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Poly[hexabromobis[μ_3-1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene]trimercury(II)]

Xiao-Feng Wang,[a] Yang Lv,[a] Taka-aki Okamura,[b] Wei-Yin Sun[a]* and Norikazu Ueyama[b]

[a]Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, People's Republic of China, and [b]Department of Macromolecular Science, Graduate School of Science, Osaka University, Toyonaka, Osaka 560-0043, Japan

Correspondence e-mail: sunwy@nju.edu.cn

Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.007 Å
R factor = 0.029
wR factor = 0.032
Data-to-parameter ratio = 20.6

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

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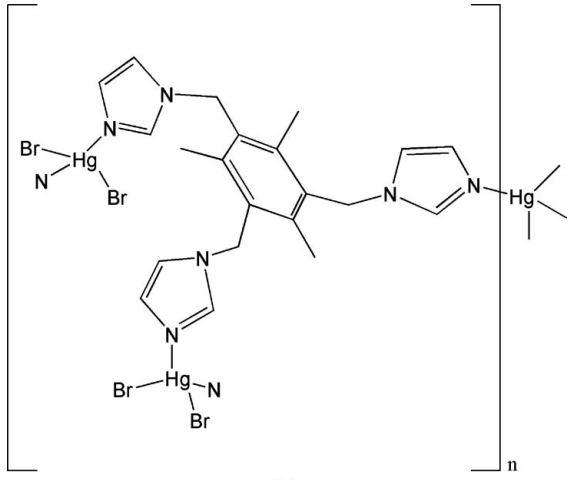
In the title compound, $[Hg_3Br_6(C_{21}H_{24}N_6)_2]_n$, the two Hg atoms (one of which lies on a twofold axis) are tetrahedrally coordinated by two Br atoms and two imidazole N atoms from two different 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene ligands. The complex has an infinite one-dimensional chain structure. The chains are further connected by C—H···Br interactions to form a three-dimensional framework.

Comment

In our previous studies, we have prepared a series of imidazole-containing tripodal ligands with flexible spacers to investigate the influence of bridging ligands on the formation of supramolecular architectures (Liu *et al.*, 2000; Fan *et al.*, 2003). As an extension of our studies on the assembly of flexible ligands, the title complex, (I), was prepared, and its structure is presented here.



(I)

The asymmetric unit of (I) consists of two mercury(II) atoms (one with site symmetry 2), three bromide ions and one 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene] (titmb) ligand (Fig. 1). Atom Hg1 (site symmetry 2), with a distorted tetrahedral geometry (Table 1), is coordinated by two imidazole N atoms from two different titmb ligands. The N12—Hg1—N12^{i} [symmetry code: (i) $1-x, y, \frac{1}{2}-z$] and Br3—Hg1—Br3^{i} bond angles are 97.9 (2) and 115.15 (6)°, respectively. Hg2 is also four coordinated with the same N_2Br_2 binding set with equivalent bond angles of 96.82 (14) and 131.40 (4)°, respectively. Overall, the bond lengths and angles involving Hg are similar to those observed in related complexes containing HgN_2Br_2 groups (Chauhan *et al.*, 2006).

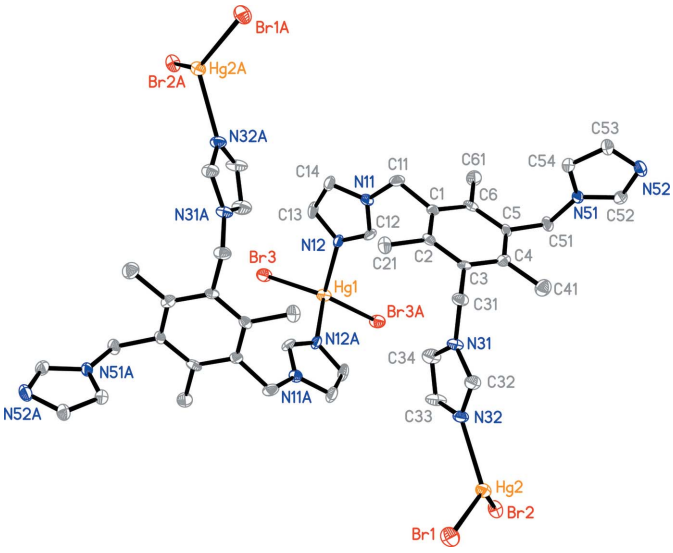


Figure 1
Structure of (I), showing 30% probability displacement ellipsoids. Atoms labeled with the suffix A are at the symmetry positions $(1-x, y, -z+\frac{1}{2})$. H atoms have been omitted.

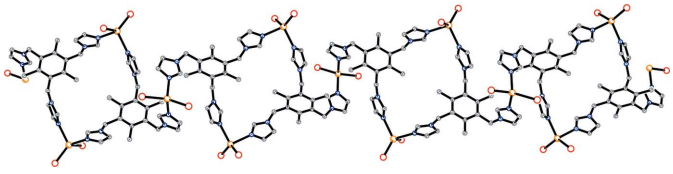


Figure 2
The one-dimensional chain structure of (I). H atoms have been omitted.

