Poly[μ-aqua-μ5-[2-(2,3,6-trichlorophenyl)acetato]-caesium]

Graham Smith

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Key indicators: single-crystal X-ray study; T = 200 K; mean ϵ(C–C) = 0.009 Å; R factor = 0.050; wR factor = 0.111; data-to-parameter ratio = 16.8.

In the structure of the title complex, [Cs(C₈H₄Cl₃O₂)(H₂O)]ₙ, the caesium salt of the commercial herbicide fenac [(2,3,6-trichlorophenyl)acetato]-caesium, the irregular eight-coordination about Cs⁺ comprises a bidentate O:Cl-chelate interaction involving a carboxylate-O atom and an ortho-related ring-substituted Cl atom, which is also bridging, a triple-bridging carboxylate-O atom and a bridging water molecule. A two-dimensional polymer is generated, lying parallel to (100), within which there are water–carboxylate O–H⋯O hydrogen-bonding interactions.

Related literature

For background information on the herbicide fenac, see: O’Neil (2001). For the structure of fenac, see: White et al. (1979). For examples of caesium complexes involving coordinating carbon-bound Cl, see: Levitskaia et al. (2000); Smith (2013).

Table 1
Selected bond lengths (Å).

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

Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SIR92 (Altomare et al., 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 2012); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2781).

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Poly[μ-aqua-μ$_5$-2-(2,3,6-trichlorophenyl)acetato]-caesium]

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1. Comment
(2,3,6-Trichlorophenyl)acetic acid (fenac) is a commercial herbicide (O’Neil, 2001) and its crystal structure (White et al., 1979) represents the only entry for this compound in the crystallographic literature. My interest in aromatic carboxylic acid herbicides and in polymeric coordination structures of the alkali metal complexes led to the preparation of the title compound, [Cs(C$_8$H$_4$Cl$_3$O$_2$)(H$_2$O)]$_n$, from the reaction of fenac with caesium hydroxide in aqueous ethanol, and the structure is reported herein.

In this structure (Fig. 1), the irregular eight-coordinate CsClO$_7$ polyhedron comprises a bidentate O:Cl-chelate interaction involving a carboxylate O-atom (O13) and an ortho-related ring substituted Cl-atom (Cl6) which is also bridging, a triple-bridging carboxylate O-atom (O12) and a bridging water molecule O1W (Table 1). A partial expansion of the asymmetric unit in the polymer structure is shown in Fig. 2, forming 4-, 7- and 8-membered cyclic associations linking Cs$^+$ ions (a triple bridge involving Cl6, O1W and O12$^\text{ii}$, extending down b). The minimum Cs···Cs$^\text{vi}$ bridging distance in the structure is 4.4336 (9) Å [for symmetry code (i), see Table 1. For code (vi): -x + 2, y + 1/2, -z + 3/2]. In the Cl bridge, the Cs—Cl bond lengths [3.646 (2) and 3.711 (2) Å] are long compared to those commonly present in the few known examples of caesium complexes having coordinating carbon-bound Cl atoms, e.g. 3.46–3.56 Å for a complex in which 1,2-dichloroethane acts as a bidentate chelate ligand (Levitskaia et al., 2000). However, I have previously reported values similar to those in the title complex in the analogous polymeric structure of caesium 4-amino-3,5,6-trichloropyridine-2-carboxylate monohydrate [3.6052 (11)– 3.7151 (11) Å], in which all three ring-substituted Cl-atoms are coordinated (Smith, 2013).

In the crystal structure of the title complex, a polymer with a sheet structure is generated which lies parallel to (100) (Fig. 3), and within which there are waterO—H···Ocarboxylate hydrogen-bonding interactions (Table 2).

2. Experimental
The title compound was synthesized by heating together under reflux for 10 minutes, 0.5 mmol of (2,3,6-trichlorophenyl)acetic acid and 0.5 mmol of CsOH in 15 ml of 10% ethanol–water. Partial room temperature evaporation of the solution gave thin colourless crystal plates of the title complex from which a specimen was cleaved for the X-ray analysis.

