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2-Chloro-*N*,*N*-dicyclohexylacetamide

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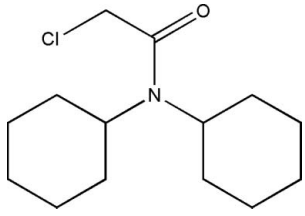
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.095; data-to-parameter ratio = 21.8.

In the title compound, $C_{14}H_{24}ClNO$, the bond lengths and angles are within normal ranges and comparable to those of related compounds. The geometry of the two cyclohexyl groups is the normal chair conformation. The crystal packing is stabilized by C—H···C short-contact interactions.

Related literature

For related literature, see: Allen *et al.* (1987); Wen *et al.* (2005); Wen, Li *et al.* (2006); Wen, Zhang *et al.* (2006); Zhang, Wen *et al.* (2006); Zhang, Xu *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{24}ClNO$
M_r = 257.79
Orthorhombic, $P2_12_12_1$
a = 10.339 (2) Å
b = 11.051 (2) Å
c = 12.358 (3) Å
V = 1412.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 113 (2) K
0.08 × 0.06 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.980, T_{max} = 0.990
17892 measured reflections
3363 independent reflections
3079 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.095
S = 1.02
3363 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), with 1436 Friedel pairs
Flack parameter: 0.01 (6)

Table 1
Selected geometric parameters (Å, °).

Cl1—C14	1.7827 (18)	N1—C7	1.476 (2)
O1—C13	1.2308 (19)	C13—C14	1.527 (2)
N1—C13	1.351 (2)		
C13—N1—C7	123.05 (13)	N1—C1—C2	113.59 (13)
C13—N1—C1	120.01 (13)	N1—C1—C6	113.46 (13)
C7—N1—C1	116.83 (13)	C2—C1—C6	111.34 (13)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O1^{i}	0.97	2.39	3.255 (2)	148

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2321).

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supplementary materials

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2-Chloro-*N*,*N*-dicyclohexylacetamide

J.-F. Liu, X.-F. Tang and Y.-H. Wen

Comment

N-Substituted-2-chloroacetamides are important intermediates in organic synthesis. They can be used in the synthesis of many derivatives such as (quinolin-8-yl-oxy)acetamide (Zhang, Xu *et al.*, 2006), 2,5-piperazinedione (Wen, Zhang *et al.*, 2006) and 2,2-(1,3,4- thiadiazolyl-2,5-dithio)diacetamide (Wen *et al.*, 2005). Here, we have synthesized and carried out the structure determination of the title compound, (I) (Fig. 1), a new *N*,*N*-disubstituted-2- chloroacetamide.

In the molecule of (I), the bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987), and comparable to those of the related compounds, 2-chloro-*N*-(4-nitrophenyl)acetamide (Wen, Li *et al.*, 2006), and 2-chloro-*N*-(4-ethoxyphenyl)acetamide (Zhang, Wen *et al.*, 2006). The C1/C7/C13/N1 andN1/C1/C14/O1 units are planar, with the dihedral angle between them 4.12 (2)°. The geometries of two cyclohexyl groups are the normal chair conformations. The crystal packing is stabilized by C9—H9···Cl short-contact interactions (Fig. 2).

Experimental

Chloroacetyl chloride (5.65 g, 0.05 mol) was added to a solution of *N*,*N*-dicyclohexylamine (9.05 g, 0.05 mol) and triethylamine (5.1 g, 0.05 mol) in benzene (60 ml) over a period of 40 min, with cooling in an ice bath, and then the mixture was stirred at room remperature for 5 h. After separation of the triethylamine hydrochloride by filtration, the organic phase was washed three times with water. The benzene layer was removed and evaporated. The title compound was obtained after drying the colorless powder at room temperature for 48 h. Colourless single cystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution over a period of 8 d.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.95–0.99 Å, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(C,*N*).

