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Iodido(1,4,7,10-tetraazacyclododecane)-zinc(II) triiodide

Jun Du, Mo Jia and Yi-Zhi Li*

State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, People's Republic of China
Correspondence e-mail: llyyjz@nju.edu.cn

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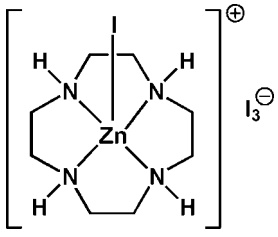
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.020 Å; R factor = 0.063; wR factor = 0.141; data-to-parameter ratio = 23.2.

The asymmetric unit of the title compound, $[ZnI(C_8H_{16}N_4)]I_3$, contains one iodido(1,4,7,10-tetraazacyclododecane)zinc(II) cation, with zinc in a slightly distorted square-pyramidal coordination geometry, and one triiodide counter-anion. The coordination number of zinc is five. In the cation, the iodine ion is located at the apex of the square pyramid and the zinc ion lies 0.846 (15) Å from the basal plane, which is made up of four N atoms. Weak intermolecular C—H···I interactions seem to be effective in stabilizing the crystal structure.

Related literature

For related literature, see: Kimura *et al.* (1997); Shionoya *et al.* (1993); Wang *et al.* (2003); Aoki & Kimura (2004).



Experimental

Crystal data

$[ZnI(C_8H_{16}N_4)]I_3$
M_r = 745.25
Triclinic, $P\overline{1}$
a = 8.596 (1) Å
b = 9.120 (1) Å
c = 12.342 (2) Å
α = 94.894 (2)°
β = 104.707 (2)°
γ = 91.295 (2)°
V = 931.5 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.94 mm^{-1}
T = 298 (2) K
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.15, T_{max} = 0.17
5025 measured reflections
3575 independent reflections
2260 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.141
S = 1.07
3575 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.65 e Å^{-3}
$\Delta\rho_{min}$ = −1.68 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: IM2013).

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supplementary materials

Acta Cryst. (2007). E**63**, m1629 [**doi:10.1107/S160053680702171X**]

Iodido(1,4,7,10-tetraazacyclododecane)zinc(II) triiodide

J. Du, M. Jia and Y.-Z. Li

Comment

The 1,4,7,10-tetraazacyclododecane (cyclen) is a multipotent ligand which has been used in chemistry, biology and many other fields not only due to its excellent coordination properties but also because of the interesting characteristics of its with metal complexes (Guo *et al.*, 1999). Previously accumulated studies about the intrinsic chemical properties of Zn^{2+} can be finely tuned by complexation with macrocyclic polyamines such as 1,5,9-triazacyclododecane and 1,4,7,10-tetraazacyclododecane (Kimura *et al.*, 2004). Kimura *et al.* and others also have investigated the coordination of imides and phosphates to Zn(II)-cyclen complexes in great detail (Kimura *et al.*, 1993; Kimura *et al.*,1997).

In this paper, we report the crystal structure of a novel Zn(II)-cyclen complex composed of a iodo-(1,4,7,10-tetraazacyclododecane)-zinc(II) cation and a triiodine anion as the counter anion. The asymmetric unit of the title compound is presented in Figure 1. In the cation each zinc atom is coordinated by four nitrogen atoms of the cyclen and one iodo ligand (Fig.1). Weak intermolecular C—H···I interactions seem to be effective in the stabilization of the whole crystal structure in which two neighboring cations are linked into a dimeric unit (C4—H4b···I1^{i}; i: 1 - *x*,1 - *y*,1 - *z*) (Fig. 2). Additional weak intermolecular C—H···I interactions (C7—H7a···I4; C5—H5b···I4^{ii}; ii: -*x*,1 - *y*,-*z*) link all of the dimers into a linear one-dimensional supramolecular structure (Fig. 2).

