Crystal Structure of 2-Cyano-*N*-cyclohexylacetamide

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The title compound, 2-cyano-*N*-cyclohexylacetamide, $C_9H_{14}N_2O$, crystallizes in the monoclinic space group $P2_1/n$ with the following unit-cell parameters: $a = 12.057(2)$, $b = 4.7671(8)$, $c = 16.327(4)$ Å, $\beta = 96.50(2)^\circ$, $Z = 4$. The crystal structure was elucidated by direct methods, and refined to a final R -value of 0.0765 for 1137 observed reflections. The cyclohexane ring adopts the *chair* conformation. Intermolecular hydrogen bonds of the type $N-H\cdots O$ and $C-H\cdots N$ have been observed.

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Aromatic compounds play important roles in the pharmaceutical and agrochemical industries as important intermediates.¹ Acetamide derivatives are used as inhibitors of peroxides and stabilizers for cellulose ester varnishes. They are also used as anticonvulsants,² antibacterials,³ and radiation-protective agents.⁴ A critical literature survey revealed that there is no substantial information on the crystal structure of cyclohexanamide derivatives. The title compound was synthesized from a mixture of aniline (0.5 M, 46.5 g) and ethyl cyanoacetate (0.5 M, 56.50 ml) heated at $160^\circ - 170^\circ\text{C}$ for 6 h. The reaction mixture was left standing at room temperature overnight. The solid obtained was washed with ethanol, dried, and then recrystallized from an acetone–water mixture (5:1 ratio).

X-ray intensity data of a crystal (size: $0.30 \times 0.20 \times 0.20$ mm) were collected at 293(2)K on an *X'calibur* CCD area-detector diffractometer equipped with graphite-monochromated Mo K_α radiation ($\lambda = 0.71073$ Å). A total number of 3364 reflections were collected, of which 1812 were found to be unique. The intensities were measured by the ω scan mode for θ ranging over 3.95 to 25.99° . Further, 1137 reflections were treated as being observed ($I > 2\sigma(I)$). Data were corrected for both Lorentz and polarization factors. The structure was solved by direct methods using SHELXS97.⁵ All non-hydrogen atoms of the molecule were located from the best E-map. A full-matrix least-squares refinement was carried out using SHELXL97.⁵ All of the hydrogen atoms (except for the N1 H atom) were geometrically fixed, and allowed to ride on the corresponding

non-H atoms with $C-H = 0.97 - 0.98$ Å and $U_{iso} = 1.2 U_{eq}(C)$. The final refinement cycles converged to $R = 0.0765$ and $wR(F^2) = 0.2067$ for 1137 observed reflections. The residual electron densities ranged from -0.195 to 0.225 eÅ⁻³. The atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 1. The bond lengths, bond angles and torsion angles are given in Table 2. An ORTEP view of the title compound with atomic labeling is shown in Fig. 2.⁶ The geometry of the molecule was calculated using WinGX⁷ and PARST⁸ software.

The bond lengths are comparable to the expected values.⁹ The

Table 1 Crystal and experimental data

Chemical formula: $C_9H_{14}N_2O$	
Formula weight: 166.22	
$T = 293(2)\text{K}$	
Crystal system: monoclinic	Space group: $P2_1/n$
$a = 12.057(2)$ Å	
$b = 4.7671(8)$ Å	$\beta = 96.50(2)^\circ$
$c = 14.327(4)$ Å	
$V = 932.4(3)$ Å ³	$Z = 4$
$D_x = 1.184$ g/cm ³	D_m (floatation) = 1.09 g/cm ³
Radiation: Mo K_α ($\lambda = 0.71073$ Å)	
$\mu(\text{Mo } K_\alpha) = 0.079$ mm ⁻¹	$F(000) = 360$
Crystal size = $0.30 \times 0.20 \times 0.20$ mm	
No. of reflections collected = 1137	
No. of independent reflections = 1812	
θ range for data collection: 3.95 to 25.99°	
Data/Restraints/parameter = 3364/0/113	
Goodness-of-fit on $F^2 = 1.152$	
R indices [$I > 2\sigma(I)$]: $R1 = 0.0765$, $wR2 = 0.2067$	
R indices (all data): $R1 = 0.1120$, $wR2 = 0.2316$	
$(\Delta\rho)_{\text{max}} = 0.000$	
$(\Delta\rho)_{\text{max}} = 0.225$ eÅ ⁻³	$(\Delta\rho)_{\text{min}} = -0.195$ eÅ ⁻³
Measurement: <i>X'calibur</i> system - Oxford diffraction make, U.K.	
Programs system: SHELXL-97, ⁵ CRYSLIS RED	
Structure determination: SHELXS-97 ⁵	
CCDC deposition number: 970744

