

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.

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Bis(dicyclohexylammonium) Trithiocarbodiglycolate Trithiocarbodiglycolic Acid

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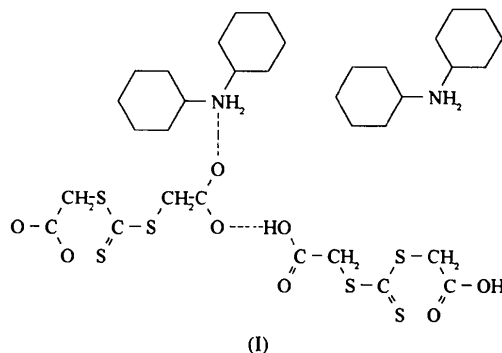
(Received 29 March 1994; accepted 3 March 1995)

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].

The cationic counterion forms hydrogen bonds to adjacent chains through the double-bonded O atoms [$N \cdots O$ 2.784 (3) and 2.889 (4) Å]. The anionic chain displays C=S double-bond distances of 1.617 (5) and 1.619 (5) Å, which are shorter than the statistical mean of 1.671 Å found in structures having the $X_2C=S$ unit (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987).

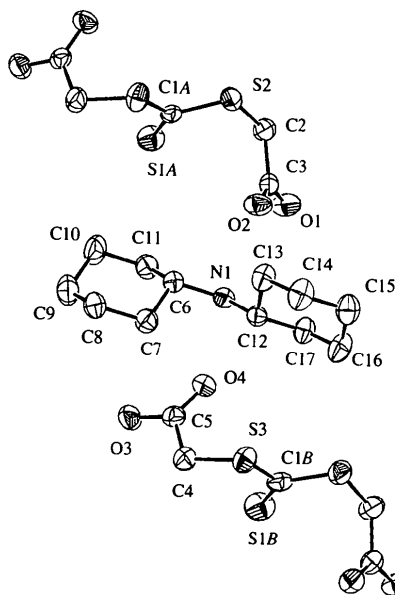
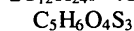
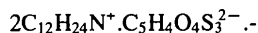


Fig. 1. Atomic labelling scheme for bis(dicyclohexylammonium) trithiocarbodiglycolate trithiocarbodiglycolic acid. The displacement ellipsoids are plotted at the 50% probability level.

Experimental

Trithiocarboglycolic acid was prepared using a modification of the literature method (Reid, 1962; Strube, 1963). The acid and an excess molar amount of dicyclohexylamine were dissolved in ethanol and the solution heated briefly. This solution afforded large yellow crystals of the dicyclohexylammonium hydrogen salt when cooled slowly. Elemental analysis: found C 50.10, H 7.25, N 3.36%; calculated for $C_{17}H_{29}NO_4S_3$ C 50.90, H 7.17, N 3.44%.

Crystal data



$$M_r = 815.18$$

Monoclinic

$P2_1/a$

$$a = 10.664 (2) \text{ \AA}$$

$$b = 9.829 (4) \text{ \AA}$$

$$c = 20.058 (2) \text{ \AA}$$

$$\beta = 105.315 (5)^\circ$$

$$V = 2027.6 (9) \text{ \AA}^3$$

$$Z = 2$$

$$D_x = 1.335 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation

$$\lambda = 0.71073 \text{ \AA}$$

Cell parameters from 25 reflections

$$\theta = 6-12^\circ$$

$$\mu = 0.387 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Cube

$$0.29 \times 0.29 \times 0.29 \text{ mm}$$

Yellow

Data collection

Enraf-Nonius CAD-4

diffractometer

$\omega-2\theta$ scans

Absorption correction:

none

7310 measured reflections

3571 independent reflections

2273 observed reflections

$$[I > 2\sigma(I)]$$

$$R_{\text{int}} = 0.0515$$

$$\theta_{\text{max}} = 24.96^\circ$$

$$h = 0 \rightarrow 12$$

$$k = -11 \rightarrow 11$$

$$l = -23 \rightarrow 22$$

3 standard reflections

frequency: 60 min

intensity decay: none

Refinement

Refinement on F^2

$$R(F) = 0.0435$$

$$wR(F^2) = 0.1186$$

$$S = 0.992$$

3571 reflections

240 parameters

$$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.0577P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$$\Delta\rho_{\text{max}} = 0.229 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.221 \text{ e \AA}^{-3}$$

