Crystal structure of bis(1-phenyl-3-methyl-4-phenylhydrazono-pyrazole-5-thionato)zinc, $Zn[SCN(Ph)NC(Me)CN(Ph)]_2$

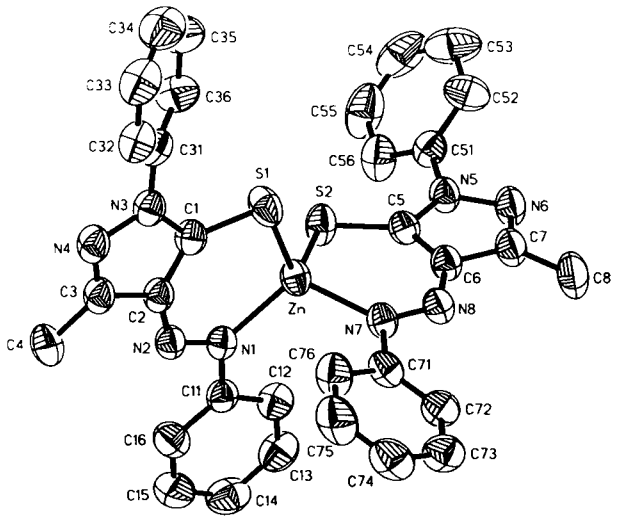
G. Hinsche, E. Uhlemann

Universität Potsdam, Institut für Anorganische Chemie, Postfach 60 15 53, D-14469 Potsdam, Germany

and F. Weller

Universität Marburg, Fachbereich Chemie, Hans Meerwein-Straße, D-35043 Marburg

Received January 20, 1997, CSD-No. 402801



Source of material: The title compound was synthesized by reaction of zinc acetate and the ligand in boiling ethanolic solution (see ref. 1). Crystals suitable for X-ray analysis were obtained by slow cooling of the solution.

In the complex the Zn atom is tetrahedrally coordinated with only little distortion. The chelate rings are nearly planar and have bond angles of 103° with the central zinc atom. The angles S–Zn–S and N–Zn–N are 110° and 111°, respectively. The distances Zn–S and Zn–N were found to be about 227 pm and 204 pm, respectively.

$C_{32}H_{26}N_8S_2Zn$, triclinic, $P\bar{1}$ (No. 2), a =9.925(1) Å, b =12.622(2) Å, c =13.274(2) Å, α =68.71(1)°, β =88.59(1)°, γ =80.92(1)°, V =1529.0 Å^3, Z=2, $R(F)$ =0.045, $R_w(F^2)$ =0.123.

Table 1. Parameters used for the X-ray data collection

Crystal:	bright orange, rhombic plate, size 0.05 x 0.21 x 0.37 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	26.82 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω
$T_{measurement}$:	293 K
$2\theta_{max}$:	133°
$N(hkl)_{unique}$:	5173
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	354
Programs:	SHELXS-96, SHELXL-96

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(41)	2*i*	0.5780	0.2467	0.7812	0.14(1)
H(42)	2*i*	0.5304	0.1744	0.8956	0.14(1)
H(43)	2*i*	0.6159	0.2726	0.8830	0.14(1)
H(81)	2*i*	0.2543	1.1953	0.4272	0.15(1)
H(82)	2*i*	0.1058	1.2062	0.4695	0.15(1)
H(83)	2*i*	0.1324	1.2693	0.3468	0.15(1)
H(12)	2*i*	0.4936	0.7700	0.4398	0.085(5)
H(13)	2*i*	0.6765	0.8105	0.3228	0.085(5)
H(14)	2*i*	0.8748	0.6721	0.3499	0.085(5)
H(15)	2*i*	0.8900	0.4932	0.4938	0.085(5)
H(16)	2*i*	0.7071	0.4527	0.6108	0.085(5)
H(32)	2*i*	0.1486	0.3180	1.0477	0.107(7)
H(33)	2*i*	–0.0683	0.3004	1.1221	0.107(7)
H(34)	2*i*	–0.2659	0.3704	1.0081	0.107(7)
H(35)	2*i*	–0.2466	0.4580	0.8197	0.107(7)
H(36)	2*i*	–0.0297	0.4757	0.7453	0.107(7)
H(52)	2*i*	–0.1771	1.0862	0.2450	0.114(7)
H(53)	2*i*	–0.3183	1.0576	0.1202	0.114(7)
H(54)	2*i*	–0.2338	0.9303	0.0321	0.114(7)
H(55)	2*i*	–0.0080	0.8314	0.0688	0.114(7)
H(56)	2*i*	0.1332	0.8600	0.1936	0.114(7)
H(72)	2*i*	0.4614	1.0228	0.6039	0.082(5)
H(73)	2*i*	0.6056	1.0158	0.7452	0.082(5)
H(74)	2*i*	0.6606	0.8435	0.8958	0.082(5)
H(75)	2*i*	0.5712	0.6782	0.9052	0.082(5)
H(76)	2*i*	0.4271	0.6852	0.7639	0.082(5)

