

**Xue-Jie Tan,* Li-Qiang Jin and
 Dian-Xiang Xing**

School of Chemical Engineering, Shandong
 Institute of Light Industry, Jinan 250100,
 People's Republic of China

Correspondence e-mail: txj@sdili.edu.cn

Key indicators

Single-crystal X-ray study
 T = 298 K
 Mean $\sigma(C-C)$ = 0.018 Å
 Disorder in main residue
 R factor = 0.054
 wR factor = 0.115
 Data-to-parameter ratio = 20.3

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

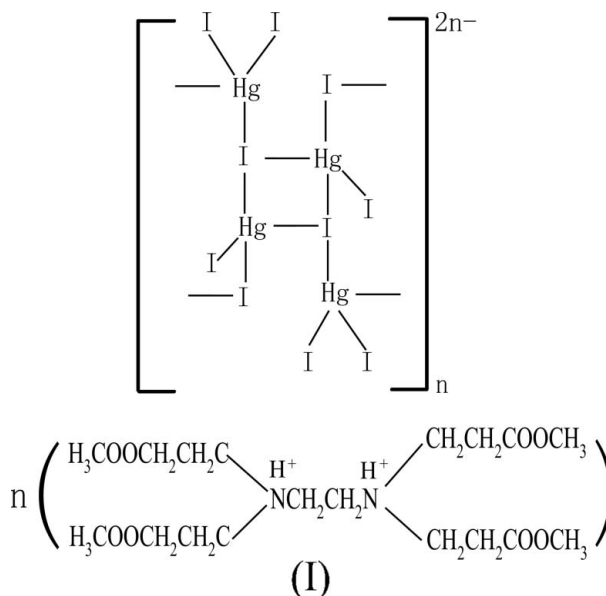
**Poly[*N,N,N',N'*-tetrakis[3-(methoxycarbonyl)-
 propyl]ethylenediammonium [tetra- μ -iodo-
 hexaiodotetramercurate(II)]]**

The title compound, $\{(C_{18}H_{34}N_2O_8)[Hg_4I_{10}]\}_n$, contains two subunits, namely doubly protonated *N,N,N',N'*-tetrakis[3-(methoxycarbonyl)propyl]ethylenediammonium cations and macroanionic pentaiododimercurate $[Hg_2I_5^-]_n$ chains. The complete cation is generated by twofold symmetry and the polymeric anion contains corner- and edge-shared HgI_3 and HgI_4 polyhedra.

Received 16 August 2006
 Accepted 1 September 2006

Comment

Currently, dendrimers are of interest because of their unusual chemical and physical properties, and also their potential application in such diverse fields as medicine, biology, chemistry, physics and materials science (Fan *et al.*, 2005). One of the major challenges in this field is to establish supramolecular structures on the basis of intermolecular interactions (Matthews *et al.*, 1998). This paper deals with the preparation and crystal structure of the title compound, (I), a new phase obtained from a polyamidoamine (PAMAM) dendrimer of generation 0.5 and dipotassium tetraiodomercurate(II).



Compound (I) is built up from two subunits: doubly protonated *N,N,N',N'*-tetrakis[3-(methoxycarbonyl)propyl]ethylenediammonium ($C_9H_{17}NO_4^{2+}$; $H_2edatpme^{2+}$) dications and macroanionic pentaiododimercurate $[Hg_2I_5^-]_n$ chains. The dication is discrete, generated from the unique atoms by twofold symmetry, and the dinuclear unit of the anion forms infinite chains *via* Hg–I covalent bonds (Table 1).

