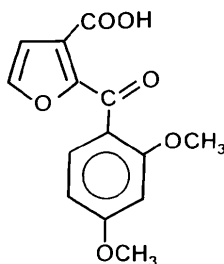


Table 3.* A view of one molecule showing the atom-numbering scheme is given in Fig. 1.



Related literature. One other keto-acid group with chelated internal unsymmetrical hydrogen bonding

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53257 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

has been reported in 4-(*o*-chloro-*N*- β -cyanoethyl-anilino)-4-oxo-but-2-enoic acid (Gonzalez-Rodriguez, Canoira, Esteban-Calderon, Martinez-Ripoll & Garcia-Blanco, 1986). There are numerous examples of symmetrical or near symmetrical internal hydrogen bonding in similar systems in dicarboxylic acid salts, *e.g.* potassium hydrogen maleate (Darlow & Cochran, 1961) and magnesium bis-(hydrogen maleate) hexahydrate (Vanhouteghem, Lenstra & Schweiss, 1987).

References

- DARLOW, S. F. & COCHRAN, W. (1961). *Acta Cryst.* **14**, 1250–1257.
 GONZALEZ-RODRIGUEZ, J., CANOIRA, L., ESTEBAN-CALDERON, C., MARTINEZ-RIPOLI, H. & GARCIA-BLANCO, S. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 199–203.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 VANHOUTEGHEM, F., LENSTRA, A. T. H. & SCHWEISS, P. (1987). *Acta Cryst.* **B43**, 523–528.

Acta Cryst. (1990). **C46**, 2489–2491

1-Cyclohexyl-6-(cyclohexylimino)-1a-phenylindano[1,2-*b*]aziridine

BY YUKIE MORI AND KOKO MAEDA*

Department of Chemistry, Faculty of Science, Ochanomizu University, Otsuka, Bunkyo-ku, Tokyo 112, Japan

AND YUJI OHASHI

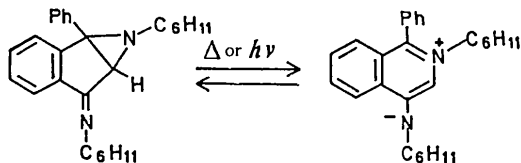
Department of Chemistry, Faculty of Science, Tokyo Institute of Technology, Ookayama, Meguro-ku, Tokyo 152, Japan

(Received 12 April 1990; accepted 22 May 1990)

Abstract. $C_{27}H_{32}N_2$, $M_r = 384.53$, monoclinic, $P2_1/n$, $a = 13.519$ (1), $b = 15.896$ (1), $c = 10.3856$ (9) Å, $\beta = 91.608$ (9)°, $V = 2231.0$ (3) Å³, $Z = 4$, $D_x = 1.145$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.34$ cm⁻¹, $F(000) = 832$, $T = 293$ K, $R = 0.048$ for 3380 unique observed reflections. The title compound was reported to exhibit photo- and thermochromism, and the colored species was revealed to be the tautomeric isoquinolinium imine. The indane moiety is planar and makes an angle of 111.32 (9)° with the aziridine ring. The two cyclohexyl groups adopt chair conformations. The aziridine C—C bond [1.515 (2) Å], which is cleaved on valence tautomerism, has a normal bond length.

* To whom correspondence should be addressed.

Experimental. The title compound was prepared by the method in the literature (Cromwell & McMaster, 1967). Recrystallization from petroleum ether gave



colorless plate-like crystals; crystal dimensions 0.5 × 0.4 × 0.3 mm, Rigaku AFC-4 diffractometer; cell parameters were determined from 21 independent 2θ values ($50 < 2\theta < 63^\circ$) with graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1.54184$ Å); intensity

Table 1. *Final atomic coordinates ($\times 10^4$) with their e.s.d.'s and equivalent isotropic temperature factors for non-H atoms*

