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## Key indicators

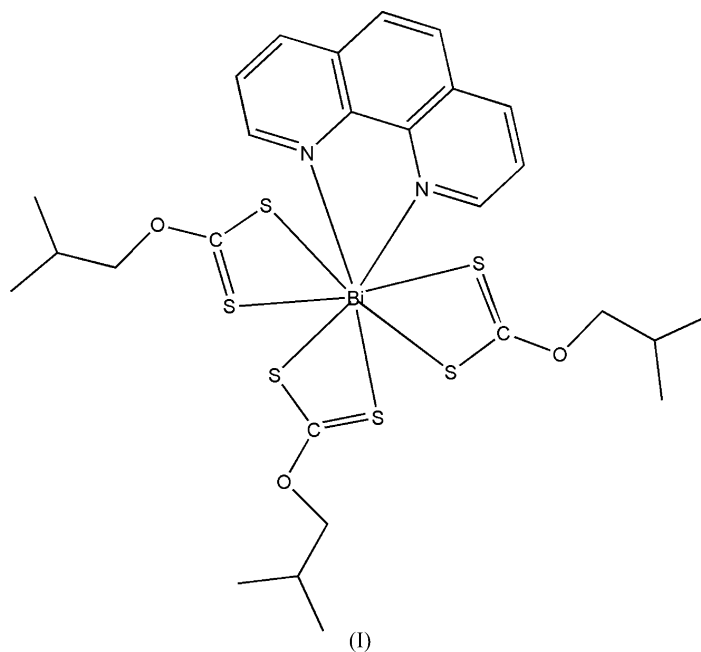
Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.015\text{ \AA}$   
Disorder in main residue  
 $R$  factor = 0.045  
 $wR$  factor = 0.119  
Data-to-parameter ratio = 15.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Tris(*O*-isobutyl dithiocarbonato- $\kappa^2\text{S,S}'$ )-  
(1,10-phenanthroline)bismuth(III)

The title compound,  $[\text{Bi}(\text{C}_5\text{H}_9\text{OS}_2)_2(\text{C}_{12}\text{H}_8\text{N}_2)]$ , is monomeric, with the Bi atom chelated by the S atoms of three anionic *O*-isobutyl dithiocarbonate ligands and the N atoms of 1,10-phenanthroline. The central Bi atom is eight-coordinate in a capped distorted pentagonal bipyramidal geometry.

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## Comment

There has recently been increasing interest in bismuth(III) coordination chemistry, particularly in the light of the role of bismuth compounds in  $^{212}\text{Bi}$  isotope therapy in cancer research (Sun *et al.*, 1997) and the use of bismuth complexes in the treatment of peptic ulcers (Baxter, 1992). In particular, the interaction of bismuth(III) salts with chelating nitrogen-donor ligands has been actively studied. In view of this, and our interest in sulfur-containing ligands (Yin *et al.*, 2006), we report here the synthesis and structure of the title compound, (I) (Fig. 1 and Table 1).



In the title complex, the Bi atom is eight-coordinated by the S atoms of bidentate *O*-isobutyl dithiocarbonate ligands and by the N atoms of 1,10-phenanthroline. Atoms S1 and N2 in axial positions, and atoms S2, S3, S4, S5 and S6 in the equatorial plane. The remaining N atom (N3) of the 1,10-phenanthroline ligand caps the S4/S6/N2 face, giving a highly distorted capped pentagonal bipyramidal coordination geometry. One Bi–S bond in each of the three *O*-isobutyl dithiocarbonate ligands is significantly longer than the other (Bi to S2, S4 and S6, mean =

2.944 Å, and to S1, S3 and S5, mean = 2.764 Å). The Bi1–N1 and Bi1–N2 distances fall in the same range as in other Bi–N complexes (Baraanyi *et al.*, 1977).

### Experimental

To a stirred solution of BiI<sub>3</sub> (0.2 mmol) in acetonitrile (20 ml), sodium *O*-isobutyl dithiocarbonate (0.6 mmol) was added. The reaction mixture was stirred for 3 h at 298 K. An orange solution was obtained and then filtered. The solvent was removed *in vacuo* and the solid product recrystallized from ethanol, giving orange–red crystals of (I) [yield 74%; m.p. 401 K (decomposition)]. Analysis calculated (%) for C<sub>15</sub>H<sub>24</sub>BiN<sub>3</sub>S<sub>6</sub>: C 38.75, H 4.22, N 3.45%; found: C 38.88, H 4.43, N 3.62%.

#### Crystal data

[Bi(C <sub>5</sub> H <sub>9</sub> OS <sub>2</sub> ) <sub>2</sub> (C <sub>12</sub> H <sub>8</sub> N <sub>2</sub> )]	Z = 8
<i>M<sub>r</sub></i> = 836.91	<i>D<sub>x</sub></i> = 1.617 Mg m <sup>−3</sup>
Monoclinic, <i>C</i> 2/ <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 17.366 (3) Å	<i>μ</i> = 5.52 mm <sup>−1</sup>
<i>b</i> = 13.130 (3) Å	<i>T</i> = 298 (2) K
<i>c</i> = 30.986 (7) Å	Block, orange–red
<i>β</i> = 103.367 (6)°	0.26 × 0.26 × 0.25 mm
<i>V</i> = 6874 (3) Å <sup>3</sup>	

