Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS
ISSN 2056-9890

# Structure of 2,2′-(5-*tert*-butyl-1,3-phenylene)bis(1-pentyl-1*H*-benzimidazol-3-ium) tetrachloridomercurate(II)

**Varsha Rani,[a] Harkesh B. Singh[a] and Ray J. Butcher[b]***

[a]Department of Chemistry, Indian Institute of Technology Bombay, Powai, Mumbai 400 076, India, and [b]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA. *Correspondence e-mail: rbutcher99@yahoo.com

Received 3 March 2017
Accepted 17 March 2017

Edited by M. Zeller, Purdue University, USA

**Keywords:** crystal structure; tetrachloridomercurate(II) salt; (benzimidazol-2-yl)benzene ligands; hydrogen bonding.

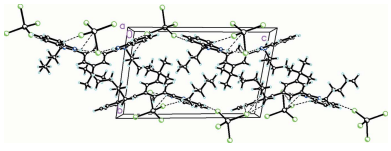
**CCDC reference**: 1538746

**Supporting information**: this article has supporting information at journals.iucr.org/e

In the title salt, $(C_{34}H_{44}N_4)[HgCl_4]$, the $[C_{34}H_{44}N_4]^{2+}$ cations and $[HgCl_4]^{2-}$ anions are linked by N—H···Cl hydrogen bonds. One of the two *n*-pentyl side chains was refined as disordered over two sets of sites, with occupancies of 0.733 (18) and 0.267 (18). The geometry around the $Hg^{II}$ atom in the $[HgCl_4]^{2-}$ anion is distorted tetrahedral, with bond angles ranging from 98.16 (3) to 120.68 (3)°. In the $[HgCl_4]^{2-}$ anion, there are two short Hg—Cl bonds [2.4120 (9) and 2.4171 (11) Å], one intermediate Hg—Cl bond [2.4716 (12) Å] and one long Hg—Cl bond [2.6579 (13) Å] for the Cl atom involved in a trifurcated hydrogen bond as an acceptor, including two N—H···Cl···H—N interactions as well as one C—H···Cl interaction. There are several C—H···Cl interactions, with C···Cl distances ranging from 3.492 (3) to 3.796 (3) Å. These link the cations and anions into a zigzag chain along the *c*-axis direction. In addition, there are Cl···Cl halogen bonds, as well as $\pi$–$\pi$ interactions, with centroid-to-centroid distances of 3.4765 (18) Å, which link one of the two benzimidazole moieties into dimeric units.

## 1. Chemical context

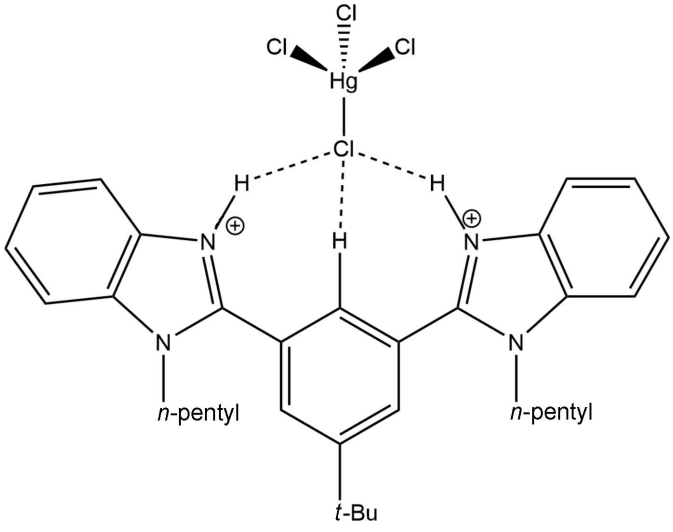
During the past few years, metallated complexes of the ligand 1,3-bis(1*H*-benzo[*d*]imidazol-2-yl)benzene have been well explored. This ligand is an ideal candidate for metalation due to the presence of two N atoms and one C atom, which bind tightly with metal atoms (Carina *et al.*, 1997; Obara *et al.*, 2006; Karlsson *et al.*, 2011; Yang *et al.*, 2012; Tam *et al.*, 2011; Gonzalez, 2014). As examples of the potential importance of this ligand, a highly phosphorescent iridium complex with bis(benzimidazol-2-yl)benzene ligand has been reported (Obara *et al.*, 2006) and helical and non helical copper(I) complexes with bis(benzimidazol-2-yl)benzene have been described (Rüttimann *et al.*, 1992). A trimeric complex has been obtained through the self assembly of cyclometalated trinuclear palladium(II) complexes (Rüttimann *et al.*, 1993). Dinuclear zinc complexes containing a (benzimidazol-2-yl)benzene based ligand have shown anticancer activity (Xie *et al.*, 2014).



A literature survey of mercury halide complexes with benzimidazole derivatives has shown that they come in two main types: polymeric, bridging either through the halide (Zhang *et al.*, 2015; Li *et al.*, 2007; Shen *et al.*, 2005;) or through alternative N atoms from the benzimidazole moieties (Xiao *et al.*, 2009, 2011; Huang *et al.*, 2006; Li *et al.*, 2007, 2012*a*,*b*; Dey *et al.*, 2013; Du *et al.*, 2011; Chen *et al.*, 2013; Su *et al.*, 2003; Xu *et al.*, 2011); or as discrete molecules (*i.e.* non-polymeric).

Reports of structurally related complex have been published recently (Rani *et al.*, 2017*a*,*b*).

An attempt was made to synthesize the compound 2,2′-(5-(*tert*-butyl)-2-(dichlorostibanyl)-1,3-phenylene)bis(1-pentyl-1*H*-benzimidazole) (**2**) from (4-(*tert*-butyl)-2,6-bis(1-pentyl-1*H*-benzimidazol-2-yl)phenyl)mercury(II) chloride; $[C_{34}H_{41}N_4HgCl]$ (**1**) using $SbCl_3$ in dry 1,4-dioxane *via* transmetallation. Related reactions (Rani *et al.*, 2017*a*,*b*) had yielded complexes containing an Hg atom bound to the ligand through Hg—N bonds. However, it was observed that the crystallization of compound **2** in MeOH at room temperature led to the formation of a bis-benzimidazolium cation; $[C_{34}H_{44}N_4]^{2+}[HgCl_4]^{2-}$, **3**. The elaborate procedure for the synthesis of complex **1** will be published elsewhere.




## 2. Structural commentary

The title compound, **3**, is a salt which contains $[C_{34}H_{44}N_4]^{2+}$ cations and $[HgCl_4]^{2-}$ anions linked by N—H···Cl hydrogen bonds. The reaction scheme leading to this product is shown in Fig. 1. The geometry around the mercury(II) atom in the $[HgCl_4]^{2-}$ anion is distorted tetrahedral with bond angles ranging from 98.16 (3) to 120.68 (3)°. In the $[HgCl_4]^{2-}$ anion, there are two short Hg—Cl bonds [Hg—Cl4, 2.4120 (9) Å; Hg—Cl3, 2.4171 (11) Å], one intermediate Hg—Cl bond [Hg—Cl2, 2.4716 (12) Å] and one long Hg—Cl bond [Hg—Cl1, 2.6579 (13) Å] for the Cl atom involved in a trifurcated bond as an acceptor including two N—H···Cl···H—N interactions as well as one C—H···Cl interaction (see Table 1), as shown in Fig. 2. Unlike a similar structure published recently containing a closely related ligand (Rani *et al.*, 2017*a*), where the Hg atom is bonded to an N atom from the benzimidazole moiety, in this instance a salt has been obtained due to the different conditions of the reaction. The structure has been published of a salt containing the tetrachloridomercurate(II) anion (Herbst *et al.*, 2013) and a closely related ligand with *n*-hexyl rather than *n*-pentyl side chains, which was the result of an attempted transmetallate reaction between Hg and Au.

