

Hexaaquacobalt(II) 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate monohydrate

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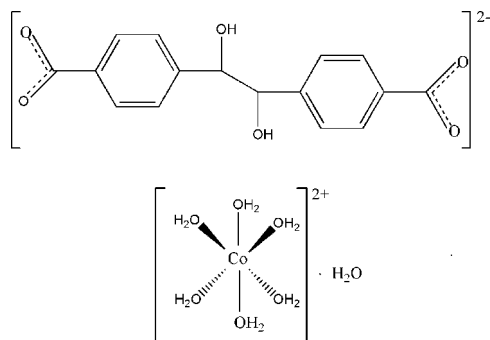
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.012$ Å; R factor = 0.075; wR factor = 0.177; data-to-parameter ratio = 13.0.

The title compound, $[\text{Co}(\text{H}_2\text{O})_6](\text{C}_{16}\text{H}_{12}\text{O}_6) \cdot \text{H}_2\text{O}$, is composed of one 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate anion lying on an inversion center, one $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ dicationic complex and a solvent water molecule located on mirror planes. In the crystal, a chain is constructed via $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds involving the carboxylate and hydroxyl groups of the organic anion; the chains are further connected into a three-dimensional framework by additional $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds between the $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ cations, solvent water molecules and the anions.

Related literature

For background to metal-organic structures and their potential applications as functional materials, see: Liang *et al.* (2000); Kondo *et al.* (2004); Lin *et al.* (2004); Fan & Hanson (2005); Laborda *et al.* (2004); Fei *et al.* (2005); Zhang *et al.* (2006).



Experimental

Crystal data

$[\text{Co}(\text{H}_2\text{O})_6](\text{C}_{16}\text{H}_{12}\text{O}_6) \cdot \text{H}_2\text{O}$
 $M_r = 485.30$
 Monoclinic, $P2_1/m$

$a = 6.0430$ (6) Å
 $b = 20.487$ (2) Å
 $c = 8.6341$ (9) Å

$\beta = 104.115$ (1)°
 $V = 1036.66$ (18) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.89$ mm⁻¹
 $T = 298$ K
 $0.38 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2007)
 $T_{\min} = 0.728$, $T_{\max} = 0.856$

5184 measured reflections
 1867 independent reflections
 1675 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.075$
 $wR(F^2) = 0.177$
 $S = 1.25$
 1867 reflections
 144 parameters

11 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.58$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

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

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|---|-------|--------------|--------------|----------------|
| $\text{O3}-\text{H3} \cdots \text{O1}^{\text{i}}$ | 0.82 | 2.00 | 2.811 (8) | 168 |
| $\text{O1W}-\text{H1W} \cdots \text{O1}^{\text{ii}}$ | 0.85 | 1.96 | 2.814 (9) | 180 |
| $\text{O1W}-\text{H2W} \cdots \text{O2}^{\text{iii}}$ | 0.85 | 1.81 | 2.665 (8) | 179 |
| $\text{O2W}-\text{H3W} \cdots \text{O3}^{\text{iv}}$ | 0.85 | 2.00 | 2.847 (8) | 180 |
| $\text{O2W}-\text{H4W} \cdots \text{O5W}^{\text{v}}$ | 0.85 | 2.19 | 3.035 (11) | 179 |
| $\text{O3W}-\text{H5W} \cdots \text{O4W}^{\text{vi}}$ | 0.85 | 1.93 | 2.778 (11) | 172 |
| $\text{O3W}-\text{H6W} \cdots \text{O5W}$ | 0.85 | 1.91 | 2.756 (13) | 171 |
| $\text{O5W}-\text{H9W} \cdots \text{O2}$ | 0.85 | 1.93 | 2.767 (10) | 169 |
| $\text{O4W}-\text{H7W} \cdots \text{O1}^{\text{vii}}$ | 0.84 | 1.88 | 2.695 (7) | 163 |
| $\text{O4W}-\text{H7W} \cdots \text{O2}^{\text{vii}}$ | 0.84 | 2.70 | 3.296 (9) | 130 |

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

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2059).