Figures

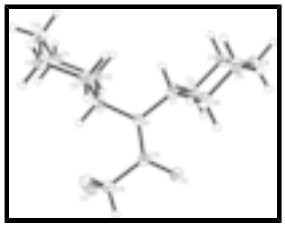

Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids.

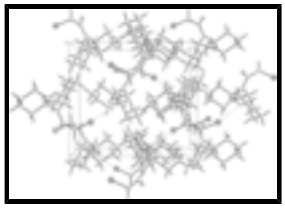

Fig. 2. The packing diagram of (I), viewed down the *a* axis, showing the short-contact interactions (dashed lines).

2-Chloro-*N*,*N*-dicyclohexylacetamide

Crystal data

$C_{14}H_{24}ClNO$	F_{000} = 560
M_r = 257.79	D_x = 1.213 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3915 reflections
a = 10.339 (2) Å	θ = 2.5–27.9º
b = 11.051 (2) Å	μ = 0.26 mm^{-1}
c = 12.358 (3) Å	T = 113 (2) K
V = 1412.1 (5) Å^3	Column, colourless
Z = 4	0.08 × 0.06 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3363 independent reflections
Radiation source: fine-focus sealed tube	3079 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 293(2) K	θ_{max} = 27.9º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.980, T_{max} = 0.990	k = −14→14
17892 measured reflections	l = −16→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.095	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
3363 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
154 parameters	Extinction correction: none

Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 1436 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.01 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.05746 (5)	0.20328 (5)	0.33226 (4)	0.03999 (15)
O1	−0.10518 (11)	0.25868 (10)	0.57080 (11)	0.0253 (3)
N1	0.03499 (12)	0.10126 (12)	0.59095 (10)	0.0172 (3)
C1	−0.04307 (15)	0.04407 (14)	0.67755 (12)	0.0173 (3)
H1	0.0069	−0.0261	0.7021	0.021*
C2	−0.17229 (16)	−0.00591 (15)	0.63806 (13)	0.0194 (3)
H2A	−0.1581	−0.0584	0.5763	0.023*
H2B	−0.2276	0.0603	0.6153	0.023*
C3	−0.23877 (16)	−0.07731 (15)	0.72882 (14)	0.0219 (4)
H3A	−0.3231	−0.1042	0.7043	0.026*
H3B	−0.1877	−0.1484	0.7459	0.026*
C4	−0.25466 (17)	−0.00027 (16)	0.83032 (15)	0.0244 (4)
H4A	−0.3142	0.0656	0.8156	0.029*
H4B	−0.2912	−0.0493	0.8878	0.029*
C5	−0.12543 (17)	0.05114 (16)	0.86687 (14)	0.0245 (4)
H5A	−0.0689	−0.0147	0.8883	0.029*
H5B	−0.1388	0.1026	0.9294	0.029*
C6	−0.06063 (17)	0.12441 (15)	0.77701 (13)	0.0219 (4)
H6A	−0.1138	0.1938	0.7587	0.026*
H6B	0.0228	0.1537	0.8015	0.026*
C7	0.14973 (16)	0.03393 (14)	0.55313 (14)	0.0185 (3)
H7	0.1866	0.0804	0.4930	0.022*
C8	0.25475 (16)	0.02467 (16)	0.63952 (15)	0.0235 (4)
H8A	0.2224	−0.0204	0.7012	0.028*
H8B	0.2787	0.1050	0.6640	0.028*
C9	0.37321 (17)	−0.03934 (16)	0.59224 (17)	0.0281 (4)
H9A	0.4093	0.0096	0.5345	0.034*
H9B	0.4386	−0.0478	0.6480	0.034*
C10	0.33861 (18)	−0.16382 (16)	0.54811 (16)	0.0301 (4)
H10A	0.4148	−0.2007	0.5163	0.036*