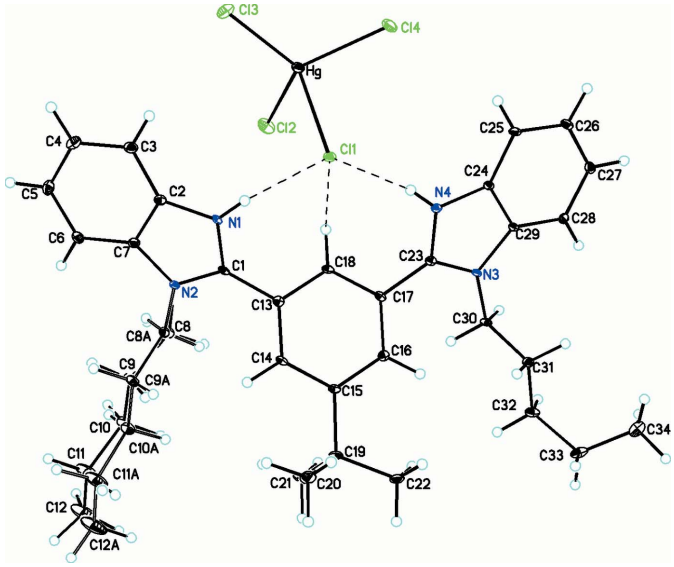



**Figure 2**
Diagram showing the atom labeling scheme, the trifurcated bond involving an N—H···Cl···H—N hydrogen bond, the C—H···Cl interactions and the disorder in one *n*-pentyl side chain. Atomic displacement parameters are at the 30% probability level.

In the ligand, the dihedral angles between the benzimidazole moieties and central phenyl ring are 40.60 (9) and 38.08 (10)°, while the angle between them is 36.04 (6)°. One of the pentyl substituents was refined as disordered over two sets of sites, with occupancies of 0.733 (18)/0.267 (18). The two pentyl side chains have adopted different conformations (for the disordered side-chain only values for the major conformation will be included) and this is illustrated by their torsion angles. For C8*A*–C12*A*, the angles involved, C1—N2—C8*A*—C9*A*, N2—C8*A*—C9*A*—C10*A*, C8*A*—C9*A*—C10*A*—C11*A*, and C9*A*—C10*A*—C11*A*—C12*A* and are 102.1 (16), −175.0 (15), 179.7 (15), and −178.1 (9)°, respectively, while

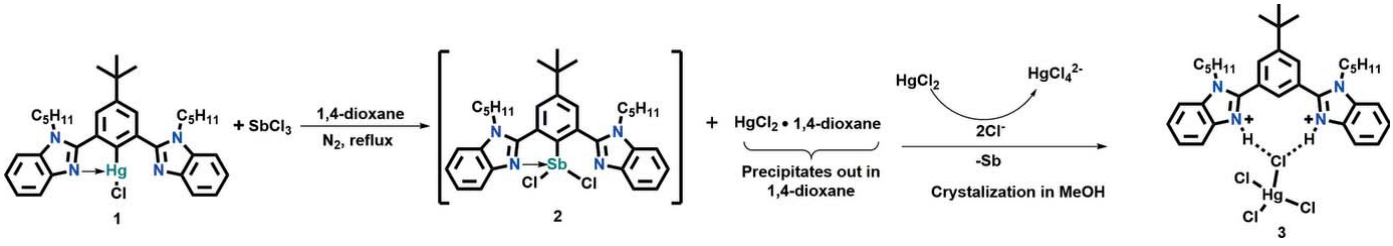



**Figure 1**
Reaction scheme showing the expected and actual products of the reaction.

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···Cl1 | 0.88 | 2.30 | 3.171 (2) | 171 |
| N4—H4$B$···Cl1 | 0.88 | 2.35 | 3.224 (2) | 170 |
| C3—H3$A$···Cl3 | 0.95 | 2.90 | 3.803 (3) | 160 |
| C6—H6$A$···Cl2$^{i}$ | 0.95 | 2.56 | 3.492 (3) | 169 |
| C18—H18$A$···Cl1 | 0.95 | 2.85 | 3.331 (4) | 113 |
| C25—H25$A$···Cl4$^{ii}$ | 0.95 | 2.96 | 3.664 (3) | 132 |
| C28—H28$A$···Cl4$^{iii}$ | 0.95 | 2.91 | 3.796 (3) | 156 |
| C30—H30$A$···Cl4$^{iii}$ | 0.99 | 2.77 | 3.627 (3) | 145 |

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+2, -y, -z+2$; (iii) $-x+1, -y, -z+2$.

for C30–C34 they are C23—N3—C30—C31, N3—C30—C31—C32, C30—C31—C32—C33, and C31—C32—C33—C34 [−105.7 (3), 175.7 (2), 173.0 (2) and −65.8 (3)°, respectively]. Thus the first side chain is in an all-*trans* conformation while the second side chain has adopted a conformation where it curls up at the end.

## 3. Supramolecular features

In addition to the inter-ionic hydrogen bonds mentioned above, there are several C—H···Cl interactions with C···Cl distances ranging from 3.492 (3) to 3.796 (3) Å (see Table1). These link the cations and anions into a zigzag chain in the *c*-axis direction, as shown in Fig. 3. There are are Cl···Cl halogen bonds [Cl4···Cl42 − x, −y, 2 − z) = 3.434 (2) Å], as shown in Fig. 4. In addition, one of the two benzimidazole moieties forms dimeric units through $\pi$–$\pi$ interactions (symmetry code $1-x, -y, 2-z$) with centroid-to-centroid distances of 3.477 (2) Å.

## 4. Database survey

A survey of the Cambridge Structural database (CSD Version 5.37) for salts containing both the benzimidazole moiety as well as the tetrachloridomercurate(II) anion gave eight hits, including a closely related ligand with *n*-hexyl rather than *n*-pentyl side chains (Herbst *et al.*, 2013).

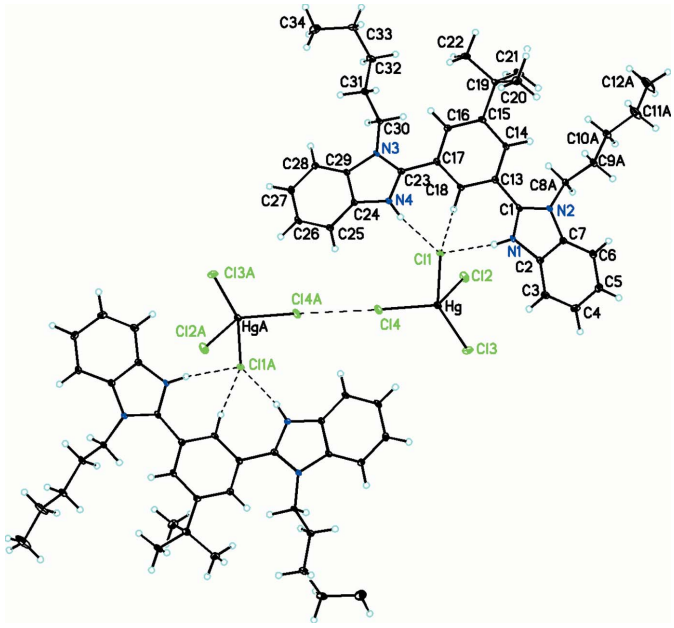



**Figure 4**
Diagram showing the Cl···Cl halogen bond.

## 5. Synthesis and crystallization

The reaction scheme is shown in Fig. 1. To a solution of **1** (0.2 g, 0.269 mmol) in dry 1,4-dioxane was added $SbCl_3$ (0.061 g, 0.269 mmol) at room temperature. The reaction mixture was refluxed for 6 h under an inert atmosphere of $N_2$ and filtered through Whatman filter paper. When the solvent was evaporated, a white-colored precipitate was obtained and purified by washing with hexane. The compound was dried under vacuum. Colourless block-shaped single crystals were

