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catena-Poly[[dibromidozinc(II)]-μ-3-(1*H*-benzimidazol-2-yl)[2,6-$^2H_2$]pyridine *N*-oxide]

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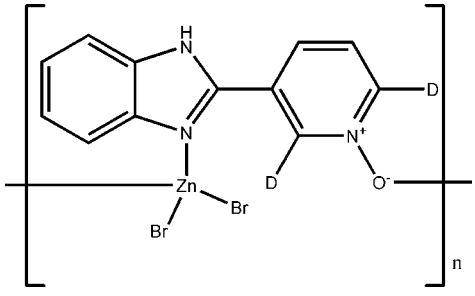
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.034; wR factor = 0.072; data-to-parameter ratio = 17.7.

In the crystal structure of the title compound, $[ZnBr_2(C_{12}H_7D_2N_3O)]_n$, the Zn atoms are coordinated by two Br atoms and by one N atom and one O atom of two symmetry-related 3-(1*H*-benzimidazol-2-yl)[2,6-$^2H_2$]pyridine *N*-oxide ligands in a slightly distorted tetrahedral geometry. The $ZnBr_2$ units are connected by the organic ligands into chains.

Related literature

For the deuteration effect (DEF) on physical properties, see: Akutagawa *et al.* (2004); Ye *et al.* (2007).



Experimental

Crystal data

$[ZnBr_2(C_{12}H_7D_2N_3O)]$
M_r = 438.42
Monoclinic, $P2_1/n$
a = 7.4071 (15) Å
b = 15.017 (3) Å
c = 12.174 (2) Å
β = 98.52 (3)°
V = 1339.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.79 mm^{-1}
T = 293 (2) K
0.2 × 0.15 × 0.1 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.261, T_{max} = 0.463
13680 measured reflections
3070 independent reflections
2538 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.072
S = 1.09
3070 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXTL*

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2082).

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Ye, Q., Zhao, H., Qu, Z.-R., Fu, D.-W., Xiong, R.-G., Cui, Y.-P., Akutagawa, T., Hong Chan, P. W. & Nakamura, T. (2007). *Angew. Chem. Int. Ed.* **46**, 6852–6856.

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supplementary materials

Acta Cryst. (2008). E**64**, m248 [**doi:10.1107/S1600536807067281**]

catena-Poly[[dibromidozinc(II)]-μ-3-(1*H*-benzimidazol-2-yl)[2,6-$^2H_2$]pyridine *N*-oxide]

H.-Z. Luo and H.-Y. Ye

Comment

Recently, investigations on the effect of deuteration onto the physical properties like permittivity has become of increasing interest (Akutagawa, *et al.*, 2004) and (Ye, *et al.*, 2007). As a part of our ongoing investigations in this field we have determined the crystal structure of the title compound, poly bis(bromo)-(3-(1*H*-benzo[*d*]imidazole-2)–2,6-dideuterium-pyridine N-oxide)-zinc(II) (I).

In the crystal structure of the title compound the zinc atoms are coordinated by two bromo atoms and by one nitrogen atom and one oxygen atom of two symmetry related 3-(1*H*-benzo[*d*]imidazole-2)–2,6-dideuterium-pyridine ligands within slightly distorted tetrahedra (Fig. 1). The $ZnBr_2$ units are connected by the ligands into chains. The dihedral angle between the 1*H*-benzo[*d*]imidazole and the pyridine ring amount to 35.87 (8) °.

Experimental

3-(1*H*-benzo[*d*]imidazole-2)–2,6-dideuterium-pyridine N-oxide (0.21 g 0.1 mmol) $ZnBr_2$ (0.027 g, 0.2 mmol), ethanol (0.8 ml) and water (0.4 ml) are transfered into a sealed Pyrex tube and heated at 100 °C for 2 d. On cooling colorless block-like crystals of the title compound are obtained, which are suitable for X-ray ananlysis.

Refinement

For the refinement of the D atoms, the atomic scattering factors for H were used. All H and D atoms were positioned with idealized geometry and were refined isotropic U_{iso}(H) = 1.2Ueq(C or N) using a riding model with d(C—H/D) = 0.93 and d(N—H) = 0.86 Å.

