STRUCTURAL CHEMISTRY

ISSN 2053-2296

A phase transition caught in mid-course: independent and concomitant analyses of the monoclinic and triclinic structures of $(^{n}Bu_4N)[Co(orotate)_2(bipy)]\cdot 3H_2O$

Miguel Castro,[a] Larry R. Falvello,[b]* Elena Forcén-Vázquez,[b] Pablo Guerra,[b] Nuha A. Al-Kenany,[b] Gema Martínez[b] and Milagros Tomás[c]*

[a]Departamento de Ciencia y Tecnología de Materiales y Fluidos, Escuela de Ingeniería y Arquitectura, Instituto de Ciencia de Materiales de Aragón (ICMA), University of Zaragoza–CSIC, María de Luna 3, Zaragoza E-50018, Spain, [b]Department of Inorganic Chemistry and Aragón Materials Science Institute (ICMA), University of Zaragoza-CSIC, Pedro Cerbuna 12, Zaragoza E-50009, Spain, and [c]Department of Inorganic Chemistry and Instituto de Síntesis Química y Catálisis Homogenea (ISQCH), University of Zaragoza–CSIC, Pedro Cerbuna 12, Zaragoza E-50009, Spain. *Correspondence e-mail: falvello@unizar.es, milagros@unizar.es

Received 12 July 2017
Accepted 24 July 2017

Edited by A. L. Spek, Utrecht University, The Netherlands

Keywords: order–disorder phase transition; crystal structure; single-crystal-to-single-crystal transformation; tetrabutylammonium disorder; orotate; cobalt; vitamin B_{13} complex.

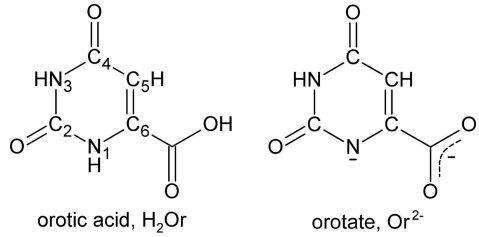
CCDC references: 1560743; 1560742; 1560741; 1560740; 1560739; 1560738

Supporting information: this article has supporting information at journals.iucr.org/c

The preparation and characterization of the $^{n}Bu_4N^+$ salts of two bis-orotate(2−) complexes of cobalt, namely bis(tetra-*n*-butylammonium) diaquabis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κ^2N^1,O^6)cobalt(II) 1.8-hydrate, $(C_{16}H_{36}N)_2[Co(C_5H_2N_2O_4)_2(H_2O)_2]\cdot 1.8H_2O$, (**1**), and tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κ^2N^1,O^6)cobalt(III) trihydrate, $(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$, (**2**), are reported. The Co^{III} complex, (**2**), which is monoclinic at room temperature, presents a conservative single-crystal-to-single-crystal phase transition below 200 K, producing a triclinic twin. The transition, which involves a conformational change in one of the ^{n}Bu groups of the cation, is reversible and can be cycled. Both end phases have been characterized structurally and the system was also characterized structurally in a two-phase intermediate state, using single-crystal diffraction techniques, with both the monoclinic and triclinic phases present. Thermal analysis allows a rough estimate of the small energy content, *viz.* 0.25 kJ mol^{-1}, for both the monoclinic-to-triclinic transformation and the reverse transition, in agreement with the nature of the structural changes involving only the $^{n}Bu_4N^+$ cation.

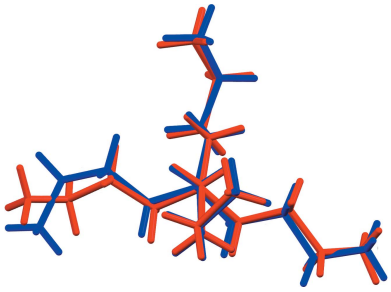
1. Introduction

We have prepared the $^{n}Bu_4N^+$ salt of one isomer of the simple transition metal complex $[Co(Or)_2(bipy)]^-$ [Or^{2-} is orotate(2−) (see Scheme 1) and bipy is 2,2′-bipyridyl] and have observed that at a temperature near 180 K it undergoes a phase transformation for which the two components can be analyzed structurally at the same time using single-crystal diffraction techniques.



Scheme 1

Orotate(2−) is the dianion of orotic acid (H_2Or or 2,6-dioxo-1,2,3,6-tetrahydropyrimidine-4-carboxylic acid), known as vitamin B_{13} (although it is understood not to be a vitamin),



OPEN ACCESS

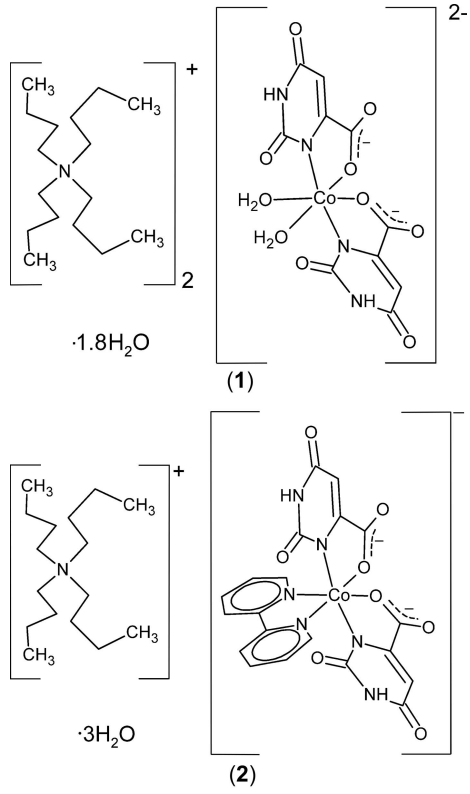
a biologically important molecule that is the precursor for the pyrimidine bases in living systems and which is important in other biological processes (Loeffler *et al.*, 2016). Orotate has been used in the preparation of a stable salt of tenofovir disoproxil, an antiviral agent used against the HIV and hepatitis B viruses (Park *et al.*, 2014). Our own interest in orotic acid and its salts arises from the five varied functional groups that gird its periphery, which make it a versatile ligand in transition-metal chemistry. It is capable of coordinating to a transition-metal atom in different ways and at the same time of participating in significant directional noncovalent interactions with its environment, including crystalline environments. We have referred to such chemical entities as 'polyfunctional ligands,' a name that reflects the presence of numerous functional groups rather than any putative mechanical or physical functionality.

Orotate complexes have been studied structurally in detail; at the time of writing, some 131 crystal structures of orotate complexes of transition metals have been recorded in the Cambridge Structural Database (CSD; Groom *et al.*, 2016), along with 15 complexes of lanthanoid elements and a single complex with a heavy rare earth element, *i.e.* uranium (Mentzafos *et al.*, 1987). In these complexes, orotate is usually found to be doubly deprotonated, at the carboxylate group and at atom N1. By far the commonest coordination mode observed for orotate is chelation through the N1 atom and one of the carboxylate O atoms.

In a separate line of development, we note that molecular solids containing the *n*-butylammonium fragment have been observed to undergo order–disorder phase transitions involving changes in the conformation of as little as one butyl arm of the cation (Willett *et al.*, 2005). Interest in the preparation of molecular materials that undergo phase transitions arises from the possibility of switching chemical and physical properties, such as spectroscopic characteristics (Falvello *et al.*, 1999), magnetic and electric properties, and others, in a controllable manner (Fujita & Awaga, 1999; Sato *et al.*, 2007; Schneemann *et al.*, 2014; Li *et al.*, 2016; Paglione & Greene, 2010; MacFarlane & Forsyth, 2001; Mason *et al.*, 2015; Nauha *et al.*, 2016; Rodríguez-Velamazán *et al.*, 2012). This phenomenon has been observed in materials of potential technological importance (Pielichowska & Pielichowski, 2014; Szaciłowski, 2008). Particularly interesting are single-crystal-to-single-crystal transformations (SCSC), which provide valuable information for the understanding of the switching of the properties of those molecular materials, since both the mother and daughter phases can be structurally characterized.

One way to obtain solids that can undergo phase transitions while maintaining their crystallinity is by using molecular fragments for which there exist potential structural changes requiring low energy and demanding little difference between the sizes and shapes of the initial and final species. Straight-chain paraffins have long been recognized as satisfying these criteria (Müller, 1932). Indeed, rotator and/or plastic phase transitions have been observed for crystals with *n*-alkylammonium salts with small anions, such as halides (Shimizu *et al.*, 1997), mainly through characterization by thermal analysis and nuclear magnetic resonance (NMR) techniques. Regarding the *n*-butyl group in particular, $^{n}Bu_4NI$, a simple salt, presents both a phase transition and ionic transport (Asayama *et al.*, 2005, 2006); however, very few di-*n*-butyl (57 structures in the CSD, only two phase transitions; Peng *et al.*, 2008; Khan *et al.*, 2015) and tri-*n*-butylammonium compounds (49 structures in the CSD, two phase transitions) have been involved in phase transitions which have been characterized by single-crystal X-ray diffraction (Asghar *et al.*, 2015, 2016).

In contrast to di- and tri-*n*-butylammonium, tetra-*n*-butylammonium is a more widely used cation, especially as a counter-ion for coordination compounds; there are more than 80 times as many structures with $^{n}Bu_4N^+$ as with $^{n}Bu_3NH^+$ or $^{n}Bu_2NH_2^+$ (4742/49/57 entries in the CSD, respectively). Yet, and in spite of the high percentage of crystal structures with this group in disorder, the number of phase transitions explicitly characterized by X-ray diffraction has also been very low for systems involving tetra-*n*-butylammonium (Czerwonka *et al.*, 1988; Excoffon *et al.*, 1991; Watase *et al.*, 2003; Willett *et al.*, 2005).



Scheme 2

The term 'partial phase transition' has been used for what are now a large number of systems concluded to have undergone phase transitions in part of the volume of a substance and not in the rest. Most, by far, of the systems observed to behave in such a way have been inorganic solids. Recent examples in which partial phase transitions have been imputed include that of a hydrogen-storage material (Luo *et al.*, 2016) and a partial transition observed in a lithium-containing spinel, *i.e.* $LiMnTiO_4$, a solid with potential relevance as a cathode material for rechargeable lithium-ion

batteries (Murphy *et al.*, 2015). As for molecular solids, a partial phase transition was proposed for the α and β polymorphs of DL-norleucine, based on molecular dynamics simulations at temperatures for which the phases are stable and metastable (van den Ende & Cuppen, 2014). We are not aware of the full structural characterization of an arrested phase transition in a molecular crystal.

In what follows, we report the preparations of $(^n\mathrm{Bu_4N})_2[\mathrm{Co(Or)_2(H_2O)_2}]\cdot 1.8\mathrm{H_2O}$, (**1**), and $(^n\mathrm{Bu_4N})[\mathrm{Co(Or)_2(bipy)}]\cdot 3\mathrm{H_2O}$, (**2**), the hydrated tetrabutylammonium salts of simple Co^{II} and Co^{III} coordination complexes. For (**2**), we report its phase transition from a room-temperature dynamically disordered monoclinic phase to a low-temperature ordered but twinned triclinic phase. Upon cycling, this phase transition was observed to halt with part of the sample in each phase. This double-phase sample was characterized structurally using single-crystal X-ray diffraction techniques based on in-house measurements, and the single composite diffraction pattern yielded two high-quality structure analyses. In addition to permitting accurate characterization of both phases, the analysis of the two-phase sample using nominally single-crystal techniques permitted the characterization of the monoclinic phase at a temperature at which in principle it would not normally exist.

2. Experimental

2.1. General

All reagents were used as received without further purification. The IR spectra of compounds (**1**) and (**2**) were recorded in the 4000–300 $\mathrm{cm^{-1}}$ range on a PerkinElmer Spectrum 100 FT–IR spectrophotometer equipped with an ATR accessory. Elemental analyses were performed on a PerkinElmer 240 Series II microanalyzer.