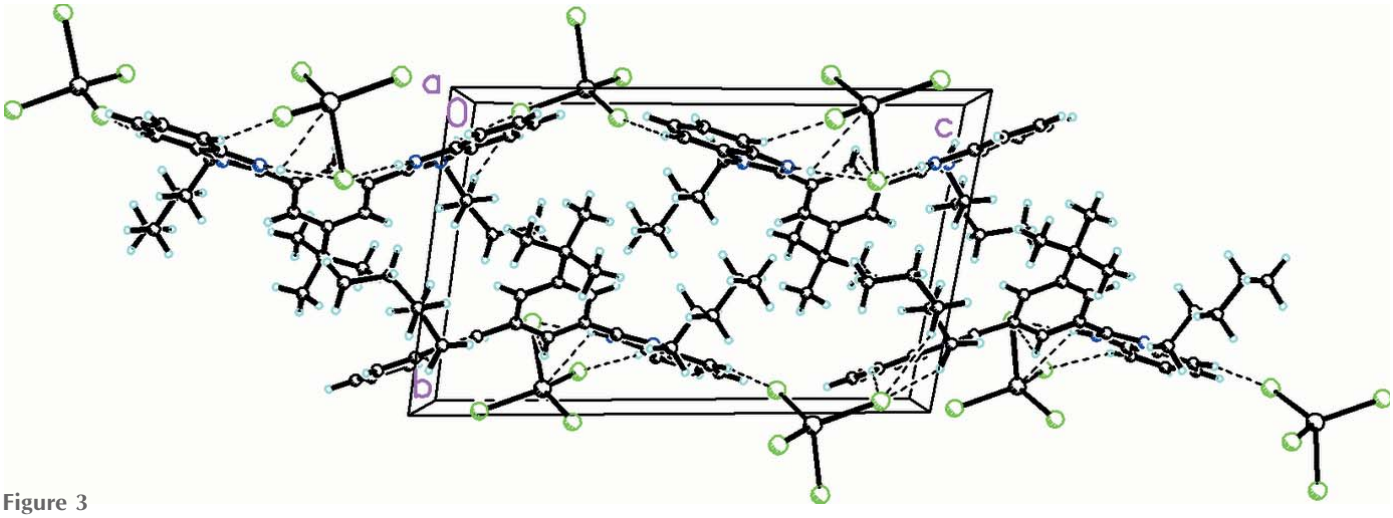



**Figure 3**
Diagram showing the C—H···Cl interactions, which link the cations and anions into a zigzag chain in the *c*-axis direction. The minor component of the pentyl disorder has been omitted for clarity. Atomic displacement parameters are at the 30% probability level.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $(C_{34}H_{44}N_4)[HgCl_4]$ |
| $M_r$ | 851.12 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 100 |
| $a, b, c$ (Å) | 9.806 (5), 11.264 (5), 17.274 (5) |
| $\alpha, \beta, \gamma$ (°) | 96.727 (5), 95.859 (5), 108.575 (5) |
| $V$ (Å$^3$) | 1776.4 (13) |
| $Z$ | 2 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 10.76 |
| Crystal size (mm) | 0.20 × 0.11 × 0.09 |
| Data collection | |
| Diffractometer | Bruker Quest CCD |
| Absorption correction | Multi-scan (*SADABS*; Sheldrick, 1996) |
| $T_{min}$, $T_{max}$ | 0.497, 0.753 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 6193, 6193, 6138 |
| $R_{int}$ | 0.038 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.020, 0.048, 1.10 |
| No. of reflections | 6193 |
| No. of parameters | 410 |
| No. of restraints | 67 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 1.36, −0.76 |

Computer programs: *APEX2* (Bruker, 2005), *SAINT* (Bruker, 2002), *SIR92* (Altomare *et al.*, 1993), *SHELXL2016* (Sheldrick, 2015) and *SHELXTL* (Sheldrick, 2008).

obtained from MeOH at room temperature, yield 64% (0.120 g).

$^1$H NMR (400 MHz, DMSO): $\delta$ 8.13 (*s*, 2H), 7.91 (*d*, $J$ = 7.2 Hz, 2H), 7.82 (*d*, $J$ = 7.2 Hz, 2H), 7.49–7.45 (*m*, 4H), 4.43 (*m*, 4H), 1.74 (*m*, 4H), 1.43 (*s*, 9H), 1.14 (*m*, 8H), 0.72 (*m*, 6H). $^{13}$C NMR (100 MHz, DMSO): 153.1, 151.4, 137.9, 134.7, 129.4, 128.5, 128.2, 124.7, 124.5, 117.8, 112.6, 45.1, 35.5, 31.3, 29.1, 28.5, 21.9, 14.1.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. One of the two *n*-pentyl side chains was refined as disordered over two sets of sites, with occupancies of 0.733 (18) and 0.267 (18) and both conformers were constrained to have similar metrical parameters using the SAME command in *SHELXL2016*. H atoms were positioned geometrically and refined as riding: N—H = 0.88 Å with $U_{iso}$(H) = 1.2$U_{eq}$(N); C—H = 0.95–0.98 Å with 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C) for methyl H atoms.

## Acknowledgements

RJB is grateful for NSF award 1205608, Partnership for Reduced Dimensional Materials, for partial funding of this research, and the Howard University Nanoscience Facility access to liquid nitrogen. The authors wish to acknowledge the assistance of Dr Matthias Zeller in the collection of diffraction data and NSF Grant DMR 1337296 for funds to purchase the X-ray diffractometer. HBS is grateful to the DST, New Delhi, for a J. C. Bose National Fellowship. VR gratefully acknowledges the Council of Scientific and Industrial Research (CSIR), New Delhi for an SRF.

## References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Bruker (2002). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carina, R. F., Williams, A. F. & Bernardinelli, G. (1997). *J. Organomet. Chem.* **548**, 45–48.
Chen, Y., Chen, C., Chen, H., Cao, T., Yue, Z., Liu, X. & Niu, Y. (2013). *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **43**, 1307–1310.
Dey, A., Mandal, S. K. & Biradha, K. (2013). *CrystEngComm*, **15**, 9769–9778.
Du, J.-L., Wei, Z.-Z. & Hu, T.-L. (2011). *Solid State Sci.* **13**, 1256–1260.
Gonzalez, A. D. (2014). *Organometallics*, **33**, 868–875.
Herbst, A., Bronner, C., Dechambenoit, P. & Wenger, O. S. (2013). *Organometallics*, **32**, 1807–1814.
Huang, M., Liu, P., Chen, Y., Wang, J. & Liu, Z. (2006). *J. Mol. Struct.* **788**, 211–217.
Karlsson, E. A., Lee, B., Åkermark, T., Johnston, E. V., Kärkäs, M. D., Sun, J., Hansson, Ö., Bäckvall, J. & Åkermark, B. (2011). *Angew. Chem. Int. Ed.* **50**, 11715–11718.
Li, J., Li, X., Lü, H., Zhu, Y., Sun, H., Guo, Y., Yue, Z., Zhao, J., Tang, M., Hou, H., Fan, Y. & Chang, J. (2012*b*). *Inorg. Chim. Acta*, **384**, 163–169.
Li, Y., Liu, Q.-K., Ma, J.-P. & Dong, Y.-B. (2012*a*). *Acta Cryst.* C**68**, m152–m155.
Li, X.-P., Zhang, J.-Y., Liu, Y., Pan, M., Zheng, S.-R., Kang, B.-S. & Su, C.-Y. (2007). *Inorg. Chim. Acta*, **360**, 2990–2996.
Obara, S., Itabashi, M., Okuda, F., Tamaki, S., Tanabe, Y., Ishii, Y., Nozaki, K. & Haga, M. (2006). *Inorg. Chem.* **45**, 8907–8921.
Rani, V., Singh, H. B. & Butcher, R. J. (2017*a*). *Acta Cryst.* E**73**, 341–344.
Rani, V., Singh, H. B. & Butcher, R. J. (2017*b*). *Acta Cryst.* E**73**, 423–428.
Rüttimann, S., Piguet, C., Bernardinelli, G., Bocquet, B. & Williams, A. F. (1992). *J. Am. Chem. Soc.* **114**, 4230–4237.
Rüttmann, S., Williams, A. F. & Bernardinelli, G. (1993). *Angew. Chem. Int. Ed. Engl.* **32**, 392–394.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Shen, Y.-H., Liu, J.-G. & Xu, D.-J. (2005). *Acta Cryst.* E**61**, m1880–m1882.
Su, C.-Y., Goforth, A. M., Smith, M. D. & zur Loye, H.-C. (2003). *Inorg. Chem.* **42**, 5685–5692.
Tam, A. Y. Y., Tsang, D. P. K., Chan, M. Y., Zhu, N. & Yam, V. W. W. (2011). *Chem. Commun.* **47**, 3383–3385.
Xiao, B., Li, W., Hou, H. & Fan, Y. (2009). *J. Coord. Chem.* **62**, 1630–1637.
Xiao, B., Yang, L.-J., Xiao, H.-Y. & Fang, S.-M. (2011). *J. Coord. Chem.* **64**, 4408–4420.
Xie, Q., Liu, S., Li, X., Wu, Q., Luo, Z., Fu, X., Cao, W., Lan, G., Li, D., Zheng, W. & Chen, T. (2014). *Dalton Trans.* **43**, 6973–6976.
Xu, C., Wang, X., Ding, D., Hou, H. & Fan, Y. (2011). *Inorg. Chem. Commun.* **14**, 1410–1413.
Yang, W. W., Zhong, Y. W., Yoshikawa, S., Shao, J. Y., Masaoka, S., Sakai, K., Yao, J. & Haga, M. (2012). *Inorg. Chem.* **51**, 890–899.
Zhang, Z., Feng, Y.-F., Wei, Q.-Y., Hu, K., Chen, Z.-L. & Liang, F.-P. (2015). *CrystEngComm*, **17**, 6724–6735.