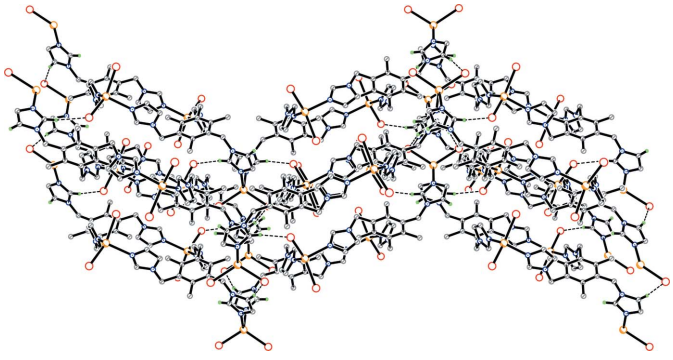
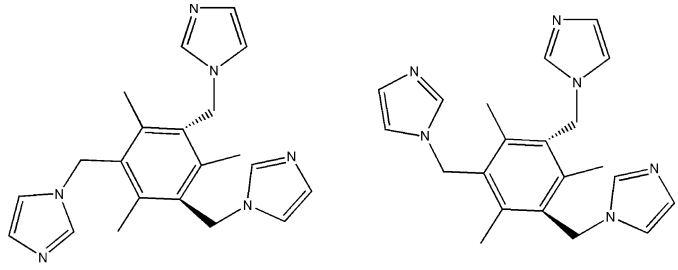


Figure 3
The three-dimensional packing diagram of (I), viewed along the *a* axis. Dashed lines indicate the C—H···Br interactions. H atoms not involved in these interactions have been omitted.

Two Hg2 atoms act as nodes to connect two titmb ligands to form a 24-membered macrocyclic ring with an Hg···Hg distance of 12.504 (7) Å; the macrocyclic units are further linked by Hg1 atoms to form an infinite one-dimensional chain as illustrated in Fig. 2. Each titmb ligand uses its three arms to link three Hg atoms in a *cis–trans–trans* configuration [see scheme below, which shows the *cis–trans–trans* (left) and *cis–cis–cis* (right) configurations] (Fan *et al.*, 2004), and the three imidazolyl rings are inclined to the central benzene ring at angles of 87.1 (2), 84.5 (2) and 89.9 (3)°, for the N11, N31 and N51 rings, respectively.



Two C—H···Br interactions (Table 2) help to consolidate the crystal packing of (I), which serve to link the one-dimensional chains to form a three-dimensional framework structure, as depicted in Fig. 3.

Experimental

A buffer layer of a solution (5 ml) of methanol and DMF (3:1) was carefully layered over a DMF solution (2 ml) of $HgBr_2$ (36.1 mg, 0.1 mmol). A solution of titmb (36.0 mg, 0.1 mmol) in methanol (2 ml) was layered over the buffer layer. Colourless crystals of (I) suitable for X-ray analysis were obtained after several days in 50% yield. The product is stable in an ambient atmosphere and is soluble in dimethylformamide or dimethyl sulfoxide. Analysis found: C 27.38, H 2.66, N 9.46%; $C_{42}H_{48}Br_6Hg_3N_{12}$ requires: C 27.99, H 2.68, N 9.33%.

Crystal data

$[Hg_3Br_6(C_{21}H_{24}N_6)_2]$
M_r = 1802.15
Orthorhombic, *Pbcn*
a = 13.927 (9) Å
b = 11.700 (10) Å
c = 31.83 (2) Å
V = 5187 (7) Å^3
Z = 4
D_x = 2.308 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 13.52 mm^{-1}
T = 200 K
Block, colourless
0.18 × 0.10 × 0.04 mm

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.220, T_{max} = 0.582
48822 measured reflections
5935 independent reflections
3388 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.032
S = 0.96
5935 reflections
288 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.003P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.04 e Å^{-3}
$\Delta\rho_{min}$ = −1.10 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—N12	2.298 (4)	Hg2—N52^{i}	2.366 (4)
Hg1—Br3	2.5515 (14)	Hg2—Br1	2.4987 (13)
Hg2—N32	2.316 (4)	Hg2—Br2	2.5609 (19)

Symmetry code: (i) $-x+1, -y, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H3···Br1^{ii}	0.95	2.76	3.703 (6)	171
C14—H5···Br3^{iii}	0.95	2.90	3.832 (6)	166

Symmetry codes: (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, z$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

All H atoms were placed in idealized locations (C—H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier). The maximum and minimujm electron-density peaks are located 0.97 and 0.49 Å, respectively, from atom Hg2.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *TEXSAN* (Rigaku/MSC, 2000); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *TEXSAN*.

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