Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of mercury(II) hydroxide chlorate(V)

Dirk Göbbels and Mathias S. Wickleder*

Institut für Anorganische Chemie, Universität zu Köln, Greinstraße 6, D-50939 Köln, Germany

Correspondence e-mail: mathias.wickleder@uni-koeln.de

Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(Cl–O) = 0.005 Å
R factor = 0.020
wR factor = 0.043
Data-to-parameter ratio = 16.0

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

Received 12 January 2004
Accepted 2 February 2004
Online 14 February 2004

$Hg(OH)ClO_3$ is built up from infinite zigzag $[Hg(OH)_{2/2}]^+$ chains along [001] and $[ClO_3]^-$ ions. These chains are connected *via* weak Hg—O interactions to O atoms of the $[ClO_3]^-$ ions, leading to layers parallel to (010). O—H···O hydrogen bonds are present between these layers.

Comment

$Hg(OH)ClO_3$ (Weiss *et al.*, 1960), whose structure is redetermined here with considerably greater precision and with the H atom located, is isotypic with $Hg(OH)BrO_3$ (Björnlund, 1971), but different from other basic Hg(OH)*X* compounds, such as $Hg(OH)NO_3$ (Ribar *et al.*, 1971; Matkovic *et al.*, 1974) or Hg(OH)F (Grdenic & Sikirica, 1973; Stalhandske, 1979; Nozik *et al.*, 1979). The structure of $Hg(OH)ClO_3$ contains infinite zigzag $[Hg(OH)_{2/2}]^+$ chains along the [001] direction (Fig. 1). The distances [Hg—O = 2.051 (2) Å] and angles [(H)O—Hg—O(H) = 177.9 (2)° and Hg—(OH)—Hg = 122.6 (2)°] in the chains are comparable with those in $Hg_2(OH)[BF_4]$ (Meyer & Göbbels, 2003) and $(Hg_2)Hg(OH)_2[ClO_4]_2$ (Wickleder, 2002). These chains are further connected *via* weak Hg—O bonds [2.733 (3) and 2.763 (3) Å] to O atoms of the $[ClO_3]^-$ ions, forming layers that are parallel to (010). Taking these weak contacts into account, the coordination number of the mercury ion is '2 + 4'. The stacking direction of the layers is [010]. The layers are connected by O—H···O hydrogen bonds (Steiner, 2002), with a 2.796 (6) Å (O)H···O distance and a 164 (3)° O—H···O angle. The $[ClO_3]^-$ ion exhibits the typical pyramidal shape

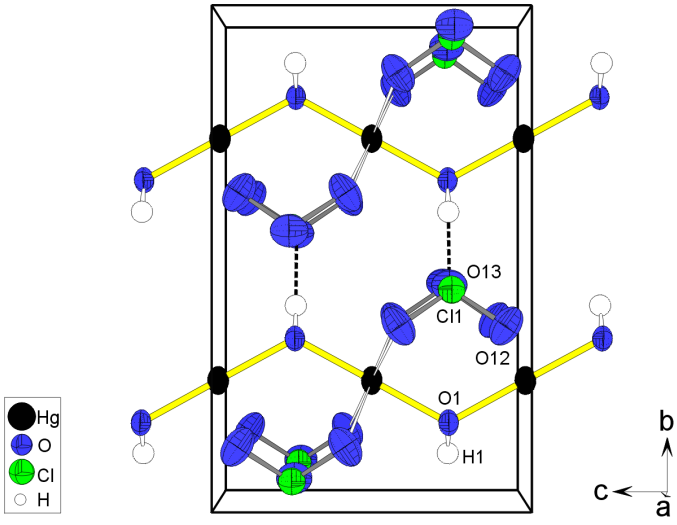

Figure 1
Projection of $Hg(OH)ClO_3$ along the *a* axis. The dashed lines show the weak O—H···O hydrogen bonds between the layers. Displacement ellipsoids are drawn at the 90% probability level.

due to the lone electron pair on the Cl atom. The distances [1.486 (4) and 1.499 (5) Å] and angles [104.8 (2) and 107.9 (3)°] within the $[ClO_3]^-$ ion are similar to those in oxochlorates(V) $M[ClO_3]_2{\cdot}H_2O$, with M = Ba or Pb (Lutz *et al.*, 1985).

