metal-organic papers

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.012 Å
R factor = 0.054
wR factor = 0.162
Data-to-parameter ratio = 16.5

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

Dibromobis(phthalazine-κN^2)zinc(II)

In the title compound, $[ZnBr_2(C_8H_6N_2)_2]$, the Zn atom is coordinated by two Br atoms and two N atoms of two different phthalazine ligands in a distorted tetrahedral arrangement. The central bond angles, N—Zn—N and Br—Zn—Br, are 105.6 (2) and 115.59 (5)°, respectively.

Comment

Phthalazine ($C_8H_6N_2$, also named 2,3-benzodiazine) is a di-azanaphthalene with two adjacent N atoms. Phthalazines, like the other members of the benzodiazine series, have found wide application as therapeutic agents. They are also widely used in industry and pharmacy and as intermediates in the synthesis of antimalarial drugs (Silva *et al.*, 1995; Tsoungas & Searcey, 2001; Sugihara *et al.*, 2000; Napoletano *et al.*, 2000; Sivakumar *et al.*, 2002). Various phthalazine compounds strongly protect against acrolein-mediated toxicity in isolated hepatocytes (Burcham *et al.*, 2002).

In this work, we have synthesized and undertaken the crystal structure determination of the title compound, $ZnBr_2(C_8H_6N_2)_2$, (I). The molecule of (I) is shown in Fig. 1. The Zn atom is coordinated tetrahedrally by two Br and two phthalazine ligands. The dihedral angle between the planes of the two phthalizene ligands is 73.9 (2)° and they are in a *cis* arrangement with respect to the N atoms. The Zn-atom coordination is slightly distorted tetrahedral. The Br1—Zn—Br2 and N1—Zn—N3 angles are 115.57 (5) and 105.4 (2)°, respectively. This range of angles is comparable to the cor-esponding values in the previously reported complexes $ZnBr_2$(benzimidazole)$_2$ [113.28 (8)–106.2 (4)°; Şahin, Ide, Kurt & Yurdakul, 2002] and $ZnBr_2$(nicotinamide)$_2$ [118.9–96.1°; Şahin, Ide, Ataç & Yurdakul, 2002]

(I)
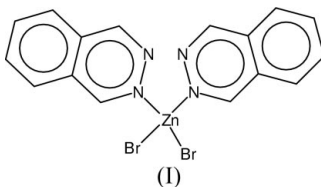
In (I), the Zn—Br1 and Zn—Br2 bond lengths are 2.3585 (13) and 2.3590 (13) Å, and the Zn—N1 and Zn—N3 bond lengths are 2.035 (6) and 2.048 (6) Å. The average Zn—Br distances reported for the related zinc complexes with distorted N—Zn—Br tetrahedral environments are 2.371 (2) Å in $ZnBr_2$(benzimidazole)$_2$ (Şahin, Ide, Kurt & Yurdakul, 2002) and 2.3592 (5) Å in $ZnBr_2$(nicotinamide)$_2$ (Şahin, Ide, Ataç & Yurdakul, 2002).

A packing diagram of the crystal structure of (I), viewed approximately along the *b* axis, is shown in Fig. 2.

Experimental

All chemicals were reagent grade (Sigma) and were used without further purification. The $ZnBr_2L_2$ complex (L = phthalazine) was obtained by addition of phthalazine (2 mmol, 0.261 g) to a saturated solution of $ZnBr_2$ (1 mmol, 0.225 g) in hot ethanol. The mixture was allowed to stand for several weeks until single crystals were formed at the bottom of the solution. Analysis (LECO,CHN-600) found: C 48.30, H 3.05, N 14.85%; calculated for Br_2L_2Zn: C 48.48, H 3.03, N 14.14%.

