Intercalated brucite-type layered cobalt(II) hydroxysulfate

Bunlawee Yotnoi, Sanchai Luachan, Timothy J. Prior and Apinpus Rujiwatra

Acta Cryst. (2009). E65, i52

This open-access article is distributed under the terms of the Creative Commons Attribution Licence http://creativecommons.org/licenses/by/2.0/uk/legalcode, which permits unrestricted use, distribution, and reproduction in any medium, provided the original authors and source are cited.

Acta Crystallographica Section E: Structure Reports Online is the IUCr’s highly popular open-access structural journal. It provides a simple and easily accessible publication mechanism for the growing number of inorganic, metal-organic and organic crystal structure determinations. The electronic submission, validation, refereeing and publication facilities of the journal ensure very rapid and high-quality publication, whilst key indicators and validation reports provide measures of structural reliability. In 2007, the journal published over 5000 structures. The average publication time is less than one month.

Crystallography Journals Online is available from journals.iucr.org
Intercalated brucite-type layered cobalt(II) hydroxysulfate

Bunlawee Yotnoi, a Sanchai Luachan, a Timothy J. Prior b and Apinpus Rujiwatra a *

a Department of Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand, and b Department of Chemistry, University of Hull, Kingston upon Hull HU6 7RX, England

Correspondence e-mail: apinpus@chiangmai.ac.th

Received 27 May 2009; accepted 12 June 2009

Key indicators: single-crystal X-ray study; T = 150 K; mean α(S–O) = 0.006 Å; disorder in main residue; R factor = 0.048; wR factor = 0.132; data-to-parameter ratio = 12.4.

In an attempt to synthesize new cobalt(II) sulfate framework structures using 1,4-diazabicyclo[2.2.2]octane as a template, crystals of poly[0.35-hexaoaquacobalt(II)][tri-μ-hydroxido-μ-sulfato-dicobalt(II)], [{Co(H2O)6}0.35[Co2(OH)3(SO4)]]n, were obtained as a mixture with [Co(H2O)6]SO4 crystals. The crystal structure can be described as being constructed from discrete brucite-type [Co4(OH)6(SO4)2] layers, each of which is built up from edge-shared [Co(OH)6] and [Co(OH)4–(OSO3)] octahedra, with partial intercalation by [Co(H2O)6]2+ ions. The absence of [Co(H2O)6]2+ cations indicates partial oxidation of cobalt(II) to cobalt(III) within the layer.

Related literature

The crystal structure of the title compound is closely related to that of Co5(OH)6(SO4)2·(H2O)4 (Ben Salah et al., 2004, 2006), which is composed of brucite-type cobalt hydroxide layers. The fundamental difference lies in the way that adjacent layers are linked; being pillared by –O–SO–Co(H2O)4–OSO4– groups in Co5(OH)6(SO4)2·(H2O)4 but partially intercalated by [Co(H2O)6]2+ ions in the title compound. For the crystal structures of layered materials of this type, see: Poudret et al. (2008). For a description of the Cambridge Structural Database, see: Allen (2002).

Experimental

Crystal data

[Co(H2O)6]0.35[Co2(OH)3(SO4)]

M r = 323.41
Trigonal, P31
a = 6.3627 (19) Å
c = 12.180 (4) Å
V = 427.0 (2) Å 3

Z = 2
Mo Kα radiation
μ = 4.80 mm –1
T = 150 K
0.21 × 0.13 × 0.03 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: multi-scan
(X-RED; Stoe & Cie, 2008)
T min = 0.415, T max = 0.862

Refinement

R[F2 > 2σ(F 2)] = 0.048
wR(F 2) = 0.132
S = 0.90
497 reflections
40 parameters

3 restraints
Only H-atom coordinates refined
Δρ max = 1.04 e Å –3
Δρ min = –1.05 e Å –3

Data collection: X-AREA (Stoe & Cie, 2008); cell refinement: X-AREA; data reduction: X-RED (Stoe & Cie, 2008); program(s) used to solve structure: SHELXS86 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 1999); software used to prepare material for publication: PLATON (Spek, 2009).

The authors thank the Thailand Research Fund, the Center for Innovation in Chemistry and the Thailand Toray Science Foundation for financial support. BY thanks the Royal Golden Jubilee PhD program and the Graduate School of Chiang Mai University for a Graduate Scholarship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2832).

