

Bis(μ -*N*-butyl-*N*-ethyldithiocarbamato-*S*:*S'*)-bis[(*N*-butyl-*N*-ethyldithiocarbamato)zinc(II)]

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Key indicators

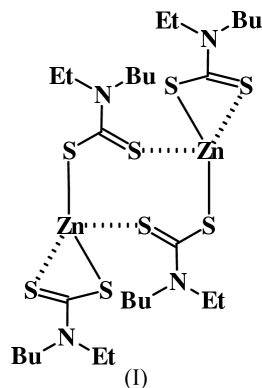
Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
Disorder in main residue
 R factor = 0.047
 wR factor = 0.144
Data-to-parameter ratio = 26.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $[(\text{C}_2\text{H}_5)(\text{C}_4\text{H}_9)\text{NCS}_2]_4\text{Zn}_2$ or $[\text{Zn}_2(\text{C}_7\text{H}_{14}\text{NS}_2)_4]$, one dithiocarbamate group chelates the Zn atom whereas the other is bonded to two Zn atoms. The coordination geometry of the Zn atom is a tetrahedron distorted towards a five-coordinate geometry intermediate between trigonal bipyramidal and a square pyramidal.

Comment

The title compound, (I), adopts a centrosymmetric dimeric structure in which the Zn atom is chelated by one dithiocarbamate anion; the other dithiocarbamate anion uses one of its two S atoms to bind to two Zn atoms through two bonds. The long bond corresponds to the typical values of 2.815 (1)–3.036 (6) Å found in zinc dithiocarbamates (Cox & Tiekink, 1997). The metal coordination geometry is tetrahedral distorted towards five-coordinate intermediate between trigonal bipyramidal and a square pyramidal. The structure is similar to those of diisopropyldithiocarbamate (Miyamae *et al.*, 1979) and diethyldithiocarbamate zinc (Tiekink, 2000).



Experimental

Carbon disulfide was added to an ethanol solution of butylethylamine at 277 K followed by an ethanol solution of zinc chloride. The mixture was vigorously stirred and then set aside. The solid that separated was isolated and then recrystallized from ethanol to afford the title compound.

Crystal data

$[\text{Zn}_2(\text{C}_7\text{H}_{14}\text{NS}_2)_4]$
 $M_r = 835.99$
Orthorhombic, *Pbca*
 $a = 14.7301$ (2) Å
 $b = 12.0254$ (2) Å
 $c = 23.7960$ (1) Å
 $V = 4215.11$ (9) Å³
 $Z = 4$
 $D_x = 1.317$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 6367 reflections
 $\theta = 1.7$ – 28.2°
 $\mu = 1.56$ mm⁻¹
 $T = 298$ (2) K
Block, colorless
 $0.46 \times 0.24 \times 0.20$ mm

Data collection

Siemens CCD area-detector
diffractometer
 ω scans
Absorption correction: empirical
(SADABS; Sheldrick, 1996)
 $T_{\min} = 0.534$, $T_{\max} = 0.746$
27 642 measured reflections

5171 independent reflections
3106 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.082$
 $\theta_{\text{max}} = 28.2^\circ$
 $h = -17 \rightarrow 19$
 $k = -14 \rightarrow 15$
 $l = -30 \rightarrow 31$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.144$
 $S = 0.96$
5171 reflections
199 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0802P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e } \text{\AA}^{-3}$

Table 1

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

Zn1—S1	2.338 (1)	Zn1—S4	2.870 (1)
Zn1—S2	2.470 (1)	Zn1—S4 ⁱ	2.386 (1)
Zn1—S3	2.331 (1)		
S1—Zn1—S2	75.7 (1)	S2—Zn1—S4 ⁱ	107.9 (1)
S1—Zn1—S3	135.6 (1)	S3—Zn1—S4	68.9 (1)
S1—Zn1—S4	95.7 (1)	S3—Zn1—S4 ⁱ	105.9 (1)
S1—Zn1—S4 ⁱ	115.8 (1)	S4—Zn1—S4 ⁱ	89.8 (1)
S2—Zn1—S3	106.3 (1)	Zn1—S4—Zn1 ⁱ	90.2 (1)
S2—Zn1—S4	162.3 (1)		

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

One of the two butyl chains is disordered in two positions; the chain was refined as two chains sharing a common C₁ atom. The occupancy was fixed at 0.5 as it refined to approximately 0.5. Pairs of 1,2- and 1,3-related C atoms were restrained by *SADI* 0.01 and *SADI* 0.02 instructions for the ordered and disordered butyl chains. The unprimed and primed atoms were restrained to have the same displacement parameters; additionally, an *ISOR* 0.02 command was imposed on the disordered atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

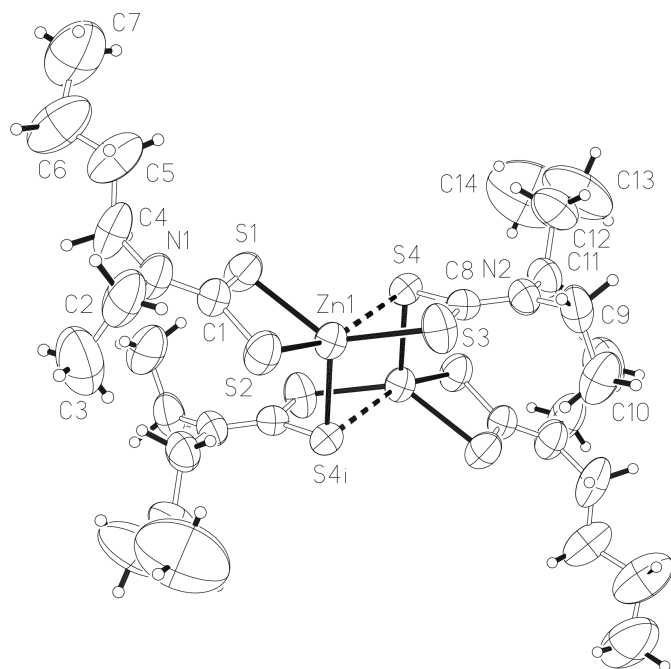


Figure 1
ORTEPII (Johnson, 1976) plot of the title compound at the 50% probability level. H atoms are shown as circles of arbitrary radii.

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References

Cox, M. J. & Tiekink, E. R. T. (1997). *Rev. Inorg. Chem.* **17**, 1–23.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Miyamae, H., Ito, M. & Iwasaki, H. (1979). *Acta Cryst.* **B35**, 1480–1482.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SAINT* and *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tiekink, E. R. T. (2000). *Z. Kristallogr. New Cryst. Struct.* **215**, 445–446.