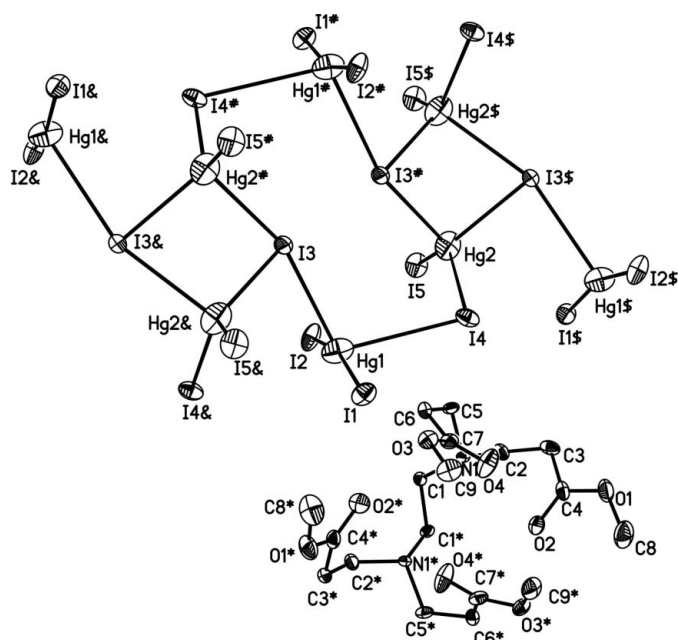


Figure 1

A 30% probability displacement ellipsoid plot of (I), with Hg1A, Hg2A and all H atoms omitted for clarity. Atoms labelled with an asterisk (*), dollar sign (\$), ampersand (&) or hash sign (#) are at the symmetry positions $(-x, y, \frac{1}{2} - z)$, $(x, -y, -\frac{1}{2} + z)$, $(x, -y, \frac{1}{2} + z)$ and $(1 - x, -y, 1 - z)$, respectively.

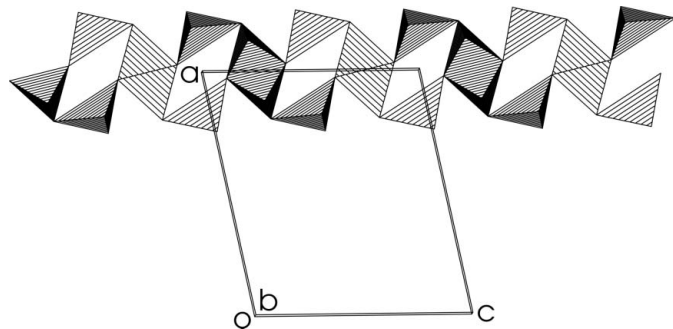


Figure 2

A polyhedral view of the corner-shared and edge-shared HgI_4 polyhedra forming infinite chains parallel to the c axis in (I).

In the asymmetric unit of (I), there is one half of the $\text{H}_2\text{edatpme}^{2+}$ dication and an $[\text{Hg}_2\text{I}_5]^-$ fragment of the anionic chain (Fig. 1). Within the cation, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds [suggested by *PLATON* (Spek, 2003)] (Table 2) cause it to adopt a ball-like structure.

In the $[-\text{Hg}_2\text{I}_5]_n^{n-}$ anionic chain, there are three different I-atom environments. The first type are terminal I atoms, which are bonded to only one Hg atom (*i.e.* I1, I2 and I5), with $\text{Hg}-\text{I}$ distances varying from 2.516 (9) (Hg2–I5) to 2.574 (8) Å (Hg1–I1). The second type are the I4 atoms, which are shared by two Hg atoms with much longer $\text{Hg}-\text{I}$ distances of 2.883 (13) and 3.11 (2) Å. The last type are the I3 atoms, which are bonded to three Hg atom neighbours with separations varying from 2.883 (9) to 3.283 (10) Å. Both Hg atoms were modelled as being disordered over adjacent sites [$\text{Hg1}\cdots\text{Hg1A} = 0.44$ (2) Å and $\text{Hg2}\cdots\text{Hg2A} = 0.38$ (2) Å]. Atoms Hg1 and Hg1A are coordinated by three I atoms within