#### Data collection

Siemens SMART CCD area-detector diffractometer	17747 measured reflections
<i>φ</i> and <i>ω</i> scans	6050 independent reflections
Absorption correction: multi-scan ( <i>SADABS</i> ; Bruker, 2000)	4260 reflections with <i>I</i> > 2σ( <i>I</i> )
<i>T<sub>min</sub></i> = 0.328, <i>T<sub>max</sub></i> = 0.339 (expected range = 0.243–0.251)	<i>R<sub>int</sub></i> = 0.061
	<i>θ<sub>max</sub></i> = 25.0°

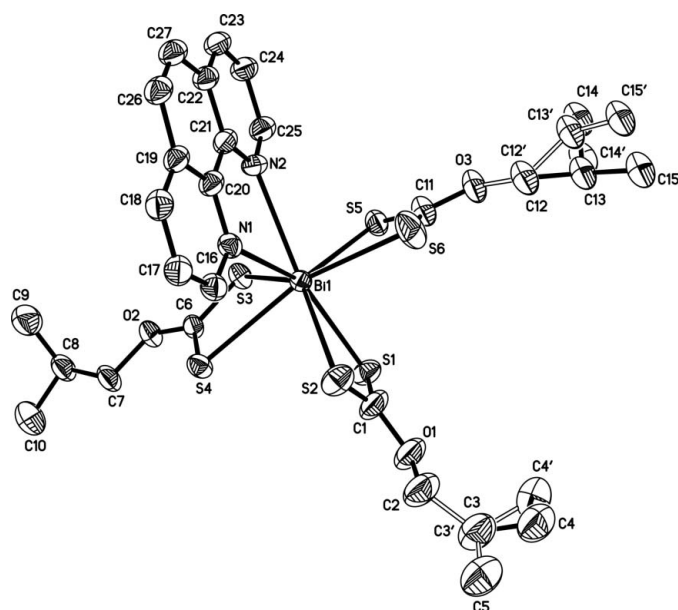
#### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 31.3228P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.119$	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
<i>S</i> = 1.02	$\Delta\rho_{max} = 0.87 \text{ e } \text{Å}^{-3}$
6050 reflections	$\Delta\rho_{min} = -1.38 \text{ e } \text{Å}^{-3}$
396 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

Bi1–S1	2.720 (3)	Bi1–N1	2.811 (7)
Bi1–N2	2.762 (7)	Bi1–S6	2.867 (3)
Bi1–S3	2.779 (2)	Bi1–S4	2.981 (2)
Bi1–S5	2.792 (3)	Bi1–S2	2.986 (3)
S1–Bi1–N2	160.63 (17)	N1–Bi1–S6	89.91 (16)
S1–Bi1–S3	91.46 (9)	S1–Bi1–S4	83.37 (8)
N2–Bi1–S3	81.85 (16)	N2–Bi1–S4	109.01 (18)
S1–Bi1–S5	79.66 (8)	S3–Bi1–S4	62.33 (7)
N2–Bi1–S5	81.12 (17)	S5–Bi1–S4	135.29 (8)
S3–Bi1–S5	77.03 (8)	N1–Bi1–S4	82.47 (15)
S1–Bi1–N1	139.45 (16)	S6–Bi1–S4	157.22 (8)
N2–Bi1–N1	58.9 (2)	S1–Bi1–S2	62.57 (8)
S3–Bi1–N1	114.62 (15)	N2–Bi1–S2	132.88 (17)
S5–Bi1–N1	134.24 (16)	S3–Bi1–S2	136.95 (9)
S1–Bi1–S6	88.74 (11)	S5–Bi1–S2	125.43 (8)
N2–Bi1–S6	84.76 (17)	N1–Bi1–S2	77.61 (16)
S3–Bi1–S6	139.50 (8)	S6–Bi1–S2	77.36 (10)
S5–Bi1–S6	63.18 (9)	S4–Bi1–S2	80.05 (8)



**Figure 1**

The molecular structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Atoms of the minor disorder components are drawn with open bonds. H atoms have been omitted for clarity.

All H atoms were positioned geometrically and treated as riding on their parent atoms: C–H = 0.93 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$  for aromatic, C–H = 0.98 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$  for CH, C–H = 0.97 Å and  $U_{iso}(H) = 1.2U_{eq}(C)$  for CH<sub>2</sub>, and C–H = 0.96 Å and  $U_{iso}(H) = 1.5U_{eq}(C)$  for CH<sub>3</sub> H atoms. Atoms C3, C4, C12, C13, C14 and C15 of two of the *O*-isobutyl dithiocarbonate ligands are each disordered over two positions. Their occupancies refined to 0.79 (3) and 0.21 (3) for C3 and C4, and 0.558 (14) and 0.442 (14) for C12, C13, C14 and C15. The deepest hole is located 0.98 Å from atom Bi1.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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### References

- Baraanyi, A. D., Cook, J. & Onyszczuk, M. (1977). *Inorg. Nucl. Chem. Lett.* **13**, 385–C394.
- Baxter, G. F. (1992). *Chem. Br.* **28**, 445–448.
- Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sun, H., Li, H. & Sadler, P. J. (1997). *Chem. Ber.* **130**, 669–708.
- Yin, H. D., Li, F., Li, F. H. & Wang, D. Q. (2006). *J. Inorg. Organomet. Polym. Mater.* **16**, 161–168.