Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*-Dicyclohexyl-2-iodoacetamide

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.023
wR factor = 0.049
Data-to-parameter ratio = 23.9

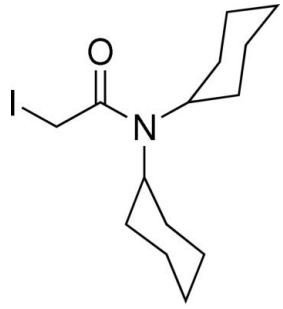
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{14}H_{24}INO$, the sum of the angles around the N atom is 359.9°, implying a planar configuration.

Received 29 September 2005
Accepted 14 October 2005
Online 22 October 2005

Comment

The title compound, (I), was synthesized in the process of investigating the reactivity of a helical foldamer (Heemstra & Moore, 2004) with iodoacetamide derivatives. The configuration around the N atom of the acetamide group is essentially planar (sum of angles = 359.9°).



(I)

Experimental

The title compound was prepared by the reaction of 2-chloro-*N*,*N*-dicyclohexylacetamide (Speziale & Hamm, 1956*a*) with 1.2 equivalents of KI in 2-butanone at reflux for 10 h (Speziale & Hamm, 1956*b*). The crude product was washed with brine and was recrystallized from diethyl ether at room temperature. Single crystals suitable for X-ray diffraction were grown at room temperature by evaporation of a diethylether/hexane solution. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.69 (*s*, 2H), 3.37 (*t*, *J* = 11.6 Hz, 1H), 2.88 (*br*, 1H), 2.41 (*br*, 2H), 1.07–1.86 (*m*, 18H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 166.4, 60.1, 56.3, 30.5, 26.3, 25.7, 25.1, 25.0, 0.0. MS (EI): *m*/*z* (%): 349 (M^+, 2.5), 222 (100), 168 (14.0), 140 (62.0), 98 (25.6), 83 (31.2), 55 (68.0). HRMS calculated for $C_{14}H_{24}INO$: 349.0903; found: 349.0907.

Crystal data

$C_{14}H_{24}INO$
M_r = 349.24
Orthorhombic, $P2_12_12_1$
a = 10.576 (2) Å
b = 11.049 (2) Å
c = 12.907 (3) Å
V = 1508.2 (5) Å^3
Z = 4
D_x = 1.538 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 939 reflections
θ = 3.1–27.7°
μ = 2.11 mm^{-1}
T = 193 (2) K
Prism, colorless
0.25 × 0.22 × 0.15 mm

Data collection

Siemens SMART/Platform CCD diffractometer
ω scans
Absorption correction: integration (*SHELXTL*/*XPREP*; Bruker, 2001)
T_{min} = 0.536, T_{max} = 0.776
14614 measured reflections
3702 independent reflections
3273 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.3°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.049
S = 1.02
3702 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 0.2165P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0010 (3)
Absolute structure: Flack (1983), 1569 Friedel pairs
Flack parameter: −0.015 (16)

H atoms were included as riding idealized contributors (C—H = 0.99 and 1.00 Å). U_{iso}(H) values were assigned as 1.2 times U_{eq}(carrier).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *XCIF* (Bruker, 2001).

The Materials Chemistry Laboratory at the University of Illinois is supported in part by grants NSF CHE 95-03145 and NSF CHE 03-43032 from the National Science Foundation. This work was supported by the Postdoctoral Fellowship Program of the Korea Science and Engineering Foundation (KOSEF, 2004).

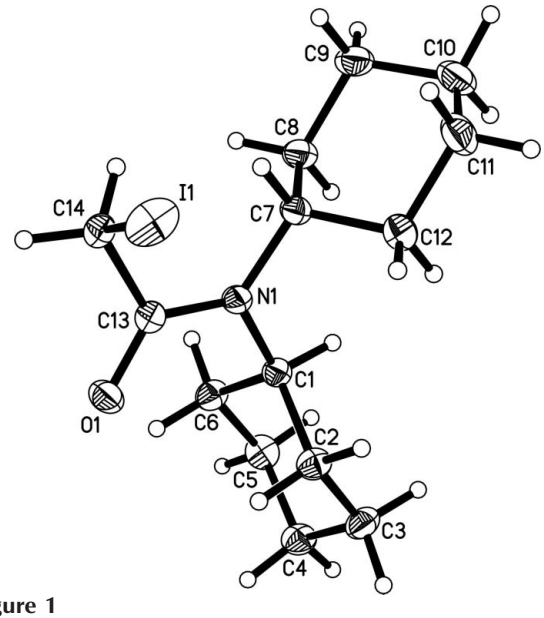


Figure 1
SHELXTL (Bruker, 2001) plot showing 35% probability ellipsoids for non-H atoms and circles of arbitrary size for H atoms.

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