Experimental

The title complex was prepared by the direct combination of 1:1 molar equivalents of cyclen, $Zn(NO_3)_2$ and KI in ethanol at room temperature for two hours. Then the solution was filtered off and placed directly in the air to evapore the solvent. Single crystals of the title compound suitable for structure analysis were obtained fromn the solution after two weeks.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.97 Å, N—H = 0.91 Å, and with U_{iso}(H) = 1.2 times U_{eq}(C or N).

Figures

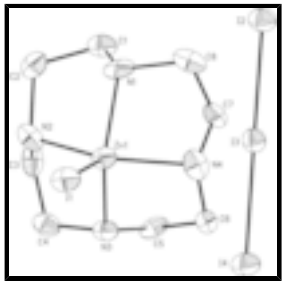

Fig. 1. View of the title complex showing the labeling of the non-H atoms. Thermal ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Fig. 2. View of the linear one-dimensional supramolecular structure down the *b* axis. Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z* (ii) -*x*, 1 - *y*, -*z*.

Iodido(1,4,7,10-tetraazacyclododecane)zinc(II) triiodide

Crystal data

$[ZnI(C_8H_{16}N_4)]I_3$	$Z = 2$
$M_r = 745.25$	$F_{000} = 676$
Triclinic, $P\bar{1}$	$D_x = 2.657$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.596$ (1) Å	Cell parameters from 1388 reflections
$b = 9.120$ (1) Å	$\theta = 2.6$–22.2°
$c = 12.342$ (2) Å	$\mu = 7.94$ mm^{-1}
$\alpha = 94.894$ (2)°	$T = 298$ (2) K
$\beta = 104.707$ (2)°	$T = 298$ (2) K, ?
$\gamma = 91.295$ (2)°	0.26 × 0.24 × 0.22 mm
$V = 931.5$ (2) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3575 independent reflections
Radiation source: sealed tube	2260 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 298(2)$ K	$\theta_{max} = 26.0^\circ$
phi and ω scans	$\theta_{min} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10 \rightarrow 10$
$T_{min} = 0.15$, $T_{max} = 0.17$	$k = -11 \rightarrow 8$
5025 measured reflections	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 1.99P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.063$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.141$	$\Delta\rho_{max} = 1.65$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -1.68$ e Å^{-3}
3575 reflections	Extinction correction: none
154 parameters	

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.1065 (17)	0.9305 (16)	0.3598 (12)	0.060 (4)
H1A	0.0278	0.8883	0.2927	0.072*
H1B	0.0728	1.0259	0.3838	0.072*
C2	0.1345 (16)	0.8207 (17)	0.4595 (11)	0.058 (4)
H2A	0.0332	0.7984	0.4769	0.070*
H2B	0.2087	0.8677	0.5270	0.070*
C3	0.0937 (17)	0.5709 (16)	0.3631 (12)	0.061 (4)
H3A	0.0108	0.6177	0.3103	0.073*
H3B	0.0455	0.5244	0.4153	0.073*
C4	0.1799 (16)	0.4582 (15)	0.3013 (12)	0.056 (3)
H4A	0.1021	0.3834	0.2572	0.067*
H4B	0.2589	0.4101	0.3560	0.067*
C5	0.1637 (15)	0.5576 (14)	0.1195 (10)	0.052 (3)
H5A	0.0671	0.6034	0.1298	0.062*
H5B	0.1333	0.4650	0.0731	0.062*
C6	0.264 (2)	0.6633 (15)	0.0640 (12)	0.067 (4)
H6A	0.3602	0.6168	0.0540	0.080*
H6B	0.1997	0.6859	−0.0089	0.080*
C7	0.1895 (17)	0.9072 (14)	0.1314 (12)	0.056 (3)
H7A	0.0869	0.8579	0.1280	0.067*
H7B	0.1807	0.9516	0.0616	0.067*
C8	0.2340 (15)	1.0297 (14)	0.2343 (13)	0.061 (4)
H8A	0.1518	1.1019	0.2278	0.073*
H8B	0.3366	1.0797	0.2388	0.073*
I1	0.64993 (10)	0.75291 (11)	0.42815 (8)	0.0584 (3)
I2	−0.33154 (12)	1.35959 (11)	0.26103 (8)	0.0616 (3)
I3	−0.30693 (10)	1.06260 (10)	0.16838 (7)	0.0498 (2)
I4	−0.28278 (12)	0.75799 (11)	0.07214 (8)	0.0637 (3)