References

- Bruker (2007). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Fan, J. & Hanson, B. E. (2005). *Inorg. Chem.* **44**, 6998–7008.
 Fei, B. L., Clerac, R., Anson, C. E. & Powell, A. K. (2005). *Dalton Trans.* pp. 1381–1386.
 Kondo, M., Irie, Y., Shimizu, Y., Miyazawa, M., Kawaguchi, H., Nakamura, A., Naito, T., Maeda, K. & Uchida, F. (2004). *Inorg. Chem.* **43**, 6139–6141.
 Laborda, S., Clerac, R., Anson, C. E. & Powell, A. K. (2004). *Inorg. Chem.* **43**, 5931–5943.
 Liang, Y. C., Cao, R., Su, W. P., Hong, M. C. & Zhang, W. J. (2000). *Angew. Chem. Int. Ed.* **39**, 3304–3307.
 Lin, P., Henderson, R. A., Harrington, R. W., Clegg, W., Wu, C. D. & Wu, X. T. (2004). *Inorg. Chem.* **43**, 181–188.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Zhang, Z. J., Xiang, S. C., Zhang, Y. F., Wu, A. Q., Cai, L. Z., Guo, G. C. & Huang, J. S. (2006). *Inorg. Chem.* **45**, 1972–1977.

supplementary materials

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Hexaaquacobalt(II) 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate monohydrate

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Comment

Metal-organic coordination polymers have been greatly developed in recent years due to their captivating structure (Kondo *et al.*, 2004; Fan *et al.*, 2005) and potential applications as functional materials in electronic (Lin *et al.*, 2004), magnetic (Laborda *et al.*, 2004; Liang *et al.*, 2000; Fei *et al.*, 2005) and optical (Zhang *et al.*, 2006) fields. Thus, we choose the ligand 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate and $\text{Co}(\text{NO}_3)_2$ under hydrothermal conditions to obtain new metal-organic complex. We report here the synthesis and structure of the title compound.

As shown in Figure 1, the title compound $[\text{Co}(\text{H}_2\text{O})_6][\text{C}_{16}\text{H}_{12}\text{O}_6]\cdot\text{H}_2\text{O}$ is composed of one 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate anion lying on inversion center, one $[\text{Co}_6\text{H}_{12}\text{O}_6]^{2+}$ dicationic complex and a solvent water molecule locating on mirror planes. The Co^{II} ion is coordinated by six water molecules in an octahedral geometry. The hydroxyl groups of the 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate anion are oriented such the the H atoms are directed away from the plane of the benzene ring. In the crystal, a one-dimensional chain is constructed via $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds interactions involving the carboxylate and hydroxyl groups of the organic anion, which was further connected into a three-dimensional framework by additional $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds formed by $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$ cations, solvent water molecules and the anions.

Experimental

A mixture of 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate (0.5 mol, 0.15 g) and $\text{Co}(\text{NO}_3)_2$ (0.5 mol, 0.14 g) in 30 ml of absolute ethanol was heated under reflux for 6 h in the presence of 1-2 drops of NaOH. The reaction mixture was cooled to room temperature for 2 h. The light-red crystal was filtered off and washed several times using absolute ethanol.

Refinement

H atoms bound to C atoms were placed at calculated positions and were treated as riding on the parent atoms, with $\text{C}-\text{H} = 0.93 \text{ \AA}$ (aromatic) and 0.98 \AA (CH) and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ H atoms of hydroxyl group and water molecules were located in a difference Fourier map and refined as riding, with $\text{O}-\text{H} = 0.85 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ for water O atoms and $1.2 U_{\text{eq}}(\text{O})$ for hydroxyl O atoms.

Figures

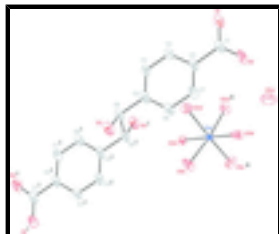


Fig. 1. The title compound, with the atom-numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids (H atoms are omitted for clarity). [Symmetry codes: (i) 2-x, -y, 1-z; (ii) x, 0.5-y, z.]

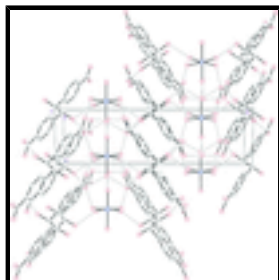


Fig. 2. The packing and hydrogen bonding of the title compound down the a axis (H atoms is not shown in the picture for clarity).