H10B	0.3093	−0.2152	0.6068	0.036*
C11	0.23278 (19)	−0.15410 (17)	0.46302 (15)	0.0298 (4)
H11A	0.2092	−0.2345	0.4386	0.036*
H11B	0.2652	−0.1093	0.4012	0.036*
C12	0.11336 (17)	−0.09055 (15)	0.50785 (14)	0.0228 (4)
H12A	0.0753	−0.1395	0.5647	0.027*
H12B	0.0497	−0.0811	0.4508	0.027*
C13	−0.00411 (15)	0.20666 (15)	0.54622 (13)	0.0187 (3)
C14	0.08384 (16)	0.26703 (14)	0.46292 (14)	0.0217 (4)
H14A	0.1735	0.2559	0.4837	0.026*
H14B	0.0663	0.3532	0.4608	0.026*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0399 (3)	0.0581 (3)	0.0220 (2)	−0.0139 (2)	−0.0001 (2)	0.0024 (2)
O1	0.0185 (6)	0.0240 (6)	0.0336 (7)	0.0053 (5)	0.0043 (5)	0.0066 (5)
N1	0.0148 (7)	0.0173 (7)	0.0196 (6)	0.0019 (5)	0.0044 (6)	0.0026 (5)
C1	0.0172 (8)	0.0171 (8)	0.0175 (7)	0.0006 (6)	0.0015 (7)	0.0026 (6)
C2	0.0187 (8)	0.0229 (8)	0.0166 (8)	−0.0003 (6)	−0.0002 (7)	−0.0010 (7)
C3	0.0214 (9)	0.0230 (9)	0.0215 (8)	−0.0039 (7)	0.0037 (7)	−0.0001 (7)
C4	0.0245 (9)	0.0275 (9)	0.0211 (8)	−0.0037 (7)	0.0073 (8)	−0.0006 (8)
C5	0.0290 (10)	0.0278 (9)	0.0165 (8)	0.0002 (7)	0.0020 (7)	−0.0017 (7)
C6	0.0208 (8)	0.0257 (9)	0.0191 (7)	−0.0024 (7)	0.0004 (7)	−0.0035 (7)
C7	0.0168 (8)	0.0174 (8)	0.0214 (8)	0.0015 (6)	0.0038 (7)	0.0005 (7)
C8	0.0159 (8)	0.0227 (9)	0.0320 (10)	−0.0004 (6)	−0.0003 (7)	−0.0005 (7)
C9	0.0183 (9)	0.0251 (9)	0.0408 (11)	0.0041 (7)	0.0035 (8)	0.0068 (8)
C10	0.0278 (10)	0.0243 (9)	0.0381 (11)	0.0083 (8)	0.0114 (9)	0.0065 (8)
C11	0.0410 (11)	0.0219 (9)	0.0264 (10)	0.0066 (8)	0.0119 (9)	−0.0019 (7)
C12	0.0253 (9)	0.0211 (8)	0.0221 (8)	−0.0003 (7)	0.0012 (7)	−0.0017 (7)
C13	0.0159 (8)	0.0195 (8)	0.0206 (8)	−0.0006 (6)	−0.0007 (6)	0.0018 (7)
C14	0.0218 (9)	0.0182 (8)	0.0250 (9)	0.0004 (6)	0.0017 (7)	0.0033 (7)

Geometric parameters (Å, °)

Cl1—C14	1.7827 (18)	C6—H6B	0.9700
O1—C13	1.2308 (19)	C7—C8	1.526 (2)
N1—C13	1.351 (2)	C7—C12	1.532 (2)
N1—C7	1.476 (2)	C7—H7	0.9800
N1—C1	1.4819 (19)	C8—C9	1.530 (2)
C1—C2	1.526 (2)	C8—H8A	0.9700
C1—C6	1.527 (2)	C8—H8B	0.9700
C1—H1	0.9800	C9—C10	1.522 (3)
C2—C3	1.534 (2)	C9—H9A	0.9700
C2—H2A	0.9700	C9—H9B	0.9700
C2—H2B	0.9700	C10—C11	1.521 (3)
C3—C4	1.525 (2)	C10—H10A	0.9700
C3—H3A	0.9700	C10—H10B	0.9700
C3—H3B	0.9700	C11—C12	1.525 (3)