N1	0.2439 (14)	0.9392 (12)	0.3441 (9)	0.060 (3)
H1	0.3093	0.9926	0.4049	0.072*
N2	0.2099 (14)	0.6659 (12)	0.4164 (10)	0.062 (3)
H2	0.2730	0.6263	0.4767	0.074*
N3	0.2664 (12)	0.5358 (11)	0.2213 (8)	0.047 (3)
H3	0.3497	0.4818	0.2102	0.056*
N4	0.3093 (13)	0.8072 (13)	0.1461 (10)	0.060 (3)
H4	0.4002	0.8507	0.1359	0.072*
Zn1	0.35735 (17)	0.74403 (17)	0.31372 (12)	0.0475 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.048 (8)	0.060 (9)	0.059 (9)	0.016 (6)	−0.006 (6)	−0.011 (7)
C2	0.041 (7)	0.086 (11)	0.043 (7)	0.008 (7)	0.004 (6)	−0.003 (7)
C3	0.057 (8)	0.070 (9)	0.052 (8)	−0.030 (7)	0.007 (6)	0.018 (7)
C4	0.052 (8)	0.043 (7)	0.064 (9)	0.002 (6)	0.000 (7)	−0.001 (6)
C5	0.047 (7)	0.056 (8)	0.044 (7)	−0.010 (6)	0.007 (6)	−0.025 (6)
C6	0.105 (13)	0.050 (8)	0.052 (8)	0.018 (8)	0.028 (8)	0.014 (7)
C7	0.056 (8)	0.048 (8)	0.063 (9)	0.016 (6)	0.010 (6)	0.019 (6)
C8	0.033 (6)	0.041 (7)	0.092 (11)	−0.002 (5)	−0.010 (6)	−0.005 (7)
I1	0.0407 (5)	0.0647 (6)	0.0574 (6)	0.0038 (4)	−0.0045 (4)	−0.0113 (4)
I2	0.0593 (6)	0.0559 (6)	0.0614 (6)	0.0094 (4)	0.0034 (4)	−0.0046 (4)
I3	0.0421 (4)	0.0550 (5)	0.0523 (5)	0.0052 (3)	0.0100 (4)	0.0096 (4)
I4	0.0624 (6)	0.0585 (6)	0.0646 (6)	0.0078 (4)	0.0107 (5)	−0.0099 (5)
N1	0.056 (7)	0.056 (7)	0.047 (6)	0.007 (5)	−0.012 (5)	−0.032 (5)
N2	0.058 (7)	0.049 (7)	0.072 (8)	0.002 (5)	0.003 (6)	0.017 (6)
N3	0.038 (5)	0.048 (6)	0.043 (6)	0.011 (4)	−0.007 (4)	−0.007 (5)
N4	0.040 (6)	0.066 (8)	0.076 (8)	−0.011 (5)	0.012 (5)	0.023 (6)
Zn1	0.0400 (8)	0.0514 (8)	0.0455 (8)	0.0024 (6)	0.0036 (6)	−0.0050 (6)

Geometric parameters (Å, °)

C1—N1	1.246 (18)	C7—N4	1.379 (16)
C1—C2	1.63 (2)	C7—C8	1.581 (19)
C1—H1A	0.9700	C7—I4	4.115 (15)
C1—H1B	0.9700	C7—H7A	0.9700
C2—N2	1.669 (18)	C7—H7B	0.9700
C2—H2A	0.9700	C8—N1	1.632 (19)
C2—H2B	0.9700	C8—H8A	0.9700
C3—N2	1.308 (16)	C8—H8B	0.9700
C3—C4	1.54 (2)	I1—Zn1	2.5483 (16)
C3—H3A	0.9700	I2—I3	2.8826 (13)
C3—H3B	0.9700	I3—I4	2.9599 (14)
C4—N3	1.581 (18)	I4—H7A	3.1705
C4—H4A	0.9700	N1—Zn1	2.100 (11)
C4—H4B	0.9700	N1—H1	0.9100
C5—N3	1.373 (16)	N2—Zn1	2.156 (12)
C5—C6	1.59 (2)	N2—H2	0.9100

