

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

Chlorido(2-formyl-6-hydroxyphenyl- $\kappa$ C<sup>1</sup>)mercury(II)Chen Xu,<sup>a\*</sup> Fei-Fei Cen,<sup>b</sup> Zhi-Qiang Wang<sup>a</sup> and Yu-Qing Zhang<sup>b</sup>

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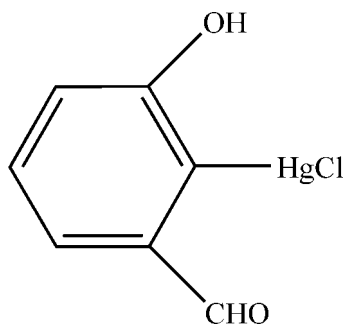
Received 1 June 2009; accepted 5 June 2009

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.018$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.114; data-to-parameter ratio = 15.8.

In the planar [r.m.s. deviation 0.0265 Å] title compound, [Hg(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)Cl], the Hg<sup>II</sup> atom shows a typical linear coordination by a C atom of a benzene ring and a Cl atom. The benzene C atom and the aldehyde O atom chelate the Hg<sup>II</sup> atom by assuming the Hg...O separation of 2.817 (9) Å as a weak intramolecular coordination bonding distance. The resulting five-membered metallacycle is nearly coplanar with the benzene ring dihedral angle 2.9 (1)°. Intermolecular O—H...O hydrogen bonds are present in the crystal structure, resulting in a one-dimensional supramolecular architecture parallel to [201].

## Related literature

For historical background and for properties of cyclometallated compounds, see: Dupont *et al.* (2005); Xu *et al.* (2009). For the properties of cyclomercurated compounds, see: Wu *et al.* (2001); Ryabov *et al.* (2003). For related structure, see: King *et al.* (2002); Zhou *et al.* (2005); Hao *et al.* (2007).



## Experimental

## Crystal data

[Hg(C <sub>7</sub> H <sub>5</sub> O <sub>2</sub> )Cl]	$V = 867.4$ (6) Å <sup>3</sup>
$M_r = 357.15$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 4.7200$ (19) Å	$\mu = 18.00$ mm <sup>-1</sup>
$b = 17.702$ (7) Å	$T = 296$ K
$c = 10.506$ (4) Å	$0.08 \times 0.01 \times 0.01$ mm
$\beta = 98.839$ (5)°	

## Data collection

Bruker SMART APEX CCD area-detector diffractometer	5002 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1595 independent reflections
$T_{\min} = 0.327$ , $T_{\max} = 0.841$	1130 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.050$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	101 parameters
$wR(F^2) = 0.114$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.94$ e Å <sup>-3</sup>
1595 reflections	$\Delta\rho_{\text{min}} = -2.32$ e Å <sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots O2^i$	0.82	1.93	2.730 (12)	165

Symmetry code: (i)  $x - 1, -y + \frac{3}{2}, z + \frac{1}{2}$ .

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

This work was supported by the Natural Science Foundation of Henan Education Department (No. 2009B150019).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2180).

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**supplementary materials**

*Acta Cryst.* (2009). E65, m754 [ doi:10.1107/S160053680902145X ]

## Chlorido(2-formyl-6-hydroxyphenyl- $\kappa C^1$ )mercury(II)

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### Comment

Cyclometallated compounds containing a metal-carbon bond stabilized by the intramolecular coordination of one or two neutral atoms have a very rich chemistry and are widely used in synthesis, catalysis and materials (Dupont *et al.*, 2005; Xu *et al.*, 2009). Among them, cyclomercurated compounds are easy to prepare through a C—H activation process and their ease in undergoing transmetalation for the synthesis of other organometallic compounds (Wu *et al.*, 2001; Ryabov *et al.*, 2003).

In the planar title compound (Fig. 1), the mercury(II) atom shows a typical linear coordination geometry with a carbon atom of the benzene ring and the chloride atom in *trans* position. O2—Hg1 distance (2.817 (9) Å) is shorter than the sum of van der Waals radii (3.29 Å) of Hg and O (King *et al.*, 2002), indicating the presence of the weak intramolecular coordination, while it is longer than those of the related Hg(II) complex (Zhou *et al.*, 2005). The C—Hg and Hg—Cl bond distances are within normal ranges. The C7—Hg1—Cl1 angle is 178.1 (3)°, slightly smaller than the ideal value of 180° in organic derivatives of mercury (Hao *et al.*, 2007). Intermolecular O—H...O hydrogen bonds are present in the crystal structure (Table 1), resulting in a one-dimensional supramolecular architecture (Fig.2).

### Experimental

The title compound was prepared from the *m*-hydroxybenzaldehyde with Hg(OAc)<sub>2</sub> and subsequent treatment with LiCl and recrystallized from dichloromethane-petroleum ether solution at room temperature to give the desired product as colorless crystals suitable for single-crystal X-ray diffraction (yield 82%; m.p 442–444 K). IR data ( $\nu_{\max}$ /cm<sup>-1</sup>): 3408, 2926, 1651, 1567, 1445, 1291, 1199, 789. NMR  $\delta$ (H) 7.18(1H,d), 7.45 (1H,t), 7.52(1H,d), 10.12(1H,s), 12.11(1H,m).

