

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

BY PETER J. S. S. VAN EIJK, WILLEM VERBOOM AND DAVID N. REINHOUDT

Laboratory of Organic Chemistry, University of Twente, POB 217, 7500 AE Enschede, The Netherlands

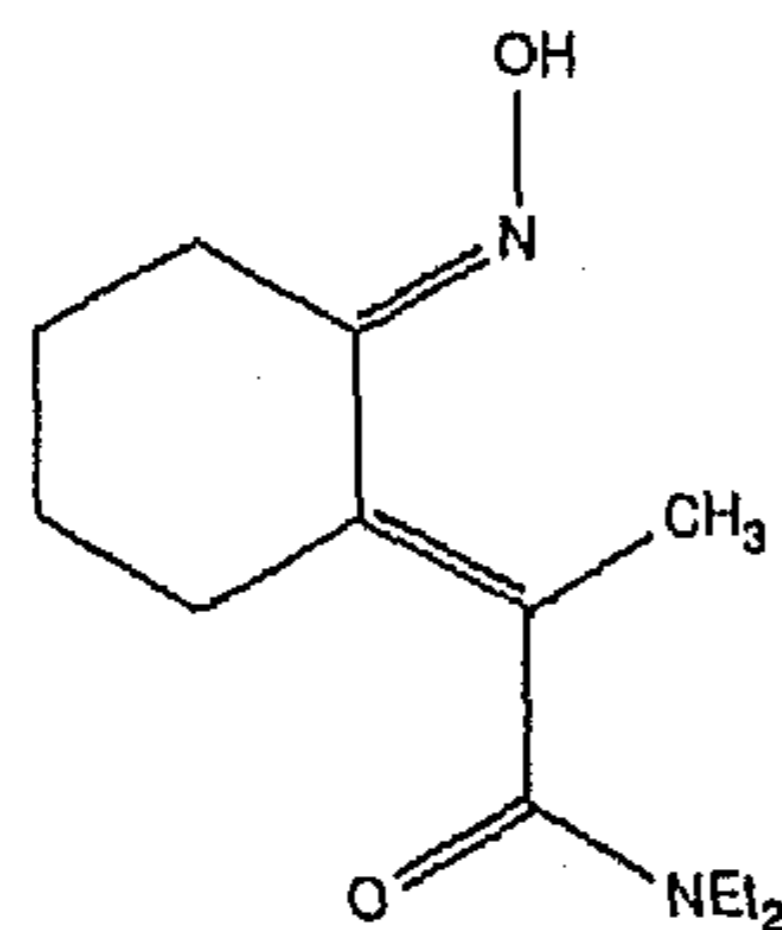
AND SYBOLT HARKEMA*

Laboratory of Chemical Physics, University of Twente, POB 217, 7500 AE Enschede, The Netherlands

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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) Å³, $Z = 4$, $D_x = 1.12$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ 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 nitrene *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.



* To whom correspondence should be addressed.

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 $6 < \theta < 14^\circ$. A total of 1827 independent reflections were collected up to $\theta = 22.5^\circ$ ($-14 \leq h \leq 13$, $0 \leq k \leq 12$, $0 \leq l \leq 10$) in the $\omega/2\theta$ scan mode [scan width (θ) 1.50° , variable scan speed between 0.01 and $0.06^\circ \text{ s}^{-1}$]; 1174 reflections were considered observed [$F_o^2 > 3\sigma(F_o^2)$]. 131 pairs of $hk0$ reflections averaged with $R_{\text{int}} = 2.6\%$. 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_{eq} of the parent atom). The number of parameters refined was 159 {scale factor, extinction parameter [final value $2.1(3) \times 10^{-7}$], positional parameters and anisotropic thermal parameters for the heavy atoms}. Refinement converged at $R = 7.2\%$, $wR = 5.3\%$, $(\Delta/\sigma)_{\text{max}} = 0.01$. The largest peak on the final difference Fourier map was $0.29 \text{ e } \text{Å}^{-3}$ (near N2, which shows large anisotropy in the thermal motion). All calculations were performed using *SDP* (B. A. Frenz & Associates,

