

N-(1-Acetyl-1-cyclohexyl)benzamide

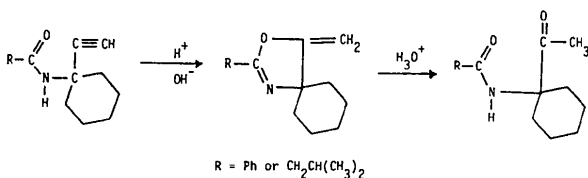
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Abstract. $C_{15}H_{19}NO_2$, $M_r = 245.3$, monoclinic, $P2_1/c$, $a = 15.453$ (3), $b = 9.004$ (2), $c = 10.220$ (2) Å, $\beta = 108.39$ (3)°, $V = 1349.4$ (5) Å³, $Z = 4$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 6.01$ cm⁻¹, $F(000) = 528$, room temperature, $R = 0.047$ for 2001 observed reflections. The structure predicted from chemical and spectral analysis is confirmed. The aromatic ring is planar and the cyclohexane ring adopts a chair conformation. Hydrogen bonds and van der Waals and stacking forces are responsible for the crystal packing.

Introduction. The synthesis of substituted propargylamines, which have assumed increased importance both with their hypotensive properties (Ryan, Easton, Dillard & Henderson, 1962) and as weed killers (Yih, Swithenbank & McRee, 1970), proceeds according to the following scheme:



In order to have a structural confirmation of the synthesis process the X-ray analysis of some products or intermediates obtained during the reaction was undertaken and the first of them is reported here.

Experimental. Colorless prismatic crystal, $0.03 \times 0.05 \times 0.21$ mm, Siemens AED single-crystal diffractometer, Ni-filtered Cu $K\alpha$, cell parameters from least-squares fit of 27 reflections accurately measured on the diffractometer, room temperature, one check reflection measured every 50 counts without significant variation, profile analysis with the Lehmann & Larsen (1974) procedure modified by Belletti, Uguzzoli, Cantoni & Pasquinelli (1979), $2 < \theta < 70^\circ$, Lp correction, absorption ignored, $-18 \leq h \leq 17$, $0 \leq k \leq 10$, $0 \leq l \leq 11$, 2832 reflections collected, 2587 unique ($R_{\text{int}} = 0.0087$), 2012 observed at $2\sigma(I)$ level, direct methods with *MULTAN80* (Main, Fiske, Hull, Lesinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement, heavy atoms anisotropic, H isotropic, located in a ΔF map, 11

reflections omitted in last cycles, 239 parameters refined, final $R = 0.047$, $wR = 0.059$ with $w = 1.0/(\sigma^2 F + 0.04514 F^2)$, $\sum w(\Delta F)^2$ minimized, $(\Delta/\sigma)_{\text{max}} = 0.035$, $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³, scattering factors of *SHELX76* (Sheldrick, 1976), all calculations performed on a Gould SEL 32/77 computer.*

Discussion. Final parameters of the molecule, illustrated in Fig. 1, are listed in Table 1. Bond distances, angles and selected torsion angles are in Table 2.

The structure established by spectral and chemical means is confirmed.

Bond lengths and angles of the aromatic ring are within expected values with mean values of 1.388 (3) Å and 119.7 (3)° respectively. This planar ring is tilted by 26.5 (1)° with respect to the C(1), C(7), N, O(1) plane.

The cyclohexane ring, rotated by 57.1 (1)° with respect to the C(8), C(14), C(15), O(2) plane, adopts the chair conformation having the spherical polar set values (Cremer & Pople, 1975) $Q = 0.57$ (2) Å, $\varphi = -118.8$ (3), $\theta = 2.64$ (2)°.

The carboxylic O(1) atom and the H attached to the N are *trans* with respect to the N–C(7) bond.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42516 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

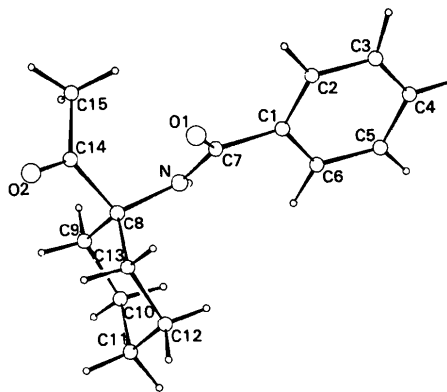


Fig. 1. Projection of the molecule down the c axis.

