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catena-Poly[[*(N,N*-diethyldithio- carbamato- κ^2 S:S')phenylbismuth(III)]- μ -chlorido]

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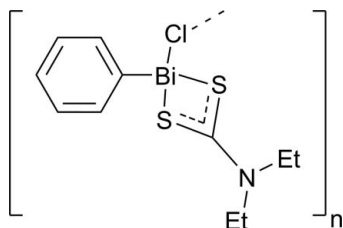
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.019$ Å;
 R factor = 0.056; wR factor = 0.149; data-to-parameter ratio = 16.6.

In the title compound, $[\text{Bi}(\text{C}_6\text{H}_5)(\text{C}_5\text{H}_{10}\text{NS}_2)\text{Cl}]_n$, the Bi atom is coordinated by two S atoms of the dithiocarbamate ligand, one C atom of the phenyl group and one Cl atom in a four-coordinated tetrahedral configuration. Molecules are linked by Cl atoms to form a zigzag chain extending in the c direction.

Related literature

For related literature see: Yin *et al.* (2003); Bardaji *et al.* (1994); Xu *et al.* (2001).



Experimental

Crystal data

$[\text{Bi}(\text{C}_6\text{H}_5)(\text{C}_5\text{H}_{10}\text{NS}_2)\text{Cl}]$
 $M_r = 469.79$

Monoclinic, $P2_1/c$

$a = 9.2029$ (9) Å

$b = 18.1432$ (17) Å

$c = 9.0779$ (8) Å

$\beta = 106.811$ (2)°

$V = 1451.0$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 12.60$ mm⁻¹

$T = 298$ (2) K

$0.23 \times 0.22 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.160$, $T_{\max} = 0.177$

(expected range = 0.064–0.071)

6443 measured reflections

2444 independent reflections

1877 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.095$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.149$

$S = 0.92$

2444 reflections

147 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 2.04$ e Å⁻³

$\Delta\rho_{\text{min}} = -2.84$ e Å⁻³

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: SU2063).

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supplementary materials

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***catena*-Poly[[*(N,N*-diethyldithiocarbamato- κ^2 S:S')phenylbismuth(III)]- μ -chlorido]**

L. Cui, H. Yin, M. Yang, L. Quan and D. Wang

Comment

Dithiocarbamates have been known as effective ligands for many years. They can form chelates (Xu *et al.*, 2001) or act as bridging ligands (Bardaji *et al.*, 1994). However, the chemistry of main-group metal complexes with dithiocarbamates has been less extensively studied, and only a few reports describing bismuth dithiocarbamate complexes have appeared (Yin *et al.*, 2003). As a continuation of our interest in sulfur-containing ligands, we report here the synthesis and structure of the title compound.

The molecular structure of the title compound is illustrated in Fig. 1. The geometry of the Bi atom is four-coordinated tetrahedral. Two Bi—S bonds distance are not similar, the Bi1—S1 bond length is 2.647 (3) Å and the Bi1—S2 bond length is 2.690 (4) Å. The molecules are linked by the chlorine atom Cl1 [Bi1—Cl1 = 2.908 (3) Å and Bi1...Cl1ⁱ = 2.920 (3); (i) = x, -y+0.5, z+0.5] to form a one-dimensional zig-zag polymer chain extending in the c direction (Fig. 2).

Experimental

A mixture of (*N,N*-diethylcarbamato-1 κ^2 S:S')sodium (0.2 mmol) and phenylbismuth dichloride (0.1 mmol) in absolute THF was heated under reflux with stirring for 24 h. Diethyl ether and hexane were added to this solution to precipitate the product, which was then recrystallized from a dichloromethane-hexane mixture (1:1 v/v). After 14 days large colorless block-shaped crystals of the title complex, suitable for X-ray diffraction analysis, were obtained.

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms, with methylene C—H distances of 0.97 Å and aromatic C—H distances of 0.93 Å. The $U_{iso}(H)$ values were set at 1.2 $U_{eq}(C)$.

Figures

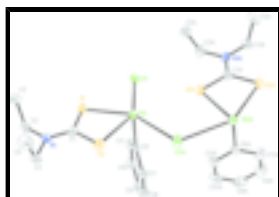


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

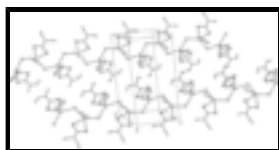


Fig. 2. The crystal packing of the title compound, viewed approximately along the *a* axis.