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
O1	0.7315 (3)	-0.4484 (4)	0.7792 (4)	13.1 (1)
O12	0.8168 (3)	0.0578 (3)	1.0631 (3)	9.03 (9)
N2	0.7611 (4)	-0.3401 (3)	0.7723 (5)	10.8 (1)
N13	0.8220 (3)	0.0841 (3)	0.8361 (4)	7.3 (1)
C3	0.6985 (3)	-0.2937 (5)	0.8754 (5)	7.9 (1)
C4	0.6227 (3)	-0.3507 (5)	0.9547 (6)	9.2 (2)
C5	0.5227 (4)	-0.2839 (5)	0.9310 (6)	9.7 (2)
C6	0.5350 (3)	-0.1551 (4)	0.9500 (5)	9.2 (1)
C7	0.6122 (3)	-0.0993 (5)	0.8651 (5)	7.9 (1)
C8	0.7136 (3)	-0.1662 (4)	0.8885 (4)	6.6 (1)
C9	0.8050 (3)	-0.1139 (4)	0.9239 (5)	7.0 (1)
C10	0.9055 (3)	-0.1811 (5)	0.9619 (6)	10.5 (2)
C11	0.8137 (3)	0.0141 (4)	0.9446 (4)	6.8 (1)
C14	0.8212 (4)	0.0385 (6)	0.6947 (5)	10.3 (2)
C15	0.9272 (4)	0.0387 (6)	0.6533 (5)	13.8 (2)
C16	0.8272 (3)	0.2130 (5)	0.8558 (5)	8.9 (1)
C17	0.7216 (4)	0.2647 (5)	0.8410 (7)	12.2 (2)

Table 2. Bond distances (\AA) and angles ($^\circ$)

O1—N2	1.283 (6)	C5—C6	1.465 (7)
O12—C11	1.241 (5)	C6—C7	1.535 (7)
N2—C3	1.484 (7)	C7—C8	1.513 (6)
N13—C11	1.330 (6)	C8—C9	1.330 (6)
N13—C14	1.457 (6)	C9—C10	1.513 (6)
N13—C16	1.461 (6)	C9—C11	1.454 (7)
C3—C4	1.494 (8)	C14—C15	1.507 (7)
C3—C8	1.451 (7)	C16—C17	1.489 (7)
C4—C5	1.497 (7)		
O1—N2—C3	95.4 (4)	C3—C8—C7	111.9 (4)
C11—N13—C14	122.8 (4)	C3—C8—C9	124.3 (5)
C11—N13—C16	119.5 (4)	C7—C8—C9	123.7 (4)
C14—N13—C16	117.6 (4)	C8—C9—C10	123.8 (4)
N2—C3—C4	132.8 (4)	C8—C9—C11	121.3 (4)
N2—C3—C8	108.9 (4)	C10—C9—C11	114.5 (4)
C4—C3—C8	118.2 (4)	O12—C11—N13	120.0 (4)
C3—C4—C5	110.2 (4)	O12—C11—C9	120.6 (5)
C4—C5—C6	113.6 (4)	N13—C11—C9	119.4 (4)
C5—C6—C7	113.9 (4)	N13—C14—C15	112.3 (4)
C6—C7—C8	110.9 (4)	N13—C16—C17	110.5 (4)

Inc., 1983). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters of non-H atoms are collected in Table 1.* Bond distances and angles are given in Table 2. Atom numbering is shown in Fig. 1 [drawn by ORTEP (Johnson, 1965)]. H1 is involved in an intermolecular hydrogen bond to O12 [O1...O12 distance 2.806 (6) \AA].

* 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 55153 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU0413]