2.2. Syntheses

2.2.1. Preparation of $(^n\mathrm{Bu_4N})_2[cis\text{-}\mathrm{Co(Or)_2(H_2O)_2}]\cdot x\mathrm{H_2O}$, (1). A mixture of $\mathrm{CoCO_3\cdot H_2O}$ (0.50 g, 3.65 mmol), orotic acid hydrate ($\mathrm{C_5H_4O_4N_2\cdot H_2O}$; 1.27 g, 7.30 mmol) and water (50 ml) was stirred for 2 h in air allowing gas evolution, then a solution of $^n\mathrm{Bu_4NOH}$ (1.5 *M*, 40%) in $\mathrm{H_2O}$ (7.15 mmol, 4.76 ml) was added. The resulting suspension was filtered and left standing for evaporation. Orange crystals of (**1**) were obtained from the filtrate after several days in 75% yield (2.48 g, 2.59 mmol). Analysis calculated (%) for $(^n\mathrm{Bu_4N})_2[cis\text{-}\mathrm{Co(Or)_2(H_2O)_2}]\cdot 1\mathrm{H_2O}$ – *i.e.* one molecule of unligated $\mathrm{H_2O}$ per formula unit – $\mathrm{C_{42}H_{82}CoN_6O_{11}}$: C 55.67, H 9.12, N 9.28; found: C 55.79, H 8.97, N 9.18. IR ($\mathrm{cm^{-1}}$): 2960 (*s*), 2876 (*m*), 1643 (*s*), 1585 (*s*), 1563 (*s*), 1464 (*s*), 1361 (*s*), 1014 (*m*), 878 (*m*), 786 (*s*). **Note**: the crystal structure determination produced a model with 1.8 free $\mathrm{H_2O}$ units per formula unit of the complex for the crystal from which the diffraction data were measured.

2.2.2. Preparation of $(^n\mathrm{Bu_4N})[\mathrm{Co(Or)_2(bipy)}]\cdot 3\mathrm{H_2O}$, (2). A mixture of orotic acid hydrate ($\mathrm{C_5H_4O_4N_2\cdot H_2O}$; 5.855 g, 33.63 mmol), $\mathrm{CoCO_3\cdot H_2O}$ (2 g, 14.60 mmol) and water (190 ml) was stirred for 2 h at 333 K. The flask was evacuated periodically by means of a water pump and then left stirring overnight. After that time, the flask was evacuated once more and then a solution of $^n\mathrm{Bu_4NOH}$ (11 ml, 16.81 mmol, 1.53 *M*, 40%) was added. The resulting suspension was stirred at room temperature for 1 h. 2,2′-Bipyridine (2.6261 g, 16.815 mmol) was added and, after 15 min, $\mathrm{H_2O_2}$ (2.06 ml, 20.2 mmol, 30%, 9.79 *M*) was added. The colour of the mixture turned to deep wine red and the suspension was filtered. Red crystals of (**2**) were obtained from the solution after 15 h in 40% yield (4.8376 g, 5.91 mmol). Posterior evaporation of the remaining solution produced more of compound (**2**), but mixed with other cobalt orotate compounds. Analysis calculated (%) for $\mathrm{C_{36}H_{54}CoN_7O_{11}}$, *cis*-(**2**): C 52.74, H 6.64, N 11.96; found: C 52.95, H 6.68, N 12.19. IR ($\mathrm{cm^{-1}}$): 3388 (*m*), 2964 (*m*), 2788 (*m*), 1642 (*s*), 1610 (*s*), 1462 (*s*), 1398 (*s*), 1351 (*s*), 1294 (*s*), 1151 (*m*), 1027 (*m*), 948 (*m*), 882 (*m*), 767 (*s*), 594 (*m*), 454 (*s*), 418 (*s*).

2.3. Thermal analysis measurements

Thermal analysis measurements were performed using a differential scanning calorimeter (DSC) Q1000 from TA Instruments equipped with a liquid-nitrogen cooling system, allowing temperatures to reach 100 K. A powder sample of approximately 10 mg mass was sealed in a nonhermetic flat aluminium capsule. Thermograms, both on heating and cooling, were performed at a scan rate of 10 K $\mathrm{min^{-1}}$. Temperature and enthalpy calibrations were made with an indium standard sample by using its melting data. Comparison with expected values shows very small changes in the onset temperature (<0.1 K) and in the enthalpy content (<1.5%). In order to determine the heat-capacity anomalies and their enthalpy contents, a smooth baseline, obtained by fitting the thermograms outside of the transition temperature range with a linear or low-degree polynomial function, was subtracted from the thermogram. In the present case, anomalies are small and diffuse, and this procedure, using a more or less arbitrary baseline, increases significantly the uncertainty in the enthalpy determination; thus, the reported values must be considered as rough estimates.

2.4. Single-crystal X-ray structure determination of compounds (1) and (2)

Single-crystal diffraction data were measured using Oxford Diffraction Xcalibur S3 four-circle diffractometers equipped with graphite-monochomated Mo $K\alpha$ radiation (λ = 0.71073 Å). Oxford Instruments CryoJetLT and CryoJetHT nitrogen-flow temperature controllers were used to maintain the samples of compound (**2**) at set temperatures. The samples were mounted on Mitegen supports and covered with Fomblin oil. Multiscan absorption correction procedures were applied to the data and used to derive error models (Blessing, 1995, 1997). The crystallographic parameters and refinement residuals for all of the structure analyses are given in Tables 1 and 2.

2.4.1. $(^n\mathrm{Bu_4N})_2[cis\text{-}\mathrm{Co(Or)_2(H_2O)_2}]\cdot x\mathrm{H_2O}$, (1). The crystal structure determination of (**1**) at room temperature produced

Table 1
Crystal data and refinement quality indicators for the structure analysis of (^{n}Bu$_4$N)$_2$[*cis*-Co(Or)$_2$(H$_2$O)$_2$]·1.8H$_2$O, (**1**).

Structure	(**1**)
CCDC reference	1560738
Formula	$C_{42}H_{80}CoN_6O_{10}\cdot 1.8H_2O$
Formula weight	920.47
Crystal	1
Crystal history	as prepared
T (K)	295 (2)
Crystal condition	single
Crystal system	triclinic
Space group	$P\overline{1}$
Z	2
H[a] (H$_2$O, N—H) located and refined	mixed: some water H located and refined (xyz and U_{iso}), some not located
Resolution[b] (Å)	0.77
No. data, total	26453
Independent data	11477
R_{int}	0.0343
Parameters	597
Restraints	8
$R1$ [$F^2 > 2\sigma(F^2)$]	0.0469
$wR2$ (all data used)	0.1034
Quality-of-fit	1.033
a (Å)	12.3630 (4)
b (Å)	12.6281 (5)
c (Å)	16.3765 (6)
α (°)	89.948 (3)
β (°)	95.460 (3)
γ (°)	96.455 (3)
V (Å^3)	2528.86 (16)
$\Delta\rho_{max}$,$\Delta\rho_{min}$ (e Å^{-3})	0.477, −0.319

Notes: (*a*) H atoms bonded to O or N atoms. Whether or not these H atoms are located and refined is an indicator for the reliability of the structure analysis. (*b*) Resolution is estimated as the minimum Bragg spacing to which data are at least 95% complete, based on the Laue group.
Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2006), *CrysAlis RED* (Oxford Diffraction, 2006), *SIR92* (Altomare *et al.*, 1994), *SHELXL2014* (Sheldrick, 2015) and *DIAMOND* (Brandenburg & Putz, 2004).

a structural model with 1.8 units of unligated water per formula unit. Structure solution and refinement were routine except for two disorder assemblies whose atomic sites were located in Fourier maps and refined with partial occupancies for the respective disorder groups. The first disorder assembly involved a γ-methylene group (C23) of one of the ^{n}Bu$_4$N$^+$ cations. Two sites were located for the C atom and their occupancies were initially refined with a constraint to a total population of 1.0. The occupancies refined to values close to $\frac{5}{6}$ and $\frac{1}{6}$, and so were fixed at these values for the final refinement. H atoms for the disordered congeners were placed at calculated positions and refined as riding atoms, with displacement-parameter constraints. The partially occupied H-atom sites included those of the adjacent CH$_2$ and CH$_3$ groups at atoms C22 and C24. For the latter, the H-atom coordinates were calculated so as to have staggered conformations with respect to atom C23. A second disorder assembly was found for the interstitial O4*W* water site. The populations of the two sites were initially refined with a constraint to a sum of 1.0. The resulting population parameter converged to a value of 0.798 (5) for O4*WA*, and so the site-occupancy factors were fixed at 0.8 (O4*WA*) and 0.2 (O4*WB*) for the final refinement. Since the remaining interstitial water site, at O3*W*, makes an impossibly short contact with an inversion-related congener of O4*WB*, O3*W* was treated as a member of the disorder assembly and also had a fixed population of 0.8 in the final refinement. The H atoms attached to O3*W*, O4*WA* and O4*WB* were not located. All of the H atoms of the ^{n}Bu$_4$N$^+$ cations were placed at calculated positions based on geometry for CH$_2$ and on local slant Fourier calculations for the CH$_3$ groups not affected by disorder. U_{iso}(H) values for methylene and methyl H atoms were constrained to 1.2 and 1.5 times the U_{eq} values of their respective carrier C atoms. H atoms of the orotate groups and ligated water molecules were located in difference Fourier maps and refined with independent coordinates and with isotropic displacement parameters constrained to 1.2U_{eq} of the carrier atom for the orotate H atoms and with H-atom U_{iso} values freely refined for the aqua ligands.

2.4.2. (^{n}Bu$_4$N)[Co(Or)$_2$(bipy)]·3H$_2$O, (2). We report five structure analyses for compound (**2**). The structure of the crystals as prepared is monoclinic, space group $P2/n$, analyzed at T = 277 K. For this analysis, *i.e.* (**2a**), all non-water H atoms were placed at calculated positions and refined as riders, with U_{iso} values set at 1.2 (nonmethyl) or 1.5 (methyl) times the U_{eq} values of the respective parent atoms. Water H atoms were located in a difference map and refined freely. The atoms of both of the independent ^{n}Bu groups of the ^{n}Bu$_4$N$^+$ cation showed increasing displacements on going from the α- to the β-C atoms, but the terminal –CH$_2$CH$_3$ group at atoms C22 and C23 showed quite pronounced transverse displacement accompanied by the shortened 'apparent' bond length of 1.182 (6) Å, normally attributed to libration.

When the same crystal is cooled to T = 100 K, it undergoes a transition to a triclinic phase (space group $P\overline{1}$), with twinning. The initial monoclinic phase, (**2a**) (Table 2), was analyzed using routine single-crystal X-ray procedures. The triclinic phase at T = 100 K, (**2b**), was treated as a 'nonmerohedral twin' (Herbst-Irmer, 2016) and the structure was refined using a combined data set (*SHELXL2014* 'HKLF 5') with the residuals given in Table 2. The twin ratios were calculated as 0.533 and 0.467 by the data integration program, and the transformation, in terms of cell-axis vectors, from the first to the second component, is:

$$\begin{pmatrix} a2 \\ b2 \\ c2 \end{pmatrix} = \begin{bmatrix} -0.9989 & 0.0010 & 0.0025 \\ -0.0445 & 1.0051 & -0.0365 \\ -0.0020 & 0.0030 & -1.0002 \end{bmatrix} \begin{pmatrix} a1 \\ b1 \\ c1 \end{pmatrix}$$

The unit cell and setting used for triclinic structure (**2b**) were chosen to correspond as closely as possible to the unit cell and setting of the initial monoclinic phase (**2a**). As a result, the triclinic cell is not the conventional reduced cell that would have been used if the structure of (**2b**) had been done independently of its monoclinic relative. The standard unit cell is a = 9.3791 (8), b = 12.9054 (8), c = 16.1290 (12) Å, α = 102.898 (6), β = 91.276 (6), γ = 91.472 (6)° and V = 1901.6 (2) Å^3. The transformation from the unit cell used to the standard reduced cell and setting, in terms of unit-cell basis vectors, is the following, in which the primed axes are those of the conventional cell:

Table 2
Crystal data and refinement quality indicators for the five determinations of the structure of (^{n}Bu$_4$N)[Co(Or)$_2$(bipy)]·3H$_2$O, (**2**).

Molecular formula $C_{36}H_{54}CoN_7O_{11}$ or $(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)_2]\cdot 3H_2O$; M_r = 819.79.

