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Supramolecular adduct of tetrachlorogallate with cucurbituril: $(H_7O_3)_4[GaCl_4]_2Cl_2\cdot C_{36}H_{36}N_{24}O_{12}\cdot 2H_2O$

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
H-atom completeness 98%
Disorder in main residue
R factor = 0.051
wR factor = 0.183
Data-to-parameter ratio = 14.4

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

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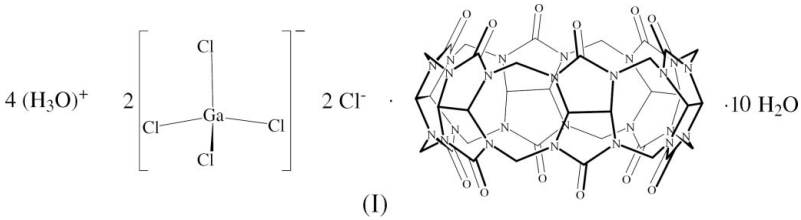
During the investigation of the Ga^{3+}–HCl–cucurbituril system, we prepared crystals of the title compound, tetrakis(hydroxonium) bis(tetrachlorogallate) dichloride cucurbituril decahydrate, by slow evaporation of an aqueous hydrochloric solution containing Ga^{3+} and cucurbituril at room temperature in air. The compound appeared to be isostructural with the cucurbituril tetrachloroferrate(III) adduct. The structure contains $H_7O_3^+$ cations, $[GaCl_4]^-$ anions, cucurbituril and water molecules. The centres of the cucurbituril molecules are arranged as body-centered packing and $[GaCl_4]^-$ anions are situated in space between them. There is a complicated network of hydrogen bonds in the structure.

Comment

Recently, during the investigation of the Fe^{3+}–HCl–cucurbituril system, we obtained a supramolecular adduct containing the tetrachloroferrate(III) anion and cucurbituril (Virovets *et al.*, 2001). Using Ga^{3+} salts, under the same conditions, we have crystallized the isostructural title compound, (I).



(I)

The structure of the title compound contains hydroxonium cations, $[GaCl_4]^-$ anions, chloride anions, cucurbituril and water molecules. Cucurbituril centres are arranged as body-centered packing and $[GaCl_4]^-$ anions are situated in space between them. One water molecule is situated near to the centre inside the cucurbituril molecule and is disordered over four positions (O1*C* and O2*C*).

The $[GaCl_4]^-$ anion lies on the mirror plane and is disordered over two orientations around the Ga1—Cl1 axis. The central Ga atoms have tetrahedral environments and the Ga—Cl distances correspond to those known from other reports (Buscher *et al.*, 1984; Gearhart *et al.*, 1975).

There are two crystallographically independent hydroxonium cations in the structure (O1*W* and O3*W*). They lie on the mirror planes, and each cation is joined with two water molecules by short hydrogen bonds (see Table 2). The O···O···O angles are 109.6 (2) and 110.9 (1)°. This corresponds to formation of $H_7O_3^+$ cations in the crystal (Wells, 1986).

There is a complicated hydrogen-bound network joining O atoms of the cucurbituril carbonyl groups, water molecules, complex anions and chloride in the structure.

Experimental

The title compound was prepared by crystallization from an aqueous hydrochloric solution containing Ga^{3+} and cucurbituril. A mixture of $Ga(NO_3)_3.8H_2O$ (0.07 g, 0.18 mmol) and cucurbituril (0.03 g, 0.03 mmol) was dissolved in 2 *M* HCl (5 ml) and was heated for 5 min. The mixture was filtered and was kept open for 1–2 weeks in a vial until some crystals appeared. Then the vial was closed and the large colourless parallelepipedal crystals were isolated by filtration after 2 weeks. Yield 96% (0.05 g).

Crystal data

$(H_7O_3)_4[GaCl_4]_2Cl_2\cdot C_{36}H_{36}N_{24}O_{12}\cdot 2H_2O$
$M_r = 1747.08$
Orthorhombic, *Cmca*
$a = 16.5665\ (10)$ Å
$b = 16.7800\ (9)$ Å
$c = 25.0342\ (10)$ Å
$V = 6959.2\ (6)$ Å^3
$Z = 4$
$D_x = 1.668$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 16$–22°
$\mu = 1.25$ mm^{-1}
$T = 293\ (2)$ K
Parallelepiped, colourless
$0.60 \times 0.35 \times 0.20$ mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: ψ scan (Sheldrick, 1990)
$T_{min} = 0.531$, $T_{max} = 0.778$
4092 measured reflections
4092 independent reflections
3008 reflections with $I > 2\sigma(I)$
$\theta_{max} = 27.5^\circ$
$h = -21 \rightarrow 0$
$k = 0 \rightarrow 21$
$l = -32 \rightarrow 0$
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.183$
$S = 1.04$
4092 reflections
285 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1074P)^2 + 10.4226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.84$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ga1—Cl1	2.1549 (14)	Ga1—Cl22	2.236 (11)
Ga1—Cl21	2.093 (9)	Ga1—Cl31	2.161 (3)
Cl1—Ga1—Cl22	107.4 (3)	Cl21—Ga1—Cl31	111.7 (5)
Cl1—Ga1—Cl31	108.34 (10)	Cl31—Ga1—Cl22^{i}	106.9 (5)
Cl21—Ga1—Cl1	112.8 (4)	Cl31—Ga1—Cl22	120.9 (5)
Cl21^{i}—Ga1—Cl22	109.60 (12)	Cl31^{i}—Cl31—Ga1	81.9 (4)
Cl21—Ga1—Cl31^{i}	97.7 (5)		

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*1···O2*W*	0.98 (2)	1.52 (4)	2.476 (5)	165 (3)
O3*W*—H1*W*3···O4*W*	1.00 (2)	1.50 (3)	2.492 (6)	172 (4)

The H atoms of the disordered water molecule were not considered.

Data collection: *XSCANS* (Bruker, 1998); cell refinement: *XSCANS*; data reduction: *SHELXTL-Plus* (Sheldrick, 1990); program(s) used to solve structure: *SHELXTL-Plus*; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1998); molecular graphics: local program; software used to prepare material for publication: local program.

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