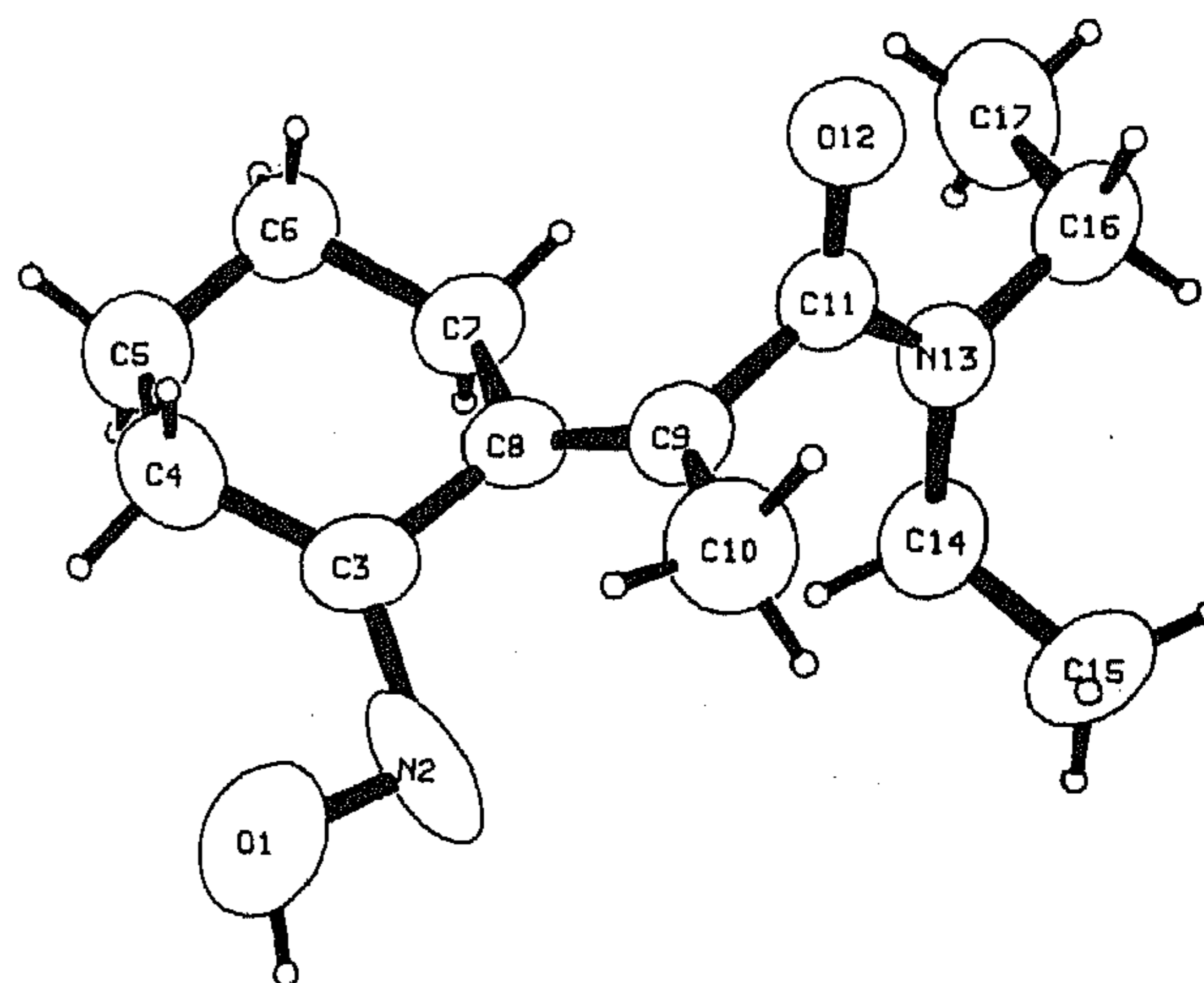


Fig. 1. View of the molecule with the atom-numbering scheme. The thermal ellipsoids of the heavy atoms have been scaled to include 30% probability.

Related literature. The preparation of the cyclic nitron precursor has been described by Pennings & Reinhoudt (1982). The crystal structure of the isomeric compound *N,N*-diethyl-6-(hydroxyimino)- α -methyl-1-cyclohexene-1-acetamide has been determined by van Eijk, Verboom, Reinhoudt & Harkema (1992). The reactions and crystal structures of related non-cyclic oximes have been reported by van Eijk, Reinhoudt, Harkema & Visser (1986) and by van Eijk, Reinhoudt, Harkema & van Hummel (1986).

References

- B. A. FRENZ & ASSOCIATES, INC. (1983). *Enraf-Nonius. SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- EIJK, P. J. S. S. VAN, REINHOUDT, D. N., HARKEMA, S. & VAN HUMMEL, G. J. (1986). *Bio-Organic Heterocycles 1986 - Synthesis, Mechanisms and Bioactivity*. In *Proceedings of the 4th FECHM Conference on Heterocycles in Bio-Organic Chemistry* (Houthalen, Belgium, 25-28 May 1986), edited by H. C. VAN DER PLAS, M. SIMONYI, F. C. ALDERWEIRELDT & J. A. LEPOIVRE. Amsterdam: Elsevier.
- EIJK, P. J. S. S. VAN, REINHOUDT, D. N., HARKEMA, S. & VISSER, R. (1986). *Recl Trav. Chim. Pays-Bas*, **105**, 103-110.
- EIJK, P. J. S. S. VAN, VERBOOM, W., REINHOUDT, D. N. & HARKEMA, S. (1992). *Acta Cryst.* **C48**, 1884-1886.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368-376.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- PENNINGS, M. L. M. & REINHOUDT, D. N. (1982). *J. Org. Chem.* **47**, 1816-1823.