Structure	(**2a**)	(**2b**)	(**2c**)	(**2d**)	(**2e**)
CCDC reference	1560739	1560740	1560741	1560742	1560743
Crystal	1	1	2	2	
Crystal history	as prepared	after one transition monoclinic to triclinic	following one full cycle monoclinic to triclinic to monoclinic	following 1.5 full cycles, monoclinic to triclinic to monoclinic to mixed monoclinic/triclinic	
T (K)	277 (1)	100 (1)	220 (1)	170 (1)	170 (1)
Crystal condition	single	twin	single	multicrystal	
Crystal system	monoclinic	triclinic	monoclinic	monoclinic	triclinic
Space group	$P2/n$	$P\overline{1}$	$P2/n$	$P2/n$	$P\overline{1}$
Z	2	2	2	2	2
H[a] (H_2O, N—H) located and refined	xyz and U_{iso} refined	no	xyz and U_{iso} refined	xyz refined and U_{iso} constrained	xyz refined and U_{iso} constrained
Resolution[b] (Å)	0.84	0.78	0.77	0.77	0.84
No. data, total	10924	15136	21720	22527	25546
Independent data	4564	15136	4673	4643	6796
R_{int}	0.0664	twin[c]	0.0499	0.1647	0.1521
Parameters	264	501	264	282	524
Restraints	0	0	0	39	0
$R1$ [$F^2 > 2\sigma(F^2)$]	0.0549	0.0949	0.0480	0.0664	0.0617
$wR2$ (all data used)	0.1100	0.2481	0.1294	0.1646	0.1608
Quality-of-fit	1.021	1.439	1.070	1.075	1.052
a (Å)	13.1679 (12)	12.9054 (8)	13.0259 (4)	13.0080 (8)	13.0155 (15)
b (Å)	9.3413 (9)	9.3791 (8)	9.3504 (3)	9.3320 (6)	9.4028 (14)
c (Å)	16.3388 (14)	16.1290 (12)	16.3308 (5)	16.3753 (12)	16.2640 (17)
α (°)	90	88.724 (6)	90	90	88.794 (11)
β (°)	102.669 (9)	102.898 (6)	103.847 (3)	104.364 (7)	103.054 (9)
γ (°)	90	88.528 (6)	90	90	88.687 (11)
V, Å^3	1960.8 (3)	1901.6 (2)	1931.24 (11)	1925.7 (2)	1937.8 (4)
$\Delta\rho_{max}$,$\Delta\rho_{min}$ (e Å^{-3})	0.478, −0.309	2.876, −0.771	1.044, −0.915	0.741, −0.731	1.630, −0.899

Notes: (*a*) H atoms bonded to O or N atoms. Whether or not these H atoms are located and refined is an indicator for the reliability of the structure analysis. (*b*) Resolution is estimated as the minimum Bragg spacing to which data are at least 95% complete, based on the Laue group. (*c*) Structure (**2b**) was refined using data from two domains in the same refinement. Traditional data merging was not performed.
Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2006), *CrysAlis RED* (Oxford Diffraction, 2006, 2009), *CrysAlis PRO* (Agilent, 2011), *SIR92* (Altomare *et al.*, 1994), *SHELXL2014* (Sheldrick, 2015) and *DIAMOND* (Brandenburg & Putz, 2004).

$$\begin{pmatrix} a' \\ b' \\ c' \end{pmatrix} = \begin{bmatrix} 0 & -1 & 0 \\ 1 & 0 & 0 \\ 0 & 0 & 1 \end{bmatrix} \begin{pmatrix} a \\ b \\ c \end{pmatrix}$$

Following the thermal analysis, which suggested that the monoclinic-to-triclinic transition occurs in the 160–220 K temperature range, we explored the diffraction pattern of a second crystal of (**2**) in the same range, beginning at the higher temperature. Firstly, the unit cell was determined at T = 220 K, confirming the exclusive presence of the monoclinic phase. The temperature was then cycled to T = 170 K and back in increments of 10 or 20 K, with an axial photo of [010] being taken at each temperature. Photos were made, in this order, for T = 220, 200, 190, 180, 170, 190, 200 and 220 K, and were taken after a 15 min interval at each temperature, except for the final T = 220 K, for which photos were taken after 20, 70 and 120 min. The photos showed progressive spot splitting as the temperature was lowered, and eventually showed the loss of mirror symmetry perpendicular to this axis. When the temperature was raised, the splitting progressively disappeared, with the axial photo returning to nearly its original appearance when the temperature had once again reached T = 220 K.

At this point, a full structure analysis was conducted at T = 220 K, *i.e.* (**2c**) (Table 2); this confirmed that the structure at this point was identical to the original monoclinic structure. The H atoms of the two independent unligated water sites were located in a difference Fourier map, and their positional and isotropic displacement parameters were refined freely.

The temperature was then lowered to T = 170 K, and a redundant sphere of data was gathered. The diffraction pattern revealed the presence of both the monoclinic and the triclinic phases. Because of the high redundancy, it was possible to isolate nearly complete data sets with reflections unique to each of the phases. Refinements were conducted routinely for both [*i.e.* monoclinic (**2d**) and triclinic (**2e**)]. For monoclinic (**2d**), the ^{n}Bu group C20—C21—C22—C23 was found to have its terminal ethyl fragment disordered two ways, with the majority component (75%) having an *anti* conformation, as in the higher-temperature monoclinic structures, and with the minor component in a *syn* conformation, as in one of the ^{n}Bu groups of the triclinic structure. Similarity restraints were applied to the Cγ—Cδ distances and to the 1,3-C$\beta\cdots$Cδ distances. Similarity restraints were also used for the anisotropic displacement parameters of the Cγ and Cδ atoms of the disordered congeners. As was described above for triclinic structure (**2b**), the unit cell that was used for (**2e**) was chosen to correspond as closely as possible to that of the monoclinic structure. The transformation from the triclinic cell

used to the conventional reduced-cell setting is the same as that given for (**2b**), and in the case of (**2e**) gives the conventional cell a = 9.4028 (14), b = 13.0155 (15), c = 16.2640 (17) Å, α = 103.054 (9), β = 91.206 (11), γ = 91.313 (11)° and V = 1937.8 (4) Å^3.

3. Results and discussion

$CoCO_3{\cdot}H_2O$ reacts with orotic acid monohydrate, $C_5H_4N_2O_4{\cdot}H_2O$, and ^{n}Bu$_4$NOH in water at room temperature, giving different products depending on the reaction conditions. When the reaction was carried out in water with Co:H_2Or:^{n}Bu$_4$NOH proportions of 1:2:2, the anionic Co^{II} derivative (^{n}Bu$_4$N)$_2$[*cis*-Co(Or)$_2$(H_2O)$_2$]·2H_2O, (**1**), was formed (Equation 1).

$$CoCO_3 + 2H_2Or + 2NBu_4OH \xrightarrow{H_2O} (NBu_4)_2[cis\text{-}Co(Or)_2(H_2O)_2] + CO_2\uparrow \quad (1)$$

The same reaction for Co:H_2Or:^{n}Bu$_4$NOH ratios of 1:2:1 in the presence of 2,2′-bipyridine (bipy) and H_2O_2 (Equation 2)

$$CoCO_3 + 2H_2Or + NBu_4OH + bipy \xrightarrow{H_2O,H_2O_2} (NBu_4)[cis\text{-}Co(Or)_2(bipy)] + CO_2\uparrow \quad (2)$$

leads to the formation of the Co^{III} salt (^{n}Bu$_4$N)[*cis*-Co(Or)$_2$(bipy)]·3H_2O, (**2**), whose properties and phase transition are the main subject of this report. Compound (**1**) is chemically isostructural with its nickel analogue (Falvello *et al.*, 2007), which was prepared using the metal chloride ($NiCl_2$) as starting material instead of the carbonate. The use of the metal carbonate instead of the chloride reduces the amount of ^{n}Bu$_4$NOH needed and eliminates the formation of residual products (^{n}Bu$_4$NCl).

3.1. Crystal structure of compound (1)

Crystals of compound (**1**) are isomorphous with the analogous Ni complex, whose structure has been discussed in detail (Falvello *et al.*, 2007). The distorted octahedral environment of atom Co1 (Fig. 1) has the two aqua ligands *cis* to each other, and the two chelating Or^{2-} ligands are disposed such that their coordinated N atoms are mutually *trans* and their ligated carboxylate O atoms *cis*. As was discussed for the corresponding Ni complex, crystallization from an environment poor in hydrogen-bonding possibilities leads to isolation of the *cis* isomer, in which two intramolecular hydrogen bonds add stability to the structure. In the absence of other hydrogen-bonding partners, (**1**) also enters into self-complementary aggregation patterns, namely an $R_2^2(8)$ interaction with the N13—H13 group as donor and atom O14 at ($-x+1$, $-y+1$, $-z+1$) as acceptor, and an $R_2^2(12)$ cycle involving the N3—H3 group and atom O17 – that is, two different orotate ligands – and the molecule at ($-x$, $-y+1$, $-z$) (see Fig. S3 in the supporting information). The hydrogen-bonded chain thus formed propagates along [101].

3.2. Crystal structures of (2)

In the monoclinic room-temperature form of compound (**2**) – we refer to this analysis of the as-prepared crystal as structure (**2a**) – the ^{n}Bu$_4$N$^+$ cation and the six-coordinate Co^{III} complex both reside on crystallographic twofold axes, as does one of the two independent unligated water molecules. The anionic six-coordinate complex (Fig. 2) presents an arrangement of orotate ligands similar to that found for Co^{II} complex (**1**), with the coordinated N1 atoms of the two ligands *trans* to each other and the coordinated carboxylate O7 atoms mutually *cis*. The chelating bipy ligand occupies the remaining two coordination sites. Except for the differences in the

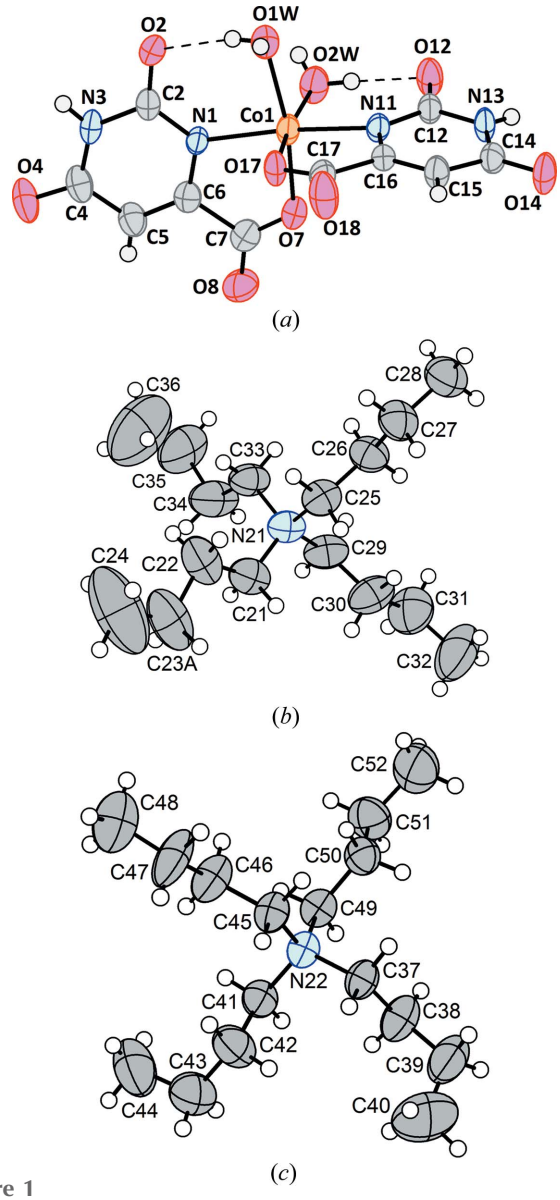


Figure 1
(*a*) The anion, (*b*) the cation centred at N21 and (*c*) the cation centred at N22 from the crystal structure of (^{n}Bu$_4$N)$_2$[*cis*-Co(Or)$_2$(H_2O)$_2$]·1.8H_2O, (**1**). In all three drawings, non-H atoms are represented by their 50% probability displacement ellipsoids. In part (*b*), the minor-disordered congener at the C23 site has been omitted, along with the corresponding H atoms.