C5—H5A	0.9700	N3—Zn1	2.151 (10)
C5—H5B	0.9700	N3—H3	0.9100
C6—N4	1.565 (18)	N4—Zn1	2.136 (12)
C6—H6A	0.9700	N4—H4	0.9100
C6—H6B	0.9700		
N1—C1—C2	101.0 (11)	H7A—C7—H7B	108.4
N1—C1—H1A	111.6	C7—C8—N1	104.3 (10)
C2—C1—H1A	111.6	C7—C8—H8A	110.9
N1—C1—H1B	111.6	N1—C8—H8A	110.9
C2—C1—H1B	111.6	C7—C8—H8B	110.9
H1A—C1—H1B	109.4	N1—C8—H8B	110.9
C1—C2—N2	108.7 (10)	H8A—C8—H8B	108.9
C1—C2—H2A	110.0	I2—I3—I4	179.70 (5)
N2—C2—H2A	110.0	I3—I4—H7A	81.9
C1—C2—H2B	110.0	I3—I4—C7	80.26 (18)
N2—C2—H2B	110.0	C1—N1—C8	108.1 (11)
H2A—C2—H2B	108.3	C1—N1—Zn1	118.8 (10)
N2—C3—C4	103.2 (12)	C8—N1—Zn1	105.5 (7)
N2—C3—H3A	111.1	C1—N1—H1	108.0
C4—C3—H3A	111.1	C8—N1—H1	108.0
N2—C3—H3B	111.1	Zn1—N1—H1	108.0
C4—C3—H3B	111.1	C3—N2—C2	110.4 (12)
H3A—C3—H3B	109.1	C3—N2—Zn1	114.6 (10)
C3—C4—N3	111.0 (10)	C2—N2—Zn1	102.5 (7)
C3—C4—H4A	109.4	C3—N2—H2	109.7
N3—C4—H4A	109.4	C2—N2—H2	109.7
C3—C4—H4B	109.4	Zn1—N2—H2	109.7
N3—C4—H4B	109.4	C5—N3—C4	112.9 (10)
H4A—C4—H4B	108.0	C5—N3—Zn1	110.3 (8)
N3—C5—C6	104.4 (10)	C4—N3—Zn1	104.0 (7)
N3—C5—H5A	110.9	C5—N3—H3	109.8
C6—C5—H5A	110.9	C4—N3—H3	109.8
N3—C5—H5B	110.9	Zn1—N3—H3	109.8
C6—C5—H5B	110.9	C7—N4—C6	113.4 (11)
H5A—C5—H5B	108.9	C7—N4—Zn1	109.0 (10)
N4—C6—C5	106.7 (10)	C6—N4—Zn1	107.4 (7)
N4—C6—H6A	110.4	C7—N4—H4	109.0
C5—C6—H6A	110.4	C6—N4—H4	109.0
N4—C6—H6B	110.4	Zn1—N4—H4	109.0
C5—C6—H6B	110.4	N1—Zn1—N4	83.6 (5)
H6A—C6—H6B	108.6	N1—Zn1—N3	132.1 (4)
N4—C7—C8	108.2 (11)	N4—Zn1—N3	80.3 (4)
N4—C7—I4	119.4 (8)	N1—Zn1—N2	80.5 (5)
C8—C7—I4	110.6 (8)	N4—Zn1—N2	134.3 (4)
N4—C7—H7A	110.1	N3—Zn1—N2	79.4 (4)
C8—C7—H7A	110.1	N1—Zn1—I1	111.8 (3)
N4—C7—H7B	110.1	N4—Zn1—I1	117.7 (3)
C8—C7—H7B	110.1	N3—Zn1—I1	115.7 (3)
I4—C7—H7B	98.0	N2—Zn1—I1	107.9 (3)