Hexaaquacobalt(II) 4,4'-(1,2-dihydroxyethane-1,2-diyl)dibenzoate monohydrate

Crystal data

[Co(H₂O)₆](C₁₆H₁₂O₆)·H₂O

$M_r = 485.30$

Monoclinic, $P2_1/m$

Hall symbol: -P 2yb

$a = 6.0430$ (6) Å

$b = 20.487$ (2) Å

$c = 8.6341$ (9) Å

$\beta = 104.115$ (1)°

$V = 1036.66$ (18) Å³

$Z = 2$

$F(000) = 506$

$D_x = 1.555$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2215 reflections

$\theta = 2.5$ – 24.0 °

$\mu = 0.89$ mm⁻¹

$T = 298$ K

Block, red

$0.38 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\min} = 0.728$, $T_{\max} = 0.856$

5184 measured reflections

1867 independent reflections

1675 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.0$ °

$h = -6 \rightarrow 7$

$k = -24 \rightarrow 24$

$l = -8 \rightarrow 10$

Refinement

| | |
|--|---|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.075$ | H-atom parameters constrained |
| $wR(F^2) = 0.177$ | $w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 7.8675P]$ |
| $S = 1.25$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1867 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 144 parameters | $\Delta\rho_{\max} = 0.58 \text{ e } \text{\AA}^{-3}$ |
| 11 restraints | $\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: <i>SHELXL</i> , $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| | Extinction coefficient: 0.0093 (18) |

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.

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|--------------|----------------------------------|
| Co1 | 0.8509 (2) | 0.2500 | 0.54019 (15) | 0.0234 (4) |
| O1 | 0.4205 (10) | 0.1418 (3) | -0.2097 (7) | 0.0505 (16) |
| O2 | 0.1731 (10) | 0.1522 (3) | -0.0588 (8) | 0.0616 (19) |
| O3 | 0.8284 (10) | -0.0725 (3) | 0.4766 (7) | 0.0536 (17) |
| H3 | 0.7734 | -0.0959 | 0.3999 | 0.080* |
| O1W | 0.8192 (10) | 0.1750 (3) | 0.6947 (7) | 0.062 (2) |
| H1W | 0.6984 | 0.1651 | 0.7232 | 0.093* |
| H2W | 0.9330 | 0.1678 | 0.7727 | 0.093* |
| O2W | 0.9112 (10) | 0.1813 (3) | 0.3752 (7) | 0.0540 (16) |
| H3W | 0.9892 | 0.1489 | 0.4198 | 0.081* |
| H4W | 0.9848 | 0.2002 | 0.3158 | 0.081* |
| O3W | 0.5046 (14) | 0.2500 | 0.4420 (9) | 0.061 (3) |
| H5W | 0.4269 | 0.2500 | 0.5118 | 0.091* |
| H6W | 0.4125 | 0.2500 | 0.3502 | 0.091* |
| O4W | 1.2136 (12) | 0.2500 | 0.6463 (9) | 0.0351 (17) |
| H7W | 1.2522 | 0.2875 | 0.6825 | 0.053* |