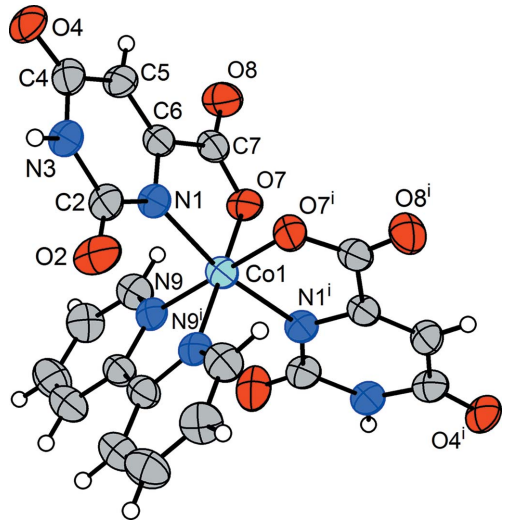


Figure 2
The anion from the monoclinic crystal structure of (NBu_4)[*cis*-$Co(Or)_2(bipy)]\cdot 3H_2O$, (**2a**). Non-H atoms are represented by their 50% probability displacement ellipsoids. [Symmetry code: (i) $-x+\frac{1}{2}, y, -z+\frac{3}{2}$.]

Co1—N and Co1—O bond lengths that accompany the change of oxidation state of the Co centre, the geometries of complex salts (**1**) and (**2**) are similar.

A narrow channel parallel to [101] and at a height of $y=\frac{1}{2}$ is occupied by ordered water molecules that act as hydrogen-bond donors and acceptors in interactions with the orotate ligands. There is one relatively weak hydrogen bond between the two free water molecules, but hydrogen bonding involving only water molecules along the water-occupied channel is not an important feature of this structure. This can be contrasted to the water wire that has been found to be a proton conductor in a molecular crystal involving a Mn^{II} citrate cubane polymer (Capelli *et al.*, 2013). In (**2a**), units of the Co^{III} complex occupy a slab perpendicular to the *b* axis (Fig. 3), and hydrogen bonding, albeit weak, joins these anions (blue in the figure) and the two independent water molecules (green) into a sheet. This sheet and the hydrophobic cations (red) are segregated into alternating layers along the *b* axis, with the cations in a layer centred at $y = 0.0$.

The $^{n}Bu_4N^+$ cation in (**2a**), which is the protagonist of the phase transition that befalls this crystal, merits a mention. At room temperature, two of the terminal ethyl fragments of the ^{n}Bu groups have their displacement ellipsoids elongated in a fashion that suggests concerted motion of this group, most likely libration in what is a typical example of dynamic disorder. This can be seen in Fig. 4, where the displacement ellipsoids for atoms C22 and C23, and their symmetry relatives, are notably more prolate, with transverse elongation, than those of the other C atoms of the ^{n}Bu chains. (When a single atomic position is modelled for sites such as these, they are not flagged as disordered entities in the CSD.)

When a crystal of compound (**2**) is cooled to 100 K, it undergoes a reversible transition to a triclinic structure, *i.e.* (**2b**), that is a minor modification of the monoclinic room-temperature structure, with the only significant difference at the molecular level being a separation of the prolate symmetry relatives of atoms C22 and C23 into fragments not related by the twofold axis. As is generally expected for a conservative monoclinic-to-triclinic transformation, the crystal becomes a twin. The structure was solved *ab initio* and refined using the usual protocol for so-called 'nonmerohedral twins' (Herbst-Irmer, 2016), with the diffraction data integrated using two orientation matrices for the two twin components, and with overlapped reflections separated as well as the software is able to do. The asymmetric unit in (**2b**) comprises one full cation, one full anion and three water molecules. The reference asymmetric unit for (**2b**) was chosen to correspond as closely as possible to that of monoclinic (**2a**), with 'A' appended to the names of the newly independent atoms – those that are related to the reference asymmetric unit by a twofold axis in the monoclinic structure. The complex anion in (**2b**) is essentially identical to that in (**2a**) (Fig. 5). It can be seen that the displacement ellipsoids for both ions behave well in (**2b**), except for effects attributable to the twinning. The $^{n}Bu_4N^+$

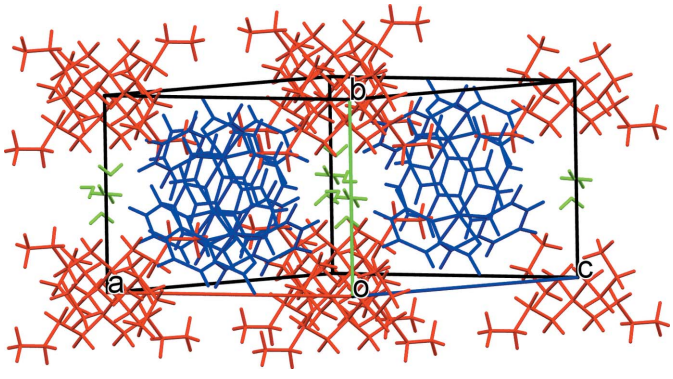


Figure 3
The packing in monoclinic $(^{n}Bu_4N)$[*cis*-$Co(Or)_2(bipy)]\cdot 3H_2O$, (**2a**), showing the separation of hydrophilic and hydrophobic fragments into layers perpendicular to [010]. Blue represents [*cis*-$Co(Or)_2(bipy)]^-$, red $^{n}Bu_4N^+$ and green H_2O.

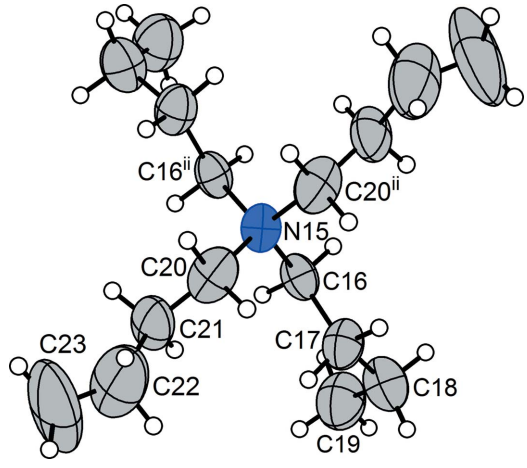


Figure 4
The $^{n}Bu_4N^+$ cation in monoclinic (**2a**), with non-H atoms represented by 50% probability displacement ellipsoids. The prolate ellipsoids for atoms C22 and C23 can be seen. [Symmetry code: (ii) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.]

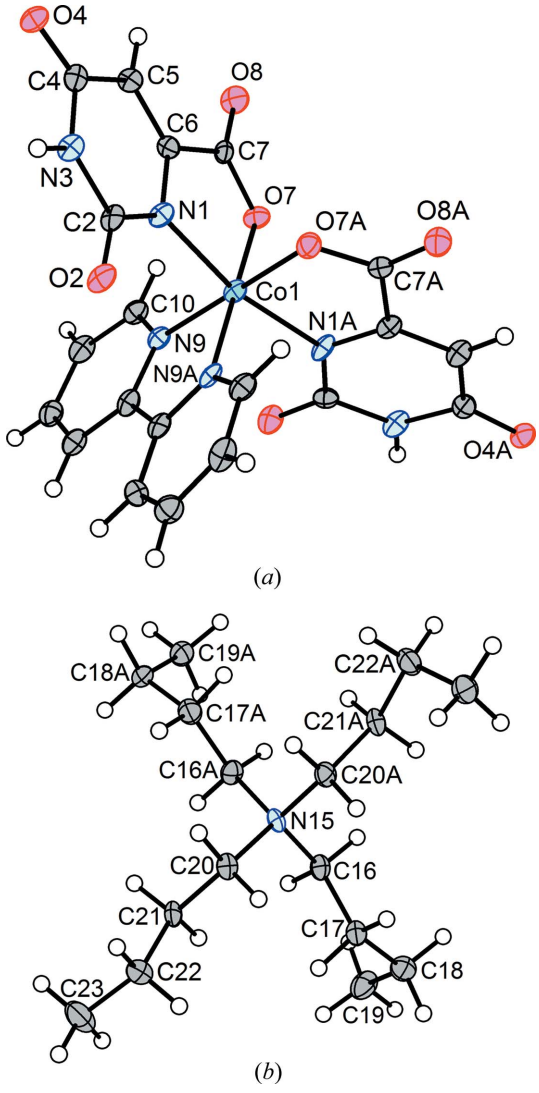


Figure 5
(*a*) The [*cis*-Co(Or)$_2$(bipy)]$^-$ anion and (*b*) the ^{n}Bu$_4$N$^+$ cation in (**2b**), with non-H atoms represented by 50% probability displacement ellipsoids in both parts.

cation is conformationally different at one extreme of one of the ^{n}Bu chains. Specifically, the newly independent terminus of the chain at C22*A*/C23*A* has been reoriented to give a *syn* conformation about the C21*A*—C22*A* bond, while the original chain at C22/C23 is still *anti* in the triclinic structure, as it was in the monoclinic mother phase. Fig. 6 shows superposed drawings of the cations from (**2a**) (red) and (**2b**) (blue). Three of the ^{n}Bu groups are almost identical in the two structures. The groups that had large prolate displacement ellipsoids (C22 and C23 at the right of the figure) are those that have segregated conformationally as indicated above.

The general features of the packing in (**2b**) (Fig. S4 in the supporting information) are essentially unchanged from the original structure (**2a**). The major features of the extended structure are a segregation of the hydrophobic cation and more hydrophilic anion layers, along with a line of water molecules weakly hydrogen bonded to the anions, running along [101].

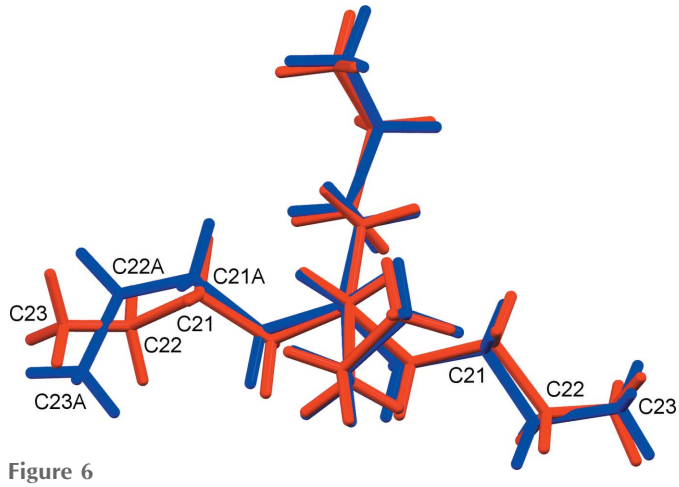


Figure 6
Superposition of the ^{n}Bu$_4$N$^+$ cations from monoclinic (**2a**) (red) and triclinic (**2b**) (blue). The two sets of atoms labelled C22 and C23 are related by symmetry in (**2a**), while C22*A* and C23*A* are at the new positions for one of these fragments in the triclinic structure.

The quality indicators for the refinement of (**2b**) are not ideal (Table 2). We use this analysis to demonstrate that the transformation has taken place under the conditions described and to establish its nature. A better refinement was achieved for triclinic (**2e**) (see below). In addition, more accurate geometrical parameters for the anion and cation are available from the refinements of monoclinic (**2a**), (**2c**) and (**2d**). Regarding the ^{n}Bu$_4$N$^+$ cation, its geometries have already been established in some 4742 previously published structure analyses recorded in the CSD.

That the original structure (**2**) is monoclinic with dynamic disorder and not triclinic without disorder and with only a slight deviation from monoclinic symmetry is clear from the fact that a transition to triclinic, accompanied by twinning, occurs on lowering the temperature. That transition, directly observable in the diffraction itself, is reversed when the temperature is raised again, and a single-domain monoclinic structure can be analyzed from the same sample after cycling

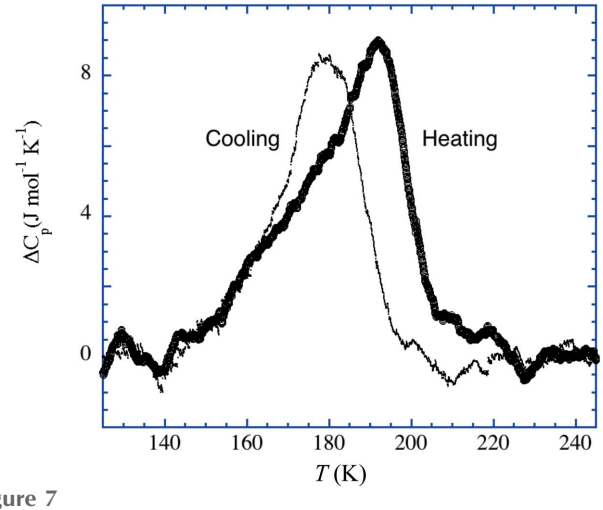


Figure 7
Heat-capacity anomalies measured by DSC for both heating (thick line) and cooling (thin line) thermograms.