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 (\AA^2)

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S1A	3/4	0.32840 (13)	1/2	0.0520 (3)
S1B	3/4	0.36004 (13)	0	0.0631 (4)
S2	0.84089 (9)	0.05733 (9)	0.46167 (4)	0.0473 (3)
S3	0.66937 (9)	0.62985 (9)	0.04536 (4)	0.0476 (2)
O1	0.8445 (2)	0.3111 (2)	0.31111 (12)	0.0521 (6)
O2	0.6960 (2)	0.1604 (2)	0.32197 (11)	0.0498 (6)
O3	0.6894 (2)	0.3920 (2)	0.20567 (12)	0.0506 (6)
O4	0.8431 (2)	0.5126 (2)	0.17547 (11)	0.0471 (6)
N1	0.4493 (3)	0.2395 (3)	0.24539 (13)	0.0335 (6)
C1A	3/4	0.1639 (4)	1/2	0.0336 (10)
C1B	3/4	0.5247 (4)	0	0.0397 (11)
C2	0.9005 (3)	0.1697 (4)	0.4072 (2)	0.0481 (9)
C3	0.8022 (3)	0.2159 (3)	0.34175 (15)	0.0372 (8)
C4	0.6228 (3)	0.5145 (4)	0.1036 (2)	0.0492 (9)
C5	0.7311 (3)	0.4719 (3)	0.1657 (2)	0.0398 (8)
C6	0.3867 (3)	0.2372 (3)	0.30438 (15)	0.0348 (7)
C7	0.2400 (3)	0.2443 (4)	0.2796 (2)	0.0431 (8)
C8	0.1787 (3)	0.2494 (4)	0.3399 (2)	0.0493 (9)
C9	0.2339 (4)	0.3654 (4)	0.3888 (2)	0.0625 (11)
C10	0.3812 (3)	0.3561 (4)	0.4137 (2)	0.0587 (10)
C11	0.4417 (3)	0.3537 (3)	0.3528 (2)	0.0440 (8)
C12	0.4058 (3)	0.1350 (3)	0.18885 (14)	0.0337 (7)
C13	0.4260 (3)	-0.0075 (3)	0.2179 (2)	0.0465 (8)
C14	0.3944 (4)	-0.1135 (4)	0.1596 (2)	0.0586 (10)
C15	0.4688 (4)	-0.0870 (4)	0.1064 (2)	0.0566 (10)
C16	0.4451 (4)	0.0559 (4)	0.0778 (2)	0.0602 (10)
C17	0.4801 (3)	0.1605 (3)	0.1355 (2)	0.0438 (8)

Table 2. Selected geometric parameters (\AA , $^\circ$)

S1A—C1A	1.617 (5)	C2—C3	1.518 (4)
S1B—C1B	1.619 (5)	C4—C5	1.517 (4)
S2—C1A	1.738 (3)	C6—C7	1.513 (4)
S2—C2	1.784 (3)	C6—C11	1.515 (4)
S3—C1B	1.746 (3)	C7—C8	1.520 (4)
S3—C4	1.790 (3)	C8—C9	1.517 (5)
O1—C3	1.266 (4)	C9—C10	1.521 (5)
O2—C3	1.224 (4)	C10—C11	1.524 (4)
O3—C5	1.283 (4)	C12—C13	1.510 (4)
O4—C5	1.225 (4)	C12—C17	1.511 (4)
N1—C6	1.504 (4)	C13—C14	1.535 (4)
N1—C12	1.510 (4)	C14—C15	1.512 (5)
C1A—S2 ⁱ	1.738 (3)	C15—C16	1.513 (5)
C1B—S3 ⁱⁱ	1.746 (3)	C16—C17	1.519 (4)