References

Allen, F. H. (2002). Acta Cryst. B58, 389–398.
Ben Salah, M., Vilminot, S., Andre, G., Richard-Plouet, M., Mhiri, T., Takagi, S. & Kurmoo, M. (2006). J. Am. Chem. Soc. 128, 7972–7981.
Ben Salah, M., Vilminot, S., Richard-Plouet, M., Andre, G., Mhiri, T. & Kurmoo, M. (2004). Chem. Commun. pp. 2548–2549.
Brandenburg, K. & Putz, H. (1999). DIAMOND. Crystal Impact GbR, Bonn, Germany.
Poudret, L., Prior, T. J., McIntyre, L. J. & Fogg, A. M. (2008). Chem. Mater. 20, 7447–7453.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Spek, A. L. (2009). Acta Cryst. D65, 148–155.
Stoe & Cie (2008). X-AREA and X-RED. Stoe & Cie, Darmstadt, Germany.
supplementary materials
Intercalated brucite-type layered cobalt(II) hydrosulfate

B. Yotnoi, S. Luanchan, T. J. Prior and A. Rujiwatra

Comment

Layered transition-metal hydroxides, in particular those exhibiting the brucite structure, have gained serious interest in both chemical and physical aspects. While the intercalation chemistry and potential application as catalysts are of the prime interest for the chemists, the physical interest primarily stems from the long-rang magnetic ordering in two dimensions. Examples of two dimensional triangular networks exhibiting such ordering are very rare, and the information on the crystal structures of layered materials of this type is yet rarer (Poudret, 2008). Thusfar there has been only one structure, Co$_5$(OH)$_6$(SO$_4$)$_2$(H$_2$O)$_4$, having the truly brucite-type magnetic layer (Ben Salah et al., 2004). The nuclear and magnetic structures, and magnetic properties of the compound were extensively reported (Ben Salah et al., 2006). The crystal structure of Co$_5$(OH)$_6$(SO$_4$)$_2$(H$_2$O)$_4$ consists of brucite-type cobalt hydroxide layers of edge-sharing octahedra, which are pillared by · · · O$_3$SO—Co(H$_2$O)$_4$—OSO$_3$··· groups. The compound exhibits ferromagnetic coupling with purely two dimensional magnetic ordering in an easy-plane magnet, which is a rare example of a single-layer magnet.

The crystal structure of [Co(H$_2$O)$_6$x][Co$_4$(SO$_4$)$_2$(OH)$_6$] (I) where x = 0.70 is closely related to that of Co$_5$(OH)$_6$(SO$_4$)$_2$(H$_2$O)$_4$ (Fig. 1), also comprised of brucite-type [Co$_4$(SO$_4$)$_2$(OH)$_6$] layers, each of which is built up from the edge-shared [Co(OH)$_6$] and [Co(OH)$_4$(OSO)$_3$] octahedra in the ab plane (Fig. 2). Two of three crystallographically distinct cobalt ions (Fig. 3), Co1 and Co2, are within the layers and located on the 1b and 3f Wyckoff sites, respectively. Unlike the Co$_5$(OH)$_6$(SO$_4$)$_2$(H$_2$O)$_4$ structure, the sulfate anion acts as a monodentate ligand and is coordinate covalently connected to the layered Co2 ion via the apical O atom, leaving the three basal O atoms pointing into the interlayered space (Fig. 3). The [Co$_4$(SO$_4$)$_2$(OH)$_6$] layers are stacked in the ABAB fashion along c, with a repeat distance of 12.180 (4) Å. In the interlayered gallery, there are intercalated discrete [Co(H$_2$O)$_6$]$^{2+}$ ions in which the Co3 is located on the 1a site. These [Co(H$_2$O)$_6$]$^{2+}$ ions are aligned in a way to maximize the hydrogen bonding interactions of O—H···O type between the aquo ligands and the basal O atoms of the layered sulfate pendants (Fig. 1). The orientation of the sulfate is also regulated by the hydrogen bonds established between the sulfate basal O atoms and the layered hydroxy groups. The Co—O bond length (2.065 (8) Å) within the hexaquo ion is in good agreement with similar cations in the Cambridge Structural Database (Allen, 2002). The mean of similar Co—O distances is 2.09 (3) Å, but is notable that the majority of these structures were collected at room temperature. 97% of the structures containing cobalt hexaquo ions are explicitly recorded at Co$^{2+}$, none are recorded as Co$^{3+}$.