Table 3
Comparison of geometric parameters (Å, °) for the variable ^{n}Bu group in the structures of (^{n}Bu$_4$N)[Co(Or)$_2$(bipy)]·3H$_2$O, (**2**).

Codes: m = monoclinic and t = triclinic.

Structure	(**2a**) (m)	(**2b**) (t)	(**2c**) (m)	(**2d**) (m)[b]	(**2e**) (t)
T (K)	277 (1)	100 (1)	220 (1)	170 (1)	170 (1)
C21—C22	1.552 (5)	1.52 (3)	1.532 (4)	1.544 (5)	1.535 (5)
C21*A*—C22*A*		1.54 (3)			1.546 (5)
C22—C23[a]	1.182 (6)	1.52 (3)	1.321 (7)	1.512 (7)	1.501 (6)
C22*A*—C23*A*		1.49 (3)			1.498 (6)
C22 principal MSDA (Å^2)	0.3296, 0.1017, 0.0544	0.0281, 0.0246, 0.0180	0.1866, 0.0627, 0.0301	0.0533, 0.0358, 0.0189	0.0440, 0.0321, 0.0267
C23 principal MSDA (Å^2)	0.5481, 0.1254, 0.0527	0.0562, 0.0307, 0.0182	0.4189, 0.0769, 0.0353	0.0778, 0.0558, 0.0267	0.0881, 0.0524, 0.0279
C22*A* principal MSDA (Å^2)		0.0343, 0.0250, 0.0153			0.0613, 0.0428, 0.0306
C23*A* principal MSDA (Å^2)		0.0339, 0.0264, 0.0179			0.0682, 0.0462, 0.0351
N15—C20—C21—C22	168.3 (3)	166.1 (17)	168.7 (2)	170.1 (3)	166.7 (3)
N15—C20*A*—C21*A*—C22*A*		164.2 (17)			163.4 (3)
C20—C21—C22—C23	150.6 (8)	174.9 (19)	164.5 (6)	173.8 (4)	173.7 (3)
C20*A*—C21*A*—C22*A*—C23*A*		68 (3)			68.6 (5)

Notes: (*a*) where severely affected by dynamic disorder, as in (**2a**) and (**2c**), these values are referred to as 'apparent distances'. (*b*) The parameters given for monoclinic (**2d**) are those of the major-disorder component, *i.e.* C20—C21—C22*A*—C23*A*.

the temperature. Clearer evidence for dynamic disorder is presented below.

3.3. Characterization of the phase transition by thermal analysis

The heat-capacity anomalies determined by differential scanning calorimetry (DSC) are shown in Fig. 7 for both heating and cooling thermograms. These small broad anomalies present their maximum temperatures at around 192 and 177 K, respectively, highlighting the first-order character of the transition, with a hysteresis of 13 K at a 10 K min^{-1} scan rate. These temperatures and the hysteresis are in agreement with the results of the X-ray diffraction measurements, which also indicated that the transition occurred roughly within the temperature range 170–200 K.

The calculated enthalpy (entropy) contents, after subtracting the baseline, are roughly 0.28 (1.56 J mol^{-1} K^{-1}) and 0.21 kJ mol^{-1} (1.23 J mol^{-1} K^{-1}) for the heating and cooling anomalies, respectively. These values are small, also in agreement with the diffraction results, which reveal that the structural changes consist of minor conformational adjustments at the periphery of the ^{n}Bu$_4$N$^+$ cation.

3.4. Arrested phase transition

As just described, DSC established more accurately the temperature range in which the transition of (**2**) from monoclinic to triclinic takes place. The heat-capacity anomalies, with maxima at 177 (cooling) and 192 K (heating), point to a first-order transition with hysteresis. On exploring this reversible phase transition further, using single-crystal diffraction with a fresh crystal, a more complex behaviour was revealed.

First, a unit-cell determination at *T* = 220 K confirmed that the crystal was monoclinic with the known cell of (**2a**). Then axial photos of [010] were used to follow the transformation accompanied by twinning as the temperature was lowered to 170 K in increments of 10 or 20 K (see *Experimental* for details). The temperature was then raised in similar increments and axial photos revealed that the spot-splitting that

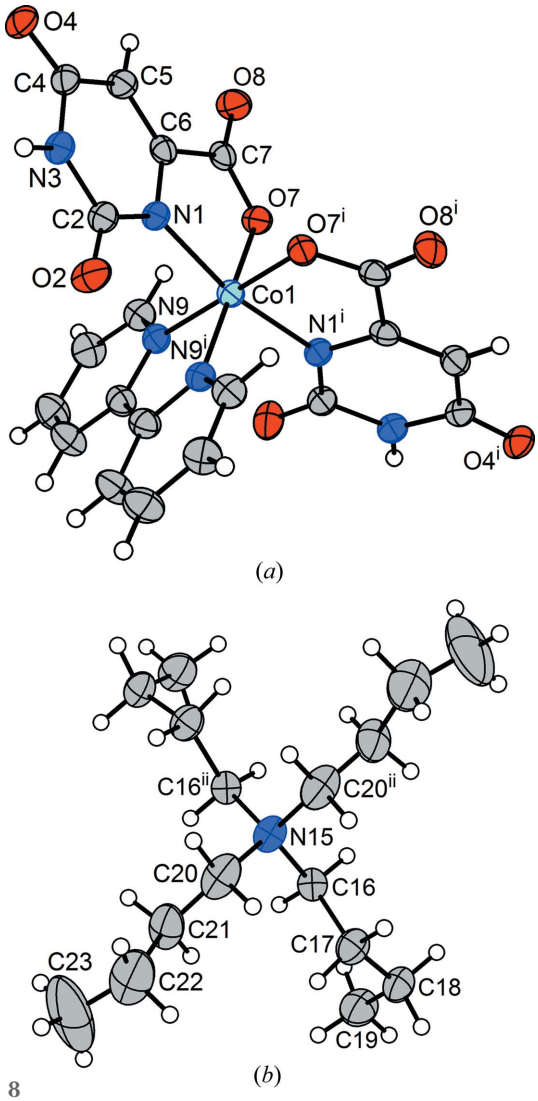


Figure 8
(*a*) The anion and (*b*) the ^{n}Bu$_4$N$^+$ cation from monoclinic form (**2c**) of (^{n}Bu$_4$N)[*cis*-Co(Or)$_2$(bipy)]·3H$_2$O following one full cycle through the phase transition. Non-H atoms are represented by 50% probability displacement ellipsoids in both parts. [Symmetry codes: (i) $-x+\frac{1}{2}, y, -z+\frac{3}{2}$; (ii) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.]

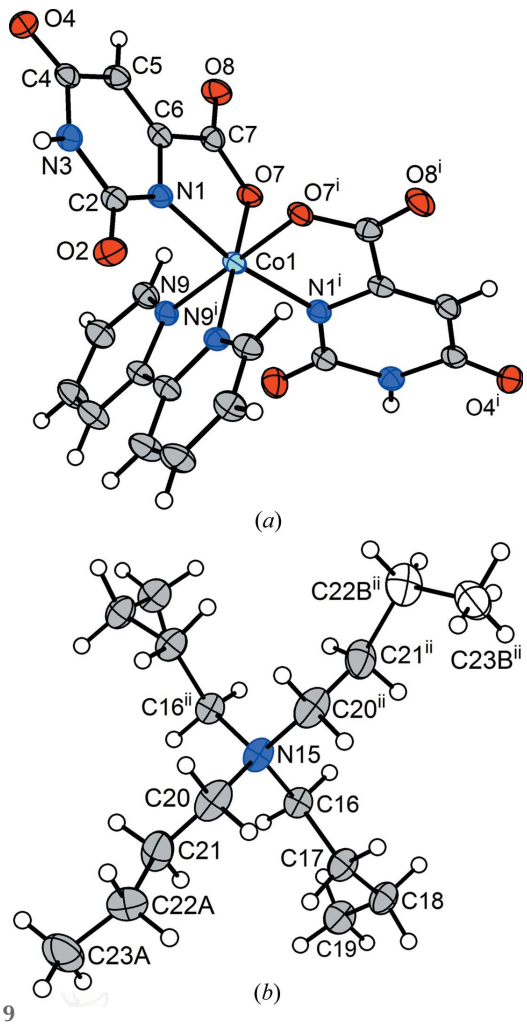


Figure 9
(*a*) The anion and (*b*) the $^{n}Bu_4N^+$ cation from monoclinic phase (**2d**) of the multicrystal of ($^{n}Bu_4N$)[*cis*-Co(Or)$_2$(bipy)]·3H$_2$O. Non-H atoms are represented by 50% probability displacement ellipsoids in both parts. For the $^{n}Bu_4N^+$cation in part (*b*), the major-disorder component (C22*A*—C23*A*) is shown for one ^{n}Bu group and the minor component (C22*B*^{ii}—C23*B*^{ii}) is shown for its symmetry relative. [Symmetry codes: (i) $-x+\frac{1}{2}, y, -z+\frac{3}{2}$; (ii) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.]

had accompanied the transformation to triclinic (**2b**) was reversed as the temperature was raised and the original monoclinic structure was restored.

A complete structure determination, (**2c**), was carried out after the crystal had been warmed again to T = 220 K, and the monoclinic structure was confirmed (Fig. 8) to be isostructural with (**2a**). Following this full cycle of the transition, there were minor indications that the crystal was not of quite the high quality that it had originally possessed – there were a number of inconsistent symmetry equivalents, and the unit-cell angles α and γ, when not constrained to their monoclinic values of 90°, refined to values of 90.168 (2) and 90.117 (2)°, respectively. Nevertheless, the structure was developed and refined to the residuals given for (**2c**) in Table 2. As an indicator of the quality of the data, we note that the H atoms of the unbound water molecules were located in a difference map and refined freely, including their isotropic displacement parameters. Except for effects that can be attributed to the difference in temperature, the structure of (**2c**) is identical to that of the near-room-temperature structure (**2a**).

After this one full cycle of the transition from monoclinic to triclinic and back, lowering the temperature again, directly, to T = 170 K, produced an arrested form of the transition, in which about one-half of the sample once again changed to the triclinic form and the rest remained in the monoclinic structure. (The temperature was lowered from 220 to 170 K over a period of several minutes and then held at T = 170 K for 4 h before the diffraction measurements commenced.) To our knowledge, a result of this entirely unexpected nature has not previously been characterized in detail for a molecular crystal. A case of several structures being characterized from the same sample has been reported recently (Aromí *et al.*, 2016). The difference in the present case is that the crystal remained stable with its two components at 170 K and, furthermore, despite a good deal of reflection overlap it was possible to isolate complete redundant individual data sets for both components using in-house data. Both the monoclinic (**2d**) (Fig. 9) and triclinic (**2e**) (Fig. 10) phases gave high-quality

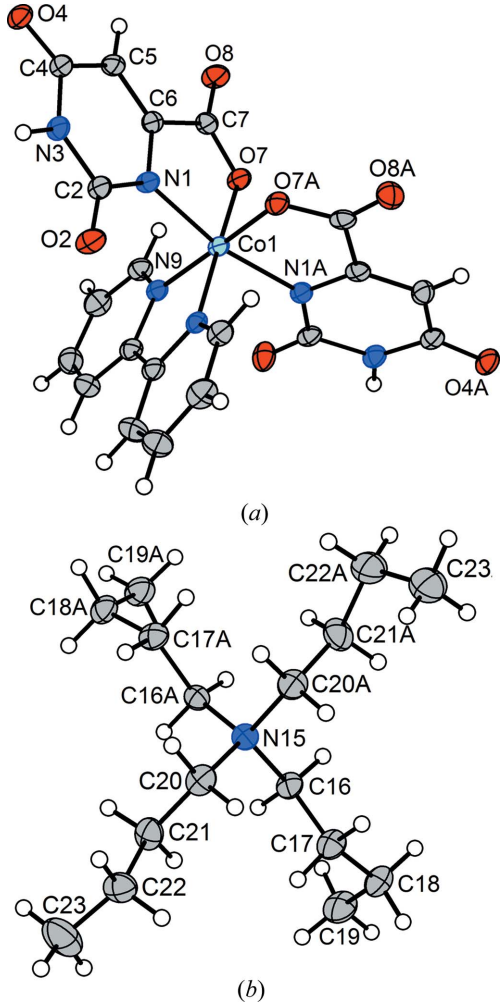


Figure 10
(*a*) The [*cis*-Co(Or)$_2$(bipy)]$^-$ anion and (*b*) the $^{n}Bu_4N^+$ cation from triclinic phase (**2e**) of the multicrystal of ($^{n}Bu_4N$)[*cis*-Co(Or)$_2$(bipy)]·3H$_2$O. Non-H atoms are represented by 50% probability displacement ellipsoids in both parts.

refinements in which the positional parameters of the H atoms attached to free water were refined freely. (The isotropic displacement parameters of these H atoms were constrained to 1.2 times U_{eq} of their bonding partners.)

