N3—C4—C5	121.4 (5)	N7—C8—H8	121 (4)
N3—C4—N9	127.3 (5)	N9—C8—H8	125 (4)
C5—C4—N9	111.3 (4)	C4—N9—C8	104.2 (5)
C4—C5—C6	124.8 (4)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*—H···*A*
N7—H7···N9^{i}	1.93 (7)	179 (5)
C8—H8···O13^{ii}	2.42 (6)	149 (4)
C8—H8···O13^{iii}	2.85 (6)	116 (4)

Symmetry codes: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$; (ii) $\frac{3}{2} - x, \frac{1}{2} + y, z - \frac{1}{2}$; (iii) $\frac{3}{2} - x, y - \frac{1}{2}, z - \frac{1}{2}$.

The intensity data were corrected for Lorentz and polarization effects, but not for absorption. All non-H-atom positions were recovered from the initial *E* map. Although subsequent difference Fourier maps yielded peaks suggestive of H-atom positions, the H atoms were placed at idealized positions. The atomic positions and anisotropic displacement parameters (ADPs) for the non-H atoms refined smoothly. The positional and isotropic displacement parameters for H atoms H7 and H8 were included in the least-squares refinement. The methyl H atoms were included in the structure-factor calculation, but not refined. These C—H distances were fixed to 0.96 Å and the isotropic displacement [*U*(H)] was set according to the expression, *U*(H) = *U*(C) + 0.01 Å^2. These parameters were updated every second cycle of the least-squares refinement. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation (Cromer, 1974). The final cycles of refinement included an extinction correction (Larson, 1970). Intermolecular geometries were analyzed using *PLATON* (Spek, 1990).

Data reduction: *NRCVAX DATRD*2 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX* and *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES* (version of January 1994).

The comments of a referee are gratefully acknowledged.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1284). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dicyclohexylammonium (*N*,*N*-Dicyclohexylthiocarbamoylthio)acetate

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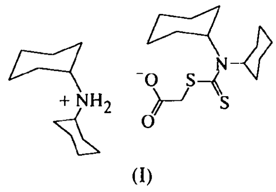
Abstract

The unit cell of dicyclohexylammonium (*N*,*N*-dicyclohexylthiocarbamoylthio)acetate contains two $C_{12}H_{24}N^+.C_{15}H_{24}NO_2S_2^-$ ion pairs in the $P2_1/c$ space group. The ammonium N atom is hydrogen bonded to the carboxyl and carbonyl O atoms, forming a O—C—O···N···O—C—O···N ring. The N-bonded cyclohexyl rings are disordered.

Comment

Dicyclohexylammonium (*N*,*N*-dimethylthiocarbamoylthio)acetate crystallizes as a monohydrate having a hydrogen-bonded network structure (Ng, 1992), whereas the present cyclohexyl analog, $[(cyclo\text{-}C_6H_{11})_2NH_2]^+.[(cyclo\text{-}C_6H_{11})_2NCS_2CH_2CO_2]^-$, (I), contains no water. Two ion pairs are held across a center of inversion by two strong hydrogen bonds [N···O 2.706 (2) and 2.757 (2) Å]. The hydrogen-bond distances are similar to those found in dicyclohexylammonium bis(*N*,*N*-dimethylthiocarbamoylthio)acetate [N···O 2.698 (2) and 2.837 (3) Å; Ng, 1996], which also exists as an ion pair.



(I)

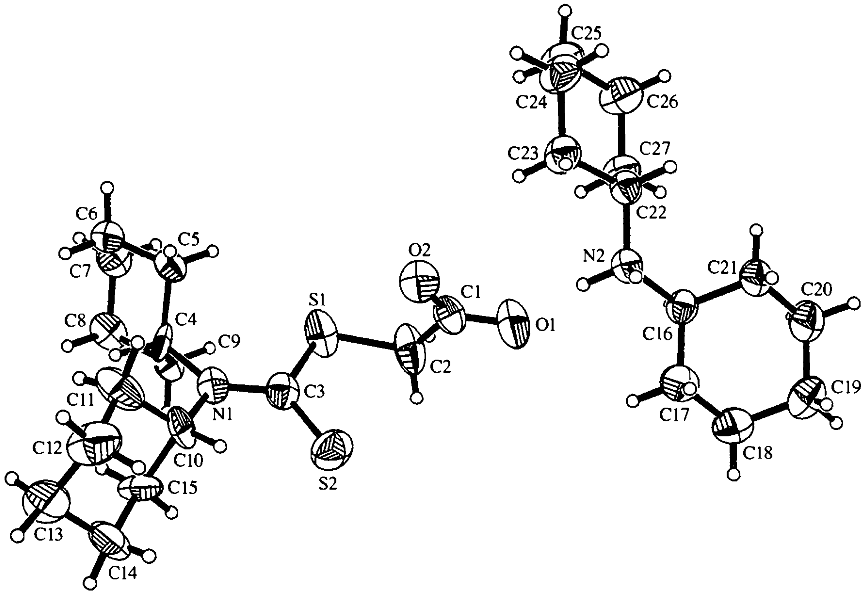


Fig. 1. *ORTEP* (in *SHELXTL/PC*; Sheldrick, 1990*b*) plot of (I) with ellipsoids plotted at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