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catena-Poly[[*(N,N*-diethyldithiocarbamato- κ^2 S:S')phenylbismuth(III)]- μ -chlorido]

Crystal data

[Bi(C ₆ H ₅)(C ₅ H ₁₀ NS ₂)Cl]	$F_{000} = 880$
$M_r = 469.79$	$D_x = 2.151 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.2029 (9) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 18.1432 (17) \text{ \AA}$	Cell parameters from 3883 reflections
$c = 9.0779 (8) \text{ \AA}$	$\theta = 2.3\text{--}28.2^\circ$
$\beta = 106.811 (2)^\circ$	$\mu = 12.60 \text{ mm}^{-1}$
$V = 1451.0 (2) \text{ \AA}^3$	$T = 298 (2) \text{ K}$
$Z = 4$	Block, colorless
	$0.23 \times 0.22 \times 0.21 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2444 independent reflections
Radiation source: fine-focus sealed tube	1877 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.095$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.160, T_{\text{max}} = 0.177$	$k = -21 \rightarrow 19$
6443 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.149$	$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2]$
$S = 0.92$	where $P = (F_o^2 + 2F_c^2)/3$
2444 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
147 parameters	$\Delta\rho_{\text{max}} = 2.04 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -2.84 \text{ e \AA}^{-3}$
	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Bi1	0.83539 (5)	0.19587 (2)	0.77837 (4)	0.0353 (2)
Cl1	0.8497 (4)	0.32758 (19)	0.6005 (3)	0.0448 (8)
N1	0.7954 (11)	-0.0056 (5)	0.4907 (10)	0.036 (2)
S1	0.8233 (4)	0.14036 (18)	0.5054 (3)	0.0435 (8)
S2	0.8036 (4)	0.0484 (2)	0.7681 (3)	0.0462 (8)
C1	0.8051 (13)	0.0546 (7)	0.5771 (12)	0.040 (3)
C2	0.7991 (16)	0.0000 (7)	0.3290 (12)	0.051 (3)
H2A	0.8309	-0.0470	0.2976	0.061*
H2B	0.8737	0.0366	0.3225	0.061*
C3	0.6464 (14)	0.0208 (8)	0.2196 (11)	0.053 (4)
H3A	0.5721	-0.0154	0.2255	0.079*
H3B	0.6540	0.0227	0.1164	0.079*
H3C	0.6163	0.0682	0.2477	0.079*
C4	0.7770 (14)	-0.0790 (7)	0.5486 (14)	0.045 (3)
H4A	0.8283	-0.0807	0.6581	0.054*
H4B	0.8252	-0.1148	0.4985	0.054*
C5	0.6136 (17)	-0.0998 (9)	0.5216 (18)	0.069 (4)
H5A	0.5645	-0.0637	0.5679	0.103*
H5B	0.6078	-0.1472	0.5667	0.103*
H5C	0.5641	-0.1020	0.4130	0.103*
C6	1.0885 (14)	0.1853 (6)	0.8490 (12)	0.035 (3)
C7	1.1620 (16)	0.1381 (8)	0.9745 (13)	0.052 (4)
H7	1.1050	0.1124	1.0271	0.063*
C8	1.3156 (18)	0.1310 (9)	1.0167 (15)	0.063 (4)
H8	1.3624	0.1002	1.0985	0.076*
C9	1.4023 (18)	0.1673 (9)	0.9431 (17)	0.064 (4)
H9	1.5074	0.1626	0.9761	0.077*
C10	1.3332 (13)	0.2117 (8)	0.8179 (18)	0.055 (4)
H10	1.3919	0.2351	0.7639	0.066*
C11	1.1787 (15)	0.2213 (7)	0.7736 (15)	0.048 (3)
H11	1.1339	0.2524	0.6915	0.058*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Bi1	0.0391 (4)	0.0378 (4)	0.0300 (3)	0.00102 (19)	0.0113 (2)	-0.00018 (16)
Cl1	0.059 (2)	0.0432 (18)	0.0374 (14)	0.0086 (16)	0.0221 (14)	0.0032 (13)

