

Synthesis and structural characterization of a novel peroxo bridged dinuclear cobalt(III) complex of succinimide showing three varieties of hydrogen bonding interactions

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Abstract. The reaction of aqueous cobaltous nitrate hexahydrate with the anion of succinimide (sucH) in the presence of excess ethylenediamine (en) in air results in the formation of a dinuclear complex μ -peroxo-bis[bis(ethylenediamine)succinimidato-cobalt(III)] dinitrate dihydrate, **1**, in good yield. Compound **1** was characterized by elemental analysis, IR, visible spectra and magnetic susceptibility studies. The explosive nature of $[\text{Co}(\text{en})_2(\text{suc})(\mu\text{-O}_2)\text{Co}(\text{en})_2(\text{suc})](\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, **1**, precluded its thermal characterization. Compound **1** crystallises in the monoclinic space group $P2_1/c$ and a half of the molecule, constitutes its asymmetric unit. In the centrosymmetric dinuclear complex **1**, two Co(III) centres are linked by a planar peroxide bridge. Each cobalt atom is surrounded by four nitrogen atoms of ethylenediamine ligands, a nitrogen atom of succinimidato anion and an oxygen atom of peroxo bridge resulting in a slightly distorted $\{\text{CoN}_5\text{O}\}$ octahedron. Due to steric hindrance between the two Co(III) centres, the peroxide bridge is planar with a Co–O–O–Co torsion angle of 180° . The dinuclear complex cation, the nitrate anion and the lattice water are involved in three varieties of H-bonding interactions namely N–H \cdots O, O–H \cdots O and C–H \cdots O.

Keywords. Ethylenediamine; succinimide; succinimidato; peroxo bridge; dinuclear; H-bonding.

1. Introduction

It is well known that complexation of dioxygen plays a vital role in many biological systems. Extensive research has focused on the coordination chemistry of naturally occurring dioxygen complexes, i.e. haemoglobin, haemerythrin, haemocyanin and their model compounds.¹ Many biological reactions involving dioxygen are very fast with synthetic caged dioxygen carriers. Cobalt(II) complexes of polyamines, amino acids, and dipeptide ligands containing dicobalt, μ -peroxo or, μ -superoxo-bridged derivatives have been studied extensively, because of their applicability to biochemical systems.² Many are available in the literature to indicate that the stabilities of dioxygen complexes increase with an increase in the number of basic nitrogens.³ In these complexes, the binding of oxygen is accompanied, in many cases, by the transfer of electrons from the metal center to the oxygen molecule, and thus the

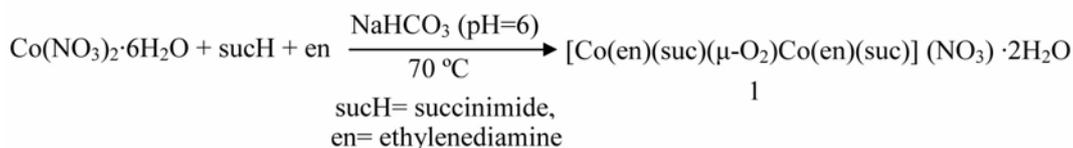
oxidation state of the metal atom changes.⁴ In view of this, peroxo-bridged dicobalt complexes are often regarded as models for biological oxygen carriers.⁵

The pyrrolidine skeleton occurs in many families of biologically important compounds. Several succinimide (pyrrolidine-2,5-dione) derivatives are important in biology due to their antiepileptic, anti-convulsive, fungicidal and other pharmacological properties.⁶ Since its first description in 1905, Yamada *et al.* synthesized the mixed ligand Cu(II) succinimide (sucH) complexes with some alkylamines (from methylamine to *n*-butylamine) to investigate electronic spectral features. Slabbert *et al.* prepared some Cu(II) and Ni(II) succinimide complexes with different alkylamines to study the vibrational spectra of the complexes.^{7,8} Various aliphatic amine derivatives of metal(II)-succinimidato complexes have been synthesized and recently some Cu(II)-succinimidato complexes have been characterized by X-ray diffraction.^{9–17} The literature survey shows that the copper complexes of succinimide

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Table 1. Crystal data and structure refinement for compound **1**.

