

Acta Cryst. (1995). **C51**, 2149–2150

Dicyclohexylammonium Chloride

SEIK WENG NG

*Institute of Advanced Studies, University of Malaya,
59100 Kuala Lumpur, Malaysia*

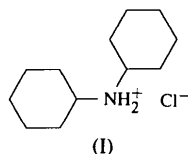
(Received 29 March 1994; accepted 3 March 1995)

Abstract

The cation in dicyclohexylammonium chloride, $C_{12}H_{24}N^+.Cl^-$, forms hydrogen bonds to two anions, resulting in a chain that propagates along the x axis.

Comment

The dicyclohexylammonium cation has been used in earlier studies to form crystalline derivatives of carboxylic acids (Ng, 1992, 1993) and these studies have been extended to the present determination of the structure of the dicyclohexylammonium salt of hydrogen chloride, (I). In the cation, the C—N—C angle is opened up to $118.6(2)^\circ$, relative to the tetrahedral angle of 109.5° , as a result of the steric bulk of the two cyclohexyl rings, both of which maintain chair conformations.



The anionic Cl atom is strongly hydrogen bonded to the cation and the two $N \cdots Cl$ contact distances [$3.145(2)$ and $3.176(2)$ Å] compare well

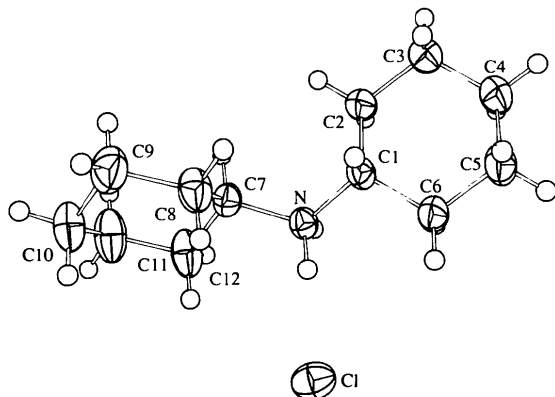


Fig. 1. ORTEP (Johnson, 1976) plot of dicyclohexylammonium chloride showing the atom-numbering scheme and 50% probability ellipsoids.

with those found in, for example, 4-substituted methylmorpholinium chlorides [$3.035(2)$ – $3.051(2)$ Å; Mentzafos & Terzis, 1993].

Experimental

The title compound was synthesized from dicyclohexylamine and concentrated hydrochloric acid. Crystals were grown from a solution of the compound in ethanol.

Crystal data

$C_{12}H_{24}N^+.Cl^-$
 $M_r = 217.77$
 Monoclinic
 $P2_1/c$
 $a = 5.394(2)$ Å
 $b = 11.725(2)$ Å
 $c = 20.062(5)$ Å
 $\beta = 95.48(2)^\circ$
 $V = 1263.1(6)$ Å³
 $Z = 4$
 $D_x = 1.145$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 9$ – 12°
 $\mu = 0.269$ mm⁻¹
 $T = 298$ K
 Cube
 $0.36 \times 0.36 \times 0.36$ mm
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: none
 4941 measured reflections
 2220 independent reflections
 1634 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0282$
 $\theta_{max} = 24.99^\circ$
 $h = 0 \rightarrow 6$
 $k = -13 \rightarrow 13$
 $l = -23 \rightarrow 23$
 3 standard reflections
 frequency: 60 min
 intensity decay: 1.4%

Refinement

Refinement on F^2
 $R(F) = 0.0398$
 $wR(F^2) = 0.1027$
 $S = 1.039$
 2220 reflections
 135 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.1659P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.196$ e Å⁻³
 $\Delta\rho_{min} = -0.165$ e Å⁻³
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Cl	0.64724 (9)	0.40724 (5)	0.29924 (3)	0.0461 (2)
N	0.1465 (3)	0.5463 (2)	0.28759 (8)	0.0330 (4)
C1	0.1219 (3)	0.5973 (2)	0.21848 (9)	0.0334 (4)
C2	-0.1166 (4)	0.6651 (2)	0.20556 (10)	0.0410 (5)
C3	-0.1503 (5)	0.7076 (2)	0.13330 (11)	0.0491 (6)
C4	-0.1383 (4)	0.6110 (2)	0.08390 (11)	0.0508 (6)
C5	0.0999 (5)	0.5442 (2)	0.09715 (10)	0.0566 (7)
C6	0.1320 (4)	0.5003 (2)	0.16902 (10)	0.0480 (6)
C7	0.1671 (4)	0.6250 (2)	0.34670 (9)	0.0356 (5)
C8	0.3979 (5)	0.6984 (2)	0.34851 (10)	0.0474 (6)
C9	0.4236 (6)	0.7720 (2)	0.41135 (12)	0.0620 (7)
C10	0.4191 (5)	0.7021 (2)	0.47445 (11)	0.0562 (7)
C11	0.1935 (5)	0.6267 (3)	0.47190 (11)	0.0670 (8)
C12	0.1683 (5)	0.5526 (2)	0.40921 (11)	0.0551 (7)