Table 3. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	2*i*	0.29844(4)	0.70768(3)	0.57610(3)	0.0570(3)	0.0388(2)	0.0425(2)	−0.0009(2)	−0.0006(2)	−0.0074(2)
S(1)	2*i*	0.15709(9)	0.62346(7)	0.70759(7)	0.0643(5)	0.0497(4)	0.0573(4)	0.0063(3)	0.0132(4)	−0.0006(3)
S(2)	2*i*	0.18961(9)	0.77194(6)	0.40989(6)	0.0799(5)	0.0388(4)	0.0499(4)	−0.0022(3)	−0.0125(4)	−0.0126(3)
N(1)	2*i*	0.4694(2)	0.5870(2)	0.6048(2)	0.052(1)	0.039(1)	0.042(1)	−0.007(1)	−0.001(1)	−0.013(1)
N(2)	2*i*	0.4890(2)	0.4875(2)	0.6818(2)	0.053(1)	0.039(1)	0.049(1)	−0.003(1)	−0.007(1)	−0.015(1)
N(3)	2*i*	0.2103(3)	0.4044(2)	0.8464(2)	0.061(2)	0.044(1)	0.042(1)	−0.009(1)	−0.001(1)	−0.008(1)
N(4)	2*i*	0.3108(3)	0.3071(2)	0.8880(2)	0.069(2)	0.043(1)	0.050(1)	−0.006(1)	−0.008(1)	−0.005(1)
N(5)	2*i*	0.0710(3)	0.9931(2)	0.3075(2)	0.057(1)	0.040(1)	0.047(1)	0.000(1)	−0.012(1)	−0.014(1)
N(6)	2*i*	0.0598(3)	1.1055(2)	0.3059(2)	0.072(2)	0.041(1)	0.066(2)	0.004(1)	−0.022(1)	−0.019(1)
N(7)	2*i*	0.3398(2)	0.8566(2)	0.5882(2)	0.056(1)	0.043(1)	0.040(1)	0.000(1)	−0.003(1)	−0.015(1)
N(8)	2*i*	0.2845(3)	0.9591(2)	0.5297(2)	0.056(1)	0.045(1)	0.049(1)	0.002(1)	−0.007(1)	−0.019(1)
C(1)	2*i*	0.2550(3)	0.4918(2)	0.7665(2)	0.058(2)	0.042(1)	0.039(1)	−0.007(1)	−0.001(1)	−0.010(1)
C(2)	2*i*	0.3927(3)	0.4487(2)	0.7526(2)	0.055(2)	0.038(1)	0.043(1)	−0.006(1)	−0.006(1)	−0.010(1)
C(3)	2*i*	0.4182(3)	0.3339(2)	0.8319(2)	0.062(2)	0.041(1)	0.049(2)	−0.005(1)	−0.011(1)	−0.007(1)
C(4)	2*i*	0.5471(4)	0.2493(3)	0.8495(3)	0.073(2)	0.049(2)	0.084(3)	0.006(2)	−0.013(2)	−0.004(2)
C(5)	2*i*	0.1538(3)	0.9156(2)	0.3895(2)	0.050(2)	0.043(1)	0.041(1)	−0.002(1)	−0.002(1)	−0.012(1)
C(6)	2*i*	0.1988(3)	0.9811(2)	0.4452(2)	0.055(2)	0.042(1)	0.046(2)	−0.000(1)	−0.007(1)	−0.015(1)
C(7)	2*i*	0.1375(3)	1.0977(3)	0.3870(3)	0.065(2)	0.045(2)	0.067(2)	0.005(1)	−0.021(2)	−0.023(1)
C(8)	2*i*	0.1595(5)	1.2014(3)	0.4096(4)	0.099(3)	0.051(2)	0.117(3)	0.016(2)	−0.052(3)	−0.040(2)
C(11)	2*i*	0.5838(2)	0.6077(2)	0.5358(1)	0.050(2)	0.047(2)	0.049(2)	−0.009(1)	0.002(1)	−0.023(1)
C(12)	2*i*	0.5748(2)	0.7133(2)	0.4509(2)	0.064(2)	0.057(2)	0.055(2)	−0.013(2)	0.007(2)	−0.021(2)
C(13)	2*i*	0.6828(2)	0.7372(2)	0.3818(2)	0.080(2)	0.068(2)	0.057(2)	−0.027(2)	0.016(2)	−0.024(2)
C(14)	2*i*	0.7998(2)	0.6555(2)	0.3978(2)	0.064(2)	0.095(3)	0.078(2)	−0.027(2)	0.020(2)	−0.049(2)
C(15)	2*i*	0.8088(2)	0.5499(2)	0.4827(2)	0.055(2)	0.079(2)	0.093(3)	−0.008(2)	0.011(2)	−0.037(2)
C(16)	2*i*	0.7008(2)	0.5260(1)	0.5518(2)	0.056(2)	0.061(2)	0.069(2)	−0.003(2)	0.005(2)	−0.023(2)
C(31)	2*i*	0.0790(2)	0.3984(2)	0.8898(2)	0.067(2)	0.049(2)	0.048(2)	−0.017(1)	0.004(1)	−0.015(1)