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
N(1)	7243 (1)	172 (1)	7940 (1)	3.1
C(2)	8111 (1)	58 (1)	7131 (2)	3.0
C(3)	8030 (1)	297 (1)	5727 (2)	2.9
C(4)	7796 (1)	1540 (1)	6923 (2)	2.9
C(5)	7618 (1)	2388 (1)	7107 (2)	3.5
C(6)	7450 (1)	2895 (1)	6037 (2)	3.8
C(7)	7452 (1)	2562 (1)	4797 (2)	3.9
C(8)	7619 (1)	1713 (1)	4611 (2)	3.4
C(9)	7805 (1)	1209 (1)	5683 (2)	2.9
C(10)	7983 (1)	854 (1)	7912 (1)	2.9
N(11)	8164 (1)	-143 (1)	4725 (1)	3.3
C(12)	8397 (1)	-1036 (1)	4864 (2)	3.1
C(13)	7732 (2)	-1535 (1)	3949 (2)	4.5
C(14)	7993 (2)	-2469 (1)	3974 (2)	4.7
C(15)	9064 (2)	-2612 (1)	3711 (3)	5.5
C(16)	9728 (2)	-2118 (1)	4615 (3)	5.6
C(17)	9478 (1)	-1180 (1)	4595 (3)	4.6
C(18)	6237 (1)	284 (1)	7388 (2)	3.3
C(19)	5779 (1)	-575 (1)	7126 (2)	4.3
C(20)	4692 (2)	-496 (2)	6703 (2)	5.1
C(21)	4104 (2)	-5 (2)	7653 (2)	5.2
C(22)	4560 (2)	849 (2)	7927 (3)	6.1
C(23)	5645 (1)	764 (1)	8353 (2)	4.8
C(24)	8540 (1)	1026 (1)	9147 (2)	3.4
C(25)	8373 (2)	557 (1)	10246 (2)	4.5
C(26)	8890 (2)	725 (2)	11393 (2)	5.8
C(27)	9581 (2)	1362 (2)	11446 (2)	6.2
C(28)	9755 (2)	1838 (2)	10368 (3)	6.1
C(29)	9244 (1)	1667 (2)	9216 (2)	4.7

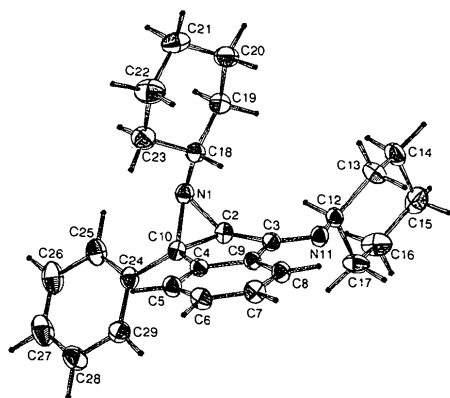


Fig. 1. ORTEP plot (Johnson, 1965) of the title compound with the atom numbering. The thermal ellipsoids enclose 30% probability.

measurements were performed with graphite-monochromated Mo *K*α radiation up to $2\theta = 50.0^\circ$ (*h*: $-16 \rightarrow 16$, *k*: $0 \rightarrow 19$, *l*: $0 \rightarrow 13$), ω - 2θ scan technique, scan speed 4° min^{-1} in θ , scan width ($1.0 + 0.35 \tan \theta$)°; background 5 s before and after each scan; three standard reflections (821, 381 and 406) were monitored every 50 reflections, no significant variation in intensities; 4069 reflections were measured, 3380 with $|F_o| > 3\sigma(|F_o|)$ were considered