The refined site occupancy of the intercalated [Co(H$_2$O)$_6$]$^{2+}$ ions and the assumption of total charge neutrality imply the partial oxidation (15%) of the layer Co$^{II}$ to Co$^{III}$. This yields an overall composition [Co$^{II}$(H$_2$O)$_6$]$_{0.70}$[Co$^{III}$$^{3+}$Co$^{II}$$^{3+}$Co$^{III}$$^{3+}$]$_{0.60}$(SO$_4$)$_2$(OH)$_6$] for I.
Experimental

In the attempt to synthesize new cobalt(II) sulfate frameworks, crystals of I were unintentionally obtained from the hydrothermal reaction of cobalt(II) sulfate heptahydrate and 1,4-diazabicyclo[2.2.2]octane in acidic aqueous solution (pH 4.4) under autogenous pressure at 453 K for 72 h.

Refinement

Hydrogen atoms were located by difference Fourier methods. The positions of these were refined subject to weak bond length restraints. Displacement parameters for the hydrogen atoms were set at 1.5 times the isotropic displacement parameter of the oxygen atom.

Prior to the refinement of site occupancy of [Co(H$_2$O)$_6$]$^{2+}$, all atoms were located using Fourier difference methods. The displacement parameters of the intercalating ion were anomalously large. There were large maxima and minima in the residual electron density: e-max = 2.59 e Å$^{-3}$ (centered on Co2); e-min = -2.58 e Å$^{-3}$. At this stage wR(F$^2$) = 0.1914.

Refinement of the occupancy of the [Co(H$_2$O)$_6$]$^{2+}$ cation resulted in a significant improvement in the quality of the fit to the data: e-max = 1.044 e Å$^{-3}$; e-min = -1.046 e Å$^{-3}$ and wR(F$^2$) = 0.132.

Careful inspection of the diffraction images did not reveal any weak reflections which might indicate ordering of the partially occupied cation.

Figures

Fig. 1. View of the crystal structure of I along the a axis showing the brucite-type [Co$_4$(SO$_4$)$_2$(OH)$_6$] layers which are intercalated by the hydrogen bonded (dash lines) [Co(H$_2$O)$_6$]$^{2+}$ ions.

Fig. 2. Polyhedral representation of the cobalt hydroxysulate layer, built up of the edge-shared [Co(OH)$_6$] and [Co(OH)$_4$(OSO$_3$)$_2$] octahedra.
poly[0.35-{hexaaquacobalt(II)} [tri-μ-hydroxido-μ-sulfato-dicobalt(II)]]

Crystal data

[Co(H₂O)₆]₀.₃₅[Co₂(OH)₃(SO₄)]  

\( Z = 2 \)

\( M_r = 323.41 \)

\( D_x = 2.515 \text{ Mg m}^{-3} \)

Trigonal, \( P3m1 \)

Hall symbol: -P 3 2\( \overline{2} \)

Cell parameters from 1764 reflections

\( a = 6.3627 \) Å

\( b = 6.3627 \) Å

\( c = 12.180 \) Å

\( \alpha = 90^\circ \)

\( \beta = 90^\circ \)

\( \gamma = 120^\circ \)

\( V = 427.0 \) (2) Å³

Data collection

Stoe IPDS2
diffрактометр

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 6.67 pixels mm⁻¹

\( T = 150 \) K

\( \omega \) scans

Absorption correction: multi-scan

\( T_{\text{min}} = 0.415, T_{\text{max}} = 0.862 \)

1663 measured reflections

497 independent reflections

325 reflections with \( I > 2\sigma(I) \)

\( R_{\text{int}} = 0.097 \)

\( \theta_{\text{max}} = 29.3^\circ \)

\( \theta_{\text{min}} = 1.7^\circ \)

\( h = -7 \rightarrow 8 \)

\( k = -7 \rightarrow 8 \)

\( l = -14 \rightarrow 16 \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.048 \)

\( wR(F^2) = 0.132 \)

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

Only H-atom coordinates refined

\( w = 1/\sigma^2(F_o^2) + (0.0839P)^2 \)

where \( P = (F_o^2 + 2F_c^2)/3 \)
supplementary materials

\[ S = 0.90 \quad (\Delta/\sigma)_{\text{max}} < 0.001 \]
\[ 497 \text{ reflections} \quad \Delta \rho_{\text{max}} = 1.04 \text{ e Å}^{-3} \]
\[ 40 \text{ parameters} \quad \Delta \rho_{\text{min}} = -1.05 \text{ e Å}^{-3} \]
\[ 3 \text{ restraints} \quad \text{Extinction correction: none} \]