| | |
|---|---|
| Empirical formula | [C ₁₆ H ₄₀ Co ₂ N ₁₀ O ₆] \cdot 2(NO ₃) \cdot 2(H ₂ O) |
| Formula weight (g mol ⁻¹) | <i>M_r</i> = 746.49 |
| Temperature (K) | 296(2) |
| Wavelength (Å) | 0.71073 |
| Crystal system | Monoclinic |
| Space group | <i>P</i> 2 ₁ / <i>c</i> |
| Unit cell dimensions (Å, °) | |
| <i>a</i> ; <i>b</i> ; <i>c</i> | 9.4769(4); 16.2143(8); 12.7866(6) |
| β | 130.984(3) |
| Volume (Å ³) | 1483.22(14) |
| <i>Z</i> | 2 |
| Density (calcd.) (g/cm ³) | 1.671 |
| Absorption coefficient (mm ⁻¹) | 1.203 |
| <i>F</i> (000) | 780 |
| Crystal size (mm ³) | 0.32 \times 0.50 \times 0.52 |
| Theta range for data collection (°) | 2.5–26.5° |
| Index ranges | –11 \rightarrow 8; 0 \rightarrow 20; 0 \rightarrow 16 |
| Reflections collected | 12116 |
| Independent reflections (<i>R</i> _{int}) | 3061 |
| Completeness to theta | 2.11–28 |
| Refinement method | Full-matrix least-squares on <i>F</i> ² |
| Data/restraints/parameters | 12116/2636/0.000 |
| Goodness-of-fit on <i>F</i> ² | 1.08 |
| Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)] | 0.0330 |
| <i>R</i> indices (all data) | 0.0849 |
| Extinction coefficient | 0 |
| Largest diff. peak and hole (eÅ ⁻³) | –0.82, 0.37 |

**Scheme 1.**

have been studied extensively, while there is no report on cobalt complexes of succinimide.

Ethylenediamine (en), its salts, and its derivatives are frequently found in drugs as well as biologically important molecules. Representative drug classes containing this moiety include the antihistamine, pyrilamine and chlorcyclazine; the ganglionic blocking agent, chlorisondamine; the antihelminthic, piperazine, and the antiarrhythmic agent, procainamide. In each of these compounds, the ethylenediamine portion plays an essential role in the biological activities of these molecules. The structural features of this moiety that confer biological activity are the electron distribution among the atoms and the stereochemistry.¹⁸ In the present report, we describe the synthesis, spectral and structural characterization of a peroxo bridged dinuclear Co(III) complex con-

taining N-donor ligands like en and succinimidato anion bonded to Co(III).

2. Experimental

2.1 Material and measurements

All chemicals used in this work were of reagent-grade commercial materials. FT-IR spectra (4000–400 cm⁻¹) were recorded on a Perkin-Elmer-Spectrum 100 FT-IR spectrophotometer with samples prepared as KBr pellets. Magnetic susceptibilities were measured using a Sherwood scientific MX1 model Gouy magnetic balance at room temperature. UV-Vis spectra were recorded with a PG-T80 + UV-Vis spectrometer. pH was measured using Eutech-Cyberscan 510 pH-meter. Elemental analysis for C,