The diffraction pattern for this final set of measurements revealed just two principal phases, one monoclinic and one triclinic. It appears that the twinning of the triclinic phase that would be expected for a clean transition was not a major feature in this case.

The nature of the phase change can be understood readily with reference to Table 3, which collects the relevant geometrical parameters for the ^{n}Bu group that changes, namely C20—C21—C22—C23. In the monoclinic structure, it is related by a twofold axis to another such chain and it is highly likely that both congeners are affected by dynamic disorder (*vide infra*). In the triclinic structure, the second congener, C20*A*—C21*A*—C22*A*—C23*A*, is not related by symmetry to the first and it is the second congener that undergoes the change. The torsion angle C20*A*—C21*A*—C22*A*—C23*A*, which in the monoclinic structure describes an *anti* conformation (Table 3), is modified to a *syn* arrangement in the triclinic structure. The base unit, *i.e.* C20—C21—C22—C23, retains its *anti* descriptor in the triclinic structure, where no disorder is evident.

Monoclinic phase (**2d**) of the multicrystal that results from the arrested transition gives a structure analysis at T = 170 K with a major component that is nearly identical – but not rigorously so – to those obtained for the monoclinic phases at T = 277 (**2a**) and 220 K (**2c**). A telling difference involves the unique ^{n}Bu group that suffers disorder at higher temperature (C20–C23, Fig. 4). Disorder is reflected in the principal mean-square displacement amplitudes (MSDA) for atoms C22 and C23 (Table 3). As is also clear from Table 3, the foreshortening of the 'apparent' C—C distance that accompanies librational disorder is pronounced at T = 277 K for (**2a**), significant but less exaggerated at 220 K for (**2c**) and observable but small at 177 K for (**2d**). Such variation with temperature discriminates between dynamic and static disorder, and is a strong indicator in this case for dynamic disorder. Being able to make this determination is one of several reasons for not restraining the terminal C—C bond length in the higher-temperature determination.

Possibly more intriguing is that the monoclinic structure (**2d**) at T = 170 K, derived from the sample after the arrested phase transition, has a minor-disordered component with one ^{n}Bu group in a *syn* conformation, as in the triclinic structure that results from the phase transition. We refrain from drawing speculative conclusions, but it may be that the first step in the phase transition is a conformational change in the affected ^{n}Bu group, and that this is then followed by the global change of the sample to the triclinic phase.

The arrested transition, which may well be fortuitous, also permits a more exact comparison between the two phases than is often the case with transitions, because it was possible to characterize the two phases at the same temperature. In actual fact, all five structural results can be superimposed quite well – anion, cation and interstitial water – except for terminal atoms C22*A* and C23*A*, which upon ordering mark the difference in the triclinic phase.

4. Concluding comments

We refer to the partially executed change from monoclinic to triclinic in this case as an *arrested phase transition*. Given that the transition proceeds to completion in both directions in the first cycle, we conclude that in the second cycle, defects are responsible for blocking the advance of the transformation following normal nucleation. We are not attempting to coin a term for this phenomenon. We note that the term 'arrested phase transition' was used in the *Abstract* of an article by Xu & Veblen (1995) describing a transition in haüyne that does not go to completion. The term 'arrested solid–solid phase transition' was also used in the *Title*, but not the text, of an article describing displacements of phase-transition temperatures or pressures in CdS nanocrystals, as compared to the bulk material, as a result of surface characteristics (Haase & Alivisatos, 1992).

The phase transition from a dynamically disordered monoclinic room-temperature structure to an ordered but twinned triclinic structure at low temperature underlines some counterintuitive features of this type of system. The room-temperature structure, and the structure to temperatures as low as 220 K, have excellent quality indicators and betray the dynamic disorder only in the displacement parameters of the affected atoms and in the apparently foreshortened bond distance at the end of one of the unique ^{n}Bu groups.

For compound (**2**), unlike what is found for most molecular crystalline systems, but not unprecedented or completely unexpected, lowering the temperature gives a decidedly worse diffraction pattern because of the twinning that accompanies the conservative symmetry-lowering transition. It is known that this occurs for some crystals, and this is a phenomenon that is worth keeping in mind when an otherwise apparently good crystal that is abruptly subjected to low temperatures displays a surprisingly poor diffraction pattern.

Acknowledgements

This work benefitted from services provided by the Servicio General de Apoyo a la Investigación, University of Zaragoza.

Funding information

Funding for this research was provided by: Ministerio de Economía y Competitividad (grant No. MAT2015-68200-C2-1-P); European Union (award No. FEDER funds); Diputación General de Aragón (grant No. E16 (M4)); Ministry of Education (Spain) under the program 'Becas y Contratos FPU' (predoctoral scholarship No. AP2009-4211 to EFV).

References

Agilent (2011). *CrysAlis PRO*. Agilent Technologies Ltd, Abingdon, Oxfordshire, England.

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Aromí, G., Beavers, C. M., Sánchez Costa, J., Craig, G. A., Mínguez Espallargas, G., Orera, A. & Roubeau, O. (2016). *Chem. Sci.* **7**, 2907–2915.
Asayama, R., Kawamura, J. & Hattori, T. (2005). *Chem. Phys. Lett.* **414**, 87–91.
Asayama, R., Kawamura, J. & Hattori, T. (2006). *Solid State Ionics*, **177**, 3245–3249.
Asghar, M. A., Ji, C., Zhou, Y., Sun, Z., Khan, T., Zhang, S., Zhao, S. & Luo, J. (2015). *J. Mater. Chem. C*, **3**, 6053–6057.
Asghar, M. A., Sun, Z., Khan, T., Ji, C., Zhang, S., Liu, S., Li, L., Zhao, S. & Luo, J. (2016). *Cryst. Growth Des.* **16**, 895–899.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Brandenburg, K. & Putz, H. (2004). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Capelli, S. C., Falvello, L. R., Forcén-Vázquez, E., McIntyre, G. J., Palacio, F., Sanz, S. & Tomás, M. (2013). *Angew. Chem. Int. Ed.* **52**, 13463–13467.
Czerwonka, J., Hodorowiez, S., Kanas, A., Samotus, A. & Sagnowski, S. (1988). *Transition Met. Chem.* **13**, 190–192.
Ende, J. A. van den & Cuppen, H. M. (2014). *Cryst. Growth Des.* **14**, 3343–3351.
Excoffon, P., Laugier, J. & Lamotte, B. (1991). *Inorg. Chem.* **30**, 3075–3081.
Falvello, L. R., Ferrer, D., Piedrafita, M., Soler, T. & Tomás, M. (2007). *CrystEngComm*, **9**, 852–855.
Falvello, L. R., Hitchman, M. A., Palacio, F., Pascual, I., Schultz, A. J., Stratemeier, H., Tomás, M., Urriolabeitia, E. P. & Young, D. M. (1999). *J. Am. Chem. Soc.* **121**, 2808–2819.
Fujita, W. & Awaga, K. (1999). *Science*, **286**, 261–262.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Haase, M. & Alivisatos, A. P. (1992). *J. Phys. Chem.* **96**, 6756–6762.
Herbst-Irmer, R. (2016). *Z. Kristallogr.* **231**, 573–581.
Khan, T., Tang, Y., Sun, Z., Zhang, S., Asghar, M. A., Chen, T., Zhao, S. & Luo, J. (2015). *Cryst. Growth Des.* **15**, 5263–5268.
Li, L., Sun, Z., Ji, C., Zhao, S. & Luo, J. (2016). *Cryst. Growth Des*. **16**, 6685–6695.
Loeffler, M., Carrey, E. A. & Zameitat, E. (2016). *Nucleosides Nucleotides Nucleic Acids*, **35**, 566–577.
Luo, J., Kang, X., Chen, C., Song, J., Luo, D. & Wang, P. (2016). *J. Phys. Chem. C*, **120**, 18386–18393.
MacFarlane, D. R. & Forsyth, M. (2001). *Adv. Mater.* **13**, 957–966.
Mason, J. A., Oktawiec, J., Taylor, M. K., Hudson, M. R., Rodriguez, J., Bachman, J. E., Gonzalez, M. I., Cervellino, A., Guagliardi, A., Brown, C. M., Llewellyn, P. L., Masciocchi, N. & Long, J. R. (2015). *Nature*, **527**, 357–361.
Mentzafos, D., Katsaros, N. & Terzis, A. (1987). *Acta Cryst.* C**43**, 1905–1908.
Müller, A. (1932). *Proc. R. Soc. London Ser. A*, **138**, 514–530.
Murphy, D. T., Schmid, S., Hester, J. R., Blanchard, P. E. R. & Miiller, W. (2015). *Inorg. Chem.* **54**, 4636–4643.
Nauha, E., Naumov, P. & Lusi, M. (2016). *CrystEngComm*, **18**, 4699–4703.
Oxford Diffraction (2006). *CrysAlis RED* and *CrysAlis CCD*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Oxford Diffraction (2009). *CrysAlis RED* and *CrysAlis PRO*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Paglione, J. & Greene, R. L. (2010). *Nat. Phys.* **6**, 645–658.
Park, S. G., Lim, J. I., Kim, Y. J., Kim, Y. D., Kim, H. S., Son, M. H., Kim, S. H., Kim, J. H. & Kwak, U. Y. (2014). Rep. Korea Patent KR 1458330 B1 20141104.
Peng, H., Ran, C., Liu, Z., Long, Y., Wang, Z., Yu, Z., Sun, H., Wei, Y., Gao, S., Chen, Z. & Chen, E.-Q. (2008). *J. Phys. Chem. C*, **112**, 11001–11006.
Pielichowska, K. & Pielichowski, K. (2014). *Prog. Mater. Sci.* **65**, 67–123.
Rodríguez-Velamazán, J. A., González, M. A., Real, J. A., Castro, M., Munñz, M. C., Gaspar, A. B., Ohtani, R., Ohba, M., Yoneda, K., Hijikata, Y., Yanai, N., Mizuno, M., Ando, H. & Kitagawa, S. (2012). *J. Am. Chem. Soc.* **134**, 5083–5089.
Sato, O., Tao, J. & Zhang, Y.-Z. (2007). *Angew. Chem. Int. Ed.* **46**, 2152–2187.
Schneemann, A., Bon, V., Schwedler, I., Senkovska, I., Kaskel, S. & Fischer, R. A. (2014). *Chem. Soc. Rev.* **43**, 6062–6096.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Shimizu, T., Tanaka, S., Onoda-Yamamuro, N., Ishimaru, S. & Ikeda, R. (1997). *J. Chem. Soc. Faraday Trans.* **93**, 321–326.
Szaciłowski, K. (2008). *Chem. Rev.* **108**, 3481–3548.
Watase, S., Kitamura, T., Kanehisa, N., Nakamoto, M., Kai, Y. & Yanagida, S. (2003). *Chem. Lett.* **32**, 1002–1003.
Willett, R. D., Gómez-García, C. J., Ramakrishna, B. L. & Twamley, B. (2005). *Polyhedron*, **24**, 2232–2237.
Xu, H. & Veblen, D. R. (1995). *Am. Mineral.* **80**, 87–93.

supporting information

Acta Cryst. (2017). C**73**, 731-742 [https://doi.org/10.1107/S2053229617010841]

A phase transition caught in mid-course: independent and concomitant analyses of the monoclinic and triclinic structures of (^{n}Bu$_4$N)[Co(orotate)$_2$(bipy)]·3H$_2$O

Miguel Castro, Larry R. Falvello, Elena Forcén-Vázquez, Pablo Guerra, Nuha A. Al-Kenany, Gema Martínez and Milagros Tomás

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006) for (1); *CrysAlis PRO* Oxford Diffraction, 2009) for (2a); *CrysAlis PRO* (Oxford Diffraction, 2009) for (2b); *CrysAlis CCD* (Oxford Diffraction, 2009) for (2c); *CrysAlis PRO* (Agilent, 2011) for (2d), (2e). Cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006) for (1); *CrysAlis PRO* Oxford Diffraction, 2009) for (2a); *CrysAlis PRO* (Oxford Diffraction, 2009) for (2b); *CrysAlis RED* (Oxford Diffraction, 2009) for (2c); *CrysAlis PRO* (Agilent, 2011) for (2d), (2e). Data reduction: *CrysAlis RED* (Oxford Diffraction, 2006) for (1); *CrysAlis PRO* Oxford Diffraction, 2009) for (2a); *CrysAlis PRO* (Oxford Diffraction, 2009) for (2b); *CrysAlis RED* (Oxford Diffraction, 2009) for (2c); *CrysAlis PRO* (Agilent, 2011) for (2d), (2e). For all structures, program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015). Molecular graphics: *DIAMOND* (Brandenburg, 2006) for (1); *DIAMOND* (Brandenburg, 1996) for (2a), (2b), (2c), (2d), (2e). Software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) for (2a), (2b), (2c), (2d), (2e).