supplementary materials

| | | | | |
|-----|-------------|-------------|--------------|-----------|
| O5W | 0.1686 (19) | 0.2500 | 0.1613 (12) | 0.099 (4) |
| H9W | 0.1690 | 0.2164 | 0.1036 | 0.148* |
| C1 | 0.3585 (14) | 0.1323 (4) | -0.0814 (10) | 0.044 (2) |
| C2 | 0.5138 (13) | 0.0948 (4) | 0.0530 (10) | 0.041 (2) |
| C3 | 0.7192 (14) | 0.0687 (4) | 0.0317 (10) | 0.045 (2) |
| H3A | 0.7632 | 0.0762 | -0.0627 | 0.054* |
| C4 | 0.8566 (15) | 0.0316 (4) | 0.1527 (10) | 0.047 (2) |
| H4 | 0.9921 | 0.0142 | 0.1380 | 0.056* |
| C5 | 0.7956 (14) | 0.0202 (4) | 0.2936 (10) | 0.043 (2) |
| C6 | 0.5928 (15) | 0.0463 (4) | 0.3163 (10) | 0.048 (2) |
| H6 | 0.5508 | 0.0393 | 0.4117 | 0.058* |
| C7 | 0.4528 (14) | 0.0831 (4) | 0.1944 (10) | 0.047 (2) |
| H7A | 0.3163 | 0.0999 | 0.2089 | 0.056* |
| C8 | 0.9495 (15) | -0.0203 (4) | 0.4267 (10) | 0.046 (2) |
| H8A | 1.0745 | -0.0386 | 0.3863 | 0.055* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|------------|------------|------------|
| Co1 | 0.0209 (7) | 0.0289 (7) | 0.0194 (7) | 0.000 | 0.0032 (5) | 0.000 |
| O1 | 0.041 (3) | 0.049 (4) | 0.053 (4) | 0.002 (3) | -0.005 (3) | 0.010 (3) |
| O2 | 0.039 (4) | 0.075 (5) | 0.062 (4) | 0.016 (3) | -0.005 (3) | 0.024 (4) |
| O3 | 0.058 (4) | 0.037 (3) | 0.054 (4) | -0.004 (3) | -0.010 (3) | 0.004 (3) |
| O1W | 0.035 (3) | 0.086 (5) | 0.058 (4) | -0.008 (3) | -0.002 (3) | 0.033 (4) |
| O2W | 0.056 (4) | 0.053 (4) | 0.050 (4) | 0.000 (3) | 0.006 (3) | -0.010 (3) |
| O3W | 0.037 (5) | 0.118 (8) | 0.024 (4) | 0.000 | -0.001 (4) | 0.000 |
| O4W | 0.033 (4) | 0.031 (4) | 0.039 (4) | 0.000 | 0.006 (3) | 0.000 |
| O5W | 0.068 (7) | 0.184 (14) | 0.046 (6) | 0.000 | 0.017 (5) | 0.000 |
| C1 | 0.039 (5) | 0.038 (5) | 0.047 (5) | -0.006 (4) | -0.009 (4) | 0.008 (4) |
| C2 | 0.036 (4) | 0.030 (4) | 0.045 (5) | -0.001 (3) | -0.011 (4) | 0.004 (4) |
| C3 | 0.045 (5) | 0.039 (5) | 0.044 (5) | 0.001 (4) | -0.003 (4) | 0.007 (4) |
| C4 | 0.042 (5) | 0.041 (5) | 0.047 (5) | 0.011 (4) | -0.008 (4) | 0.004 (4) |
| C5 | 0.040 (5) | 0.029 (4) | 0.047 (5) | -0.001 (3) | -0.014 (4) | 0.002 (4) |
| C6 | 0.044 (5) | 0.050 (5) | 0.041 (5) | -0.001 (4) | -0.007 (4) | 0.008 (4) |
| C7 | 0.034 (4) | 0.045 (5) | 0.054 (5) | 0.002 (4) | -0.003 (4) | 0.009 (4) |
| C8 | 0.044 (5) | 0.032 (4) | 0.048 (5) | 0.000 (4) | -0.013 (4) | 0.006 (4) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------------------|------------|---------|------------|
| Co1—O3W | 2.058 (8) | O4W—H7W | 0.8413 |
| Co1—O1W ⁱ | 2.074 (6) | O5W—H9W | 0.8500 |
| Co1—O1W | 2.074 (6) | C1—C2 | 1.511 (10) |
| Co1—O2W | 2.097 (6) | C2—C7 | 1.380 (12) |
| Co1—O2W ⁱ | 2.097 (6) | C2—C3 | 1.404 (12) |
| Co1—O4W | 2.159 (7) | C3—C4 | 1.392 (11) |
| O1—C1 | 1.268 (10) | C3—H3A | 0.9300 |
| O2—C1 | 1.251 (11) | C4—C5 | 1.374 (12) |
| O3—C8 | 1.420 (10) | C4—H4 | 0.9300 |