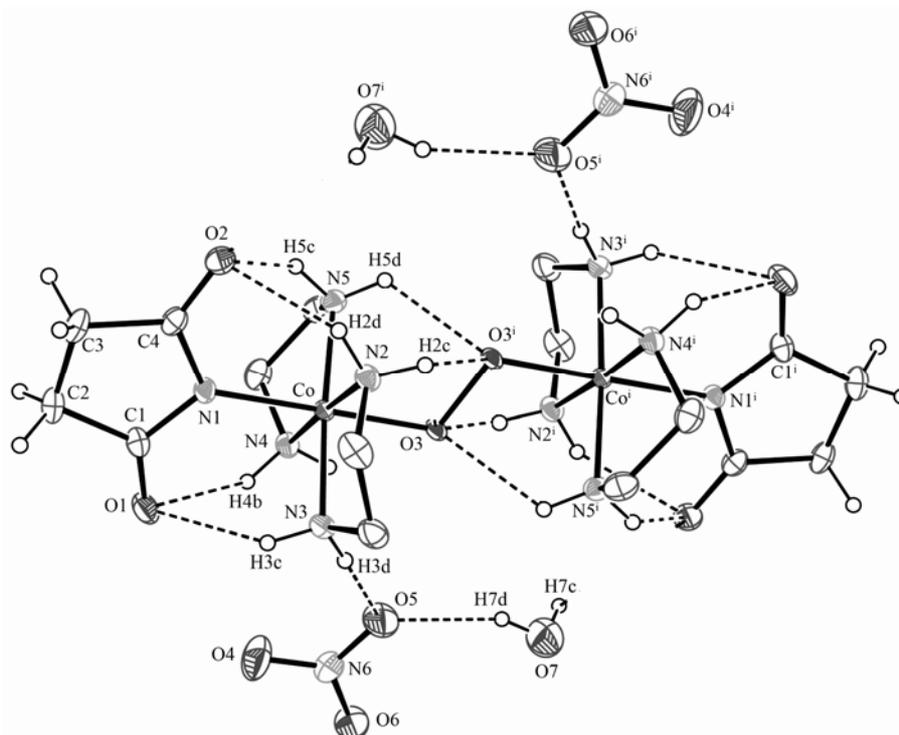


Figure 1. An ORTEP view of compound **1** showing the crystallographic labelling. Ellipsoids are drawn at 30% probability level and some H atoms are omitted for clarity. Intramolecular hydrogen bonding is shown by broken lines. (Symmetry code: $I = [1 - x, 1 - y, 1 - z]$).

H and N were performed using a Carlo Erba 1106 microanalyser.

2.2 Synthesis of μ -peroxo-bis[bis(ethylenediamine)succinimidato-cobalt(III)] dinitrate dihydrate **1**

To a solution of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (2.91 g, 10 mmol) in water (20 ml), succinimide (1.00 g, 10.1 mmol) was added and the reaction mixture was heated to 70°C . The pH was adjusted to 6 by adding 1% NaHCO_3 solution. An excess of ethylenediamine (approximately 2 ml) was added to the hot solution and stirred at 70°C for 30 min. The colour of the solution changed from red violet to brown. The reaction mixture was slowly cooled to room temperature and left undisturbed in open air. After several days dark-brown crystals were collected, rinsed with water, followed by cold ethanol. Yield of dry product (3.4 g, 45%).

Caution: The complex explodes when heating and hence not studied by thermal method.

Analysis: Calc. for $\text{C}_{16}\text{H}_{44}\text{Co}_2\text{N}_{12}\text{O}_{14}$: C, 25.74; H, 5.94; N, 22.52%. Found: C, 25.77; H, 5.90; N, 22.57%.

IR (KBr, $\bar{\nu}$, cm^{-1} selected peaks): 3437 *w*, 3300 *m*, 3235 *m*, 1884 *m*, 1775 *w*, 1455 *m*, 1430 *m*, 886 *m*, 532 *m*.

UV-Vis : [λ_{max} in nm, (ϵ , $\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 400 (2000) and 800 (50) (in H_2O).

2.3 X-ray crystallography

Intensity data were collected on an Image Plate diffraction System (IPDS 2) from STOE using graphite-monochromated Mo- $K\alpha$ radiation. Data collection and cell refinement was performed using X-Area,¹⁹ while X-Red32;¹⁹ was used for data reduction. The structure was solved using Sir2004;²⁰ and refinement was done against F^2 using SHELXL-97.²¹ OrtepIII²² was used for molecular graphics while the publication material was prepared using Wingx.²³ All non-hydrogen atoms were refined anisotropically. The C–H hydrogen atoms were positioned with idealized geometry and refined using a riding model. The N–H and O–H hydrogen atoms were located in the difference map, their bond lengths set to ideal values and afterwards they were refined isotropic using a riding model. The technical details of data acquisi-

Table 2. Selected bond lengths (Å) and angles (°).