### Refinement

H atoms attached to C atoms of the title compound were placed in geometrically idealized positions and treated as riding with C—H distances constrained to 0.93–0.96 Å, and with  $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ .

### Figures

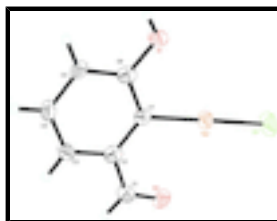


Fig. 1. The molecular structure of the title compound with displacement ellipsoids at the 30% probability level.

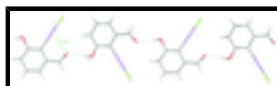


Fig. 2. Partial view of the crystal packing showing the formation of the one-dimensional chain structure formed by the intermolecular O—H...O hydrogen bonds.

## Chlorido(2-formyl-6-hydroxyphenyl- $\kappa$ C<sup>1</sup>)mercury(II)

### Crystal data

[Hg(C <sub>7</sub> H <sub>5</sub> O <sub>2</sub> )Cl]	$F_{000} = 640$
$M_r = 357.15$	$D_x = 2.735 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 4.7200 (19) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 17.702 (7) \text{ \AA}$	Cell parameters from 1175 reflections
$c = 10.506 (4) \text{ \AA}$	$\theta = 2.3\text{--}22.3^\circ$
$\beta = 98.839 (5)^\circ$	$\mu = 18.00 \text{ mm}^{-1}$
$V = 867.4 (6) \text{ \AA}^3$	$T = 296 \text{ K}$
$Z = 4$	Block, colourless
	$0.08 \times 0.01 \times 0.01 \text{ mm}$

### Data collection

Bruker SMART APEX CCD area-detector diffractometer	1595 independent reflections
Radiation source: fine-focus sealed tube	1130 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.050$
$T = 296 \text{ K}$	$\theta_{\text{max}} = 25.5^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -5 \rightarrow 5$
$T_{\text{min}} = 0.327$ , $T_{\text{max}} = 0.841$	$k = -21 \rightarrow 21$
5002 measured reflections	$l = -12 \rightarrow 12$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 3.2348P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1595 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
101 parameters	$\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -2.32 \text{ e \AA}^{-3}$
	Extinction correction: none

### 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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Hg1	0.26063 (12)	0.66539 (3)	0.30597 (6)	0.0470 (2)
O1	-0.160 (2)	0.6917 (5)	0.5036 (10)	0.050 (2)
H1	-0.2948	0.6960	0.5439	0.074*
O2	0.468 (2)	0.7878 (5)	0.1758 (9)	0.052 (2)
Cl1	0.4684 (11)	0.5580 (2)	0.2416 (5)	0.0833 (14)
C1	0.345 (3)	0.8403 (7)	0.2208 (14)	0.043 (3)
H1A	0.3802	0.8888	0.1928	0.052*
C2	0.151 (3)	0.8330 (7)	0.3132 (13)	0.042 (3)
C3	0.039 (3)	0.8998 (7)	0.3574 (14)	0.051 (4)
H3	0.0837	0.9460	0.3235	0.061*
C4	-0.137 (3)	0.8974 (7)	0.4509 (13)	0.048 (4)
H4	-0.2123	0.9420	0.4788	0.058*
C5	-0.204 (3)	0.8276 (7)	0.5043 (13)	0.044 (3)
H5	-0.3193	0.8257	0.5683	0.053*
C6	-0.092 (3)	0.7611 (7)	0.4591 (12)	0.042 (3)
C7	0.087 (2)	0.7635 (7)	0.3644 (11)	0.032 (3)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Hg1	0.0477 (4)	0.0392 (3)	0.0583 (4)	0.0027 (3)	0.0213 (3)	-0.0048 (3)
O1	0.047 (6)	0.048 (5)	0.060 (6)	-0.006 (4)	0.027 (5)	0.003 (5)
O2	0.047 (6)	0.058 (6)	0.056 (6)	-0.003 (5)	0.028 (5)	-0.011 (5)