| | | | |
|--|------------|---------------------------|------------|
| O3—H3 | 0.8200 | C5—C6 | 1.394 (12) |
| O1W—H1W | 0.8500 | C5—C8 | 1.534 (10) |
| O1W—H2W | 0.8500 | C6—C7 | 1.399 (11) |
| O2W—H3W | 0.8500 | C6—H6 | 0.9300 |
| O2W—H4W | 0.8500 | C7—H7A | 0.9300 |
| O3W—H5W | 0.8501 | C8—C8 ⁱⁱ | 1.513 (16) |
| O3W—H6W | 0.8500 | C8—H8A | 0.9800 |
| O3W—Co1—O1W ⁱ | 91.2 (2) | O2—C1—C2 | 117.2 (8) |
| O3W—Co1—O1W | 91.2 (2) | O1—C1—C2 | 119.3 (8) |
| O1W ⁱ —Co1—O1W | 95.6 (4) | C7—C2—C3 | 119.0 (7) |
| O3W—Co1—O2W | 92.7 (2) | C7—C2—C1 | 121.2 (8) |
| O1W ⁱ —Co1—O2W | 173.2 (3) | C3—C2—C1 | 119.7 (8) |
| O1W—Co1—O2W | 89.9 (3) | C4—C3—C2 | 119.6 (8) |
| O3W—Co1—O2W ⁱ | 92.7 (2) | C4—C3—H3A | 120.2 |
| O1W ⁱ —Co1—O2W ⁱ | 89.9 (3) | C2—C3—H3A | 120.2 |
| O1W—Co1—O2W ⁱ | 173.2 (3) | C5—C4—C3 | 121.2 (8) |
| O2W—Co1—O2W ⁱ | 84.3 (4) | C5—C4—H4 | 119.4 |
| O3W—Co1—O4W | 179.3 (3) | C3—C4—H4 | 119.4 |
| O1W ⁱ —Co1—O4W | 88.3 (2) | C4—C5—C6 | 119.6 (7) |
| O1W—Co1—O4W | 88.3 (2) | C4—C5—C8 | 120.7 (8) |
| O2W—Co1—O4W | 87.8 (2) | C6—C5—C8 | 119.7 (8) |
| O2W ⁱ —Co1—O4W | 87.8 (2) | C5—C6—C7 | 119.5 (8) |
| C8—O3—H3 | 109.5 | C5—C6—H6 | 120.3 |
| Co1—O1W—H1W | 125.7 | C7—C6—H6 | 120.3 |
| Co1—O1W—H2W | 117.0 | C2—C7—C6 | 121.1 (8) |
| H1W—O1W—H2W | 108.4 | C2—C7—H7A | 119.4 |
| Co1—O2W—H3W | 112.7 | C6—C7—H7A | 119.4 |
| Co1—O2W—H4W | 107.9 | O3—C8—C8 ⁱⁱ | 107.0 (9) |
| H3W—O2W—H4W | 108.4 | O3—C8—C5 | 111.8 (7) |
| Co1—O3W—H5W | 112.9 | C8 ⁱⁱ —C8—C5 | 112.1 (8) |
| Co1—O3W—H6W | 138.9 | O3—C8—H8A | 108.6 |
| H5W—O3W—H6W | 108.2 | C8 ⁱⁱ —C8—H8A | 108.6 |
| Co1—O4W—H7W | 108.6 | C5—C8—H8A | 108.6 |
| O2—C1—O1 | 123.5 (8) | | |
| O2—C1—C2—C7 | 0.1 (12) | C4—C5—C6—C7 | 0.7 (12) |
| O1—C1—C2—C7 | -179.7 (8) | C8—C5—C6—C7 | -179.9 (7) |
| O2—C1—C2—C3 | 177.1 (8) | C3—C2—C7—C6 | 0.6 (13) |
| O1—C1—C2—C3 | -2.8 (12) | C1—C2—C7—C6 | 177.5 (7) |
| C7—C2—C3—C4 | 0.1 (12) | C5—C6—C7—C2 | -1.0 (13) |
| C1—C2—C3—C4 | -176.9 (7) | C4—C5—C8—O3 | -126.6 (9) |
| C2—C3—C4—C5 | -0.4 (13) | C6—C5—C8—O3 | 54.0 (10) |
| C3—C4—C5—C6 | 0.0 (13) | C4—C5—C8—C8 ⁱⁱ | 113.3 (11) |
| C3—C4—C5—C8 | -179.4 (7) | C6—C5—C8—C8 ⁱⁱ | -66.1 (12) |