3. Refinement
Carbon-bound hydrogen atoms were placed in calculated positions [aromatic C—H = 0.93 Å and methylene C—H = 0.97 Å] and allowed to ride in the refinement, with $U_{	ext{w}}$(H) = 1.2$U_{	ext{eq}}$(C). Hydrogen atoms of the coordinating water molecule were located in a difference-Fourier synthesis but were subsequently allowed to ride, with $U_{	ext{w}}$(H) = 1.5$U_{	ext{eq}}$(O). A large maximum residual electron density peak was present (2.176 e$^-$/Å$^3$) located at 0.82 Å from Cs1. A short O1W···O1W$^\text{ii}$ non-bonding contact [2.804 (8) Å] across an inversion centre was also found.
Computing details

Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO (Agilent, 2012); data reduction: CrysAlis PRO (Agilent, 2012); program(s) used to solve structure: SIR92 (Altomare et al., 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 2012); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON (Spek, 2009).

Figure 1

The molecular configuration and atom-numbering scheme for the title compound, with non-H atoms drawn as 40% probability displacement ellipsoids. [For symmetry codes, see Table 1.]
Figure 2
A partial expansion of the Cs⁺ coordination in the polymer generated by cyclic links through carboxylate, chlorine and water bridges. Ligand H-atoms are omitted. [For symmetry code (vi): -x + 2, y + 1/2, -z + 3/2. For other codes, see Fig. 1 and Table 1.]
Figure 3
The packing of the sheet structure in the unit cell viewed down $b$.

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Crystal data

$[\text{Cs(C}_8\text{H}_4\text{Cl}_3\text{O}_2)(\text{H}_2\text{O})]$  
$M_r = 389.39$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 17.0606$ (12) Å

$b = 4.9834$ (3) Å

$c = 13.9283$ (10) Å

$β = 98.127$ (6)°

$V = 1172.29$ (14) Å$^3$

$Z = 4$

$F(000) = 736$

$D_x = 2.206$ Mg m$^{-3}$

Mo $Kα$ radiation, $λ = 0.71073$ Å

Cell parameters from 2248 reflections

$θ = 3.3$–28.0°

$μ = 3.82$ mm$^{-1}$

$T = 200$ K

Plate, colourless

$0.20 × 0.15 × 0.07$ mm

Data collection

Oxford Diffraction Gemini-S CCD-detector
diffractometer

Detector resolution: 16.077 pixels mm$^{-1}$

ω scans

Radiation source: Enhance (Mo) X-ray source

Absorption correction: multi-scan

Graphite monochromator

(CrysAlis PRO; Agilent, 2012)
supplementary materials

$T_{\text{min}} = 0.582, T_{\text{max}} = 0.980$

7585 measured reflections
2284 independent reflections
1873 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\text{max}} = 26.0^\circ, \theta_{\text{min}} = 3.4^\circ$

$h = -20\rightarrow 21$

$k = -6 \rightarrow 6$

$l = -17 \rightarrow 12$

Refinement

Refinement on $F^2$
Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.111$

$S = 1.09$

2284 reflections
136 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 9.056P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta \sigma)_{\text{max}} = 0.001$

$\Delta \rho_{\text{max}} = 2.18 \text{ e Å}^{-3}$

$\Delta \rho_{\text{min}} = -1.86 \text{ e Å}^{-3}$

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2\sigma(F^2)$ 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 ($\text{Å}^2$)