| | | | |
|------------------------|------------|--------------------|------------|
| Bond distances | | | |
| Co1–O3 | 1.897(1) | N1–C1 | 1.374(2) |
| Co1–N1 | 1.974(2) | N1–C4 | 1.371(2) |
| Co1–N2 | 1.938(2) | O1–C1 | 1.225(3) |
| Co1–N3 | 1.957(2) | O2–C4 | 1.231(4) |
| Co1–N4 | 1.944(2) | O3–O3 ⁱ | 1.499(2) |
| Co1–N5 | 1.949(2) | | |
| Bond angles | | | |
| Co1–O3–O3 ⁱ | 109.72(12) | N2–Co1–N3 | 85.79(7) |
| O3–Co1–N1 | 179.35(6) | N2–Co1–N4 | 177.48(7) |
| O3–Co1–N2 | 89.33(6) | N2–Co1–N5 | 95.77(7) |
| O3–Co1–N3 | 88.09(6) | N3–Co1–N4 | 92.42(7) |
| O3–Co1–N4 | 88.82(6) | N3–Co1–N5 | 175.55(7) |
| O3–Co1–N5 | 87.77(6) | N4–Co1–N5 | 85.88(7) |
| N1–Co1–N2 | 91.32(7) | N1–C1–C2 | 111.29(18) |
| N1–Co1–N3 | 91.89(7) | N1–C4–C3 | 111.48(18) |
| N1–Co1–N4 | 90.53(7) | O1–C1–N1 | 124.39(19) |
| N1–Co1–N5 | 92.24(7) | O2–C4–N1 | 124.18(19) |

i: 1 – x, 1 – y, 1 – z

tion and some selected refinement results are summarised in table 1.

3. Results and discussion

3.1 Synthesis and spectral studies

The reaction of a Co(II) salt in air in the presence of N-donor ligands resulting in the formation of Co(III) complexes is well documented in the literature. Thus in the present work the reaction of cobaltous nitrate with such and en resulted in the formation of the peroxo bridged title compound **1** in good yield (scheme 1). The formula of **1** was arrived at based on its analytical, spectral and structural data. Based on analytical data, charge considerations, **1** was formulated as a dinuclear Co(III) compound. The observed diamagnetic nature of **1** is consistent with the presence of Co(III). The UV–Vis. spectrum in water showed a charge transfer band at 400 nm and a band at 800 nm assignable for a $d \rightarrow d$ transition as expected for octahedrally surrounded Co(III) ions. Interestingly, the weak signal at 800 nm disappeared after 15 min in water and DMSO solutions. This result can be attributed to the decomposition of **1** in solution.

The IR spectrum of pure such shows a signal at 3375 cm^{-1} , assignable to the N–H vibration and a C=O peak at 1775 cm^{-1} . In the IR spectrum of **1**, no signal is observed at around 3375 cm^{-1} indicating the presence of such in its anionic (suc). This is fur-

ther supported by the shifting of the C–N-vibration of such from 1430 to 1455 cm^{-1} . The $\nu(\text{C}=\text{O})$ signal remains unshifted indicating that the C=O unit is non-coordinated to the metal.^{7,8,17} The peaks at 3300 and 3235 cm^{-1} are assigned to the vibrations for the NH₂ groups of en. These results are in agreement with previous reports.^{7–12,16–17} The presence of water in **1** is evidenced by the strong signal at 3437 cm^{-1} . The observed signals at 886 and 532 cm^{-1} are assigned for the vibrations of the peroxo moiety which is in accordance with literature reports^{24,25}.

3.2 Crystal structure description

Compound **1** crystallises in the centrosymmetric monoclinic space group $P2_1/c$ and its structure consists of a complex cation of **1** namely μ -peroxo-bis[succinimidato-bis(ethylenediamine)cobalt(III)]²⁺ located on an inversion centre, a nitrate anion and a lattice water (figure 1). The centre of inversion is situated at the midpoint of the O–O bond with the (O–O)²⁻ functioning as a bridge between the symmetry related Co(III) ions. In view of this special position for the complex cation a half of the molecule, constitutes its asymmetric unit. Each cobalt atom is surrounded in an octahedral fashion by four nitrogen atoms from two en ligands, a nitrogen atom of an anionic suc ligand and an oxygen atom of peroxo-bridge resulting in a {CoN₅O} octahedron. The trans O–Co–N and N–Co–N angles are very close to the ideal value and vary between $175.55(7)$ and

Table 3. Hydrogen bond geometry for compound **1**.