Fig. 1 Structure of 2-cyano-*N*-cyclohexylacetamide.

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Table 2 Bond lengths (Å), bond angles (°) and torsion angles (°) for non-hydrogen atoms (e.s.d.'s are given in parentheses)

Bond lengths			
O1-C7	1.224(3)	N1-C7	1.304(4)
N1-C1	1.445(4)	N2-C9	1.126(4)
C1-C6	1.491(5)	C1-C2	1.506(5)
C2-C3	1.512(5)	C3-C4	1.498(6)
C4-C5	1.496(6)	C5-C6	1.506(5)
C7-C8	1.505(4)	C8-C9	1.439(5)
Bond angles			
C7-N1-C1	124.4(3)	N1-C1-C6	111.6(3)
N1-C1-C2	111.0(3)	C6-C1-C2	110.9(3)
C1-C2-C3	110.4(3)	C4-C3-C2	112.0(3)
C5-C4-C3	111.5(3)	C4-C5-C6	111.5(3)
C1-C6-C5	111.3(3)	O1-C7-N1	123.9(3)
O1-C7-C8	120.9(3)	N1-C7-C8	115.2(3)
C9-C8-C7	111.6(3)	N2-C9-C8	178.5(4)
Torsion angles			
C7-N1-C1-C6	-126.5(4)	C7-N1-C1-C2	109.3(4)
N1-C1-C2-C3	-179.0(3)	C6-C1-C2-C3	56.3(4)
C1-C2-C3-C4	-55.1(5)	C2-C3-C4-C5	54.0(5)
C3-C4-C5-C6	-53.8(5)	N1-C1-C6-C5	178.9(3)
C2-C1-C6-C5	-56.9(4)	C4-C5-C6-C1	55.6(5)
C1-N1-C7-O1	0.0(6)	C1-N1-C7-C8	178.4(3)
O1-C7-C8-C9	-8.3(5)	N1-C7-C8-C9	173.2(3)

Fig. 2 ORTEP view of the molecule showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

cyclohexane ring adopts the *chair* conformation with the best mirror plane passing through atoms C1 and C4, and bisecting the bonds C1-C6 and C3-C4 with the asymmetry parameter [$\Delta C_s(C1) = 0.570$ and $\Delta C_s(C1-C6) = 1.044$]. The double-bond C7=O1 is confirmed by its distance of 1.224(3) Å, and is significantly larger than the standard value of 1.192 Å. The lengthening of the double-bond C7=O1 could be due to the involvement of O1 (as acceptor) in the formation of the intermolecular N1-H1...O1 interaction.

A packing view of the molecules in the unit cell viewed down the *b*-axis is shown in Fig. 3. The packing of the title compound

Fig. 3 Molecular packing of the title compound viewed down the *b*-axis.

Table 3 Hydrogen-bonding geometry (e.s.d.'s are given in parentheses)

D-H...A	D-H(Å)	H...A(Å)	D...A(Å)	D-H...A(°)
C1-H1...O1	0.98	2.41	2.808	104
N1-H1'...O1 ⁱ	0.78(4)	2.08(4)	2.852(4)	168(3)
C8-H8A...N2 ⁱⁱ	0.97	2.61	3.381(5)	136
Symmetry code:		(i) $x, y-1, z$	(ii) $-x+1/2, y-1/2, -z+3/2$	

involves intermolecular N-H...O and C-H...N hydrogen bonds, which results in a virtual dimeric structure. The one-dimensional chain structure, which is constructed by N-H...O hydrogen bonds, runs along the *b*-axis. The geometry of intra- and intermolecular interactions is given in Table 3.

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