| x   | y   | z   | U_{eq}^* | U_{eq} |
|-----|-----|-----|----------|--------|
| Cs1 | 0.91683 (3) | 1.08611 (9) | 0.65098 (4) | 0.0524 (2) |
| Cl2 | 0.66412 (12) | 1.1809 (4) | 0.23490 (12) | 0.0501 (6) |
| Cl3 | 0.53476 (12) | 1.4225 (4) | 0.34892 (17) | 0.0616 (8) |
| Cl6 | 0.76993 (11) | 0.5765 (4) | 0.54801 (14) | 0.0508 (6) |
| O1W | 1.0140 (3) | 0.5882 (12) | 0.5977 (4) | 0.065 (2) |
| O12 | 0.8947 (3) | 0.8961 (12) | 0.2855 (4) | 0.0529 (19) |
| O13 | 0.8658 (3) | 1.0892 (13) | 0.4175 (5) | 0.072 (2) |
| C1  | 0.7124 (3) | 0.8850 (12) | 0.3931 (4) | 0.0274 (17) |
| C2  | 0.6586 (4) | 1.0773 (13) | 0.3521 (4) | 0.0326 (19) |
| C3  | 0.6013 (4) | 1.1852 (14) | 0.4022 (5) | 0.0367 (19) |
| C4  | 0.5961 (4) | 1.1051 (15) | 0.4948 (5) | 0.040 (2) |
| C5  | 0.6479 (4) | 0.9137 (15) | 0.5385 (5) | 0.039 (2) |
| C6  | 0.7052 (4) | 0.8101 (13) | 0.4877 (5) | 0.0322 (19) |
| C11 | 0.7748 (4) | 0.7685 (14) | 0.3401 (5) | 0.036 (2) |
| C12 | 0.8505 (4) | 0.9352 (12) | 0.3479 (4) | 0.0307 (19) |
| H4  | 0.55790 | 1.17900 | 0.52840 | 0.0480* |
| H5  | 0.64430 | 0.85520 | 0.60120 | 0.0470* |
| H11A | 0.75320 | 0.75000 | 0.27210 | 0.0430* |
| H11B | 0.78800 | 0.59030 | 0.36530 | 0.0430* |
| H11W | 1.06400 | 0.68180 | 0.60200 | 0.0970* |
| H12W | 1.02500 | 0.45100 | 0.63200 | 0.0970* |
supplementary materials

**Atomic displacement parameters (Å²)**

|     | \(U_{11}^{\text{ij}}\) | \(U_{22}^{\text{ij}}\) | \(U_{33}^{\text{ij}}\) | \(U_{12}^{\text{ij}}\) | \(U_{13}^{\text{ij}}\) | \(U_{23}^{\text{ij}}\) |
|-----|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
| Cs1 | 0.0581 (3)             | 0.0302 (3)             | 0.0667 (4)             | −0.0028 (2)            | 0.0012 (2)             | 0.0010 (2)             |
| Cl2 | 0.0655 (12)            | 0.0500 (11)            | 0.0322 (9)             | −0.0108 (9)            | −0.0020 (8)            | 0.0071 (8)             |
| Cl3 | 0.0487 (12)            | 0.0503 (12)            | 0.0787 (15)            | 0.0179 (9)             | −0.0157 (10)           | −0.0073 (11)           |
| Cl6 | 0.0477 (11)            | 0.0492 (11)            | 0.0530 (11)            | 0.0041 (9)             | −0.0016 (8)            | 0.0152 (9)             |
| O1W | 0.067 (4)              | 0.073 (4)              | 0.061 (3)              | −0.041 (3)             | 0.031 (3)              | −0.027 (3)             |
| O12 | 0.039 (3)              | 0.075 (4)              | 0.049 (3)              | −0.016 (3)             | 0.021 (2)              | −0.026 (3)             |
| O13 | 0.061 (4)              | 0.081 (4)              | 0.083 (4)              | −0.042 (3)             | 0.041 (3)              | −0.050 (4)             |
| C1  | 0.025 (3)              | 0.026 (3)              | 0.031 (3)              | −0.006 (3)             | 0.003 (2)              | −0.004 (3)             |
| C2  | 0.035 (4)              | 0.032 (3)              | 0.029 (3)              | −0.011 (3)             | −0.002 (3)             | −0.004 (3)             |
| C3  | 0.022 (3)              | 0.034 (3)              | 0.051 (4)              | 0.003 (3)              | −0.006 (3)             | −0.011 (3)             |
| C4  | 0.032 (4)              | 0.051 (4)              | 0.039 (4)              | −0.001 (3)             | 0.011 (3)              | −0.017 (3)             |
| C5  | 0.042 (4)              | 0.047 (4)              | 0.030 (3)              | −0.009 (3)             | 0.013 (3)              | −0.005 (3)             |
| C6  | 0.025 (3)              | 0.030 (3)              | 0.039 (4)              | −0.002 (3)             | −0.004 (3)             | −0.003 (3)             |
| C11 | 0.035 (4)              | 0.035 (4)              | 0.038 (4)              | −0.003 (3)             | 0.010 (3)              | −0.010 (3)             |
| C12 | 0.038 (4)              | 0.026 (3)              | 0.029 (3)              | 0.001 (3)              | 0.008 (3)              | −0.005 (3)             |