# supporting information

*Acta Cryst.* (2017). E**73**, 560-563 [https://doi.org/10.1107/S2056989017004303]

## Structure of 2,2′-(5-*tert*-butyl-1,3-phenylene)bis(1-pentyl-1*H*-benzimidazol-3-ium) tetrachloridomercurate(II)

**Varsha Rani, Harkesh B. Singh and Ray J. Butcher**

### Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

### 2,2′-(5-*tert*-Butyl-1,3-phenylene)bis(1-pentyl-1*H*-benzimidazol-3-ium) tetrachloridomercurate(II)

*Crystal data*

$(C_{34}H_{44}N_4)[HgCl_4]$
$M_r = 851.12$
Triclinic, $P\overline{1}$
$a = 9.806$ (5) Å
$b = 11.264$ (5) Å
$c = 17.274$ (5) Å
$\alpha = 96.727$ (5)°
$\beta = 95.859$ (5)°
$\gamma = 108.575$ (5)°
$V = 1776.4$ (13) Å$^3$
$Z = 2$
$F(000) = 848$
$D_x = 1.591$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9629 reflections
$\theta = 2.6$–$61.2°$
$\mu = 10.76$ mm$^{-1}$
$T = 100$ K
Block, colourless
0.20 × 0.11 × 0.09 mm

*Data collection*

Bruker Quest CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.497$, $T_{max} = 0.753$
6193 measured reflections
6193 independent reflections
6138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 66.6°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.048$
$S = 1.10$
6193 reflections
410 parameters
67 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0123P)^2 + 4.1059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.76$ e Å$^{-3}$

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Hg | 0.92160 (2) | 0.04332 (2) | 0.77777 (2) | 0.01320 (4) | |
| Cl1 | 0.90325 (6) | 0.27221 (6) | 0.81804 (3) | 0.01173 (12) | |
| Cl2 | 0.67643 (7) | −0.05962 (7) | 0.70059 (4) | 0.02181 (15) | |
| Cl3 | 1.13497 (7) | 0.09387 (7) | 0.71258 (4) | 0.02149 (14) | |
| Cl4 | 0.92562 (7) | −0.03357 (7) | 0.90227 (4) | 0.02064 (14) | |
| N1 | 0.7288 (2) | 0.2251 (2) | 0.64598 (12) | 0.0103 (4) | |
| H1A | 0.786478 | 0.240862 | 0.691195 | 0.012* | |
| N2 | 0.5412 (2) | 0.2067 (2) | 0.55973 (12) | 0.0093 (4) | |
| N3 | 0.4506 (2) | 0.2061 (2) | 0.96891 (12) | 0.0083 (4) | |
| N4 | 0.6596 (2) | 0.2112 (2) | 0.93340 (12) | 0.0093 (4) | |
| H4B | 0.731909 | 0.222420 | 0.905661 | 0.011* | |
| C1 | 0.5974 (3) | 0.2376 (2) | 0.63675 (15) | 0.0093 (5) | |
| C2 | 0.7605 (3) | 0.1834 (2) | 0.57342 (15) | 0.0107 (5) | |
| C3 | 0.8791 (3) | 0.1515 (3) | 0.55187 (16) | 0.0145 (5) | |
| H3A | 0.959906 | 0.157791 | 0.589417 | 0.017* | |
| C4 | 0.8719 (3) | 0.1104 (3) | 0.47260 (17) | 0.0175 (6) | |
| H4A | 0.950066 | 0.087255 | 0.455158 | 0.021* | |
| C5 | 0.7527 (3) | 0.1016 (3) | 0.41676 (17) | 0.0182 (6) | |
| H5A | 0.753552 | 0.074077 | 0.362738 | 0.022* | |
| C6 | 0.6347 (3) | 0.1318 (3) | 0.43830 (16) | 0.0160 (6) | |
| H6A | 0.553716 | 0.125044 | 0.400717 | 0.019* | |
| C7 | 0.6410 (3) | 0.1728 (2) | 0.51834 (16) | 0.0112 (5) | |
| C8 | 0.388 (7) | 0.196 (6) | 0.529 (4) | 0.011 (2) | 0.267 (18) |
| H8A | 0.324342 | 0.184164 | 0.568393 | 0.013* | 0.267 (18) |
| H8B | 0.347222 | 0.123574 | 0.487932 | 0.013* | 0.267 (18) |
| C9 | 0.383 (3) | 0.295 (4) | 0.477 (3) | 0.0131 (15) | 0.267 (18) |
| H9A | 0.441717 | 0.290281 | 0.434405 | 0.016* | 0.267 (18) |
| H9B | 0.425475 | 0.380907 | 0.509106 | 0.016* | 0.267 (18) |
| C10 | 0.226 (3) | 0.274 (2) | 0.4416 (15) | 0.0146 (14) | 0.267 (18) |
| H10A | 0.185328 | 0.190262 | 0.407582 | 0.018* | 0.267 (18) |
| H10B | 0.166182 | 0.274397 | 0.484450 | 0.018* | 0.267 (18) |
| C11 | 0.218 (2) | 0.377 (2) | 0.3929 (14) | 0.0331 (16) | 0.267 (18) |
| H11A | 0.261683 | 0.461609 | 0.426420 | 0.040* | 0.267 (18) |
| H11B | 0.274690 | 0.375184 | 0.348875 | 0.040* | 0.267 (18) |
| C12 | 0.061 (2) | 0.359 (2) | 0.3600 (16) | 0.045 (2) | 0.267 (18) |
| H12A | 0.059390 | 0.417668 | 0.322680 | 0.067* | 0.267 (18) |
| H12B | 0.008610 | 0.375105 | 0.403260 | 0.067* | 0.267 (18) |
| H12C | 0.013309 | 0.271338 | 0.332829 | 0.067* | 0.267 (18) |
| C8A | 0.396 (2) | 0.198 (2) | 0.5222 (15) | 0.011 (2) | 0.733 (18) |