C1A—S2—C2	103.2 (2)	N1—C6—C7	112.1 (2)
C1B—S3—C4	103.0 (2)	N1—C6—C11	108.4 (2)
C6—N1—C12	117.9 (2)	C7—C6—C11	111.5 (3)
S1A—C1A—S2	127.04 (12)	C6—C7—C8	111.3 (3)
S1A—C1A—S2 ⁱ	127.04 (12)	C9—C8—C7	111.4 (3)
S2—C1A—S2 ⁱ	105.9 (2)	C8—C9—C10	110.9 (3)
S1B—C1B—S3	126.28 (12)	C9—C10—C11	110.9 (3)
S1B—C1B—S3 ⁱⁱ	126.28 (12)	C6—C11—C10	110.6 (3)
S3—C1B—S3 ⁱⁱ	107.4 (2)	N1—C12—C13	110.9 (2)
C3—C2—S2	116.0 (2)	N1—C12—C17	107.7 (2)
O2—C3—O1	126.1 (3)	C13—C12—C17	112.2 (3)
O2—C3—C2	121.0 (3)	C12—C13—C14	110.9 (3)
O1—C3—C2	112.8 (3)	C15—C14—C13	111.7 (3)
C5—C4—S3	115.4 (2)	C14—C15—C16	111.2 (3)
O4—C5—O3	125.8 (3)	C15—C16—C17	110.9 (3)
O4—C5—C4	122.7 (3)	C12—C17—C16	111.0 (3)
O3—C5—C4	111.5 (3)		

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

H atoms were allowed to ride on their parent C atoms with a displacement factor 1.5 times that of the parent atom. The acidic and ammonium H 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 152/91).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1016). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(diethyldithiocarbamato)(4-methoxyphenyl)tellurium(IV), *p*-MeOC₆H₄Te(S₂CNEt₂)₃, and the Triclinic Modification of Tris(diethyldithiocarbamato)phenyl-tellurium(IV), PhTe(S₂CNEt₂)₃

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Abstract

The structures of the tellurium(IV) complexes [Te(C₅H₁₀NS₂)₃(C₇H₇O)], (1), and [Te(C₅H₁₀NS₂)₃(C₆H₅)], (2) (the triclinic modification), have been investigated. In both structures the coordination of the Te atom is distorted pentagonal bipyramidal, with four S atoms from two near symmetrically bidentate dithiocarbamate ligands [Te—S 2.625(1)–2.889(1) Å] and a fifth S atom from the third unsymmetrically bidentate dithiocarbamate ligand [Te—S 2.585(1)–2.602(1) Å] in equatorial positions. The aryl group is axial [Te—C 2.148(5)–2.160(3) Å]; the second axial position is occupied by the second S atom of the unsymmetrically bidentate dithiocarbamate ligand [Te···S 3.235(2)–3.241(1) Å], the *trans* angle C—Te···S being 144.6–147.2(1)°. In the structure of (2), the two crystallographically independent molecules have different orientations of the ethyl groups in all three ligands and slightly differing phenyl group orientations.

Comment

In the crystal structures of both (1) and (2), the Te atom has distorted pentagonal bipyramidal coordination geometry (Figs. 1 and 2); two dithiocarbamate groups behave as near symmetrically bidentate chelate ligands and are situated in the equatorial plane together with another strongly coordinated S atom from the third dithiocarbamate group. The second S atom of this group seems to be weakly coordinated and is axial, but with significant deviation from an ideal position *trans* to the aryl ligand, which occupies the second axial position.

Detailed investigation shows that the equatorial bidentate dithiocarbamate ligands are not quite symmetrically coordinated: the Te—S bonds quasi *trans* to the unique unsymmetrically coordinated dithio-