| D–H...A | D–H | H...A | D...A | D–H...A |
|----------------------------|----------|-----------|----------|---------|
| N2–H2d...O2 | 0.90 | 2.14 | 2.886(2) | 139.9 |
| N3–H3c...O1 | 0.90 | 2.33 | 2.997(2) | 130.6 |
| N4–H4b...O1 | 0.90 | 2.13 | 2.852(2) | 136.8 |
| N4–H4a...O5 | 0.90 | 2.71 | 3.272(3) | 121.9 |
| N5–H5c...O2 | 0.90 | 2.35 | 3.041(2) | 133.4 |
| N3–H3d...O5 | 0.90 | 2.15 | 3.036(3) | 166.7 |
| O7–H7d...O5 | 0.822(9) | 2.134(12) | 2.905(4) | 156(2) |
| N2–H2c...O6 ⁱⁱ | 0.90 | 2.47 | 3.142(3) | 131.7 |
| C3–H3b...O6 ⁱⁱⁱ | 0.97 | 2.69 | 3.639(3) | 166.9 |
| C2–H2a...O5 ⁱⁱⁱ | 0.97 | 2.76 | 3.602(3) | 145.9 |
| N2–H2c...O3 ⁱ | 0.90 | 2.16 | 2.774(2) | 124.5 |
| N5–H5d...O3 ⁱ | 0.90 | 2.17 | 2.750(2) | 122.0 |
| N4–H4a...O6 ^{iv} | 0.90 | 2.26 | 3.039(3) | 144.1 |
| O7–H7c...O4 ^{iv} | 0.816(9) | 2.528(18) | 3.171(4) | 137(2) |
| N5–H5c...O1 ^v | 0.90 | 2.48 | 3.074(2) | 124.0 |
| C3–H3a...O5 ^v | 0.97 | 2.79 | 3.726(3) | 162.1 |
| C2–H2b...O6 ^v | 0.97 | 2.76 | 3.632(3) | 150.1 |
| N3–H3c...O2 ^{vi} | 0.90 | 2.54 | 3.157(2) | 126.3 |

i: 1 – x, 1 – y, 1 – z; ii: 1 + x, y, z; iii: 1 – x, 1/2 + y, 3/2 – z; iv: –x, 1 – y, 1 – z; v: x, 3/2 – y, –1/2 + z; vi: x, 3/2 – y, 1/2 + z

179.35(6)° while the *cis* O–Co–N and N–Co–N angles range from 85.88(7) to 95.77(7)° indicating a distorted octahedron. The succinimide rings exhibit an approximately planar structure and are perpendicular to the Co–N4 equatorial plane formed by four nitrogen atoms of two en ligands, with the largest deviation from the plane being that of atom C2 [0.035(3) Å]. The observed bond angles of suc anion indicate the presence of an anionic suc and also electron delocalization in the π -conjugated system (table 2).^{11,17}

The two en ligands in the asymmetric unit are in the $\delta\delta$ conformation. The Co–N_{en} bond lengths are found as 1.938(3), 1.957(2), 1.949(2) and 1.944(2) Å (table 2).²⁶ The short distances across the bridge were those of C6...C8ⁱ and C8...C6ⁱ, and are both 3.83 Å. This is less than twice the van der Waals radius of C–H (2.0 Å) in the synthesized complex and the suc and en ligands have *trans*-configuration with respect to the μ -peroxo bridge.

For the charge balance, +2 charges on the binuclear cations require that the O₂-bridge is a peroxide group. Due to the steric hindrance between the two +3 charged Co(III) centres, the peroxide bridge is planar with a Co–O–O–Co torsion angle 180°. The O–O distance in bridge for **1** is 1.499(2) Å and is comparable with the reported values for O–O bond in μ -peroxo-*bis*[nitro-*bis*(ethylenediamine) cobalt(III)]²⁺ [1.529(9) Å], H₂O₂ (1.47–1.49 Å) and BaO₂ (1.49 Å).²² The Co–O distance of 1.897(1) Å is

slightly longer than that found in μ -peroxo-*bis*[nitro-*bis*(ethylenediamine)cobalt(III)]²⁺ [1.887(6) Å].²²

The Co–N_{succin.} bond length is 1.974(2) Å, and it is slightly longer than that in μ -peroxo-*bis*[nitro-*bis*(ethylenediamine)cobalt(III)]²⁺ with a Co–N_{nitro} distance of 1.940(8) Å.^{26,27}