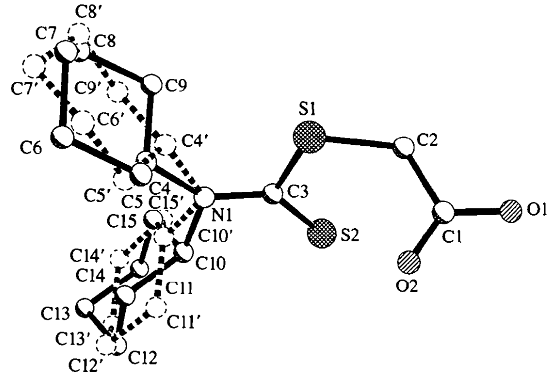


Fig. 2. Plot of the title anion showing the disorder in the N-bonded cyclohexyl rings. H atoms are not shown.

Experimental

(*N*,*N*-Dicyclohexylthiocarbamoylthio)acetic acid was synthesized from dicyclohexylamine, carbon disulfide and sodium chloroacetate, duplicating the method for (*N*,*N*-dimethylthiocarbamoylthio)acetic acid (Pluijgers & van der Kerk, 1961). Equimolar quantities of the acid and dicyclohexylamine were heated in a small volume of ethanol and the salt separated on cooling the filtered solution.

Crystal data

$C_{12}H_{24}N^+.C_{15}H_{24}NO_2S_2^-$
$M_r = 496.79$
Monoclinic
$P2_1/c$
$a = 9.2523\,(7)$ Å
$b = 30.116\,(1)$ Å
$c = 10.2195\,(7)$ Å
$\beta = 95.203\,(3)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14–15°$
$\mu = 0.213$ mm^{-1}
$T = 298\,(2)$ K
Block

$V = 2835.9\,(3)$ Å^3
$Z = 4$
$D_x = 1.164$ Mg m^{-3}
D_m not measured

0.44 × 0.44 × 0.44 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.887$, $T_{max} = 0.911$
5288 measured reflections
4966 independent reflections

3645 reflections with $I > 2\sigma(I)$
$R_{int} = 0.0123$
$\theta_{max} = 24.98°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 35$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0453$
$wR(F^2) = 0.1249$
$S = 1.040$
4966 reflections
414 parameters
H atoms: riding with $U(\mathrm{H}) = 1.5U_{eq}(\mathrm{C})$; ammonium H atoms were located and refined

$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.2946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.243$ e Å^{-3}
$\Delta\rho_{min} = -0.140$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S1	0.27492 (6)	0.13738 (2)	0.68776 (7)	0.0630 (2)
S2	0.53582 (8)	0.16493 (2)	0.86079 (6)	0.0661 (2)
O1	0.2778 (2)	0.05463 (5)	0.9858 (2)	0.0670 (5)
O2	0.4016 (2)	0.05290 (5)	0.8090 (2)	0.0560 (4)

