *tert*-butoxide (van Eijk, Reinhoudt, Harkema & van Hummel, 1986) in THF under reflux. The crystal structure was solved to determine the conformation of the reaction product.



(I)

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O1	0.7841 (3)	0.1084 (1)	0.7122 (2)	6.56 (5)
N2	0.6532 (3)	0.1057 (1)	0.7993 (2)	5.09 (5)
C3	0.4922 (4)	0.0649 (1)	0.7663 (2)	4.92 (6)
C4	0.4515 (5)	0.0238 (2)	0.6521 (3)	6.93 (8)
C5*A*	0.333 (1)	−0.0408 (4)	0.6612 (7)	6.4 (2)
C5*B*	0.2374 (9)	−0.0075 (3)	0.6232 (5)	7.6 (1)
C6	0.1513 (6)	−0.0347 (2)	0.7212 (3)	7.78 (9)
C7	0.1909 (5)	0.0116 (2)	0.8292 (3)	6.48 (7)
C8	0.3422 (4)	0.0583 (1)	0.8500 (2)	4.76 (6)
C9	0.3703 (4)	0.1068 (1)	0.9565 (2)	4.89 (6)
C10	0.2283 (5)	0.0906 (2)	1.0468 (3)	6.51 (7)
C11	0.3074 (4)	0.1754 (1)	0.9075 (2)	4.86 (5)
O12	0.1406 (3)	0.18380 (9)	0.8241 (2)	6.03 (5)
N13	0.4236 (4)	0.2273 (1)	0.9620 (3)	7.42 (7)
C14	0.3464 (7)	0.2942 (2)	0.9177 (5)	10.8 (1)
C15	0.205 (1)	0.3218 (3)	0.9804 (7)	17.8 (3)
C16*A*	0.659 (1)	0.2235 (3)	1.0310 (8)	8.8 (2)
C16*B*	0.585 (1)	0.2230 (4)	1.0881 (8)	9.4 (2)
C17*A*	0.679 (1)	0.2207 (4)	1.1692 (7)	10.3 (2)
C17*B*	0.802 (1)	0.2313 (5)	1.065 (1)	12.8 (3)

Table 2. *Bond distances* (Å) *and angles* (°)

O1—N2	1.407 (3)	C9—C10	1.523 (4)
N2—C3	1.286 (3)	C9—C11	1.511 (4)
C3—C4	1.488 (4)	C11—O12	1.227 (3)
C3—C8	1.473 (4)	C11—N13	1.339 (3)
C4—C5*A*	1.522 (8)	N13—C14	1.485 (4)
C4—C5*B*	1.442 (7)	C14—C15	1.37 (1)
C5*A*—C6	1.449 (9)	N13—C16*A*	1.482 (6)
C5*B*—C6	1.435 (7)	N13—C16*B*	1.519 (8)
C6—C7	1.498 (5)	C16*A*—C17*A*	1.52 (1)
C7—C8	1.319 (4)	C16*B*—C17*B*	1.44 (1)
C8—C9	1.517 (4)		

O1—N2—C3	111.7 (3)	C8—C9—C10	113.3 (2)
N2—C3—C4	124.5 (3)	C8—C9—C11	110.3 (3)
N2—C3—C8	116.6 (2)	C10—C9—C11	107.2 (2)
C4—C3—C8	118.9 (2)	O12—C11—N13	119.9 (2)
C3—C4—C5*A*	114.3 (4)	O12—C11—C9	120.0 (2)
C3—C4—C5*B*	113.4 (3)	N13—C11—C9	119.8 (3)
C4—C5*A*—C6	113.7 (5)	C11—N13—C14	118.3 (3)
C4—C5*B*—C6	119.8 (4)	C11—N13—C16*A*	123.5 (3)
C5*A*—C6—C7	115.2 (4)	C11—N13—C16*B*	122.6 (4)
C5*B*—C6—C7	110.2 (3)	C14—N13—C16*A*	114.9 (4)
C6—C7—C8	124.5 (3)	C14—N13—C16*B*	116.7 (4)
C3—C8—C7	119.3 (3)	N13—C14—C15	113.8 (5)
C3—C8—C9	117.5 (3)	N13—C16*A*—C17*A*	111.0 (7)
C7—C8—C9	123.2 (3)	N13—C16*B*—C17*B*	105.8 (7)

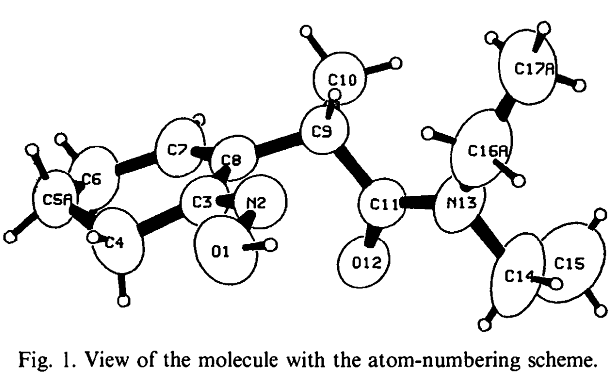

Fig. 1. View of the molecule with the atom-numbering scheme.