Figures

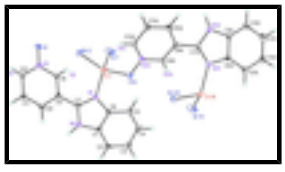


Fig. 1. Crystal structure of the title compound with the atomic numbering scheme and displacement ellipsoids drawn at the 30% probability level. The H and D atoms are shown as spheres of arbitrary size. Symmetry code: A = 1.5 + *x*, -1/2 + *y*,1.5 + *z*.

catena-Poly[[dibromidozinc(II)]-μ-3-(1*H*-benzimidazol-2-yl)[2,6-\ $^2H_2$]pyridine N-oxide]

Crystal data

$[ZnBr_2(C_{12}H_7D_2N_3O)]$	$F(000) = 840$
$M_r = 438.42$	$D_x = 2.175$ Mg m^{-3}

Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 12253 reflections
$a = 7.4071$ (15) Å	$\theta = 3.0–27.5°$
$b = 15.017$ (3) Å	$\mu = 7.79$ mm^{-1}
$c = 12.174$ (2) Å	$T = 293$ K
$\beta = 98.52$ (3)°	Block, colorless
$V = 1339.2$ (5) Å^3	0.2 × 0.15 × 0.1 mm
$Z = 4$	

Data collection

Rigaku Mercury2 diffractometer	3070 independent reflections
Radiation source: fine-focus sealed tube	2538 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.058$
Detector resolution: 13.6612 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
ω scans	$h = -9\rightarrow9$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$k = -19\rightarrow19$
$T_{min} = 0.261$, $T_{max} = 0.463$	$l = -15\rightarrow15$
13680 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.072$	$w = 1/[\sigma^2(F_o^2) + (0.0204P)^2 + 0.5716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
3070 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.57$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0021 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-

factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.18024 (5)	0.76280 (2)	0.56995 (3)	0.02687 (12)
Br1	0.43934 (6)	0.68154 (3)	0.53737 (4)	0.05495 (15)
Br2	0.05935 (5)	0.71851 (2)	0.73359 (3)	0.03347 (12)
C1	0.2625 (4)	1.0321 (2)	0.4904 (3)	0.0270 (7)
C2	0.2551 (5)	1.0978 (2)	0.4096 (3)	0.0348 (8)
H2B	0.2762	1.1574	0.4277	0.042*
C3	0.2151 (5)	1.0700 (3)	0.3022 (3)	0.0376 (9)
H3A	0.2073	1.1119	0.2455	0.045*
C4	0.1855 (5)	0.9800 (3)	0.2749 (3)	0.0376 (9)
H4A	0.1610	0.9638	0.2004	0.045*
C5	0.1914 (5)	0.9149 (2)	0.3545 (3)	0.0333 (8)
H5A	0.1712	0.8554	0.3357	0.040*
C6	0.2292 (4)	0.9425 (2)	0.4644 (3)	0.0255 (7)
C7	0.2883 (4)	0.9521 (2)	0.6446 (3)	0.0242 (7)
C8	0.4011 (4)	0.8528 (2)	0.8000 (3)	0.0258 (7)
D2	0.4465	0.8148	0.7503	0.031*
C9	0.3176 (4)	0.9318 (2)	0.7629 (3)	0.0235 (7)
C10	0.2589 (4)	0.9895 (2)	0.8391 (3)	0.0290 (8)
H10A	0.2069	1.0440	0.8160	0.035*
C11	0.2781 (5)	0.9656 (2)	0.9485 (3)	0.0326 (8)
H11A	0.2391	1.0038	1.0003	0.039*
C12	0.3550 (4)	0.8850 (2)	0.9822 (3)	0.0312 (8)
D1	0.3645	0.8679	1.0562	0.037*
N1	0.2441 (3)	0.89347 (17)	0.5628 (2)	0.0246 (6)
N2	0.2984 (4)	1.03547 (18)	0.6041 (2)	0.0299 (6)
H2A	0.3232	1.0828	0.6431	0.036*
N3	0.4165 (4)	0.83101 (18)	0.9080 (2)	0.0282 (6)
O1	0.4873 (3)	0.75130 (15)	0.9391 (2)	0.0386 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0340 (2)	0.0198 (2)	0.0252 (2)	−0.00331 (15)	−0.00104 (17)	−0.00046 (15)
Br1	0.0509 (3)	0.0317 (2)	0.0885 (4)	0.00727 (18)	0.0311 (2)	0.0069 (2)
Br2	0.0438 (2)	0.0292 (2)	0.0269 (2)	−0.00230 (15)	0.00361 (15)	0.00400 (14)
C1	0.0279 (18)	0.0246 (18)	0.0273 (19)	−0.0010 (13)	0.0006 (14)	0.0017 (14)
C2	0.044 (2)	0.0230 (19)	0.038 (2)	−0.0001 (15)	0.0108 (16)	0.0038 (15)
C3	0.037 (2)	0.042 (2)	0.035 (2)	0.0040 (17)	0.0088 (16)	0.0182 (17)
C4	0.041 (2)	0.048 (3)	0.024 (2)	−0.0048 (17)	0.0054 (16)	0.0033 (17)
C5	0.041 (2)	0.030 (2)	0.028 (2)	−0.0040 (16)	0.0037 (15)	−0.0001 (15)
C6	0.0269 (17)	0.0226 (18)	0.0269 (19)	−0.0018 (13)	0.0038 (13)	0.0011 (14)
C7	0.0261 (17)	0.0191 (17)	0.0267 (18)	0.0004 (13)	0.0015 (13)	0.0009 (13)