C(32)	2*i*	0.0676(2)	0.3467(2)	1.0010(1)	0.083(2)	0.058(2)	0.048(2)	−0.018(2)	0.008(2)	−0.020(2)
C(33)	2*i*	−0.0604(3)	0.3363(2)	1.0449(1)	0.105(3)	0.077(2)	0.060(2)	−0.031(2)	0.026(2)	−0.029(2)
C(34)	2*i*	−0.1770(2)	0.3776(3)	0.9776(2)	0.087(3)	0.097(3)	0.094(3)	−0.032(2)	0.039(3)	−0.042(3)
C(35)	2*i*	−0.1657(2)	0.4294(2)	0.8664(2)	0.069(2)	0.096(3)	0.089(3)	−0.024(2)	0.006(2)	−0.028(2)
C(36)	2*i*	−0.0377(2)	0.4397(2)	0.8225(1)	0.068(2)	0.079(2)	0.056(2)	−0.022(2)	0.002(2)	−0.017(2)
C(51)	2*i*	−0.0092(2)	0.9757(2)	0.2306(2)	0.063(2)	0.046(2)	0.040(1)	−0.011(1)	−0.005(1)	−0.009(1)
C(52)	2*i*	−0.1425(2)	1.0340(2)	0.2089(2)	0.058(2)	0.093(3)	0.059(2)	−0.005(2)	−0.008(2)	−0.028(2)
C(53)	2*i*	−0.2258(2)	1.0172(3)	0.1352(2)	0.070(2)	0.134(4)	0.065(2)	−0.034(3)	−0.012(2)	−0.023(3)
C(54)	2*i*	−0.1759(3)	0.9420(3)	0.0833(2)	0.124(4)	0.098(3)	0.066(2)	−0.056(3)	−0.024(2)	−0.018(2)
C(55)	2*i*	−0.0426(3)	0.8837(2)	0.1049(2)	0.149(4)	0.064(2)	0.066(2)	−0.020(3)	−0.026(3)	−0.026(2)
C(56)	2*i*	0.0407(2)	0.9005(2)	0.1786(2)	0.099(3)	0.054(2)	0.056(2)	−0.003(2)	−0.013(2)	−0.020(2)
C(71)	2*i*	0.4312(2)	0.8546(2)	0.6712(1)	0.055(2)	0.058(2)	0.044(2)	0.005(1)	−0.008(1)	−0.022(1)
C(72)	2*i*	0.4839(2)	0.9522(2)	0.6656(1)	0.060(2)	0.064(2)	0.053(2)	−0.006(2)	−0.005(1)	−0.019(2)
C(73)	2*i*	0.5691(2)	0.9481(2)	0.7490(2)	0.066(2)	0.082(2)	0.065(2)	−0.012(2)	−0.009(2)	−0.033(2)
C(74)	2*i*	0.6015(3)	0.8464(2)	0.8379(2)	0.074(2)	0.095(3)	0.068(2)	0.007(2)	−0.024(2)	−0.036(2)
C(75)	2*i*	0.5488(3)	0.7488(2)	0.8434(2)	0.108(3)	0.075(2)	0.060(2)	0.013(2)	−0.036(2)	−0.019(2)
C(76)	2*i*	0.4636(3)	0.7529(2)	0.7601(2)	0.094(3)	0.057(2)	0.054(2)	0.000(2)	−0.021(2)	−0.018(2)

References

1. Hinsche, G.; Uhlemann, E.; Zeigan, E.; Engelhardt, G.: Ligandeigenschaften von 1-Phenyl-3-methyl-4-phenylazo-pyrazol-5-thion. Z. Chem. **21** (1981) 414-415.
2. Hansen, L.; Sieler, J.; Richter, R.; Hinsche, G.; Uhlemann, E.: The Crystal and Molecular Structure of 1-Phenyl-3-methyl-4-phenylhydrazono-pyrazole-5- thione. Acta Chem. Scand. **A38** (1984) 331-334.
3. Hinsche, G.; Uhlemann, E.; Weller, F.: Strukturelle Unterschiede von Ruthenium(III)-Chelaten mit SN- und SO-Liganden. Strukturen von Tris(1-phenyl-3-methyl-4-phenylhydrazono-pyrazol-5-thionato)ruthenium(III) und Tris(thiodibenzoylmethanato)ruthenium(III). Z. Naturforsch **51b** (1996) 1355-1358.
4. Sheldrick, G. M.: SHELXS-96, β-Test Version 02. Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. **A46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-96, β-Test Version 02. A Program for Refining Crystal Structures. University of Göttingen, Germany 1996.