supplementary materials

N1	0.035 (6)	0.034 (6)	0.039 (5)	0.008 (5)	0.009 (4)	0.003 (4)
S1	0.064 (2)	0.0374 (18)	0.0303 (13)	-0.0063 (16)	0.0152 (13)	0.0013 (12)
S2	0.061 (2)	0.043 (2)	0.0348 (14)	-0.0054 (17)	0.0142 (14)	0.0066 (13)
C1	0.040 (7)	0.043 (8)	0.037 (6)	0.002 (6)	0.010 (5)	0.005 (5)
C2	0.073 (10)	0.043 (8)	0.039 (6)	0.014 (7)	0.022 (6)	-0.003 (5)
C3	0.060 (10)	0.048 (9)	0.046 (7)	0.008 (7)	0.008 (6)	0.003 (5)
C4	0.039 (8)	0.030 (7)	0.063 (7)	-0.001 (6)	0.012 (6)	0.002 (6)
C5	0.073 (11)	0.052 (10)	0.093 (10)	-0.010 (9)	0.042 (9)	0.009 (8)
C6	0.041 (8)	0.032 (7)	0.037 (6)	0.009 (5)	0.019 (5)	-0.002 (5)
C7	0.058 (9)	0.057 (10)	0.043 (6)	0.021 (7)	0.016 (6)	0.005 (6)
C8	0.069 (11)	0.068 (11)	0.051 (7)	0.031 (9)	0.016 (7)	0.004 (7)
C9	0.046 (9)	0.067 (11)	0.071 (9)	0.006 (8)	0.000 (8)	-0.028 (9)
C10	0.012 (7)	0.059 (10)	0.094 (10)	-0.009 (6)	0.016 (7)	-0.006 (8)
C11	0.055 (9)	0.039 (8)	0.055 (7)	-0.012 (7)	0.023 (6)	0.000 (6)

Geometric parameters (Å, °)

Bi1—C6	2.238 (13)	C4—C5	1.500 (17)
Bi1—S1	2.647 (3)	C4—H4A	0.9700
Bi1—S2	2.690 (4)	C4—H4B	0.9700
Bi1—Cl1	2.908 (3)	C5—H5A	0.9600
Bi1—Cl1 ⁱ	2.920 (3)	C5—H5B	0.9600
Cl1—Bi1 ⁱⁱ	2.920 (3)	C5—H5C	0.9600
N1—C1	1.332 (15)	C6—C11	1.383 (16)
N1—C4	1.459 (14)	C6—C7	1.429 (16)
N1—C2	1.481 (13)	C7—C8	1.360 (19)
S1—C1	1.714 (13)	C7—H7	0.9300
S2—C1	1.742 (10)	C8—C9	1.35 (2)
C2—C3	1.516 (15)	C8—H8	0.9300
C2—H2A	0.9700	C9—C10	1.39 (2)
C2—H2B	0.9700	C9—H9	0.9300
C3—H3A	0.9600	C10—C11	1.373 (16)
C3—H3B	0.9600	C10—H10	0.9300
C3—H3C	0.9600	C11—H11	0.9300
C6—Bi1—S1	89.7 (3)	N1—C4—C5	112.7 (11)
C6—Bi1—S2	91.1 (3)	N1—C4—H4A	109.0
S1—Bi1—S2	67.33 (9)	C5—C4—H4A	109.0
C6—Bi1—Cl1	91.1 (3)	N1—C4—H4B	109.0
S1—Bi1—Cl1	77.84 (9)	C5—C4—H4B	109.0
S2—Bi1—Cl1	145.09 (8)	H4A—C4—H4B	107.8
C6—Bi1—Cl1 ⁱ	87.6 (3)	C4—C5—H5A	109.5
S1—Bi1—Cl1 ⁱ	149.27 (10)	C4—C5—H5B	109.5
S2—Bi1—Cl1 ⁱ	82.11 (8)	H5A—C5—H5B	109.5
Cl1—Bi1—Cl1 ⁱ	132.80 (6)	C4—C5—H5C	109.5
Bi1—Cl1—Bi1 ⁱⁱ	116.11 (11)	H5A—C5—H5C	109.5
C1—N1—C4	122.1 (9)	H5B—C5—H5C	109.5
C1—N1—C2	120.7 (10)	C11—C6—C7	117.6 (12)