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

supplementary materials

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| O3—H3...O1 ⁱⁱⁱ | 0.82 | 2.00 | 2.811 (8) | 168 |
| O1W—H1W...O1 ^{iv} | 0.85 | 1.96 | 2.814 (9) | 180 |
| O1W—H2W...O2 ^v | 0.85 | 1.81 | 2.665 (8) | 179 |
| O2W—H3W...O3 ⁱⁱ | 0.85 | 2.00 | 2.847 (8) | 180 |
| O2W—H4W...O5W ^{vi} | 0.85 | 2.19 | 3.035 (11) | 179 |
| O3W—H5W...O4W ^{vii} | 0.85 | 1.93 | 2.778 (11) | 172 |
| O3W—H6W...O5W | 0.85 | 1.91 | 2.756 (13) | 171 |
| O5W—H9W...O2 | 0.85 | 1.93 | 2.767 (10) | 169 |
| O4W—H7W...O1 ^{viii} | 0.84 | 1.88 | 2.695 (7) | 163 |
| O4W—H7W...O2 ^{viii} | 0.84 | 2.70 | 3.296 (9) | 130 |

Symmetry codes: (iii) $-x+1, -y, -z$; (iv) $x, y, z+1$; (v) $x+1, y, z+1$; (ii) $-x+2, -y, -z+1$; (vi) $x+1, y, z$; (vii) $x-1, y, z$; (viii) $x+1, -y+1/2, z+1$.

Fig. 1

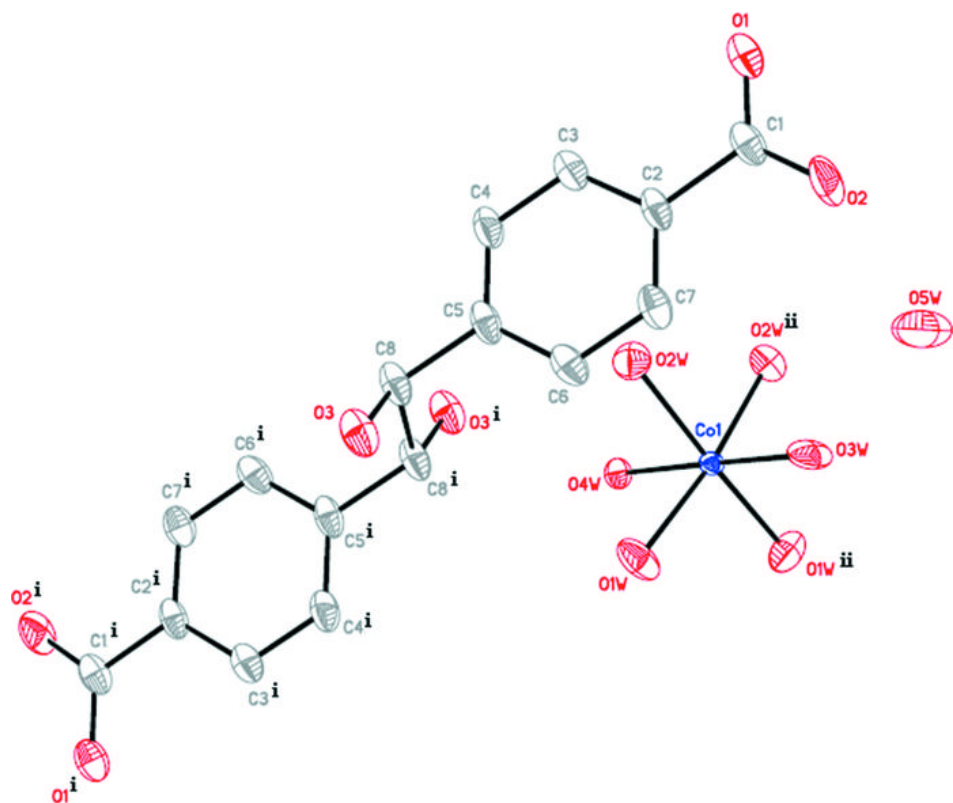


Fig. 2