A non-planar arrangement (the Co–O–O–Co torsion angle < 180°) occurs only when the charges on the two centres are neutralized by extensively hydrogen bonded counter ions according to Fritch.²⁸ When the μ -peroxo-*bis*[pentaamminecobalt(III)]⁴⁺ ion is crystallized as the thiocyanate salt,²⁸ the cation participates in only two hydrogen bonds, and the peroxo bridge is planar. In the sulphate salt of the same cation, however, nearly all the amine hydrogen atoms participate in hydrogen bonds, effectively neutralizing the charges on the cobalt centres, and the peroxo-bridge has a dihedral angle of 146°. ²⁸ The μ -peroxo-*bis*[(ethylenediamine)(diethylenetriamine) cobalt(III)]⁴⁺ cationic complex charge balanced by perchlorate counter anions, exhibits six hydrogen bonds, four of which are intramolecular. Based on this observation it was reported that perchlorate counter ions were isolated from the cation and were thus ineffective in neutralizing the concentration of charge; leading to a planar peroxide bridge.²⁸ Interestingly the planar arrangements (the Co–O–O–Co torsion angle = 180°) were explained as a result of steric hindrance by Shibahara in μ -peroxo-*bis*[nitro-*bis*(ethylenediamine) cobalt(III)]²⁺ cation

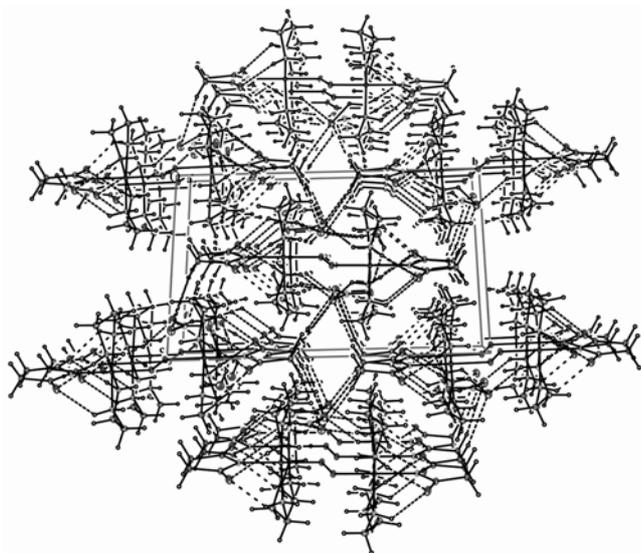


Figure 2. A view of crystal packing diagram of **1**. H-bonds are shown in broken lines.

(nitrate was counter ion, and two hydrogen bonds were observed) and a similar arrangement was reported in μ -peroxo-bis[acetonitrile-bis(ethylenediamine)-cobalt(III)]⁴⁺ (tetratrilate was counter ion with two hydrogen bonds).^{26,27} The planar arrangement in μ -peroxo-bis[nitro-bis(ethylenediamine)cobalt(III)]²⁺ and the complex **1**, in which the charges on the cobalt centres are compensated by negatively charged anionic ligands like nitro and succinimidato anion, can be attributed to the steric hindrance to peroxo bridges.

An analysis of the crystal structure reveals that the dinuclear complex cation, the nitrate anion and the lattice water are involved in several H-bonding interactions many of which are intramolecular (figure 1). All the oxygen atoms in **1** namely O1, O2 from suc, O3 of bridging peroxide, O4, O5 and O6 of nitrate and O7 of lattice water function as H-acceptors while the H atoms attached to the amine groups of en, H atoms of lattice water and H atoms attached to C2 and C3 of suc ligand function as H-donors resulting in three varieties of H-bonding interactions namely N–H...O, O–H...O and C–H...O and the geometrical details of these weak interactions are given in table 3.

The H-bonding network is extended along *a* axis with the aid of N2–H2c...O6.

The complex molecules extend along the *b* axis via H-bonding to each other with the aid of the weak C–H...O interactions (table 3). These hydrogen bonds

form rings, which appear like a cube and extend along the three axes and can be described as R₂²(7) in Etter's notation.²⁹ A view of the crystallographic packing of compound **1** showing its supramolecular architecture is displayed in figure 2.

4. Conclusions

In the present report we have described the synthesis, spectral characteristics and structural features of a new peroxo bridged dinuclear Co(III) compound showing three varieties of H-bonding interactions.

Supplementary material

Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as the supplementary publication No. CCDC 682427. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk

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