

Bis[*N*-*n*-butyl-*N*-(3,5-di-*tert*-butyl-2-hydroxybenzyl)dithiocarbamato- κ^2 S,S']zinc(II)

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Both zinc dithiocarbamates and sterically hindered phenols are known to exhibit antioxidant activity. As part of an investigation on the effect of both moieties being combined in one molecule, the title compound, $[\text{Zn}(\text{C}_{17}\text{H}_{26}\text{NOS}_2)_2]$, has been prepared and its crystal structure determined.

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Key indicators

Single-crystal X-ray study

$T = 293 \text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.038

wR factor = 0.101

Data-to-parameter ratio = 16.4

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

Comment

Zinc dithiocarbamates have been investigated as antioxidant additives to plastics (Al-Malaika *et al.*, 1986) and hydrocarbon-based lubricants (Shelkova *et al.*, 1990) and their mechanism of action has received some attention. In an investigation to optimize the antioxidant efficiency of additives to vegetable oils to be utilized as lubricants the zinc dithiocarbamate moiety was combined with a sterically hindered phenol (Becker & Knorr, 1996; Becker *et al.*, 1996). The preparation followed a known pathway for insertion of CS_2 into 4-hydroxyphenylmethylamines (Fitton *et al.*, 1969) with subsequent alkaline hydrolysis and treatment of the dithiocarbamic acid with ZnSO_4 . Due to the bulky groups in *ortho* positions the phenolic hydroxy groups do not form hydrogen bonds.

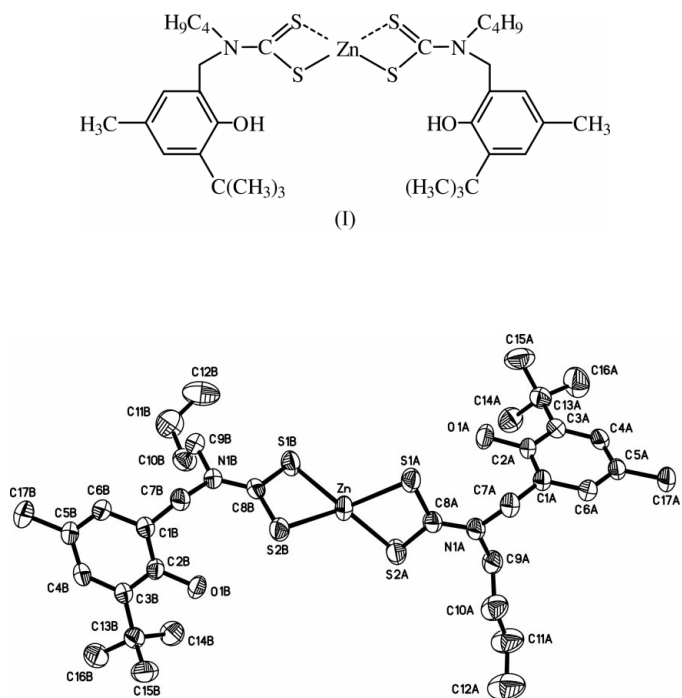


Figure 1
 The molecular structure of (I), showing 50% probability displacement ellipsoids (*SHELXTL*; Sheldrick, 1999). H atoms have been omitted for clarity.

Experimental

A solution of bis(3,5-di-*tert*-butyl-2-hydroxybenzyl)-*n*-butylamine in ethanol was treated with sodium hydroxide solution and excess CS₂ and stirred for 2 h at ambient temperature. After addition of water, a yellow oil could be separated with diethyl ether. The diethyl ether solution was stirred for 3 h with aqueous ZnSO₄. The precipitate formed after evaporation of organic solvent was recrystallized from *n*-hexane/ethanol to yield colourless crystals, m.p. 436 K. IR (cm⁻¹): 3300, 2970, 2935, 1475, 1425, 1385, 1355, 1335, 1290, 1235, 1210, 1195, 1160, 1125, 1095, 1085, 965, 925, 875, 855, 810, 790, 760, 735, 715, 675, 640.

Crystal data

[Zn(C₁₇H₂₆NOS₂)₂]
 $M_r = 714.39$
 Triclinic, $P\bar{1}$
 $a = 8.936$ (3) Å
 $b = 10.762$ (2) Å
 $c = 19.740$ (3) Å
 $\alpha = 80.63$ (1)°
 $\beta = 88.39$ (2)°
 $\gamma = 84.11$ (2)°
 $V = 1863.0$ (8) Å³

$Z = 2$
 $D_x = 1.273$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 12$ – 20°
 $\mu = 0.91$ mm⁻¹
 $T = 293$ (2) K
 Prism, colourless
 $0.35 \times 0.28 \times 0.25$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 2θ – ω scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.683$, $T_{\max} = 0.796$
 6539 measured reflections
 6517 independent reflections
 5755 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.008$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = -23 \rightarrow 23$
 3 standard reflections
 frequency: 120 min
 intensity decay: 1.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.101$
 $S = 1.07$
 6517 reflections
 397 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.9487P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.44$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0021 (5)

The hydroxyl H atoms were refined freely and all other H atoms were refined as riding.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Software*; data reduction: *PROCESS* in *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXL97*.

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