Primary atom site location: structure-invariant direct methods

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 (Å\(^2\))

|      | \( x \)  | \( y \)  | \( z \)  | \( U_{iso} \) or \( U_{eq} \) |
|------|----------|----------|----------|-----------------------------|
| Co2  | 0.5000   | 0.0000   | 0.5000   | 0.0170 (3)                  |
| Co1  | 0.0000   | 0.0000   | 0.5000   | 0.0163 (5)                  |
| S1   | 0.6667   | 0.3333   | 0.2679 (2)| 0.0214 (6)                  |
| O1   | 0.1717 (3)| -0.1717 (3)| 0.4215 (3)| 0.0164 (9)                  |
| O2   | 0.6667   | 0.3333   | 0.3913 (6)| 0.0188 (15)                 |
| O3   | 0.5403 (5)| 0.0807 (9)| 0.2298 (4)| 0.0292 (11)                 |
| Co3  | 0.0000   | 0.0000   | 0.0000   | 0.0250 (11)                 |
| O4   | 0.1574 (8)| 0.3148 (16)| 0.0920 (7)| 0.039 (2)                   |
| H1   | 0.188 (9) | -0.188 (9)| 0.355 (3) | 0.059*                      |
| H4   | 0.073 (9) | 0.353 (18)| 0.128 (5) | 0.059*                      |

Atomic displacement parameters (Å\(^2\))

|      | \( U^{11} \) | \( U^{22} \) | \( U^{33} \) | \( U^{12} \) | \( U^{13} \) | \( U^{23} \) |
|------|--------------|--------------|--------------|--------------|--------------|--------------|
| Co2  | 0.0146 (5)   | 0.0156 (6)   | 0.0211 (6)   | 0.0078 (3)   | 0.0001 (2)   | 0.0002 (4)   |
| Co1  | 0.0144 (6)   | 0.0144 (6)   | 0.0202 (9)   | 0.0072 (3)   | 0.0000       | 0.0000       |
| S1   | 0.0220 (9)   | 0.0220 (9)   | 0.0203 (12)  | 0.0110 (4)   | 0.0000       | 0.0000       |
| O1   | 0.0177 (16)  | 0.0177 (16)  | 0.0167 (18)  | 0.0111 (17)  | -0.0002 (8)  | 0.0002 (8)   |
| O2   | 0.017 (2)    | 0.017 (2)    | 0.023 (4)    | 0.0083 (11)  | 0.0000       | 0.0000       |
| O3   | 0.034 (2)    | 0.023 (2)    | 0.027 (2)    | 0.0116 (12)  | -0.0034 (10) | -0.0069 (19) |
| Co3  | 0.0256 (13)  | 0.0256 (13)  | 0.0238 (17)  | 0.0128 (7)   | 0.0000       | 0.0000       |
| O4   | 0.034 (3)    | 0.043 (5)    | 0.044 (4)    | 0.022 (2)    | -0.0080 (18) | -0.016 (4)   |

Geometric parameters (Å, °)