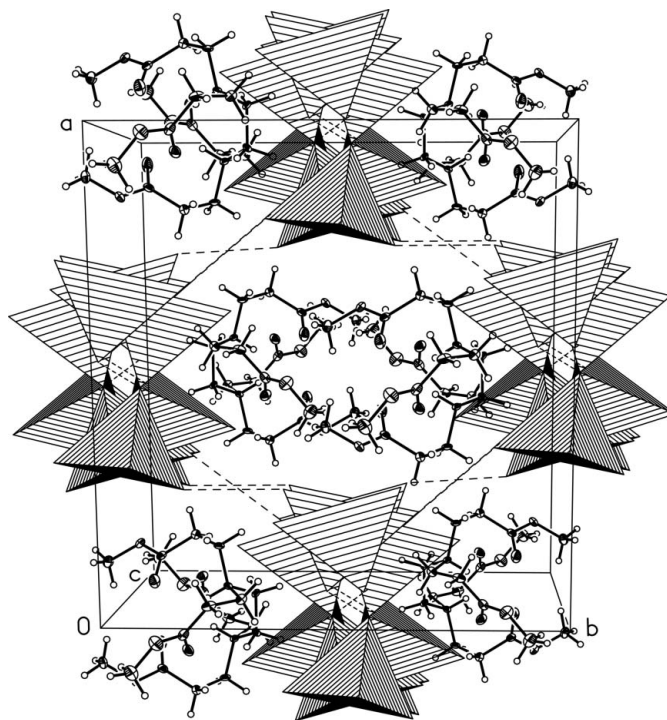


Figure 3

The packing of (I), viewed along the c axis, with close $\text{I}\cdots\text{I}$ contacts indicated by dashed lines (20% displacement ellipsoids for the organic component).

3.4 Å; in both cases, the two closer I atoms are roughly opposite to each other. Atoms Hg2 and Hg2A are surrounded by a very distorted tetrahedron of I atoms within 3.4 Å. If much longer $\text{Hg}\cdots\text{I}$ contacts up to 4 Å are considered as bonds, then Hg1 and Hg1A are four-coordinate but in an irregular geometry (Fig. 2). As in many Hg^{II} iodides, the disordered Hg atoms are mobile inside the I-atom substructure (Hostettler & Schwarzenbach, 2002), which contributes to the long-range $\text{Hg}-\text{I}$ bond distances and larger displacement parameters of Hg than of I.

The corner-shared and edge-shared HgI_4 polyhedra form infinite chains parallel to the c axis (Fig. 2), with the Hg_2I_4 tetrahedra sharing an edge. The $\text{I}\cdots\text{I}$ distances between adjacent chains are shorter than in normal HgI_2 crystals [3.811 (5) and 4.057 (6) Å in (I), compared with 4.361 (3), 4.641 (3) and 4.136 (3) Å in orange and red HgI_2 (Hostettler *et al.*, 2002)], which implies that there may be bonding interactions between the $[-\text{Hg}_2\text{I}_5]_n^{n-}$ anion chains.

The $\text{H}_2\text{edatpme}^{2+}$ dications occupy spaces between $[-\text{Hg}_2\text{I}_5]_n^{n-}$ anion chains (Fig. 3). *PLATON* (Spek 2003) suggests that there are no solvent-accessible voids in the structure of (I).

Experimental

The PAMAM 0.5 G dendrimer was synthesized using ethylenediamine and methyl acrylate as raw materials by means of Michael addition reactions. When an aqueous 0.1 M PAMAM 0.5 G solution and an aqueous 0.1 M K_2HgI_4 solution were mixed together, a khaki

precipitate appeared. After filtration, a light-yellow solution was obtained, and yellow crystals of (I) grew from this solution after several days. Elemental analysis, calculated for $C_9H_{17}Hg_2I_5NO_4$: C 8.73, H 1.38, N 1.13%; found: C 8.01, H 1.61, N 0.96%.