Crystal data

$[ZnBr_2(C_8H_6N_2)_2]$	$Z = 2$
$M_r = 485.49$	$D_x = 1.898$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.3664 (10)$ Å	Cell parameters from 1730
$b = 8.2971 (10)$ Å	reflections
$c = 14.4141 (18)$ Å	$\theta = 2.5-26.3°$
$\alpha = 85.660 (10)°$	$\mu = 6.15$ mm^{-1}
$\beta = 75.231 (11)°$	$T = 293 (2)$ K
$\gamma = 88.387 (10)°$	Prism, colourless
$V = 849.40 (19)$ Å^3	$0.4 \times 0.3 \times 0.2$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.083$
	$\theta_{max} = 26.3°$
Non-profiled ω scans	$h = -9 \rightarrow 9$
Absorption correction: ψ scan	$k = -10 \rightarrow 0$
(North et al., 1968)	$l = -17 \rightarrow 17$
$T_{min} = 0.122$, $T_{max} = 0.290$	3 standard reflections
3675 measured reflections	frequency: 120 min
3431 independent reflections	intensity decay: 1%
1730 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2]$
$wR(F^2) = 0.162$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{max} < 0.001$
3431 reflections	$\Delta\rho_{max} = 0.74$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -1.10$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br2—Zn	2.3590 (13)	N1—N2	1.375 (9)
Zn—N1	2.035 (6)	N3—C16	1.310 (9)
Zn—N3	2.048 (6)	N3—N4	1.373 (8)
Zn—Br1	2.3585 (13)	N4—C9	1.310 (10)
N1—C1	1.312 (9)	N2—C8	1.294 (10)
N1—Zn—N3	105.6 (2)	C1—N1—N2	121.1 (6)
N1—Zn—Br1	107.73 (18)	C1—N1—Zn	125.8 (5)
N3—Zn—Br1	112.1 (2)	N2—N1—Zn	113.1 (4)
N1—Zn—Br2	110.78 (19)	C16—N3—Zn	123.8 (5)
N3—Zn—Br2	104.57 (19)	N4—N3—Zn	114.5 (4)
Br1—Zn—Br2	115.59 (5)		
N3—Zn—N1—C1	−136.8 (6)	N3—Zn—N1—N2	45.8 (6)
Br1—Zn—N1—C1	−16.8 (7)	Br1—Zn—N1—N2	165.8 (5)
Br2—Zn—N1—C1	110.5 (6)	Br2—Zn—N1—N2	−66.9 (5)

H atoms were placed in calculated positions (C—H = 0.93 Å) and included in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows*
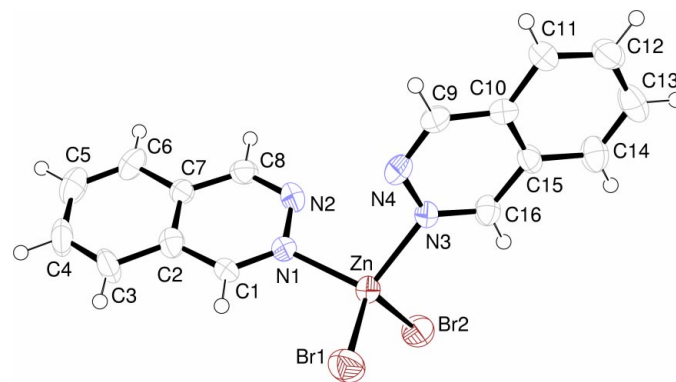

Figure 1
The structure of the molecule of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids (Farrugia, 1997).
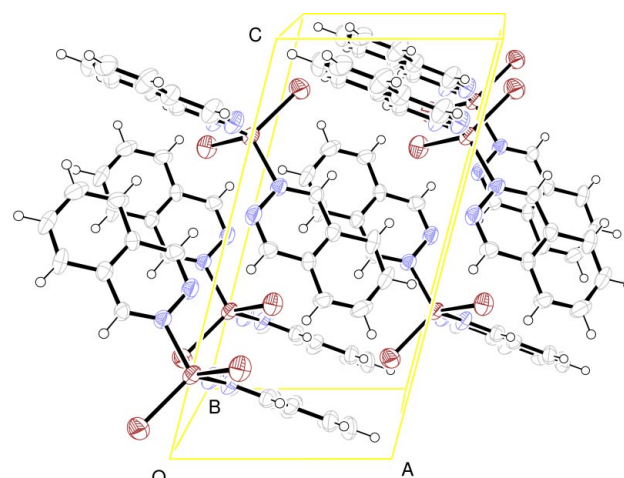

Figure 2
Packing diagram of the crystal structure of (I), viewed approximately along the *b* axis (Farrugia, 1997).

(Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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