N1	0.4944 (2)	0.18132 (6)	0.6035 (2)	0.0499 (4)
N2	0.3223 (2)	−0.02538 (6)	1.1032 (2)	0.0385 (4)
C1	0.3158 (2)	0.07010 (7)	0.8805 (2)	0.0464 (5)
C2	0.2458 (3)	0.11444 (8)	0.8448 (3)	0.0672 (7)
C3	0.4467 (2)	0.16351 (7)	0.7116 (2)	0.0443 (5)
C4†	0.4336 (6)	0.1863 (3)	0.4670 (6)	0.040 (2)
C5†	0.396 (1)	0.1430 (3)	0.394 (1)	0.041 (2)
C6†	0.352 (1)	0.1561 (3)	0.2498 (9)	0.055 (2)
C7†	0.2176 (9)	0.1857 (3)	0.2458 (8)	0.062 (2)
C8†	0.257 (1)	0.2285 (4)	0.3232 (9)	0.050 (2)
C9†	0.2993 (6)	0.2162 (2)	0.4684 (5)	0.042 (1)
C4′†	0.3820 (7)	0.1833 (3)	0.4831 (6)	0.045 (2)
C5′†	0.420 (1)	0.1498 (4)	0.380 (1)	0.059 (4)
C6′†	0.305 (1)	0.1457 (3)	0.264 (1)	0.080 (3)
C7′†	0.261 (1)	0.1901 (3)	0.2049 (8)	0.069 (2)
C8′†	0.222 (1)	0.2236 (4)	0.307 (1)	0.066 (4)
C9′†	0.337 (1)	0.2277 (2)	0.4228 (8)	0.082 (2)
C10†	0.6554 (6)	0.1945 (2)	0.6253 (7)	0.042 (2)
C11†	0.7446 (9)	0.1799 (3)	0.5105 (7)	0.061 (2)
C12†	0.904 (1)	0.1942 (4)	0.555 (2)	0.089 (5)
C13†	0.9047 (9)	0.2450 (4)	0.5420 (7)	0.071 (3)
C14†	0.817 (1)	0.2607 (4)	0.654 (1)	0.070 (3)
C15†	0.657 (1)	0.2455 (3)	0.623 (2)	0.060 (4)
C10′†	0.6293 (7)	0.2037 (2)	0.5866 (9)	0.059 (2)
C11′†	0.7447 (8)	0.1702 (3)	0.5723 (9)	0.075 (3)
C12′†	0.885 (1)	0.1910 (4)	0.532 (2)	0.080 (4)
C13′†	0.926 (1)	0.2338 (4)	0.599 (1)	0.106 (4)
C14′†	0.807 (2)	0.2674 (4)	0.611 (1)	0.088 (5)
C15′†	0.663 (1)	0.2484 (3)	0.645 (2)	0.064 (4)
C16	0.2513 (2)	−0.01666 (6)	1.2271 (2)	0.0407 (5)
C17	0.3392 (3)	0.01768 (8)	1.3071 (2)	0.0585 (6)
C18	0.2706 (3)	0.02832 (9)	1.4344 (2)	0.0720 (7)
C19	0.2476 (3)	−0.01305 (9)	1.5130 (2)	0.0739 (8)
C20	0.1617 (3)	−0.04737 (9)	1.4305 (2)	0.0680 (7)
C21	0.2335 (3)	−0.05847 (7)	1.3066 (2)	0.0532 (6)
C22	0.2592 (2)	−0.06181 (6)	1.0154 (2)	0.0386 (4)
C23	0.3407 (2)	−0.06333 (8)	0.8933 (2)	0.0496 (5)
C24	0.2819 (3)	−0.10060 (9)	0.8023 (2)	0.0609 (6)
C25	0.1196 (3)	−0.09669 (9)	0.7688 (2)	0.0664 (7)
C26	0.0394 (2)	−0.09350 (9)	0.8915 (2)	0.0626 (7)
C27	0.0976 (2)	−0.05567 (8)	0.9792 (2)	0.0486 (5)

† Site occupancy = 0.50.

Table 2. *Selected geometric parameters (Å, °)*

S1—C2	1.789 (2)	N1—C10	1.538 (6)
S1—C3	1.770 (2)	N1—C4′	1.539 (6)
S2—C3	1.667 (2)	N1—C10′	1.442 (6)
O1—C1	1.252 (3)	N2—C16	1.501 (2)
O2—C1	1.240 (3)	N2—C22	1.501 (3)
N1—C3	1.338 (3)	N2—O1	2.706 (2)
N1—C4	1.464 (6)	N2—O2^{i}	2.757 (2)
C2—S1—C3	104.7 (1)	S2—C3—S1	120.1 (1)
C3—N1—C4	134.5 (3)	N1—C4—C5	115.5 (6)
C3—N1—C10	111.8 (3)	N1—C4—C9	107.0 (4)
C3—N1—C4′	115.2 (3)	C9′—C4′—N1	120.0 (6)
C3—N1—C10′	129.8 (4)	C5′—C4′—N1	110.1 (6)
C16—N2—C22	117.1 (2)	C15—C10—N1	105.6 (6)
C4—N1—C10	113.2 (4)	C11—C10—N1	113.0 (6)
C4′—N1—C10′	114.4 (4)	C11′—C10′—N1	109.4 (6)
O1—C1—O2	126.5 (2)	C15′—C10′—N1	121.7 (7)
O2—C1—C2	120.6 (2)	C17—C16—N2	108.8 (2)
O1—C1—C2	112.9 (2)	C21—C16—N2	112.6 (2)
C1—C2—S1	117.5 (2)	C23—C22—N2	108.6 (2)
N1—C3—S2	124.8 (2)	C27—C22—N2	112.2 (2)
N1—C3—S1	115.1 (2)		

Symmetry code: (i) $1 - x, -y, 2 - z$.

The two cyclohexyl rings bonded to the thiocarbamoyl N atom are both disordered over two positions and were refined with site occupancies of 0.5, subject to constraints in bond distances (1,2-related C atoms 1.54±0.01, 1,3-related C atoms 2.52±0.02 and N—C 1.45±0.01 Å).

Data collection: *CAD*-4 *VAX*/*PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX*/*PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Polysulfonylamines. LXXXII. Oxalyl Chloride Dimesylamide†

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Abstract

The molecule of the previously unknown title compound, *N*,*N*-dimesyloxamoyl chloride, $C_4H_6ClNO_6S_2$, is characterized by a long C_{sp^2}—N_{sp^2} bond [1.408 (2) Å]

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† Part LXXXI: Wirth, Henschel, Blaschette & Jones (1997).

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