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

N(1)—C(2)	1.474 (2)	C(12)—C(17)	1.513 (3)
N(1)—C(10)	1.476 (2)	C(13)—C(14)	1.526 (3)
N(1)—C(18)	1.471 (2)	C(14)—C(15)	1.499 (3)
C(2)—C(3)	1.508 (2)	C(15)—C(16)	1.502 (4)
C(2)—C(10)	1.515 (2)	C(16)—C(17)	1.528 (4)
C(3)—C(9)	1.482 (2)	C(18)—C(19)	1.521 (3)
C(3)—N(11)	1.271 (2)	C(18)—C(23)	1.507 (3)
C(4)—C(5)	1.384 (2)	C(19)—C(20)	1.527 (3)
C(4)—C(9)	1.391 (2)	C(20)—C(21)	1.503 (3)
C(4)—C(10)	1.514 (2)	C(21)—C(22)	1.515 (4)
C(5)—C(6)	1.387 (3)	C(22)—C(23)	1.526 (4)
C(6)—C(7)	1.393 (3)	C(24)—C(25)	1.387 (3)
C(7)—C(8)	1.383 (2)	C(24)—C(29)	1.395 (3)
C(8)—C(9)	1.389 (2)	C(25)—C(26)	1.390 (4)
C(10)—C(24)	1.495 (2)	C(26)—C(27)	1.377 (4)
N(11)—C(12)	1.460 (2)	C(27)—C(28)	1.378 (4)
C(12)—C(13)	1.515 (3)	C(28)—C(29)	1.391 (4)

C(2)—N(1)—C(10)	61.8 (1)	C(3)—N(11)—C(12)	119.3 (1)
C(2)—N(1)—C(18)	122.3 (1)	N(11)—C(12)—C(13)	108.9 (1)
C(10)—N(1)—C(18)	121.5 (1)	N(11)—C(12)—C(17)	109.6 (2)
N(1)—C(2)—C(3)	118.9 (1)	C(13)—C(12)—C(17)	111.4 (2)
N(1)—C(2)—C(10)	59.2 (1)	C(12)—C(13)—C(14)	111.5 (2)
C(3)—C(2)—C(10)	107.6 (1)	C(13)—C(14)—C(15)	111.7 (2)
C(2)—C(3)—C(9)	106.6 (1)	C(14)—C(15)—C(16)	111.7 (2)
C(2)—C(3)—N(11)	130.2 (1)	C(15)—C(16)—C(17)	112.0 (3)
C(9)—C(3)—N(11)	123.2 (1)	C(12)—C(17)—C(16)	111.1 (2)
C(5)—C(4)—C(9)	120.1 (1)	N(1)—C(18)—C(19)	109.1 (1)
C(5)—C(4)—C(10)	129.3 (1)	N(1)—C(18)—C(23)	107.7 (1)
C(9)—C(4)—C(10)	110.5 (1)	C(19)—C(18)—C(23)	110.6 (2)
C(4)—C(5)—C(6)	118.7 (2)	C(18)—C(19)—C(20)	111.2 (2)
C(5)—C(6)—C(7)	121.0 (2)	C(19)—C(20)—C(21)	112.1 (2)
C(6)—C(7)—C(8)	120.3 (2)	C(20)—C(21)—C(22)	111.6 (2)
C(7)—C(8)—C(9)	118.5 (2)	C(21)—C(22)—C(23)	111.0 (3)
C(3)—C(9)—C(4)	110.4 (1)	C(18)—C(23)—C(22)	112.1 (2)
C(3)—C(9)—C(8)	128.4 (1)	C(10)—C(24)—C(25)	121.2 (2)
C(4)—C(9)—C(8)	121.2 (1)	C(10)—C(24)—C(29)	120.3 (2)
N(1)—C(10)—C(2)	59.0 (1)	C(25)—C(24)—C(29)	118.5 (2)
N(1)—C(10)—C(4)	116.3 (1)	C(24)—C(25)—C(26)	120.8 (2)
N(1)—C(10)—C(24)	116.3 (1)	C(25)—C(26)—C(27)	120.0 (3)
C(2)—C(10)—C(4)	104.9 (1)	C(26)—C(27)—C(28)	120.2 (3)
C(2)—C(10)—C(24)	123.3 (1)	C(27)—C(28)—C(29)	120.0 (3)
C(4)—C(10)—C(24)	121.4 (1)	C(24)—C(29)—C(28)	120.5 (2)

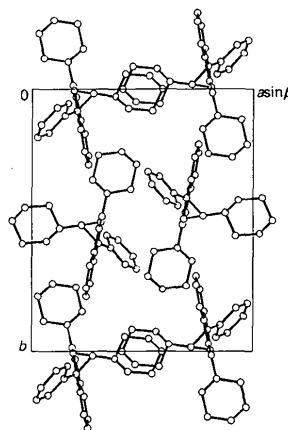


Fig. 2. Crystal structure viewed along the *c* axis.