C8	0.0300 (18)	0.0231 (18)	0.0225 (18)	−0.0030 (13)	−0.0019 (13)	−0.0046 (13)
C9	0.0239 (16)	0.0234 (18)	0.0220 (17)	−0.0039 (13)	−0.0007 (13)	−0.0014 (13)
C10	0.0313 (19)	0.0235 (19)	0.032 (2)	0.0028 (14)	0.0031 (15)	−0.0008 (14)
C11	0.038 (2)	0.032 (2)	0.027 (2)	0.0069 (15)	0.0039 (15)	−0.0022 (15)
C12	0.038 (2)	0.032 (2)	0.0216 (18)	0.0032 (15)	−0.0007 (14)	−0.0005 (15)
N1	0.0303 (15)	0.0196 (14)	0.0218 (15)	−0.0021 (11)	−0.0024 (11)	−0.0004 (11)
N2	0.0447 (18)	0.0187 (15)	0.0247 (16)	−0.0034 (12)	0.0002 (13)	−0.0023 (11)
N3	0.0295 (15)	0.0227 (15)	0.0284 (16)	0.0003 (11)	−0.0092 (12)	−0.0007 (12)
O1	0.0521 (16)	0.0247 (14)	0.0333 (15)	0.0125 (11)	−0.0125 (12)	−0.0008 (10)

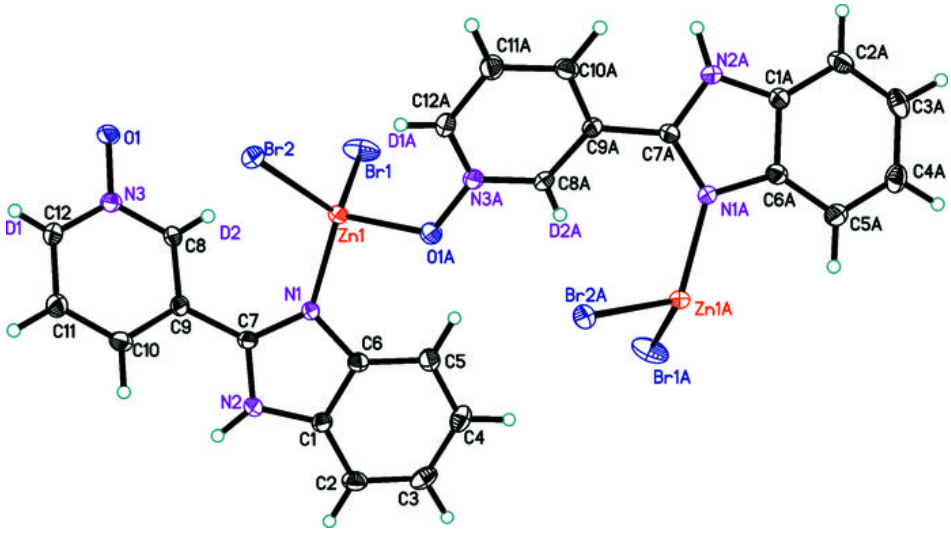
Geometric parameters (Å, °)