## supplementary materials

C11	0.098 (4)	0.044 (2)	0.117 (4)	0.016 (2)	0.045 (3)	-0.010 (2)
C1	0.033 (7)	0.043 (7)	0.054 (8)	-0.002 (6)	0.007 (6)	0.005 (7)
C2	0.029 (6)	0.053 (8)	0.043 (7)	-0.008 (6)	0.007 (6)	0.000 (6)
C3	0.058 (9)	0.034 (7)	0.070 (10)	0.012 (6)	0.038 (8)	0.012 (7)
C4	0.056 (9)	0.035 (7)	0.056 (9)	0.006 (6)	0.012 (7)	-0.010 (6)
C5	0.040 (8)	0.053 (8)	0.044 (8)	0.005 (7)	0.020 (6)	-0.007 (7)
C6	0.027 (7)	0.054 (8)	0.045 (8)	-0.012 (6)	0.007 (6)	0.000 (6)
C7	0.023 (6)	0.043 (7)	0.026 (6)	-0.004 (5)	-0.008 (5)	0.001 (5)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Hg1—C7	2.052 (12)	C2—C3	1.403 (17)
Hg1—C11	2.288 (4)	C3—C4	1.382 (17)
O1—C6	1.370 (14)	C3—H3	0.9300
O1—H1	0.8200	C4—C5	1.412 (18)
O2—C1	1.229 (14)	C4—H4	0.9300
C1—C2	1.440 (18)	C5—C6	1.402 (17)
C1—H1A	0.9300	C5—H5	0.9300
C2—C7	1.394 (16)	C6—C7	1.403 (16)
C7—Hg1—C11	178.1 (3)	C3—C4—H4	119.9
C6—O1—H1	109.5	C5—C4—H4	119.9
O2—C1—C2	125.4 (12)	C6—C5—C4	118.9 (11)
O2—C1—H1A	117.3	C6—C5—H5	120.6
C2—C1—H1A	117.3	C4—C5—H5	120.6
C7—C2—C1	122.4 (12)	O1—C6—C7	117.8 (11)
C7—C2—C3	120.1 (12)	O1—C6—C5	121.2 (11)
C1—C2—C3	117.3 (11)	C7—C6—C5	121.0 (12)
C4—C3—C2	120.6 (12)	C2—C7—C6	119.2 (11)
C4—C3—H3	119.7	C2—C7—Hg1	120.8 (9)
C2—C3—H3	119.7	C6—C7—Hg1	119.9 (9)
C3—C4—C5	120.2 (11)		
O2—C1—C2—C7	2(2)	C3—C2—C7—C6	0.8 (19)
O2—C1—C2—C3	177.6 (14)	C1—C2—C7—Hg1	-1.9 (18)
C7—C2—C3—C4	-1(2)	C3—C2—C7—Hg1	-177.8 (10)
C1—C2—C3—C4	-176.7 (14)	O1—C6—C7—C2	177.3 (12)
C2—C3—C4—C5	1(2)	C5—C6—C7—C2	-1.1 (19)
C3—C4—C5—C6	-1(2)	O1—C6—C7—Hg1	-4.2 (16)
C4—C5—C6—O1	-177.0 (12)	C5—C6—C7—Hg1	177.4 (10)
C4—C5—C6—C7	1(2)	C11—Hg1—C7—C2	46 (10)
C1—C2—C7—C6	176.6 (12)	C11—Hg1—C7—C6	-132 (10)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	1.93	2.730 (12)	165

Symmetry codes: (i)  $x-1, -y+3/2, z+1/2$ .

Fig. 1

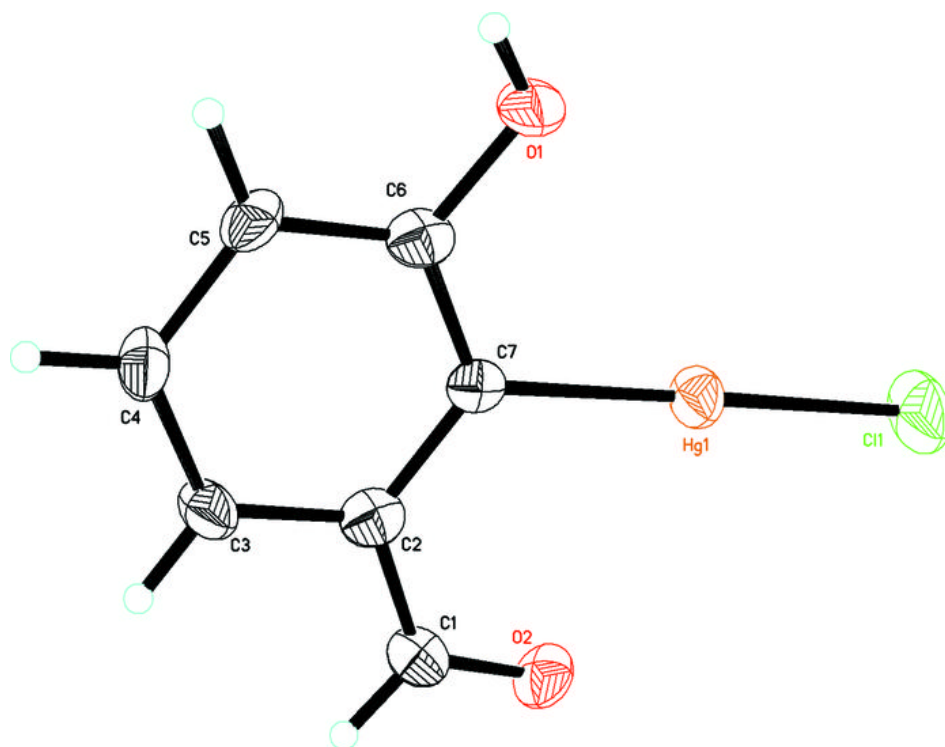


Fig. 2