|       | Co2—O1      | S1—O3\(^{\text{ix}}\) | Co2—O1      |
|-------|--------------|------------------------|--------------|
|       | 2.047 (3)    | 1.467 (5)              | 1.467 (5)    |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|
| Co2—O1               | 2.047 (3)    | S1—O2                | 1.502 (7)    |
| Co2—O1               | 2.047 (3)    | O1—Co2               | 2.047 (3)    |
| Co2—O1               | 2.047 (3)    | O1—H1                | 0.83 (3)     |
| Co2—O2               | 2.264 (4)    | O2—Co2               | 2.264 (4)    |
| Co2—O2               | 2.264 (4)    | O2—Co2               | 2.264 (4)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| Co1—O1               | 2.121 (4)    | Co3—O4               | 2.065 (8)    |
| S1—O3                | 1.467 (5)    | O4—H4                | 0.82 (3)     |
| S1—O3-viii           | 1.467 (5)    | O3-viii—S1—O3-xvii   | 110.49 (19)  |
| O1—Co2—O1            | 180.000 (1)  | O3-viii—S1—O3-xvii   | 110.49 (19)  |
| O1—Co2—O1            | 97.8 (2)     | O3—S1—O2             | 108.4 (2)    |
| O1—Co2—O1            | 82.2 (2)     | O3-viii—S1—O2        | 108.4 (2)    |
| O1—Co2—O1            | 82.2 (2)     | O3—S1—O2             | 108.4 (2)    |
| O1—Co2—O1            | 97.8 (2)     | Co2—O1—Co2-xvii      | 102.00 (17)  |
| O1—Co2—O1            | 180.0 (2)    | Co2—O1—Co1           | 99.51 (14)   |
| O1—Co2—O2            | 95.83 (13)   | Co2-xv—O1—Co1        | 99.51 (14)   |
| O1—Co2—O2            | 84.17 (13)   | Co2—O1—H1            | 112 (4)      |
| O1—Co2—O2            | 84.17 (13)   | Co2—O1—H1            | 112 (4)      |
| O1—Co2—O2            | 84.17 (13)   | Co2—O1—H1            | 112 (4)      |
| O1—Co2—O2            | 95.83 (13)   | Co2—O1—H1            | 112 (4)      |
| O1—Co2—O2            | 95.83 (13)   | Co2—O1—H1            | 112 (4)      |
| O1—Co2—O2            | 84.17 (13)   | S1—O2—Co2-xvii       | 125.79 (14)  |
| O1—Co2—O2            | 84.17 (13)   | S1—O2—Co2            | 125.79 (14)  |
| O1—Co2—O2            | 84.17 (13)   | Co2-xvii—O2—O2       | 89.3 (2)     |
| O1—Co2—O2            | 84.17 (13)   | S1—O2—Co2-xvii       | 125.79 (14)  |
| O1—Co2—O2            | 95.83 (13)   | Co2—O2—Co2-xvii      | 89.3 (2)     |
| O1—Co2—O2            | 180.0        | Co2—O2—Co2-xvii      | 89.3 (2)     |
| O1—Co1—O1            | 180.0 (2)    | Co2—O2—Co2-xvii      | 89.3 (2)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4            | 180.0 (4)    |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 86.7 (4)     |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4-xvii      | 86.7 (4)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 86.7 (4)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 78.77 (13)   | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 93.3 (4)     |
| O1—Co1—O1            | 101.23 (13)  | O4—Co3—O4-xvii      | 93.3 (4)     |
supplementary materials

| Bond                  | Distance (Å) | Angle (°)  |
|-----------------------|--------------|------------|
| O1\(^{\text{vi}}\)-Co1-O1\(^{\text{vii}}\) | 78.77 (13)   |            |
| O1-Co1-O1\(^{\text{vii}}\)         | 180.0 (2)    |            |
| O3-S1-O3\(^{\text{viii}}\)        | 110.49 (19)  |            |
| O3-S1-O3\(^{\text{ix}}\)          | 110.49 (19)  |            |
| O4\(^{\text{iv}}\)-Co3-O4\(^{\text{viii}}\) | 180.0 (4)    |            |
| O4\(^{\text{v}}\)-Co3-O4\(^{\text{viii}}\) | 86.7 (4)     |            |
| O4\(^{\text{vii}}\)-Co3-O4\(^{\text{viii}}\) | 93.3 (4)     |            |

Symmetry codes: (i) \(-x+1, -y, -z+1\); (ii) \(-x+y+1, -x, z\); (iii) \(x-y, x, -z+1\); (iv) \(-x+y, -x, z\); (v) \(y, -x+y, -z+1\); (vi) \(-y, x-y, z\); (vii) \(-x, -y, -z+1\); (viii) \(-y+1, x-y, z\); (ix) \(-x+y+1, -x+1, z\); (x) \(-y, x-y-1, z\); (xi) \(-x, -y, -z\); (xii) \(-x+y, -z\); (xiii) \(x-y, x, -z\).

**Hydrogen-bond geometry (Å, °)**

| Type        | D—H  | H···A   | D···A   | D—H···A |
|-------------|------|--------|--------|---------|
| O1—H1···O3\(^{\text{v}}\) | 0.83 (3) | 2.54 (5) | 3.125 (6) | 129 (4) |
| O1—H1···O3  | 0.83 (3) | 2.54 (5) | 3.125 (6) | 129 (4) |
| O4—H4···O3\(^{\text{vi}}\) | 0.82 (3) | 1.90 (9) | 2.712 (1) | 170 (7) |

Symmetry codes: (x) \(-y, x-y-1, z\); (vi) \(-y, x-y, z\).
Fig. 1
supplementary materials

Fig. 2