Crystal data

$(C_{18}H_{34}N_2O_8)[Hg_4I_{10}]$
 $M_r = 2477.84$
 Monoclinic, $C2/c$
 $a = 18.580$ (5) Å
 $b = 16.805$ (4) Å
 $c = 14.864$ (4) Å
 $\beta = 100.632$ (5)°
 $V = 4561$ (2) Å³
 $Z = 4$
 $D_x = 3.608$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 20.23$ mm⁻¹
 $T = 298$ (2) K
 Block, yellow
 $0.11 \times 0.10 \times 0.06$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{min} = 0.122, T_{max} = 0.290$
 11521 measured reflections
 4237 independent reflections
 2702 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.062$
 $\rho_{max} = 25.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.115$
 $S = 0.98$
 4237 reflections
 209 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0369P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 1.66$ e Å⁻³
 $\Delta\rho_{min} = -1.32$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Hg1–I2	2.553 (8)	Hg2–I3 ⁱ	2.883 (9)
Hg1–I1	2.574 (8)	Hg2–I4	2.883 (13)
Hg1–I3	3.283 (10)	Hg2–I3 ⁱⁱ	3.086 (14)
Hg1A–I2	2.570 (14)	Hg2A–I5	2.645 (6)
Hg1A–I1	2.631 (17)	Hg2A–I4	2.673 (6)
Hg1A–I4	3.11 (2)	Hg2A–I3 ⁱ	2.794 (4)
Hg2–I5	2.516 (9)	Hg2A–I3 ⁱⁱ	3.383 (5)
I2–Hg1–I1	163.6 (3)	I5–Hg2–I3 ⁱⁱ	116.0 (7)
I2–Hg1–I3	99.3 (3)	I3 ⁱ –Hg2–I3 ⁱⁱ	89.4 (3)
I1–Hg1–I3	94.6 (3)	I4–Hg2–I3 ⁱⁱ	94.4 (2)
I2–Hg1a–I1	154.8 (10)	I5–Hg2a–I4	130.18 (15)
I2–Hg1a–I4	106.5 (8)	I5–Hg2a–I3 ⁱ	120.32 (14)
I1–Hg1a–I4	97.3 (5)	I4–Hg2a–I3 ⁱ	107.8 (2)
I5–Hg2–I3 ⁱ	121.7 (3)	I5–Hg2a–I3 ⁱⁱ	103.6 (2)
I5–Hg2–I4	126.5 (4)	I3 ⁱ –Hg2a–I3 ⁱⁱ	85.2 (2)
I3 ⁱ –Hg2–I4	100.0 (4)	I4–Hg2a–I3 ⁱⁱ	92.0 (2)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1–H10 ^{..} ·O2	0.91	2.25	2.876 (14)	125
N1–H10 ^{..} ·O4	0.91	2.08	2.771 (13)	132
C6–H6A ^{..} ·O2 ⁱⁱⁱ	0.97	2.44	3.150 (15)	129

Symmetry code: (iii) $-x, y, -z + \frac{1}{2}$.

The site occupancies of Hg1 and Hg1A refined to 0.65 (3) and 0.35 (3), respectively. For Hg2 and Hg2A, refined occupancies of 0.35 (3) and 0.65 (3), respectively (sum constrained to unity in each case). The N-bound H atom was located in a difference map and refined as riding in its as-found relative position, with $U_{iso}(H) = 1.2U_{eq}(N)$. The C-bound H atoms were fixed geometrically (C–H = 0.93–0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl C})$. The maximum and minimum electron-density peaks are located near I atoms.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL/NT (Bruker, 2000); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL/NT; software used to prepare material for publication: SHELXL97.

The authors thank Professor Liu Lian-Dong (School of Chemistry, Shandong Normal University) for his support and for helpful discussion.

References

Bruker (2000). SMART, SAINT, SADABS and SHELXTL/NT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Fan, F.-R. F., Mazzitelli, C. L., Brodbelt, J. S. & Bard, A. J. (2005). *Anal. Chem.* **77**, 4413–4422.
 Hostettler, M., Birkedal, H. & Schwarzenbach, D. (2002). *Acta Cryst.* **B58**, 903–913.
 Hostettler, M. & Schwarzenbach, D. (2002). *Acta Cryst.* **B58**, 914–920.
 Matthews, O. A., Shipway, A. N. & Stoddart, J. F. (1998). *Progr. Polym. Sci.* **23**, 1–56.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.