Table 2. Selected geometric parameters (Å, °)

N—C7	1.498 (3)	C7—C8	1.511 (3)
N—C1	1.504 (2)	C7—C12	1.513 (3)
C1—C2	1.513 (3)	C8—C9	1.524 (3)
C1—C6	1.514 (3)	C9—C10	1.511 (3)
C2—C3	1.527 (3)	C10—C11	1.501 (3)
C3—C4	1.511 (3)	C11—C12	1.523 (3)
C4—C5	1.507 (3)	Cl···N	3.145 (2)
C5—C6	1.525 (3)	Cl···N ⁱ	3.176 (2)
C7—N—C1	118.6 (2)	N—C7—C12	107.8 (2)
N—C1—C2	111.4 (2)	C8—C7—C12	111.1 (2)
N—C1—C6	107.5 (2)	C7—C8—C9	110.6 (2)
C2—C1—C6	111.4 (2)	C10—C9—C8	112.2 (2)
C1—C2—C3	110.9 (2)	C11—C10—C9	111.7 (2)
C4—C3—C2	111.7 (2)	C10—C11—C12	111.8 (2)
C5—C4—C3	111.3 (2)	C7—C12—C11	111.0 (2)
C4—C5—C6	111.1 (2)	C7—N···Cl	105.19 (11)
C1—C6—C5	111.0 (2)	C1—N···Cl	105.91 (11)
N—C7—C8	111.6 (2)		

Symmetry code: (i) 1 + x, y, z.

H atoms bonded to C atoms were generated and allowed to ride on their parent atoms. H atoms bonded to N atoms were located and refined.

Data collection: *CAD-4 VAX/PC Fortran System* (Enraf-Nonius, 1988). Cell refinement: *CAD-4 VAX/PC Fortran System*. Data reduction: *Xtal3.0* (Hall & Stewart, 1990). Program(s) used to solve structure: *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

The author thanks the University of Malaya for supporting this research (PJP 280/94).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1015). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Enraf-Nonius (1988). *CAD-4 VAX/PC Fortran System. Operator's Guide to the Enraf-Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System*. Enraf-Nonius, Delft, The Netherlands.
- Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Mentzafos, D. & Terzis, A. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 7–11.
- Ng, S. W. (1992). *J. Crystallogr. Spectrosc. Res.* **22**, 615–618.
- Ng, S. W. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 73–75.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). **C51**, 2150–2152

Bis(dicyclohexylammonium) Trithiocarbodiglycolate Trithiocarbodiglycolic Acid

SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

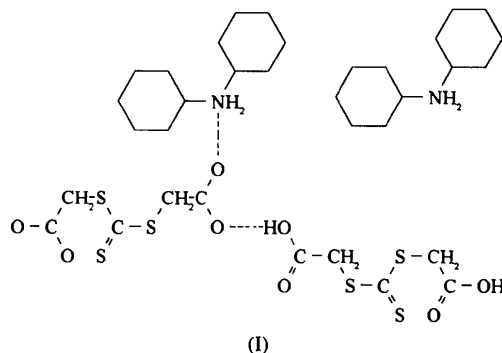
(Received 29 March 1994; accepted 3 March 1995)

Abstract

The [(O₂CCH₂S)₂CS]²⁻ and [(HO₂CCH₂S)₂CS] entities of bis(dicyclohexylammonium) trithiocarbodiglycolate trithiocarbodiglycolic acid, 2C₁₂H₂₄N⁺.C₅H₄O₄S₃²⁻.C₅H₆O₄S₃, form a hydrogen-bonded chain parallel to *c*. The carbonyl O atoms of the acid moiety are additionally hydrogen bonded to the counterion, giving rise to a three-dimensional network.

Comment

A dicarboxylic acid, when treated with an equimolar amount of dicyclohexylamine in ethanol, affords the dicyclohexylammonium hydrogen dicarboxylate, which can then be used in a condensation reaction with an organotin hydroxide or oxide to yield the corresponding (dicarboxylato)organostannate. Thus, oxalic acid dihydrate, when reacted with this amine, afforded the anticipated dicyclohexylammonium hydrogen oxalate (Ng, 1995), which when condensed with triphenyltin hydroxide yielded dicyclohexylammonium oxalatotriphenylstannate (Ng, Kumar Das, Hossain, Goerlitz & van der Helm, 1993). The title compound, (I), did not give a stannate when reacted with triphenyltin hydroxide; the reason for the absence of the reaction between the acid and the hydroxide is not apparent from the crystal structure.



The anionic chain of the title compound consists of [(O₂CCH₂S)₂CS]²⁻ dianions hydrogen bonded to neutral [(HO₂CCH₂S)₂CS] molecules [O···O 2.448 (3) Å].