C4—C5	1.521 (2)	C11—H11A	0.9700
C4—H4A	0.9700	C11—H11B	0.9700
C4—H4B	0.9700	C12—H12A	0.9700
C5—C6	1.529 (2)	C12—H12B	0.9700
C5—H5A	0.9700	C13—C14	1.527 (2)
C5—H5B	0.9700	C14—H14A	0.9700
C6—H6A	0.9700	C14—H14B	0.9700
C13—N1—C7	123.05 (13)	N1—C7—H7	106.8
C13—N1—C1	120.01 (13)	C8—C7—H7	106.8
C7—N1—C1	116.83 (13)	C12—C7—H7	106.8
N1—C1—C2	113.59 (13)	C7—C8—C9	109.47 (15)
N1—C1—C6	113.46 (13)	C7—C8—H8A	109.8
C2—C1—C6	111.34 (13)	C9—C8—H8A	109.8
N1—C1—H1	105.9	C7—C8—H8B	109.8
C2—C1—H1	105.9	C9—C8—H8B	109.8
C6—C1—H1	105.9	H8A—C8—H8B	108.2
C1—C2—C3	110.16 (13)	C10—C9—C8	111.50 (15)
C1—C2—H2A	109.6	C10—C9—H9A	109.3
C3—C2—H2A	109.6	C8—C9—H9A	109.3
C1—C2—H2B	109.6	C10—C9—H9B	109.3
C3—C2—H2B	109.6	C8—C9—H9B	109.3
H2A—C2—H2B	108.1	H9A—C9—H9B	108.0
C4—C3—C2	111.26 (14)	C11—C10—C9	110.66 (15)
C4—C3—H3A	109.4	C11—C10—H10A	109.5
C2—C3—H3A	109.4	C9—C10—H10A	109.5
C4—C3—H3B	109.4	C11—C10—H10B	109.5
C2—C3—H3B	109.4	C9—C10—H10B	109.5
H3A—C3—H3B	108.0	H10A—C10—H10B	108.1
C5—C4—C3	111.00 (14)	C10—C11—C12	111.34 (15)
C5—C4—H4A	109.4	C10—C11—H11A	109.4
C3—C4—H4A	109.4	C12—C11—H11A	109.4
C5—C4—H4B	109.4	C10—C11—H11B	109.4
C3—C4—H4B	109.4	C12—C11—H11B	109.4
H4A—C4—H4B	108.0	H11A—C11—H11B	108.0
C4—C5—C6	111.54 (14)	C11—C12—C7	110.34 (15)
C4—C5—H5A	109.3	C11—C12—H12A	109.6
C6—C5—H5A	109.3	C7—C12—H12A	109.6
C4—C5—H5B	109.3	C11—C12—H12B	109.6
C6—C5—H5B	109.3	C7—C12—H12B	109.6
H5A—C5—H5B	108.0	H12A—C12—H12B	108.1
C1—C6—C5	109.20 (13)	O1—C13—N1	123.76 (15)
C1—C6—H6A	109.8	O1—C13—C14	117.90 (15)
C5—C6—H6A	109.8	N1—C13—C14	118.31 (14)
C1—C6—H6B	109.8	C13—C14—Cl1	110.29 (12)
C5—C6—H6B	109.8	C13—C14—H14A	109.6
H6A—C6—H6B	108.3	Cl1—C14—H14A	109.6
N1—C7—C8	112.58 (13)	C13—C14—H14B	109.6
N1—C7—C12	111.78 (13)	Cl1—C14—H14B	109.6
C8—C7—C12	111.71 (14)	H14A—C14—H14B	108.1