| | | | | | |
|---|---|---|---|---|---|
| H8AA | 0.336219 | 0.197140 | 0.562771 | 0.013* | 0.733 (18) |
| H8AB | 0.355259 | 0.119230 | 0.486591 | 0.013* | 0.733 (18) |
| C9A | 0.3993 (11) | 0.3134 (11) | 0.4833 (9) | 0.0131 (15) | 0.733 (18) |
| H9AA | 0.457776 | 0.317383 | 0.439655 | 0.016* | 0.733 (18) |
| H9AB | 0.445820 | 0.391532 | 0.522333 | 0.016* | 0.733 (18) |
| C10A | 0.2453 (8) | 0.3062 (7) | 0.4513 (5) | 0.0146 (14) | 0.733 (18) |
| H10C | 0.199418 | 0.227719 | 0.412483 | 0.018* | 0.733 (18) |
| H10D | 0.187130 | 0.301002 | 0.495179 | 0.018* | 0.733 (18) |
| C11A | 0.2417 (7) | 0.4193 (8) | 0.4123 (5) | 0.0331 (16) | 0.733 (18) |
| H11C | 0.290625 | 0.498002 | 0.450600 | 0.040* | 0.733 (18) |
| H11D | 0.297192 | 0.422840 | 0.367341 | 0.040* | 0.733 (18) |
| C12A | 0.0887 (7) | 0.4149 (10) | 0.3828 (6) | 0.045 (2) | 0.733 (18) |
| H12D | 0.093526 | 0.487533 | 0.355660 | 0.067* | 0.733 (18) |
| H12E | 0.035429 | 0.418294 | 0.427505 | 0.067* | 0.733 (18) |
| H12F | 0.038428 | 0.335987 | 0.346036 | 0.067* | 0.733 (18) |
| C13 | 0.5319 (3) | 0.2776 (2) | 0.70281 (15) | 0.0096 (5) | |
| C14 | 0.4612 (3) | 0.3667 (2) | 0.69900 (15) | 0.0106 (5) | |
| H14A | 0.448566 | 0.397654 | 0.650880 | 0.013* | |
| C15 | 0.4086 (3) | 0.4111 (2) | 0.76422 (15) | 0.0099 (5) | |
| C16 | 0.4336 (3) | 0.3674 (2) | 0.83453 (15) | 0.0100 (5) | |
| H16A | 0.402656 | 0.399307 | 0.880457 | 0.012* | |
| C17 | 0.5035 (3) | 0.2771 (2) | 0.83886 (15) | 0.0090 (5) | |
| C18 | 0.5510 (3) | 0.2304 (2) | 0.77264 (15) | 0.0092 (5) | |
| H18A | 0.595734 | 0.167208 | 0.774895 | 0.011* | |
| C19 | 0.3288 (3) | 0.5074 (2) | 0.75636 (16) | 0.0139 (5) | |
| C20 | 0.4329 (4) | 0.6272 (3) | 0.73242 (19) | 0.0228 (7) | |
| H20A | 0.382906 | 0.689130 | 0.726752 | 0.034* | |
| H20B | 0.463592 | 0.604517 | 0.682141 | 0.034* | |
| H20C | 0.518497 | 0.664220 | 0.773123 | 0.034* | |
| C21 | 0.1958 (3) | 0.4478 (3) | 0.69226 (18) | 0.0243 (7) | |
| H21A | 0.131693 | 0.369865 | 0.706788 | 0.036* | |
| H21B | 0.227761 | 0.427582 | 0.641934 | 0.036* | |
| H21C | 0.142904 | 0.507698 | 0.687084 | 0.036* | |
| C22 | 0.2771 (3) | 0.5445 (3) | 0.83319 (17) | 0.0162 (6) | |
| H22A | 0.208836 | 0.468930 | 0.847978 | 0.024* | |
| H22B | 0.228501 | 0.606900 | 0.825887 | 0.024* | |
| H22C | 0.360949 | 0.581275 | 0.874997 | 0.024* | |
| C23 | 0.5353 (3) | 0.2327 (2) | 0.91271 (15) | 0.0088 (5) | |
| C24 | 0.6564 (3) | 0.1682 (2) | 1.00576 (15) | 0.0091 (5) | |
| C25 | 0.7573 (3) | 0.1319 (2) | 1.05176 (15) | 0.0112 (5) | |
| H25A | 0.847917 | 0.133746 | 1.036094 | 0.013* | |
| C26 | 0.7169 (3) | 0.0929 (2) | 1.12160 (16) | 0.0127 (5) | |
| H26A | 0.782433 | 0.068395 | 1.155286 | 0.015* | |
| C27 | 0.5816 (3) | 0.0886 (2) | 1.14430 (15) | 0.0126 (5) | |
| H27A | 0.558408 | 0.060958 | 1.192707 | 0.015* | |
| C28 | 0.4818 (3) | 0.1234 (2) | 1.09797 (15) | 0.0104 (5) | |
| H28A | 0.390052 | 0.119514 | 1.112863 | 0.013* | |
| C29 | 0.5231 (3) | 0.1643 (2) | 1.02846 (15) | 0.0086 (5) | |