Bis(tetra-*n*-butylammonium) diaquabis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(II) 1.8-hydrate (1)

Crystal data

$(C_{16}H_{36}N)_2[Co(C_5H_2N_2O_4)_2(H_2O)_2]\cdot 1.8H_2O$
$M_r = 920.47$
Triclinic, $P\overline{1}$
$a = 12.3630\ (4)$ Å
$b = 12.6281\ (5)$ Å
$c = 16.3765\ (6)$ Å
$\alpha = 89.948\ (3)°$
$\beta = 95.460\ (3)°$
$\gamma = 96.455\ (3)°$
$V = 2528.86\ (16)$ Å^3
$Z = 2$
$F(000) = 998$
$D_x = 1.209$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6999 reflections
$\theta = 3.0–25.8°$
$\mu = 0.40$ mm^{-1}
$T = 295$ K
Block, pink
0.64 × 0.23 × 0.21 mm

Data collection

Four-circle CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.3990 pixels mm^{-1}
ω–scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)
$T_{min} = 0.918$, $T_{max} = 1.060$
26453 measured reflections
11477 independent reflections
5630 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$

θ_{max} = 28.8°, θ_{min} = 3.0°
h = −16→16
k = −16→17
l = −17→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.103
S = 1.03
11477 reflections
597 parameters
8 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Co1	0.20175 (3)	0.43431 (3)	0.16986 (2)	0.03937 (11)	
N1	0.09812 (14)	0.37157 (15)	0.06684 (10)	0.0347 (5)	
C2	0.08484 (19)	0.4090 (2)	−0.01027 (14)	0.0384 (6)	
O2	0.14990 (13)	0.48231 (14)	−0.03637 (9)	0.0491 (4)	
N3	−0.00245 (17)	0.36443 (17)	−0.06261 (12)	0.0431 (6)	
H3	−0.0094 (19)	0.3938 (18)	−0.1065 (14)	0.052*	
C4	−0.0785 (2)	0.2826 (2)	−0.04318 (16)	0.0469 (7)	
O4	−0.15753 (14)	0.25296 (14)	−0.09406 (11)	0.0655 (6)	
C5	−0.0581 (2)	0.2423 (2)	0.03679 (17)	0.0480 (7)	
H5	−0.100 (2)	0.1925 (19)	0.0508 (15)	0.058*	
C6	0.02638 (19)	0.28832 (19)	0.08722 (14)	0.0379 (6)	
C7	0.0479 (2)	0.2483 (2)	0.17510 (15)	0.0446 (6)	
O7	0.12237 (13)	0.30265 (13)	0.22071 (9)	0.0495 (4)	
O8	−0.00910 (15)	0.16763 (14)	0.19568 (11)	0.0623 (5)	
N11	0.29056 (14)	0.49621 (15)	0.27986 (10)	0.0364 (5)	
C12	0.38820 (18)	0.47242 (19)	0.31490 (14)	0.0395 (6)	
O12	0.45193 (13)	0.42412 (15)	0.27771 (10)	0.0558 (5)	
N13	0.41833 (15)	0.50245 (17)	0.39507 (11)	0.0416 (5)	
H13	0.4692 (18)	0.4783 (19)	0.4123 (14)	0.050*	
C14	0.35858 (19)	0.55668 (19)	0.44405 (14)	0.0419 (6)	
O14	0.39047 (13)	0.57424 (14)	0.51786 (10)	0.0601 (5)	
C15	0.2611 (2)	0.5881 (2)	0.40234 (14)	0.0435 (6)	
H15	0.2177 (17)	0.6266 (18)	0.4307 (14)	0.052*	
C16	0.23150 (17)	0.55545 (18)	0.32420 (13)	0.0344 (5)	
C17	0.12363 (19)	0.5833 (2)	0.27890 (15)	0.0440 (6)	
O17	0.09452 (12)	0.53487 (12)	0.21053 (9)	0.0440 (4)	
O18	0.07422 (14)	0.64881 (16)	0.31114 (11)	0.0694 (6)	

O1W	0.26760 (15)	0.56604 (19)	0.09725 (13)	0.0497 (5)	
H1A	0.238 (2)	0.622 (2)	0.1085 (16)	0.066 (11)*	
H1B	0.239 (2)	0.552 (2)	0.0560 (16)	0.055 (10)*	
O2W	0.33023 (17)	0.34423 (17)	0.14115 (16)	0.0542 (6)	
H2A	0.375 (2)	0.358 (2)	0.1818 (18)	0.077 (11)*	
H2B	0.353 (3)	0.361 (3)	0.1023 (19)	0.083 (14)*	
N21	0.23782 (17)	0.99328 (17)	0.26754 (14)	0.0598 (6)	
C21	0.1836 (2)	0.9936 (2)	0.1819 (2)	0.0736 (9)	
H21A	0.1149	1.0236	0.1835	0.088*	
H21B	0.1662	0.9203	0.1633	0.088*	
C22	0.2468 (3)	1.0533 (3)	0.1195 (2)	0.0993 (12)	
H22A	0.3132	1.0211	0.1131	0.119*	0.8333
H22B	0.2670	1.1267	0.1371	0.119*	0.8333
H22C	0.3235	1.0439	0.1317	0.119*	0.1667
H22D	0.2400	1.1286	0.1256	0.119*	0.1667
C23A	0.1744 (5)	1.0489 (4)	0.0376 (3)	0.122 (2)	0.8333
H23A	0.1509	0.9750	0.0228	0.183*	0.8333
H23B	0.1096	1.0834	0.0447	0.183*	0.8333
C23B	0.213 (3)	1.0209 (16)	0.0295 (9)	0.122 (2)	0.1667
H23C	0.2529	0.9626	0.0161	0.183*	0.1667
H23D	0.1357	0.9940	0.0245	0.183*	0.1667
C24	0.2285 (6)	1.0992 (4)	−0.0279 (3)	0.235 (3)	
H24A	0.1795	1.0940	−0.0772	0.352*	0.8333
H24B	0.2505	1.1728	−0.0143	0.352*	0.8333
H24C	0.2919	1.0644	−0.0362	0.352*	0.8333
H24D	0.2045	1.0703	−0.0816	0.352*	0.1667
H24E	0.1873	1.1567	−0.0169	0.352*	0.1667
H24F	0.3048	1.1251	−0.0253	0.352*	0.1667
C25	0.2593 (2)	1.1072 (2)	0.30037 (18)	0.0644 (8)	
H25A	0.3150	1.1453	0.2701	0.077*	
H25B	0.1930	1.1410	0.2888	0.077*	
C26	0.2953 (2)	1.1206 (2)	0.39049 (18)	0.0710 (9)	
H26A	0.2361	1.0915	0.4216	0.085*	
H26B	0.3569	1.0805	0.4042	0.085*	
C27	0.3277 (3)	1.2352 (2)	0.41481 (19)	0.0771 (9)	
H27A	0.3868	1.2642	0.3834	0.093*	
H27B	0.2661	1.2752	0.4009	0.093*	
C28	0.3639 (3)	1.2502 (3)	0.5044 (2)	0.0995 (12)	
H28A	0.3058	1.2221	0.5359	0.149*	
H28B	0.3822	1.3248	0.5165	0.149*	
H28C	0.4269	1.2134	0.5182	0.149*	
C29	0.1629 (2)	0.9241 (2)	0.3187 (2)	0.0755 (9)	
H29A	0.1483	0.8538	0.2936	0.091*	
H29B	0.2010	0.9163	0.3726	0.091*	
C30	0.0551 (3)	0.9652 (3)	0.3293 (2)	0.0916 (11)	
H30A	0.0683	1.0383	0.3491	0.110*	
H30B	0.0120	0.9645	0.2766	0.110*	
C31	−0.0075 (3)	0.8987 (3)	0.3885 (3)	0.1171 (13)	

H31A	−0.0208	0.8259	0.3680	0.141*
H31B	0.0369	0.8985	0.4406	0.141*
C32	−0.1142 (3)	0.9371 (4)	0.4021 (3)	0.161 (2)
H32A	−0.1020	1.0101	0.4204	0.241*
H32B	−0.1483	0.8941	0.4430	0.241*
H32C	−0.1610	0.9319	0.3517	0.241*
C33	0.3469 (2)	0.9478 (2)	0.2710 (2)	0.0746 (9)
H33A	0.3951	0.9932	0.2389	0.090*
H33B	0.3793	0.9517	0.3274	0.090*
C34	0.3442 (3)	0.8371 (3)	0.2414 (2)	0.1099 (14)
H34A	0.3166	0.8327	0.1839	0.132*
H34B	0.2946	0.7907	0.2717	0.132*
C35	0.4576 (4)	0.7993 (4)	0.2519 (3)	0.1389 (19)
H35A	0.4906	0.8148	0.3073	0.167*
H35B	0.4513	0.7228	0.2432	0.167*
C36	0.5213 (5)	0.8485 (5)	0.1977 (5)	0.247 (4)
H36A	0.5180	0.8047	0.1495	0.371*
H36B	0.5955	0.8603	0.2221	0.371*
H36C	0.4959	0.9157	0.1831	0.371*
N22	0.76367 (16)	0.35627 (17)	0.27448 (12)	0.0513 (6)
C37	0.7465 (2)	0.3209 (2)	0.18504 (15)	0.0584 (7)
H37A	0.7966	0.3664	0.1546	0.070*
H37B	0.7658	0.2489	0.1815	0.070*
C38	0.6322 (2)	0.3232 (3)	0.14380 (18)	0.0823 (10)
H38A	0.6188	0.3965	0.1341	0.099*
H38B	0.5798	0.2921	0.1800	0.099*
C39	0.6156 (3)	0.2625 (3)	0.0632 (2)	0.1051 (13)
H39A	0.5547	0.2879	0.0301	0.126*
H39B	0.6803	0.2791	0.0344	0.126*
C40	0.5949 (5)	0.1500 (4)	0.0696 (3)	0.196 (3)
H40A	0.6631	0.1197	0.0754	0.294*
H40B	0.5518	0.1216	0.0210	0.294*
H40C	0.5559	0.1326	0.1166	0.294*
C41	0.6889 (2)	0.2868 (2)	0.32626 (16)	0.0610 (8)
H41A	0.6999	0.3142	0.3821	0.073*
H41B	0.6137	0.2936	0.3059	0.073*
C42	0.7045 (3)	0.1700 (3)	0.3278 (2)	0.0882 (10)
H42A	0.7787	0.1618	0.3500	0.106*
H42B	0.6942	0.1417	0.2723	0.106*
C43	0.6242 (3)	0.1073 (3)	0.3798 (3)	0.1077 (12)
H43A	0.5508	0.1227	0.3613	0.129*
H43B	0.6272	0.0318	0.3709	0.129*
C44	0.6448 (4)	0.1303 (3)	0.4672 (3)	0.1395 (17)
H44A	0.7203	0.1248	0.4847	0.209*
H44B	0.5994	0.0802	0.4967	0.209*
H44C	0.6284	0.2012	0.4779	0.209*
C45	0.88369 (19)	0.3471 (2)	0.30173 (16)	0.0582 (7)
H45A	0.8975	0.2742	0.2923	0.070*