Zn1—O1^{i}	1.987 (2)	C7—N1	1.333 (4)
Zn1—N1	2.023 (3)	C7—N2	1.352 (4)
Zn1—Br1	2.3569 (7)	C7—C9	1.457 (4)
Zn1—Br2	2.3960 (7)	C8—N3	1.343 (4)
C1—N2	1.371 (4)	C8—C9	1.383 (4)
C1—C2	1.388 (5)	C8—D2	0.9300
C1—C6	1.397 (4)	C9—C10	1.385 (4)
C2—C3	1.362 (5)	C10—C11	1.367 (5)
C2—H2B	0.9300	C10—H10A	0.9300
C3—C4	1.402 (5)	C11—C12	1.373 (5)
C3—H3A	0.9300	C11—H11A	0.9300
C4—C5	1.372 (5)	C12—N3	1.343 (4)
C4—H4A	0.9300	C12—D1	0.9300
C5—C6	1.389 (4)	N2—H2A	0.8600
C5—H5A	0.9300	N3—O1	1.339 (3)
C6—N1	1.397 (4)	O1—Zn1^{ii}	1.987 (2)
O1^{i}—Zn1—N1	102.35 (10)	N2—C7—C9	123.1 (3)
O1^{i}—Zn1—Br1	108.47 (8)	N3—C8—C9	119.8 (3)
N1—Zn1—Br1	107.08 (8)	N3—C8—D2	120.1
O1^{i}—Zn1—Br2	108.89 (8)	C9—C8—D2	120.1
N1—Zn1—Br2	115.04 (8)	C8—C9—C10	119.2 (3)
Br1—Zn1—Br2	114.22 (3)	C8—C9—C7	119.7 (3)
N2—C1—C2	132.1 (3)	C10—C9—C7	121.1 (3)
N2—C1—C6	105.4 (3)	C11—C10—C9	119.3 (3)
C2—C1—C6	122.5 (3)	C11—C10—H10A	120.3
C3—C2—C1	116.3 (3)	C9—C10—H10A	120.3
C3—C2—H2B	121.9	C10—C11—C12	120.1 (3)
C1—C2—H2B	121.9	C10—C11—H11A	120.0
C2—C3—C4	121.8 (3)	C12—C11—H11A	120.0
C2—C3—H3A	119.1	N3—C12—C11	119.9 (3)
C4—C3—H3A	119.1	N3—C12—D1	120.0
C5—C4—C3	122.1 (3)	C11—C12—D1	120.0
C5—C4—H4A	119.0	C7—N1—C6	105.8 (3)
C3—C4—H4A	119.0	C7—N1—Zn1	129.9 (2)
C4—C5—C6	116.7 (3)	C6—N1—Zn1	123.9 (2)
C4—C5—H5A	121.6	C7—N2—C1	108.7 (3)

C6—C5—H5A	121.6	C7—N2—H2A	125.6
C5—C6—N1	130.5 (3)	C1—N2—H2A	125.6
C5—C6—C1	120.6 (3)	O1—N3—C12	120.5 (3)
N1—C6—C1	108.9 (3)	O1—N3—C8	117.9 (3)
N1—C7—N2	111.1 (3)	C12—N3—C8	121.6 (3)
N1—C7—C9	125.8 (3)	N3—O1—Zn1^{ii}	121.83 (19)

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) *x*+1/2, −*y*+3/2, *z*+1/2.

Fig. 1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N3
C12
D1
C8
D2
C11
C10
C9
C7
N2
C1
C2
C3
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C6
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Br1
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