| | | | | |
|---|---|---|---|---|
| C30 | 0.3016 (3) | 0.2106 (2) | 0.96844 (15) | 0.0097 (5) |
| H30A | 0.239454 | 0.132290 | 0.984765 | 0.012* |
| H30B | 0.260795 | 0.213677 | 0.914165 | 0.012* |
| C31 | 0.2987 (3) | 0.3254 (2) | 1.02351 (15) | 0.0116 (5) |
| H31A | 0.366003 | 0.403763 | 1.009825 | 0.014* |
| H31B | 0.331720 | 0.319240 | 1.078541 | 0.014* |
| C32 | 0.1447 (3) | 0.3319 (3) | 1.01653 (17) | 0.0153 (6) |
| H32A | 0.117316 | 0.347650 | 0.962901 | 0.018* |
| H32B | 0.076545 | 0.248586 | 1.023004 | 0.018* |
| C33 | 0.1271 (3) | 0.4344 (3) | 1.07662 (18) | 0.0209 (6) |
| H33A | 0.198659 | 0.517088 | 1.071842 | 0.025* |
| H33B | 0.028751 | 0.439720 | 1.063744 | 0.025* |
| C34 | 0.1476 (4) | 0.4122 (3) | 1.16171 (19) | 0.0294 (7) |
| H34A | 0.123242 | 0.475748 | 1.196056 | 0.044* |
| H34B | 0.249058 | 0.419258 | 1.177643 | 0.044* |
| H34C | 0.083626 | 0.327163 | 1.166161 | 0.044* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Hg | 0.01221 (6) | 0.01533 (7) | 0.01315 (6) | 0.00693 (5) | −0.00075 (4) | 0.00220 (4) |
| Cl1 | 0.0100 (3) | 0.0128 (3) | 0.0126 (3) | 0.0058 (2) | −0.0012 (2) | −0.0006 (2) |
| Cl2 | 0.0157 (3) | 0.0210 (3) | 0.0217 (3) | 0.0007 (3) | −0.0083 (3) | 0.0010 (3) |
| Cl3 | 0.0168 (3) | 0.0241 (4) | 0.0258 (4) | 0.0097 (3) | 0.0075 (3) | 0.0008 (3) |
| Cl4 | 0.0176 (3) | 0.0293 (4) | 0.0191 (3) | 0.0108 (3) | 0.0016 (3) | 0.0121 (3) |
| N1 | 0.0104 (10) | 0.0146 (11) | 0.0073 (10) | 0.0067 (9) | −0.0015 (8) | 0.0027 (8) |
| N2 | 0.0096 (10) | 0.0109 (11) | 0.0087 (10) | 0.0056 (9) | −0.0003 (8) | 0.0019 (8) |
| N3 | 0.0082 (10) | 0.0088 (10) | 0.0088 (10) | 0.0046 (8) | −0.0010 (8) | 0.0016 (8) |
| N4 | 0.0084 (10) | 0.0107 (11) | 0.0102 (11) | 0.0046 (9) | 0.0018 (8) | 0.0024 (8) |
| C1 | 0.0114 (12) | 0.0061 (12) | 0.0102 (12) | 0.0030 (10) | −0.0008 (10) | 0.0033 (9) |
| C2 | 0.0105 (12) | 0.0097 (12) | 0.0120 (13) | 0.0042 (10) | 0.0005 (10) | 0.0016 (10) |
| C3 | 0.0109 (13) | 0.0167 (14) | 0.0173 (14) | 0.0064 (11) | 0.0010 (11) | 0.0040 (11) |
| C4 | 0.0144 (13) | 0.0195 (15) | 0.0212 (15) | 0.0082 (12) | 0.0064 (11) | 0.0026 (12) |
| C5 | 0.0231 (15) | 0.0205 (15) | 0.0123 (14) | 0.0094 (12) | 0.0044 (11) | 0.0002 (11) |
| C6 | 0.0189 (14) | 0.0208 (15) | 0.0102 (13) | 0.0105 (12) | −0.0012 (11) | 0.0020 (11) |
| C7 | 0.0124 (13) | 0.0101 (12) | 0.0124 (13) | 0.0052 (10) | 0.0017 (10) | 0.0022 (10) |
| C8 | 0.007 (3) | 0.0168 (16) | 0.008 (5) | 0.0046 (19) | −0.001 (3) | 0.002 (2) |
| C9 | 0.009 (2) | 0.016 (4) | 0.014 (3) | 0.003 (2) | −0.002 (2) | 0.005 (3) |
| C10 | 0.008 (2) | 0.017 (4) | 0.017 (3) | 0.003 (3) | −0.0042 (17) | 0.003 (3) |
| C11 | 0.015 (2) | 0.037 (4) | 0.050 (4) | 0.009 (3) | −0.003 (2) | 0.026 (3) |
| C12 | 0.021 (3) | 0.046 (5) | 0.070 (5) | 0.010 (3) | −0.007 (3) | 0.036 (4) |
| C8A | 0.007 (3) | 0.0168 (16) | 0.008 (5) | 0.0046 (19) | −0.001 (3) | 0.002 (2) |
| C9A | 0.009 (2) | 0.016 (4) | 0.014 (3) | 0.003 (2) | −0.002 (2) | 0.005 (3) |
| C10A | 0.008 (2) | 0.017 (4) | 0.017 (3) | 0.003 (3) | −0.0042 (17) | 0.003 (3) |
| C11A | 0.015 (2) | 0.037 (4) | 0.050 (4) | 0.009 (3) | −0.003 (2) | 0.026 (3) |
| C12A | 0.021 (3) | 0.046 (5) | 0.070 (5) | 0.010 (3) | −0.007 (3) | 0.036 (4) |
| C13 | 0.0081 (12) | 0.0084 (12) | 0.0105 (12) | 0.0014 (10) | −0.0007 (10) | 0.0004 (10) |
| C14 | 0.0112 (12) | 0.0098 (12) | 0.0113 (13) | 0.0039 (10) | −0.0007 (10) | 0.0043 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0094 (12) | 0.0067 (12) | 0.0135 (13) | 0.0028 (10) | 0.0006 (10) | 0.0024 (10) |
| C16 | 0.0102 (12) | 0.0077 (12) | 0.0111 (13) | 0.0022 (10) | 0.0010 (10) | 0.0003 (10) |
| C17 | 0.0075 (12) | 0.0072 (12) | 0.0101 (12) | 0.0004 (10) | −0.0019 (9) | 0.0018 (10) |
| C18 | 0.0068 (12) | 0.0076 (12) | 0.0128 (13) | 0.0025 (10) | −0.0012 (10) | 0.0023 (10) |
| C19 | 0.0182 (14) | 0.0101 (13) | 0.0174 (14) | 0.0095 (11) | 0.0030 (11) | 0.0042 (10) |
| C20 | 0.0355 (18) | 0.0146 (14) | 0.0269 (16) | 0.0148 (13) | 0.0148 (14) | 0.0104 (12) |
| C21 | 0.0270 (16) | 0.0317 (17) | 0.0223 (16) | 0.0234 (14) | −0.0040 (13) | 0.0036 (13) |
| C22 | 0.0211 (14) | 0.0128 (13) | 0.0211 (15) | 0.0118 (12) | 0.0075 (12) | 0.0060 (11) |
| C23 | 0.0097 (12) | 0.0047 (12) | 0.0106 (12) | 0.0017 (10) | −0.0005 (10) | −0.0001 (9) |
| C24 | 0.0122 (12) | 0.0054 (12) | 0.0090 (12) | 0.0037 (10) | −0.0013 (10) | −0.0007 (9) |
| C25 | 0.0107 (12) | 0.0100 (13) | 0.0136 (13) | 0.0060 (10) | −0.0012 (10) | 0.0004 (10) |
| C26 | 0.0156 (13) | 0.0093 (13) | 0.0132 (13) | 0.0063 (11) | −0.0044 (10) | 0.0009 (10) |
| C27 | 0.0185 (14) | 0.0093 (12) | 0.0099 (13) | 0.0047 (11) | 0.0010 (10) | 0.0021 (10) |
| C28 | 0.0132 (13) | 0.0079 (12) | 0.0099 (12) | 0.0042 (10) | 0.0011 (10) | −0.0006 (10) |
| C29 | 0.0100 (12) | 0.0057 (12) | 0.0091 (12) | 0.0033 (10) | −0.0030 (9) | −0.0011 (9) |
| C30 | 0.0049 (12) | 0.0120 (13) | 0.0128 (13) | 0.0037 (10) | −0.0008 (9) | 0.0031 (10) |
| C31 | 0.0096 (12) | 0.0130 (13) | 0.0124 (13) | 0.0050 (10) | −0.0003 (10) | 0.0014 (10) |
| C32 | 0.0121 (13) | 0.0146 (14) | 0.0204 (14) | 0.0070 (11) | −0.0007 (11) | 0.0027 (11) |
| C33 | 0.0188 (15) | 0.0177 (15) | 0.0300 (17) | 0.0121 (12) | 0.0044 (12) | 0.0011 (12) |
| C34 | 0.0379 (19) | 0.0280 (17) | 0.0275 (17) | 0.0191 (15) | 0.0084 (14) | −0.0012 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Hg—Cl4 | 2.4120 (9) | C11A—H11C | 0.9900 |
| Hg—Cl3 | 2.4171 (11) | C11A—H11D | 0.9900 |
| Hg—Cl2 | 2.4716 (12) | C12A—H12D | 0.9800 |
| Hg—Cl1 | 2.6579 (13) | C12A—H12E | 0.9800 |
| Cl4—Cl4$^{i}$ | 3.4343 (16) | C12A—H12F | 0.9800 |
| N1—C1 | 1.336 (3) | C13—C14 | 1.393 (4) |
| N1—C2 | 1.386 (3) | C13—C18 | 1.393 (4) |
| N1—H1A | 0.8800 | C14—C15 | 1.392 (4) |
| N2—C1 | 1.347 (3) | C14—H14A | 0.9500 |
| N2—C7 | 1.390 (3) | C15—C16 | 1.391 (4) |
| N2—C8A | 1.468 (17) | C15—C19 | 1.536 (3) |
| N2—C8 | 1.51 (5) | C16—C17 | 1.400 (4) |
| N3—C23 | 1.340 (3) | C16—H16A | 0.9500 |
| N3—C29 | 1.401 (3) | C17—C18 | 1.388 (4) |
| N3—C30 | 1.477 (3) | C17—C23 | 1.464 (4) |
| N4—C23 | 1.339 (3) | C18—H18A | 0.9500 |
| N4—C24 | 1.392 (3) | C19—C22 | 1.528 (4) |
| N4—H4B | 0.8800 | C19—C21 | 1.533 (4) |
| C1—C13 | 1.460 (4) | C19—C20 | 1.540 (4) |
| C2—C3 | 1.394 (4) | C20—H20A | 0.9800 |
| C2—C7 | 1.396 (4) | C20—H20B | 0.9800 |
| C3—C4 | 1.380 (4) | C20—H20C | 0.9800 |
| C3—H3A | 0.9500 | C21—H21A | 0.9800 |
| C4—C5 | 1.407 (4) | C21—H21B | 0.9800 |
| C4—H4A | 0.9500 | C21—H21C | 0.9800 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.379 (4) | C22—H22A | 0.9800 |
| C5—H5A | 0.9500 | C22—H22B | 0.9800 |
| C6—C7 | 1.394 (4) | C22—H22C | 0.9800 |
| C6—H6A | 0.9500 | C24—C29 | 1.392 (4) |
| C8—C9 | 1.524 (16) | C24—C25 | 1.396 (4) |
| C8—H8A | 0.9599 | C25—C26 | 1.384 (4) |
| C8—H8B | 0.9600 | C25—H25A | 0.9500 |
| C9—C10 | 1.532 (15) | C26—C27 | 1.409 (4) |
| C9—H9A | 0.9900 | C26—H26A | 0.9500 |
| C9—H9B | 0.9900 | C27—C28 | 1.382 (4) |
| C10—C11 | 1.529 (15) | C27—H27A | 0.9500 |
| C10—H10A | 0.9900 | C28—C29 | 1.389 (4) |
| C10—H10B | 0.9900 | C28—H28A | 0.9500 |
| C11—C12 | 1.526 (15) | C30—C31 | 1.523 (4) |
| C11—H11A | 0.9900 | C30—H30A | 0.9900 |
| C11—H11B | 0.9900 | C30—H30B | 0.9900 |
| C12—H12A | 0.9800 | C31—C32 | 1.528 (4) |
| C12—H12B | 0.9800 | C31—H31A | 0.9900 |
| C12—H12C | 0.9800 | C31—H31B | 0.9900 |
| C8A—C9A | 1.525 (8) | C32—C33 | 1.524 (4) |
| C8A—H8AA | 0.9599 | C32—H32A | 0.9900 |
| C8A—H8AB | 0.9600 | C32—H32B | 0.9900 |
| C9A—C10A | 1.527 (6) | C33—C34 | 1.524 (5) |
| C9A—H9AA | 0.9900 | C33—H33A | 0.9900 |
| C9A—H9AB | 0.9900 | C33—H33B | 0.9900 |
| C10A—C11A | 1.518 (6) | C34—H34A | 0.9800 |
| C10A—H10C | 0.9900 | C34—H34B | 0.9800 |
| C10A—H10D | 0.9900 | C34—H34C | 0.9800 |
| C11A—C12A | 1.518 (6) | | |