Reflections were measured at 293 K on an Enraf–Nonius CAD-4 diffractometer, using graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were determined by least squares from repeated measurements of 25 reflections with $6 < \theta < 13°$. A total of 2401 independent reflections were collected up to $\theta = 25°$ ($-7 \leq h \leq 7$, $0 \leq k \leq 24$, $0 \leq l \leq 13$) in the $\omega/2\theta$ scan mode {scan width (θ) $[1.0 + 0.34\tan(\theta)]°$, scan speed $0.06° \text{ s}^{-1}$}; 1524 reflections were considered observed [$F_o^2 > 3\sigma(F_o^2)$]. 124 pairs of $hk0$ reflections averaged with $R_{\text{int}} = 0.8\%$. The intensities of three standard reflections, measured every hour, showed no significant decrease. No absorption correction was applied.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined with full-matrix least squares. Weights for each reflection in the refinement (on F) were calculated from $w = 4F_o^2/\sigma^2(F^2)$, $\sigma^2(I) + (pF_o^2)^2$; the value of the instability factor p was determined as 0.05.

In the course of refinement disorder was found in one of the C atoms of the cyclohexene ring (C5) and in one of the ethyl groups (C16 and C17). The atoms were refined in two positions with partial occupancies of 0.40/0.60 and 0.50/0.50, respectively. H atoms were placed in calculated positions (bond distance 0.95 Å) and treated as riding atoms. The H atom connected to O1 was found from a difference Fourier synthesis. The number of parameters refined was 186 {scale factor, isotropic extinction factor [final value $7(5) \times 10^{-7}$], positional parameters and anisotropic thermal parameters for the heavy atoms and positional and isotropic thermal parameters for H1}. Refinement converged at $R = 5.9\%$, $wR = 8.7\%$, $(\Delta/\sigma)_{\max} = 0.02$. The largest peak on the final difference Fourier map was 0.34 e Å^{-3}. All calculations were performed using *SDP* (B. A. Frenz & Associates, Inc., 1983). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–98). Atomic parameters of the non-H atoms are collected in Table 1.* Bond distances and angles are given in Table 2. Atom numbering is shown in Fig. 1. H1 is involved in an intermolecular hydrogen bond to O12 [O1···O12 distance 2.738 (3) Å, O1—H1···O12 angle 173 (3)°].

Related literature. The preparation of the cyclic nitrone precursor has been described by Pennings &

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55152 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU0411]

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N,*N*-Diethyl-2-[2-(hydroxyimino)cyclohexylidene]propanamide

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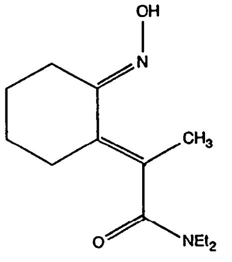
Abstract. $C_{13}H_{22}N_2O_2$, $M_r = 238.3$, monoclinic, $P2_1/c$, $a = 13.123$ (7), $b = 11.240$ (8), $c = 9.666$ (6) Å, $\beta = 99.22$ (5)°, $V = 1407$ (2) Å^3, $Z = 4$, $D_x = 1.12$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.71$ cm^{-1}, $F(000) = 520$, $T = 293$ K, final $R = 0.072$ for 1174 observed reflections. Molecules are connected by O—H···O hydrogen bonds.

Experimental. The reaction of the cyclic nitrone *N*,*N*-diethyl-8-methyl-7-azabicyclo[4.2.0]oct-6-ene-8-carboxamide 7-oxide with potassium *tert*-butoxide (van Eijk, Reinhoudt, Harkema & van Hummel, 1986) yields two isomeric products, dependent on the reaction conditions used. The present compound is formed as the major product of the reaction in DMF at 273 K. The crystal structure of the second isomer has also been determined (van Eijk, Verboom, Reinhoudt & Harkema, 1992). The two compounds differ in the position of the C═C double bond.



Reflections were measured at 293 K on an Enraf–Nonius CAD-4 diffractometer, using graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were determined by least squares from repeated measurements of 25 reflections with $6 < \theta < 14°$. A total of 1827 independent reflections were collected up to $\theta = 22.5°$ ($-14 \le h \le 13$, $0 \le k \le 12$, $0 \le l \le 10$) in the $\omega/2\theta$ scan mode [scan width (θ) 1.50°, variable scan speed between 0.01 and 0.06° s^{-1}]; 1174 reflections were considered observed [$F_o^2 > 3\sigma(F_o^2)$]. 131 pairs of *hk*0 reflections averaged with $R_{\rm int} = 2.6\%$. The intensities of three standard reflections, measured every hour, were used to correct for decay (maximum correction 0.9%). No absorption correction was applied.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined with full-matrix least squares. Unit weights were used. H atoms were placed in calculated positions and treated as riding atoms with isotropic thermal parameters (1.2 times the value of $B_{\rm eq}$ of the parent atom). The number of parameters refined was 159 {scale factor, extinction parameter [final value 2.1 (3) × 10^{-7}], positional parameters and anisotropic thermal parameters for the heavy atoms}. Refinement converged at $R = 7.2\%$, $wR = 5.3\%$, $(\Delta/\sigma)_{\rm max} = 0.01$. The largest peak on the final difference Fourier map was 0.29 e Å^{-3} (near N2, which shows large anisotropy in the thermal motion). All calculations were performed using *SDP* (B. A. Frenz & Associates,

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