Fig. 1

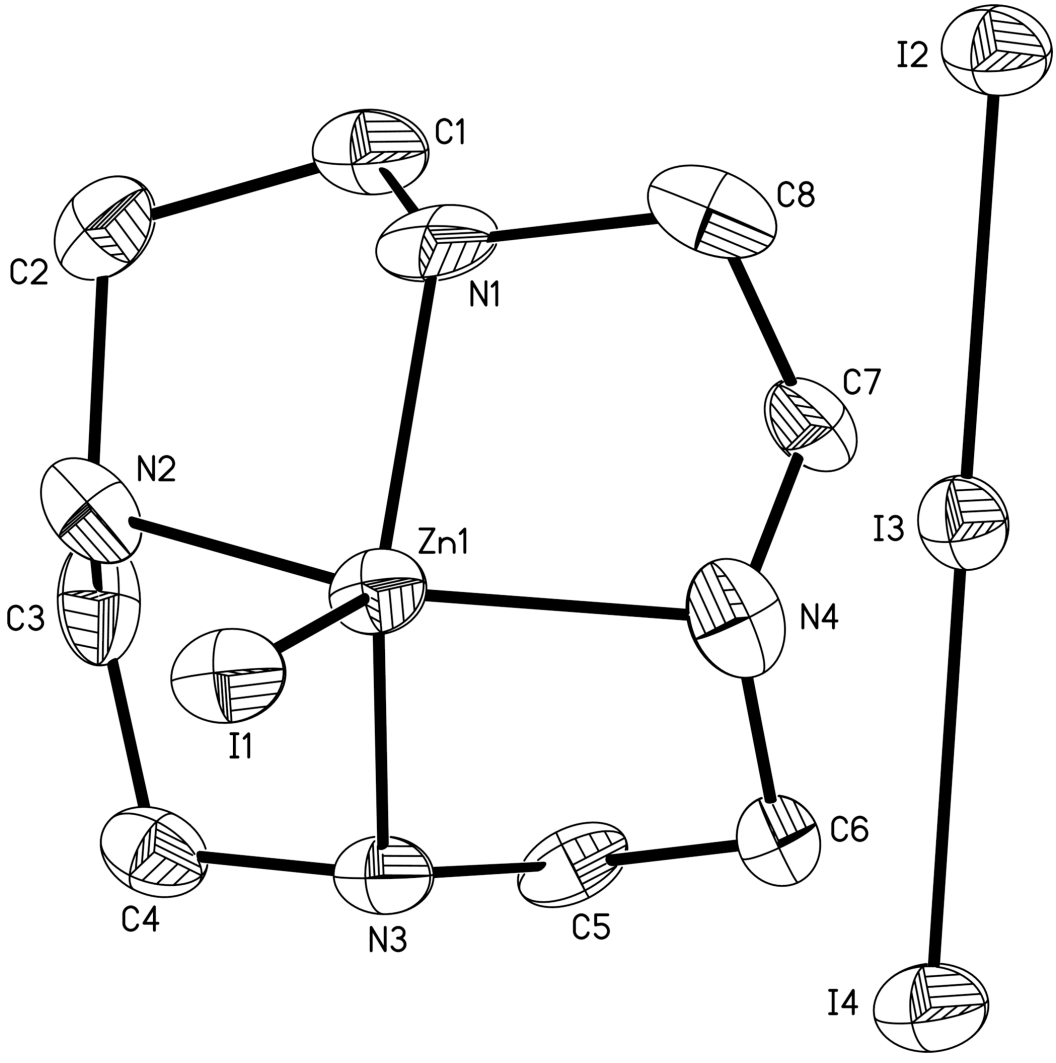

Fig. 2