C13—N1—C1—C2	69.27 (18)	C1—N1—C7—C12	59.52 (18)
C7—N1—C1—C2	−107.01 (15)	N1—C7—C8—C9	−176.61 (13)
C13—N1—C1—C6	−59.22 (19)	C12—C7—C8—C9	56.66 (18)
C7—N1—C1—C6	124.51 (15)	C7—C8—C9—C10	−56.9 (2)
N1—C1—C2—C3	172.81 (13)	C8—C9—C10—C11	57.0 (2)
C6—C1—C2—C3	−57.63 (18)	C9—C10—C11—C12	−56.2 (2)
C1—C2—C3—C4	55.47 (19)	C10—C11—C12—C7	55.71 (19)
C2—C3—C4—C5	−55.04 (19)	N1—C7—C12—C11	176.38 (14)
C3—C4—C5—C6	56.40 (19)	C8—C7—C12—C11	−56.45 (18)
N1—C1—C6—C5	−172.10 (12)	C7—N1—C13—O1	173.80 (15)
C2—C1—C6—C5	58.27 (18)	C1—N1—C13—O1	−2.2 (2)
C4—C5—C6—C1	−57.46 (19)	C7—N1—C13—C14	−8.2 (2)
C13—N1—C7—C8	116.68 (17)	C1—N1—C13—C14	175.78 (14)
C1—N1—C7—C8	−67.17 (17)	O1—C13—C14—Cl1	−98.04 (16)
C13—N1—C7—C12	−116.62 (16)	N1—C13—C14—Cl1	83.83 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14A···O1^{i}	0.97	2.39	3.255 (2)	148

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1.

Fig. 1

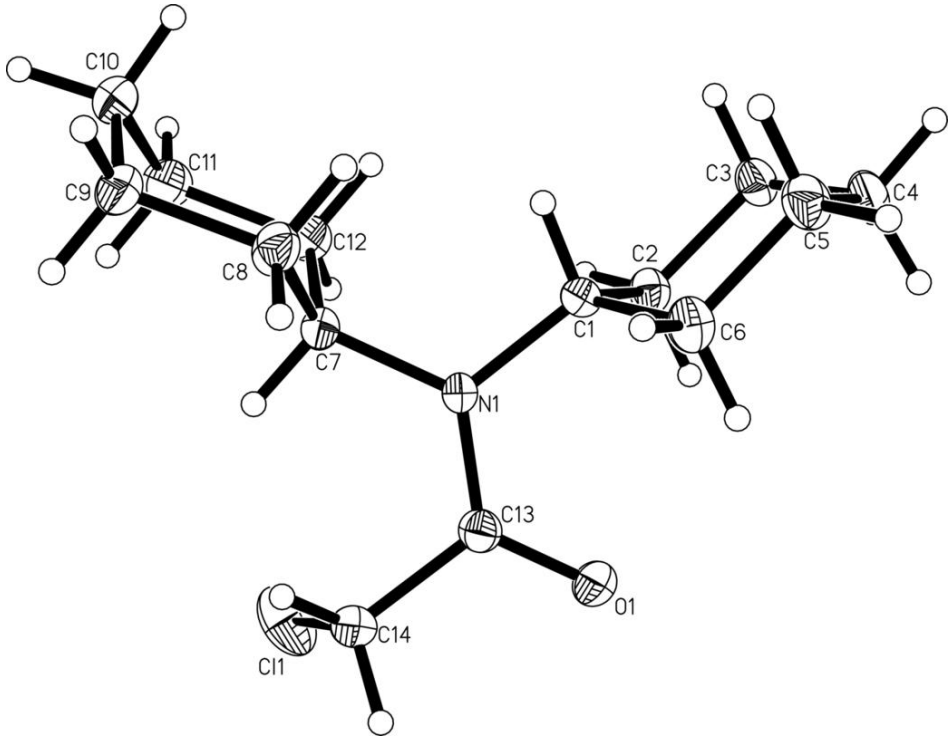

Fig. 2