| | | | |
|---|---|---|---|
| Cl4—Hg—Cl3 | 120.68 (3) | H12D—C12A—H12E | 109.5 |
| Cl4—Hg—Cl2 | 108.75 (3) | C11A—C12A—H12F | 109.5 |
| Cl3—Hg—Cl2 | 120.54 (4) | H12D—C12A—H12F | 109.5 |
| Cl4—Hg—Cl1 | 102.32 (3) | H12E—C12A—H12F | 109.5 |
| Cl3—Hg—Cl1 | 101.25 (3) | C14—C13—C18 | 120.3 (2) |
| Cl2—Hg—Cl1 | 98.16 (3) | C14—C13—C1 | 121.9 (2) |
| Hg—Cl4—Cl4$^{i}$ | 146.10 (4) | C18—C13—C1 | 117.7 (2) |
| C1—N1—C2 | 109.6 (2) | C15—C14—C13 | 121.4 (2) |
| C1—N1—H1A | 125.2 | C15—C14—H14A | 119.3 |
| C2—N1—H1A | 125.2 | C13—C14—H14A | 119.3 |
| C1—N2—C7 | 108.4 (2) | C16—C15—C14 | 117.8 (2) |
| C1—N2—C8A | 128.6 (11) | C16—C15—C19 | 122.9 (2) |
| C7—N2—C8A | 122.9 (12) | C14—C15—C19 | 119.2 (2) |
| C1—N2—C8 | 123 (3) | C15—C16—C17 | 121.2 (2) |
| C7—N2—C8 | 128 (3) | C15—C16—H16A | 119.4 |
| C23—N3—C29 | 108.6 (2) | C17—C16—H16A | 119.4 |
| C23—N3—C30 | 127.4 (2) | C18—C17—C16 | 120.3 (2) |
| C29—N3—C30 | 123.9 (2) | C18—C17—C23 | 117.5 (2) |

| | | | |
|---|---|---|---|
| C23—N4—C24 | 109.2 (2) | C16—C17—C23 | 122.2 (2) |
| C23—N4—H4B | 125.4 | C17—C18—C13 | 118.9 (2) |
| C24—N4—H4B | 125.4 | C17—C18—H18A | 120.6 |
| N1—C1—N2 | 109.0 (2) | C13—C18—H18A | 120.6 |
| N1—C1—C13 | 122.6 (2) | C22—C19—C21 | 108.6 (2) |
| N2—C1—C13 | 128.3 (2) | C22—C19—C15 | 112.1 (2) |
| N1—C2—C3 | 132.0 (2) | C21—C19—C15 | 108.5 (2) |
| N1—C2—C7 | 106.0 (2) | C22—C19—C20 | 109.1 (2) |
| C3—C2—C7 | 122.0 (2) | C21—C19—C20 | 109.8 (2) |
| C4—C3—C2 | 115.9 (2) | C15—C19—C20 | 108.7 (2) |
| C4—C3—H3A | 122.1 | C19—C20—H20A | 109.5 |
| C2—C3—H3A | 122.1 | C19—C20—H20B | 109.5 |
| C3—C4—C5 | 122.2 (3) | H20A—C20—H20B | 109.5 |
| C3—C4—H4A | 118.9 | C19—C20—H20C | 109.5 |
| C5—C4—H4A | 118.9 | H20A—C20—H20C | 109.5 |
| C6—C5—C4 | 121.8 (3) | H20B—C20—H20C | 109.5 |
| C6—C5—H5A | 119.1 | C19—C21—H21A | 109.5 |
| C4—C5—H5A | 119.1 | C19—C21—H21B | 109.5 |
| C5—C6—C7 | 116.2 (3) | H21A—C21—H21B | 109.5 |
| C5—C6—H6A | 121.9 | C19—C21—H21C | 109.5 |
| C7—C6—H6A | 121.9 | H21A—C21—H21C | 109.5 |
| N2—C7—C6 | 131.1 (2) | H21B—C21—H21C | 109.5 |
| N2—C7—C2 | 107.1 (2) | C19—C22—H22A | 109.5 |
| C6—C7—C2 | 121.8 (2) | C19—C22—H22B | 109.5 |
| N2—C8—C9 | 112 (4) | H22A—C22—H22B | 109.5 |
| N2—C8—H8A | 113.5 | C19—C22—H22C | 109.5 |
| C9—C8—H8A | 116.9 | H22A—C22—H22C | 109.5 |
| N2—C8—H8B | 109.3 | H22B—C22—H22C | 109.5 |
| C9—C8—H8B | 96.3 | N4—C23—N3 | 109.2 (2) |
| H8A—C8—H8B | 107.2 | N4—C23—C17 | 122.9 (2) |
| C8—C9—C10 | 110 (2) | N3—C23—C17 | 127.9 (2) |
| C8—C9—H9A | 109.6 | N4—C24—C29 | 106.4 (2) |
| C10—C9—H9A | 109.6 | N4—C24—C25 | 131.6 (2) |
| C8—C9—H9B | 109.6 | C29—C24—C25 | 122.0 (2) |
| C10—C9—H9B | 109.6 | C26—C25—C24 | 115.8 (2) |
| H9A—C9—H9B | 108.1 | C26—C25—H25A | 122.1 |
| C11—C10—C9 | 111.3 (18) | C24—C25—H25A | 122.1 |
| C11—C10—H10A | 109.4 | C25—C26—C27 | 122.0 (2) |
| C9—C10—H10A | 109.4 | C25—C26—H26A | 119.0 |
| C11—C10—H10B | 109.4 | C27—C26—H26A | 119.0 |
| C9—C10—H10B | 109.4 | C28—C27—C26 | 121.7 (2) |
| H10A—C10—H10B | 108.0 | C28—C27—H27A | 119.1 |
| C12—C11—C10 | 111.1 (15) | C26—C27—H27A | 119.1 |
| C12—C11—H11A | 109.4 | C27—C28—C29 | 116.3 (2) |
| C10—C11—H11A | 109.4 | C27—C28—H28A | 121.8 |
| C12—C11—H11B | 109.4 | C29—C28—H28A | 121.8 |
| C10—C11—H11B | 109.4 | C28—C29—C24 | 122.1 (2) |
| H11A—C11—H11B | 108.0 | C28—C29—N3 | 131.4 (2) |