**Geometric parameters (Å, °)**

|     | \(d_{\text{ij}}\) | \(d_{\text{ij}}\) | \(d_{\text{ij}}\) | \(d_{\text{ij}}\) | \(d_{\text{ij}}\) | \(d_{\text{ij}}\) |
|-----|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| Cs1—Cl6 | 3.711 (2) | O1W—H12W | 0.8400 |
| Cs1—O1W | 3.131 (6) | C1—C2 | 1.392 (9) |
| Cs1—O13 | 3.246 (7) | C1—C11 | 1.496 (9) |
| Cs1—Cl6i | 3.646 (2) | C1—C6 | 1.392 (9) |
| Cs1—O1Wi | 3.148 (6) | C2—C3 | 1.387 (9) |
| Cs1—O12ii | 3.213 (5) | C3—C4 | 1.365 (10) |
| Cs1—O12ii | 3.103 (6) | C4—C5 | 1.382 (10) |
| Cs1—O12vi | 3.242 (6) | C5—C6 | 1.385 (10) |
| Cl2—C2 | 1.727 (6) | C11—C12 | 1.527 (10) |
| Cl3—C3 | 1.732 (7) | C4—H4 | 0.9300 |
| Cl6—C6 | 1.737 (7) | C5—H5 | 0.9300 |
| O12—C12 | 1.244 (8) | C11—H11A | 0.9700 |
| O13—C12 | 1.235 (9) | C11—H11B | 0.9700 |
| O1W—H11W | 0.9700 | |
| Cl6—Cs1—O1W | 73.58 (10) | Cs1—a—O12—Cs1vi | 89.15 (14) |
| Cl6—Cs1—O13 | 62.95 (11) | Cs1—a—O12—Cs1vi | 86.76 (13) |
| Cl6—Cs1—Cl6i | 85.27 (4) | Cs1—O12—Cs1vi | 103.50 (16) |
| Cl6—Cs1—O1Wi | 143.35 (11) | Cs1—O13—C12 | 141.3 (5) |
| Cl6—Cs1—O12ii | 136.07 (11) | Cs1—O1W—H12W | 126.00 |
| Cl6—Cs1—O12ii | 64.54 (11) | H11W—O1W—H12W | 103.00 |
| Cl6—Cs1—O12vi | 129.83 (10) | Cs1—O1W—H11W | 95.00 |
| O1W—Cs1—O13 | 80.93 (15) | Cs1—a—O1W—H11W | 149.00 |
| Cl6—Cs1—O1W | 142.70 (11) | C2—C1—C11 | 122.6 (5) |
| O1W—Cs1—O1Wi | 150.07 (14) | C6—C1—C11 | 121.8 (5) |
| O1W—Cs1—O12ii | 62.90 (14) | C2—C1—C6 | 115.6 (5) |
| O1W—Cs1—O12vi | 69.09 (14) | C12—C2—C1 | 118.2 (5) |
| O1W—Cs1—O12vi | 151.22 (14) | C12—C2—C3 | 119.7 (5) |
| Cl6—Cs1—O13 | 62.00 (11) | C1—C2—C3 | 122.1 (5) |