The site symmetries are Hg 2, and O1, H, Cl1 and O11 *m*.

Experimental

Red HgO was dissolved with heating in 10 ml of 10% $HClO_3$ until a saturated solution was obtained. Colourless rod-shaped crystals were obtained upon cooling and filtration. These crystals were handled under air and an appropriate specimen was sealed in a glass capillary for the X-ray investigation.

Crystal data

$Hg(OH)ClO_3$
M_r = 301.05
Orthorhombic, *Pbcm*
a = 4.6375 (6) Å
b = 11.4064 (19) Å
c = 7.1965 (11) Å
V = 380.67 (10) Å^3
Z = 4
D_x = 5.253 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2000 reflections
θ = 1.8–29.6°
μ = 40.99 mm^{-1}
T = 170 (2) K
Rod, colourless
0.13 × 0.07 × 0.02 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1999)
T_{min} = 0.012, T_{max} = 0.116
9295 measured reflections
592 independent reflections
457 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 30.0°
$h = -6 \rightarrow 6$
$k = -16 \rightarrow 16$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.043
S = 0.97
592 reflections
37 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 2.03 e Å^{-3}
$\Delta\rho_{min}$ = −1.21 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0265 (9)

Table 1
Selected geometric parameters (Å, °).

Hg1—O1^{i}	2.051 (2)	Cl1—O12^{iii}	1.486 (4)
O1—H1	0.92 (8)	Cl1—O11	1.499 (5)
Cl1—O12^{ii}	1.486 (4)		
O1^{i}—Hg1—O1	177.9 (2)	O12^{ii}—Cl1—O12^{iii}	107.9 (3)
Hg1^{iv}—O1—Hg1	122.6 (2)	O12^{ii}—Cl1—O11	104.81 (18)
Hg1^{iv}—O1—H1	112.8 (15)	O12^{iii}—Cl1—O11	104.81 (18)

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

The maximum and minimum electron-density residuals are located 0.93 and 0.80 Å, respectively, from Hg1.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL*97.

References

Björnlund, G. (1971). *Acta Chem. Scand.* **25**, 1645–1654.
Brandenburg, K. (2001). *DIAMOND*. Version 2.1e. Crystal Impact GbR, Bonn, Germany.
Grdenic, D. & Sikirica, M. (1973). *Inorg. Chem.* **12**, 544–546.
Lutz, H. D., Alici, E. & Buchmeier, W. (1985). *Z. Anorg. Allg. Chem.* **535**, 31–38.
Matkovic, B., Ribar, B., Prelesnik, B. & Herak, R. (1974). *Inorg. Chem.* **13**, 3006–3008.
Meyer, G. & Göbbels, D. (2003). *Z. Anorg. Allg. Chem.* **629**, 933–935.
Nozik, Yu. Z., Fykin, L. E., Bukin, V. I. & Laptasch, N. M. (1979). *Koord. Khim.* **5**, 276–278.
Ribar, B., Matkovic, B., Sljukic, M. & Gabela, F. (1971). *Z. Kristallogr.* **134**, 311–318.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Stalhandske, C. (1979). *Acta Cryst.* B**35**, 949–951.
Steiner, T. (2002). *Angew. Chem.* **114**, 50–80.
Stoe & Cie (1999). *X-SHAPE*. Version 1.06. Stoe and Cie, Darmstadt, Germany.
Stoe & Cie (2001). *X-AREA* (Version 1.15) and *X-RED*32 (Version 1.22). Stoe & Cie, Darmstadt, Germany.
Weiss, A., Lyng, S. & Weiss, A. (1960). *Z. Naturforsch. Teil B*, **15**, 678.
Wickleder, M. S. (2002). *Z. Anorg. Allg. Chem.* **628**, 1459–1460.