C4—N1—C2	117.2 (9)	C11—C6—Bi1	122.8 (9)
C1—S1—Bi1	88.4 (4)	C7—C6—Bi1	119.5 (9)
C1—S2—Bi1	86.5 (4)	C8—C7—C6	119.7 (13)
N1—C1—S1	121.2 (8)	C8—C7—H7	120.2
N1—C1—S2	121.0 (9)	C6—C7—H7	120.2
S1—C1—S2	117.8 (7)	C7—C8—C9	122.0 (14)
N1—C2—C3	112.5 (10)	C7—C8—H8	119.0
N1—C2—H2A	109.1	C9—C8—H8	119.0
C3—C2—H2A	109.1	C8—C9—C10	119.4 (15)
N1—C2—H2B	109.1	C8—C9—H9	120.3
C3—C2—H2B	109.1	C10—C9—H9	120.3
H2A—C2—H2B	107.8	C11—C10—C9	120.2 (14)
C2—C3—H3A	109.5	C11—C10—H10	119.9
C2—C3—H3B	109.5	C9—C10—H10	119.9
H3A—C3—H3B	109.5	C6—C11—C10	121.0 (13)
C2—C3—H3C	109.5	C6—C11—H11	119.5
H3A—C3—H3C	109.5	C10—C11—H11	119.5
H3B—C3—H3C	109.5		
C6—Bi1—C11—Bi1 ⁱⁱ	90.5 (3)	C1—N1—C2—C3	-81.2 (14)
S1—Bi1—C11—Bi1 ⁱⁱ	1.09 (12)	C4—N1—C2—C3	97.5 (13)
S2—Bi1—C11—Bi1 ⁱⁱ	-2.9 (2)	C1—N1—C4—C5	89.4 (14)
C11 ⁱ —Bi1—C11—Bi1 ⁱⁱ	178.29 (5)	C2—N1—C4—C5	-89.2 (13)
C6—Bi1—S1—C1	91.2 (5)	S1—Bi1—C6—C11	60.6 (10)
S2—Bi1—S1—C1	-0.1 (4)	S2—Bi1—C6—C11	127.9 (10)
C11—Bi1—S1—C1	-177.6 (4)	C11—Bi1—C6—C11	-17.2 (10)
C11 ⁱ —Bi1—S1—C1	6.4 (5)	C11 ⁱ —Bi1—C6—C11	-150.0 (10)
C6—Bi1—S2—C1	-89.1 (5)	S1—Bi1—C6—C7	-117.6 (9)
S1—Bi1—S2—C1	0.1 (4)	S2—Bi1—C6—C7	-50.3 (9)
C11—Bi1—S2—C1	4.3 (4)	C11—Bi1—C6—C7	164.6 (9)
C11 ⁱ —Bi1—S2—C1	-176.6 (4)	C11 ⁱ —Bi1—C6—C7	31.8 (9)
C4—N1—C1—S1	-178.9 (8)	C11—C6—C7—C8	1.0 (19)
C2—N1—C1—S1	-0.3 (15)	Bi1—C6—C7—C8	179.3 (10)
C4—N1—C1—S2	2.4 (15)	C6—C7—C8—C9	0(2)
C2—N1—C1—S2	-179.0 (8)	C7—C8—C9—C10	-2(2)
Bi1—S1—C1—N1	-178.6 (10)	C8—C9—C10—C11	3(2)
Bi1—S1—C1—S2	0.1 (7)	C7—C6—C11—C10	0.1 (19)
Bi1—S2—C1—N1	178.6 (10)	Bi1—C6—C11—C10	-178.2 (10)
Bi1—S2—C1—S1	-0.1 (6)	C9—C10—C11—C6	-2(2)

Symmetry codes: (i) $x, -y+1/2, z+1/2$; (ii) $x, -y+1/2, z-1/2$.

Fig. 1

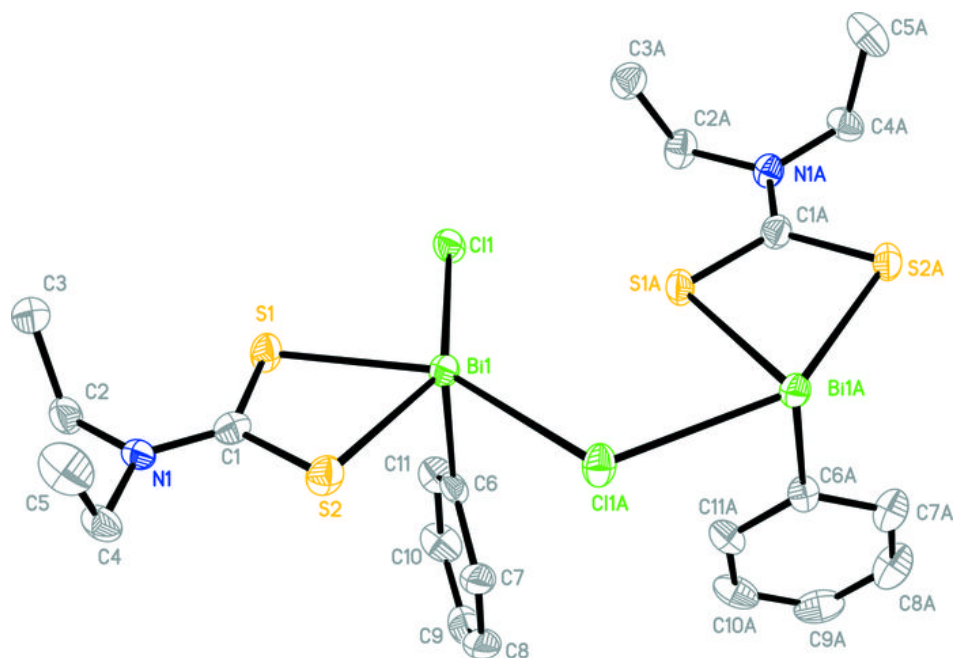


Fig. 2