| | | | |
|---|---|---|---|
| C11—C12—H12A | 109.5 | C24—C29—N3 | 106.6 (2) |
| C11—C12—H12B | 109.5 | N3—C30—C31 | 111.9 (2) |
| H12A—C12—H12B | 109.5 | N3—C30—H30A | 109.2 |
| C11—C12—H12C | 109.5 | C31—C30—H30A | 109.2 |
| H12A—C12—H12C | 109.5 | N3—C30—H30B | 109.2 |
| H12B—C12—H12C | 109.5 | C31—C30—H30B | 109.2 |
| N2—C8A—C9A | 112.4 (13) | H30A—C30—H30B | 107.9 |
| N2—C8A—H8AA | 107.8 | C30—C31—C32 | 110.1 (2) |
| C9A—C8A—H8AA | 106.1 | C30—C31—H31A | 109.6 |
| N2—C8A—H8AB | 108.8 | C32—C31—H31A | 109.6 |
| C9A—C8A—H8AB | 113.3 | C30—C31—H31B | 109.6 |
| H8AA—C8A—H8AB | 108.3 | C32—C31—H31B | 109.6 |
| C8A—C9A—C10A | 110.8 (7) | H31A—C31—H31B | 108.1 |
| C8A—C9A—H9AA | 109.5 | C33—C32—C31 | 113.9 (2) |
| C10A—C9A—H9AA | 109.5 | C33—C32—H32A | 108.8 |
| C8A—C9A—H9AB | 109.5 | C31—C32—H32A | 108.8 |
| C10A—C9A—H9AB | 109.5 | C33—C32—H32B | 108.8 |
| H9AA—C9A—H9AB | 108.1 | C31—C32—H32B | 108.8 |
| C11A—C10A—C9A | 113.0 (6) | H32A—C32—H32B | 107.7 |
| C11A—C10A—H10C | 109.0 | C34—C33—C32 | 114.2 (2) |
| C9A—C10A—H10C | 109.0 | C34—C33—H33A | 108.7 |
| C11A—C10A—H10D | 109.0 | C32—C33—H33A | 108.7 |
| C9A—C10A—H10D | 109.0 | C34—C33—H33B | 108.7 |
| H10C—C10A—H10D | 107.8 | C32—C33—H33B | 108.7 |
| C12A—C11A—C10A | 113.2 (5) | H33A—C33—H33B | 107.6 |
| C12A—C11A—H11C | 108.9 | C33—C34—H34A | 109.5 |
| C10A—C11A—H11C | 108.9 | C33—C34—H34B | 109.5 |
| C12A—C11A—H11D | 108.9 | H34A—C34—H34B | 109.5 |
| C10A—C11A—H11D | 108.9 | C33—C34—H34C | 109.5 |
| H11C—C11A—H11D | 107.7 | H34A—C34—H34C | 109.5 |
| C11A—C12A—H12D | 109.5 | H34B—C34—H34C | 109.5 |
| C11A—C12A—H12E | 109.5 | | |

| | | | |
|---|---|---|---|
| C2—N1—C1—N2 | −0.7 (3) | C19—C15—C16—C17 | 178.4 (2) |
| C2—N1—C1—C13 | 178.7 (2) | C15—C16—C17—C18 | 0.8 (4) |
| C7—N2—C1—N1 | 0.2 (3) | C15—C16—C17—C23 | 177.9 (2) |
| C8A—N2—C1—N1 | 175.5 (10) | C16—C17—C18—C13 | 2.0 (4) |
| C8—N2—C1—N1 | 173 (3) | C23—C17—C18—C13 | −175.4 (2) |
| C7—N2—C1—C13 | −179.2 (2) | C14—C13—C18—C17 | −2.5 (4) |
| C8A—N2—C1—C13 | −3.9 (11) | C1—C13—C18—C17 | 173.2 (2) |
| C8—N2—C1—C13 | −7 (3) | C16—C15—C19—C22 | −1.9 (4) |
| C1—N1—C2—C3 | −177.2 (3) | C14—C15—C19—C22 | 179.3 (2) |
| C1—N1—C2—C7 | 0.9 (3) | C16—C15—C19—C21 | −121.9 (3) |
| N1—C2—C3—C4 | 178.6 (3) | C14—C15—C19—C21 | 59.4 (3) |
| C7—C2—C3—C4 | 0.8 (4) | C16—C15—C19—C20 | 118.8 (3) |
| C2—C3—C4—C5 | 0.3 (4) | C14—C15—C19—C20 | −60.0 (3) |
| C3—C4—C5—C6 | −1.0 (5) | C24—N4—C23—N3 | 0.3 (3) |
| C4—C5—C6—C7 | 0.7 (4) | C24—N4—C23—C17 | −179.1 (2) |

| | | | |
|---|---|---|---|
| C1—N2—C7—C6 | 178.9 (3) | C29—N3—C23—N4 | −0.5 (3) |
| C8A—N2—C7—C6 | 3.3 (10) | C30—N3—C23—N4 | −176.8 (2) |
| C8—N2—C7—C6 | 7 (3) | C29—N3—C23—C17 | 178.8 (2) |
| C1—N2—C7—C2 | 0.4 (3) | C30—N3—C23—C17 | 2.5 (4) |
| C8A—N2—C7—C2 | −175.3 (9) | C18—C17—C23—N4 | 37.0 (3) |
| C8—N2—C7—C2 | −172 (2) | C16—C17—C23—N4 | −140.3 (3) |
| C5—C6—C7—N2 | −178.0 (3) | C18—C17—C23—N3 | −142.3 (3) |
| C5—C6—C7—C2 | 0.4 (4) | C16—C17—C23—N3 | 40.4 (4) |
| N1—C2—C7—N2 | −0.8 (3) | C23—N4—C24—C29 | 0.1 (3) |
| C3—C2—C7—N2 | 177.6 (2) | C23—N4—C24—C25 | 179.3 (3) |
| N1—C2—C7—C6 | −179.5 (2) | N4—C24—C25—C26 | −179.7 (3) |
| C3—C2—C7—C6 | −1.2 (4) | C29—C24—C25—C26 | −0.6 (4) |
| C1—N2—C8—C9 | 115 (5) | C24—C25—C26—C27 | 0.9 (4) |
| C7—N2—C8—C9 | −74 (6) | C25—C26—C27—C28 | −0.2 (4) |
| N2—C8—C9—C10 | 175 (4) | C26—C27—C28—C29 | −0.8 (4) |
| C8—C9—C10—C11 | 177 (4) | C27—C28—C29—C24 | 1.1 (4) |
| C9—C10—C11—C12 | −178 (3) | C27—C28—C29—N3 | −179.8 (2) |
| C1—N2—C8A—C9A | 102.1 (16) | N4—C24—C29—C28 | 178.9 (2) |
| C7—N2—C8A—C9A | −83 (2) | C25—C24—C29—C28 | −0.4 (4) |
| N2—C8A—C9A—C10A | −175.0 (15) | N4—C24—C29—N3 | −0.4 (3) |
| C8A—C9A—C10A—C11A | 179.7 (15) | C25—C24—C29—N3 | −179.7 (2) |
| C9A—C10A—C11A—C12A | −178.1 (9) | C23—N3—C29—C28 | −178.6 (3) |
| N1—C1—C13—C14 | 136.1 (3) | C30—N3—C29—C28 | −2.2 (4) |
| N2—C1—C13—C14 | −44.6 (4) | C23—N3—C29—C24 | 0.6 (3) |
| N1—C1—C13—C18 | −39.6 (4) | C30—N3—C29—C24 | 177.0 (2) |
| N2—C1—C13—C18 | 139.8 (3) | C23—N3—C30—C31 | −105.7 (3) |
| C18—C13—C14—C15 | 0.3 (4) | C29—N3—C30—C31 | 78.5 (3) |
| C1—C13—C14—C15 | −175.2 (2) | N3—C30—C31—C32 | 175.7 (2) |
| C13—C14—C15—C16 | 2.3 (4) | C30—C31—C32—C33 | 173.0 (2) |
| C13—C14—C15—C19 | −178.8 (2) | C31—C32—C33—C34 | −65.8 (3) |
| C14—C15—C16—C17 | −2.9 (4) | | |

Symmetry code: (i) −*x*+2, −*y*, −*z*+2.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···Cl1 | 0.88 | 2.30 | 3.171 (2) | 171 |
| N4—H4*B*···Cl1 | 0.88 | 2.35 | 3.224 (2) | 170 |
| C3—H3*A*···Cl3 | 0.95 | 2.90 | 3.803 (3) | 160 |
| C6—H6*A*···Cl2$^{ii}$ | 0.95 | 2.56 | 3.492 (3) | 169 |
| C18—H18*A*···Cl1 | 0.95 | 2.85 | 3.331 (4) | 113 |
| C25—H25*A*···Cl4$^{i}$ | 0.95 | 2.96 | 3.664 (3) | 132 |
| C28—H28*A*···Cl4$^{iii}$ | 0.95 | 2.91 | 3.796 (3) | 156 |
| C30—H30*A*···Cl4$^{iii}$ | 0.99 | 2.77 | 3.627 (3) | 145 |

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