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sup-6
| Bond/Angle | Dist./Angle | Bond/Angle | Dist./Angle |
|------------|-------------|------------|-------------|
| O1Wi—Cs1—O13 | 80.54 (15) | C2—C3—C4 | 120.4 (6) |
| O12ii—Cs1—O13 | 113.08 (14) | Cl3—C3—C4 | 118.6 (5) |
| O12iii—Cs1—O13 | 124.78 (15) | Cl3—C3—C2 | 121.0 (5) |
| O12iv—Cs1—O13 | 122.59 (15) | C3—C4—C5 | 119.7 (6) |
| Cl6—Cs1—O1Wi | 74.34 (10) | C4—C5—C6 | 119.1 (6) |
| Cl6—Cs1—O12ii | 134.05 (11) | Cl6—C6—C5 | 116.7 (5) |
| Cl6—Cs1—O12iii | 128.39 (10) | C1—C6—C5 | 123.2 (6) |
| Cl6—Cs1—O12iv | 64.16 (10) | Cl6—C6—C1 | 120.2 (5) |
| O1Wi—Cs1—O12ii | 60.21 (14) | C1—C11—C12 | 114.1 (5) |
| O1Wi—Cs1—O12iii | 150.59 (14) | O12—C12—C11 | 117.1 (6) |
| O1Wi—Cs1—O12iv | 67.16 (14) | O13—C12—C11 | 118.5 (6) |
| O12ii—Cs1—O12iii | 93.30 (14) | O12—C12—O13 | 124.3 (7) |
| O12ii—Cs1—O12iv | 90.73 (14) | C3—C4—H4 | 120.00 |
| O12iii—Cs1—O12iv | 103.50 (15) | C5—C4—H4 | 120.00 |
| Cs1—Cl6—C6 | 94.4 (2) | C4—C5—H5 | 120.00 |
| Cs1—Cl6—Cs1v | 85.27 (4) | C6—C5—H5 | 120.00 |
| Cs1v—Cl6—C6 | 173.7 (2) | C1—C11—H11A | 109.00 |
| Cs1—O1W—Cs1v | 105.07 (15) | C1—C11—H11B | 109.00 |
| Cs1ii—O12—C12 | 119.0 (4) | C12—C11—H11A | 109.00 |
| Cs1vi—O12—C12 | 132.9 (4) | C12—C11—H11B | 109.00 |
| Cs1vii—O12—C12 | 114.3 (4) | H11A—C11—H11B | 108.00 |

Supplementary materials

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O12ii—Cs1—O13—C12 −161.3 (7) C11—C1—C2—C3 −179.6 (6)
Cl6—Cs1—Cl6i—Cs1i 180.00 (4) C11—C1—C6—Cl6 0.3 (9)
O1W—Cs1—Cl6i—Cs1i −125.21 (17) C11—C1—C6—C5 −179.8 (6)
O13—Cs1—Cl6i—Cs1i −118.22 (12) C2—C1—C11—C12 85.3 (7)
Cl6—Cs1—O1Wii—Cs1i 97.39 (19) C6—C1—C11—C12 −93.7 (7)
O1W—Cs1—O1Wii—Cs1i 179.98 (16) C12—C2—C3—Cl3 0.2 (8)
O13—Cs1—O1Wii—Cs1i 102.15 (17) C12—C2—C3—C4 179.9 (6)
Cl6—Cs1—O12iii—Cs1vii −157.48 (7) C1—C2—C3—Cl3 −179.6 (5)
Cl6—Cs1—O12iii—Cs1v −62.1 (5) C1—C2—C3—C4 0.2 (10)
O1W—Cs1—O12iii—Cs1v −166.04 (19) C2—C3—C4—C5 −0.5 (11)
O1W—Cs1—O12iii—Cs1vii 53.6 (5) C2—C3—C4—C5 179.2 (6)
O13—Cs1—O12iii—Cs1vii 128.20 (15) C3—C4—C5—C6 1.1 (11)
O13—Cs1—O12iii—Cs1v −12.2 (5) C4—C5—C6—Cl6 178.4 (6)
Cl6—Cs1—O12iii—Cs1v 52.02 (11) C4—C5—C6—C1 −1.5 (11)
Cl6—Cs1—O12iii—Cs1vii −91.1 (6) C1—C11—C12—O12 −160.0 (6)
O1W—Cs1—O12iii—Cs1vii −29.16 (14) C1—C11—C12—O12 23.4 (9)

Symmetry codes: (i) x, y+1, z; (ii) −x+2, −y+2, −z+1; (iii) x, −y+3/2, z+1/2; (iv) x, −y+5/2, z+1/2; (v) x, y, −z; (vi) x, −y+3/2, z−1/2; (vii) x, −y+5/2, z−1/2; (viii) −x+2, y+1/2, −z+3/2.

Hydrogen-bond geometry (Å, °)

\begin{tabular}{cccccc}
D—H···A & D—H & H···A & D···A & D—H···A \\
\hline
O1W—H11W···O13ii & 0.97 & 1.70 & 2.638 (8) & 161 \\
O1W—H12W···O13ix & 0.84 & 2.40 & 3.191 (8) & 158 \\
C11—H11A···Cl2 & 0.97 & 2.64 & 3.026 (7) & 104 \\
C11—H11B···Cl6 & 0.97 & 2.61 & 3.062 (7) & 109 \\
\end{tabular}

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