observed and used for structure determination; corrections for Lorentz and polarization effects, absorption was ignored; direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference Fourier

map calculations, full-matrix least squares (*SHELX76*; Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms and isotropic ones for H atoms, $w = [\sigma^2(|F_o|) + 0.0040|F_o|^2]^{-1}$; all H atoms were located on difference Fourier map; final $R = 0.048$ and $wR = 0.057$ for 3380 observed reflections; $(\Delta/\sigma)_{\max} = 0.08$; final difference map showed $\Delta\rho < 0.13 \text{ e } \text{\AA}^{-3}$; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV); calculations were carried out on an IBM 4381-R24 computer at this university. Final atomic coordinates are listed in Table 1 and selected bond distances and angles in Table 2.* The molecule with

* Lists of anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms, bond distances and angles involving H atoms, least-squares-planes data and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53199 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the atom numbering is shown in Fig. 1. The crystal structure is shown in Fig. 2.

Related literature. Mechanistic study of the valence tautomerism: Lown & Matsumoto (1970), and Padwa & Vega (1975).

References

- CROMWELL, N. H. & MCMASTER, M. C. (1967). *J. Org. Chem.* **32**, 2145–2148.
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
 LOWN, J. W. & MATSUMOTO, K. (1970). *Chem. Commun.* pp. 692–693.
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 PADWA, A. & VEGA, E. (1975). *J. Org. Chem.* **40**, 175–181.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). **C46**, 2491–2493

N,N'-Dicyclohexyl-*N*-4-phenylbutyrylurea

BY RICHARD G. BALL

Merck Sharp & Dohme Research Laboratories, PO Box 2000, Rahway, New Jersey 07065, USA

AND R. STANLEY BROWN AND ANDREW J. BENNET

Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada T6G 2G2

(Received 21 November 1989; accepted 15 March 1990)

Abstract. $\text{C}_{23}\text{H}_{34}\text{N}_2\text{O}_2$, $M_r = 370.54$, monoclinic, $P2_1/c$, $a = 11.0578$ (8), $b = 23.740$ (3), $c = 8.8991$ (9) Å, $\beta = 111.117$ (8)°, $V = 2179.2$ Å³, $Z = 4$, $D_x = 1.129 \text{ Mg m}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 0.53 \text{ mm}^{-1}$, $F(000) = 808$, $T = 296 \text{ K}$, $R(F) = 0.059$ for 3060 observed [$I \geq 3\sigma(I)$] reflections. Bond distances and angles within the molecule are normal. There is one close intermolecular hydrogen-bonding distance between O(1) and N(1) of 2.841 (1) Å. The C(1)—N(1)—C(7)—O(1) and C(8)—N(2)—C(14)—O(2) torsion angles are -10.6 (3) and -10.7 (3)° respectively.

Experimental. The title compound was prepared as a side product in a reaction to prepare *N*-methyl-*N*-(2-methylphenyl)-4-phenylbutanamide by reacting 4-phenylbutyric acid with dicyclohexylcarbodiimide and 2-methyl-*N*-methylaniline in acetonitrile. Re-

crystallization from ethanol/water yielded diffraction-quality crystals.

Crystal $0.13 \times 0.21 \times 0.44 \text{ mm}$. Enraf–Nonius CAD-4 diffractometer. Lattice parameters determined using 24 reflections with $18 < 2\theta < 40^\circ$. Lorentz–polarization correction applied; absorption correction using empirical method (absorption surface) (Walker & Stuart, 1983). Maximum–minimum correction coefficients applied to F_o were 1.2219 and 0.6630. Intensity measurements in range $0 < 2\theta < 140^\circ$ (index limits: $h + 10$, $k + 28$, $l \pm 13$). Intensity standards: two reflections remeasured every 60 min of X-ray exposure time showed changes in intensity of $< 0.1\%$. No decay correction. Total reflections measured: 4520; unique: 4242; 1182 unobserved [$I < 3\sigma(I)$]. R factor for averaging equivalent reflections 0.020. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). Full-matrix least-