Table 1. Fractional atomic coordinates ($\times 10^4$) and B_{eq} (\AA^2)
$$B_{eq} = \frac{1}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{eq}
O(1)	2765 (1)	2831 (2)	1897 (1)	4.32
O(2)	439 (1)	2084 (2)	539 (2)	6.45
N(1)	2516 (1)	1776 (1)	-207 (1)	3.31
C(1)	3980 (1)	2932 (2)	926 (1)	3.37
C(2)	4416 (1)	4121 (2)	1735 (2)	4.49
C(3)	5315 (2)	4472 (3)	1843 (2)	5.85
C(4)	5778 (1)	3634 (3)	1165 (2)	5.90
C(5)	5352 (1)	2455 (3)	348 (3)	5.82
C(6)	4453 (1)	2107 (2)	218 (2)	4.46
C(7)	3037 (1)	2521 (2)	911 (2)	3.24
C(8)	1631 (1)	1140 (2)	-257 (1)	3.46
C(9)	1162 (1)	509 (2)	-1718 (2)	4.08
C(10)	1643 (1)	-838 (2)	-2057 (2)	4.64
C(11)	1766 (2)	-2048 (2)	-966 (3)	5.34
C(12)	2263 (2)	-1440 (3)	461 (2)	5.22
C(13)	1758 (1)	-118 (2)	813 (2)	4.41
C(14)	991 (1)	2371 (2)	-49 (2)	4.37
C(15)	1030 (2)	3860 (3)	-654 (3)	5.62
H(N)	2648 (13)	1762 (22)	-1041 (21)	
H(2)	4038 (15)	4793 (25)	2119 (24)	
H(3)	5512 (16)	5496 (26)	2432 (25)	
H(4)	6398 (20)	3917 (29)	1147 (28)	
H(5)	5704 (22)	1915 (33)	-72 (31)	
H(6)	4112 (17)	1256 (27)	-541 (26)	
H(9)	559 (17)	272 (25)	-1728 (23)	
H(91)	1093 (14)	1276 (23)	-2428 (24)	
H(10)	1264 (18)	-1239 (29)	-2991 (27)	
H(101)	2249 (17)	-590 (26)	-2045 (23)	
H(11)	2158 (23)	-2875 (38)	-1224 (36)	
H(111)	1146 (19)	-2456 (27)	-940 (25)	
H(12)	2298 (20)	-2242 (33)	1029 (31)	
H(121)	2849 (16)	-1101 (25)	495 (23)	
H(13)	2118 (19)	300 (28)	1730 (30)	
H(131)	1143 (16)	-395 (24)	838 (23)	
H(151)	482 (19)	4472 (29)	-648 (26)	
H(152)	885 (25)	3716 (39)	-1701 (37)	
H(153)	1650 (24)	4345 (38)	-72 (36)	

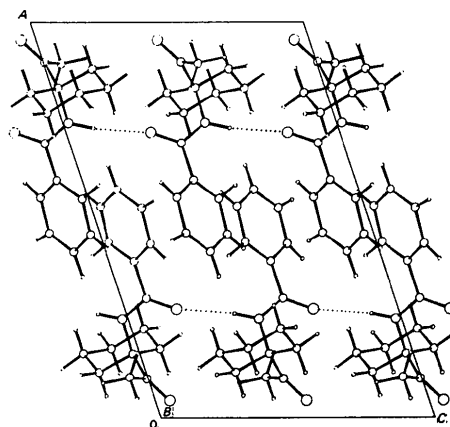


Fig. 2. Packing of the molecules.

The molecules are joined in the crystal by means of $O(1)\cdots H(N)$ bonds [$O(1)\cdots H(N^1) = 2.20$ (2), $O(1)\cdots N^1 = 3.12$ (1) \AA , $O(1)\cdots H(N)-N = 167.3^\circ$; (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$]. This bond, together with the $O(2)\cdots H(91)$, $O(1)\cdots H(13)$ and $O(2)\cdots H(131)$ interactions of 2.49 (2), 2.47 (3) and 2.46 (2) \AA , respectively, is mainly responsible with van der Waals and stacking forces for the packing in the crystal which is represented in Fig. 2.

Table 2. Bond distances (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

O(1)-C(7)	1.240 (3)	N-H(N)	0.94 (2)
O(2)-C(14)	1.216 (3)	C(2)-H(2)	1.00 (3)
N-C(7)	1.350 (2)	C(3)-H(3)	1.09 (2)
N-C(8)	1.469 (2)	C(4)-H(4)	1.00 (3)
C(1)-C(2)	1.390 (2)	C(5)-H(5)	0.93 (4)
C(1)-C(6)	1.394 (3)	C(6)-H(6)	1.10 (2)
C(1)-C(7)	1.499 (2)	C(9)-H(9)	0.95 (3)
C(2)-C(3)	1.395 (4)	C(9)-H(91)	0.98 (2)
C(3)-C(4)	1.369 (4)	C(10)-H(10)	1.02 (2)
C(4)-C(5)	1.382 (3)	C(10)-H(101)	0.96 (3)
C(5)-C(6)	1.389 (2)	C(11)-H(11)	1.04 (4)
C(8)-C(9)	1.548 (2)	C(11)-H(111)	1.03 (3)
C(8)-C(13)	1.543 (2)	C(12)-H(12)	0.95 (3)
C(8)-C(14)	1.544 (3)	C(12)-H(121)	0.95 (3)
C(9)-C(10)	1.519 (3)	C(13)-H(13)	1.00 (3)
C(10)-C(11)	1.527 (3)	C(13)-H(131)	0.99 (3)
C(11)-C(12)	1.520 (3)	C(15)-H(151)	1.01 (3)
C(12)-C(13)	1.527 (3)	C(15)-H(152)	1.03 (4)
C(14)-C(15)	1.486 (3)	C(15)-H(153)	1.05 (3)
C(7)-N-C(8)	121.6 (1)	N-C(8)-C(13)	110.7 (1)
C(6)-C(1)-C(7)	122.0 (1)	N-C(8)-C(9)	108.9 (1)
C(2)-C(1)-C(7)	118.9 (1)	C(13)-C(8)-C(14)	111.9 (1)
C(2)-C(1)-C(6)	119.0 (1)	C(9)-C(8)-C(14)	105.7 (1)
C(1)-C(2)-C(3)	120.4 (2)	C(9)-C(8)-C(13)	109.3 (1)
C(2)-C(3)-C(4)	120.1 (2)	C(8)-C(9)-C(10)	113.5 (2)
C(3)-C(4)-C(5)	120.2 (2)	C(9)-C(10)-C(11)	111.1 (2)
C(4)-C(5)-C(6)	120.2 (2)	C(10)-C(11)-C(12)	110.6 (2)
C(1)-C(6)-C(5)	120.2 (2)	C(11)-C(12)-C(13)	111.5 (2)
C(1)-C(6)-C(7)	120.2 (2)	C(8)-C(13)-C(12)	111.4 (2)
N-C(7)-C(1)	116.7 (2)	O(2)-C(14)-C(8)	119.7 (2)
O(1)-C(7)-C(1)	120.9 (2)	C(8)-C(14)-C(15)	118.6 (2)
O(1)-C(7)-N	122.4 (2)	O(2)-C(14)-C(15)	121.6 (2)
N-C(8)-C(14)	110.2 (1)	C(7)-N-C(8)-C(9)	174.1 (2)
C(2)-C(1)-C(7)-O(1)	-25.1 (3)	C(7)-N-C(8)-C(13)	-65.7 (2)
C(2)-C(1)-C(7)-N	156.4 (2)	C(7)-N-C(8)-C(14)	58.6 (2)
C(6)-C(1)-C(7)-O(1)	151.2 (3)	N-C(8)-C(14)-C(15)	36.5 (2)
C(6)-C(1)-C(7)-N	-27.4 (2)	N-C(8)-C(14)-O(2)	-147.2 (2)
C(1)-C(7)-N-C(8)	171.6 (1)		
O(1)-C(7)-N-C(8)	-6.9 (3)		

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