H45B	0.9280	0.3923	0.2668	0.070*	
C46	0.9219 (2)	0.3762 (3)	0.38930 (18)	0.0854 (10)	
H46A	0.8671	0.3470	0.4244	0.102*	
H46B	0.9306	0.4531	0.3957	0.102*	
C47	1.0296 (2)	0.3338 (3)	0.41564 (19)	0.1104 (14)	
H47A	1.0184	0.2567	0.4111	0.132*	
H47B	1.0815	0.3591	0.3772	0.132*	
C48	1.0779 (3)	0.3617 (3)	0.4968 (2)	0.1252 (15)	
H48A	1.1019	0.4367	0.4997	0.188*	
H48B	1.1394	0.3225	0.5099	0.188*	
H48C	1.0249	0.3445	0.5354	0.188*	
C49	0.7370 (2)	0.4690 (2)	0.28632 (16)	0.0594 (8)	
H49A	0.6595	0.4707	0.2707	0.071*	
H49B	0.7504	0.4868	0.3443	0.071*	
C50	0.7992 (2)	0.5534 (2)	0.23948 (17)	0.0655 (8)	
H50A	0.7825	0.5399	0.1811	0.079*	
H50B	0.8771	0.5513	0.2528	0.079*	
C51	0.7693 (3)	0.6623 (3)	0.2605 (2)	0.0906 (11)	
H51A	0.6908	0.6628	0.2505	0.109*	
H51B	0.7900	0.6773	0.3183	0.109*	
C52	0.8257 (3)	0.7479 (3)	0.2103 (2)	0.1191 (14)	
H52A	0.9035	0.7479	0.2204	0.179*	
H52B	0.8058	0.8160	0.2253	0.179*	
H52C	0.8038	0.7342	0.1531	0.179*	
O3W	0.49608 (19)	0.5714 (2)	0.13512 (17)	0.0920 (9)	0.8
O4WA	0.6439 (2)	0.6139 (3)	0.02116 (16)	0.1055 (10)	0.8
O4WB	0.5571 (11)	0.5212 (12)	−0.0190 (10)	0.146 (7)	0.2

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0373 (2)	0.0502 (2)	0.02996 (18)	0.00898 (16)	−0.00465 (14)	−0.00130 (15)
N1	0.0340 (11)	0.0434 (12)	0.0260 (11)	0.0068 (10)	−0.0029 (9)	0.0026 (9)
C2	0.0375 (14)	0.0450 (16)	0.0330 (14)	0.0122 (13)	−0.0038 (12)	−0.0057 (12)
O2	0.0523 (11)	0.0611 (12)	0.0309 (9)	−0.0020 (9)	−0.0011 (8)	0.0042 (8)
N3	0.0470 (13)	0.0520 (15)	0.0285 (11)	0.0094 (12)	−0.0100 (11)	−0.0038 (10)
C4	0.0453 (16)	0.0425 (17)	0.0510 (17)	0.0100 (14)	−0.0111 (14)	−0.0122 (13)
O4	0.0634 (12)	0.0567 (12)	0.0679 (13)	0.0015 (10)	−0.0318 (11)	−0.0117 (10)
C5	0.0481 (18)	0.0406 (17)	0.0520 (17)	0.0016 (13)	−0.0083 (14)	−0.0016 (14)
C6	0.0385 (14)	0.0374 (15)	0.0395 (14)	0.0141 (12)	0.0004 (12)	−0.0021 (12)
C7	0.0485 (17)	0.0463 (17)	0.0415 (16)	0.0151 (14)	0.0055 (13)	0.0007 (13)
O7	0.0507 (11)	0.0602 (12)	0.0353 (10)	0.0050 (9)	−0.0056 (8)	0.0065 (8)
O8	0.0744 (13)	0.0510 (12)	0.0590 (12)	−0.0030 (10)	0.0060 (10)	0.0129 (10)
N11	0.0309 (11)	0.0517 (13)	0.0275 (10)	0.0113 (10)	−0.0007 (9)	0.0002 (9)
C12	0.0343 (14)	0.0520 (17)	0.0314 (14)	0.0067 (12)	−0.0036 (11)	−0.0012 (12)
O12	0.0400 (10)	0.0909 (14)	0.0401 (10)	0.0292 (10)	−0.0037 (8)	−0.0139 (9)
N13	0.0343 (12)	0.0614 (15)	0.0291 (11)	0.0161 (11)	−0.0094 (9)	−0.0067 (10)
C14	0.0412 (15)	0.0499 (16)	0.0325 (14)	0.0023 (12)	−0.0035 (12)	−0.0062 (12)

O14	0.0578 (11)	0.0905 (14)	0.0323 (10)	0.0242 (10)	−0.0134 (9)	−0.0159 (9)
C15	0.0396 (15)	0.0583 (18)	0.0334 (14)	0.0144 (13)	−0.0033 (12)	−0.0091 (12)
C16	0.0317 (13)	0.0402 (15)	0.0304 (13)	0.0020 (11)	−0.0003 (10)	0.0012 (11)
C17	0.0396 (15)	0.0556 (18)	0.0374 (15)	0.0125 (13)	−0.0014 (12)	0.0034 (13)
O17	0.0382 (9)	0.0583 (11)	0.0352 (10)	0.0159 (8)	−0.0110 (8)	−0.0058 (8)
O18	0.0584 (12)	0.0924 (16)	0.0610 (12)	0.0422 (11)	−0.0153 (10)	−0.0255 (11)
O1W	0.0463 (12)	0.0574 (15)	0.0441 (13)	0.0073 (10)	−0.0043 (10)	0.0021 (10)
O2W	0.0532 (13)	0.0690 (14)	0.0422 (13)	0.0198 (10)	−0.0018 (12)	−0.0069 (12)
N21	0.0582 (15)	0.0462 (15)	0.0738 (17)	0.0112 (12)	−0.0057 (13)	0.0026 (12)
C21	0.080 (2)	0.056 (2)	0.084 (2)	0.0194 (17)	−0.010 (2)	−0.0041 (17)
C22	0.136 (3)	0.094 (3)	0.070 (2)	0.030 (2)	0.000 (2)	−0.009 (2)
C23A	0.219 (7)	0.071 (3)	0.085 (3)	0.058 (3)	0.002 (4)	−0.011 (3)
C23B	0.219 (7)	0.071 (3)	0.085 (3)	0.058 (3)	0.002 (4)	−0.011 (3)
C24	0.438 (11)	0.186 (6)	0.084 (4)	0.048 (6)	0.030 (5)	0.041 (4)
C25	0.066 (2)	0.0374 (18)	0.089 (2)	−0.0003 (14)	0.0081 (17)	0.0016 (15)
C26	0.076 (2)	0.064 (2)	0.070 (2)	−0.0019 (17)	0.0008 (17)	0.0030 (17)
C27	0.089 (2)	0.056 (2)	0.084 (2)	−0.0017 (17)	0.0090 (19)	−0.0104 (17)
C28	0.096 (3)	0.109 (3)	0.087 (3)	−0.017 (2)	0.008 (2)	−0.018 (2)
C29	0.066 (2)	0.052 (2)	0.103 (3)	−0.0038 (17)	−0.0043 (19)	0.0116 (18)
C30	0.065 (2)	0.076 (2)	0.132 (3)	−0.0008 (19)	0.012 (2)	0.010 (2)
C31	0.112 (3)	0.107 (3)	0.131 (4)	−0.013 (3)	0.033 (3)	0.004 (3)
C32	0.118 (4)	0.151 (4)	0.219 (6)	−0.011 (3)	0.087 (4)	−0.036 (4)
C33	0.061 (2)	0.062 (2)	0.099 (2)	0.0144 (16)	−0.0115 (17)	−0.0138 (18)
C34	0.084 (3)	0.084 (3)	0.160 (4)	0.029 (2)	−0.024 (2)	−0.044 (3)
C35	0.109 (4)	0.096 (4)	0.219 (6)	0.030 (3)	0.030 (4)	−0.047 (4)
C36	0.199 (7)	0.205 (7)	0.365 (12)	0.087 (6)	0.092 (7)	−0.012 (7)
N22	0.0450 (13)	0.0639 (16)	0.0471 (13)	0.0136 (11)	0.0068 (11)	−0.0029 (11)
C37	0.0605 (18)	0.071 (2)	0.0450 (16)	0.0150 (15)	0.0053 (14)	−0.0087 (14)
C38	0.070 (2)	0.111 (3)	0.066 (2)	0.0229 (19)	−0.0092 (17)	−0.0101 (19)
C39	0.093 (3)	0.146 (4)	0.069 (2)	−0.001 (3)	−0.011 (2)	−0.013 (2)
C40	0.270 (7)	0.114 (4)	0.185 (5)	−0.021 (4)	−0.020 (5)	−0.049 (4)
C41	0.0517 (17)	0.076 (2)	0.0566 (18)	0.0057 (15)	0.0134 (14)	0.0017 (15)
C42	0.104 (3)	0.076 (3)	0.086 (3)	0.003 (2)	0.028 (2)	0.0088 (19)
C43	0.094 (3)	0.107 (3)	0.116 (3)	−0.011 (2)	0.006 (3)	0.017 (3)
C44	0.166 (4)	0.150 (4)	0.105 (4)	0.014 (3)	0.028 (3)	0.046 (3)
C45	0.0423 (16)	0.081 (2)	0.0535 (17)	0.0174 (14)	0.0050 (13)	−0.0001 (15)
C46	0.068 (2)	0.123 (3)	0.064 (2)	0.019 (2)	−0.0045 (17)	−0.014 (2)
C47	0.059 (2)	0.210 (4)	0.060 (2)	0.022 (2)	−0.0134 (18)	−0.009 (3)
C48	0.119 (3)	0.165 (4)	0.095 (3)	0.043 (3)	−0.004 (3)	0.005 (3)
C49	0.0542 (17)	0.067 (2)	0.0601 (18)	0.0186 (15)	0.0070 (15)	−0.0072 (15)
C50	0.0647 (19)	0.066 (2)	0.067 (2)	0.0124 (16)	0.0035 (16)	0.0050 (16)
C51	0.101 (3)	0.069 (2)	0.103 (3)	0.018 (2)	0.007 (2)	0.009 (2)
C52	0.162 (4)	0.083 (3)	0.112 (3)	0.026 (3)	−0.005 (3)	0.004 (2)
O3W	0.0657 (17)	0.106 (2)	0.105 (2)	0.0078 (15)	0.0156 (16)	0.0155 (17)
O4WA	0.099 (2)	0.171 (3)	0.0554 (17)	0.051 (2)	0.0136 (16)	0.0135 (19)
O4WB	0.122 (11)	0.155 (13)	0.195 (16)	0.088 (10)	0.099 (11)	0.130 (12)

Geometric parameters (Å, º)

Co1—O7	2.0511 (16)	C30—C31	1.496 (4)
Co1—O17	2.0884 (15)	C30—H30A	0.9700
Co1—N1	2.1163 (17)	C30—H30B	0.9700
Co1—N11	2.1200 (17)	C31—C32	1.492 (5)
Co1—O2W	2.142 (2)	C31—H31A	0.9700
Co1—O1W	2.1726 (19)	C31—H31B	0.9700
N1—C2	1.350 (3)	C32—H32A	0.9600
N1—C6	1.360 (3)	C32—H32B	0.9600
C2—O2	1.259 (3)	C32—H32C	0.9600
C2—N3	1.380 (3)	C33—C34	1.476 (4)
N3—C4	1.375 (3)	C33—H33A	0.9700
N3—H3	0.81 (2)	C33—H33B	0.9700
C4—O4	1.246 (3)	C34—C35	1.526 (5)
C4—C5	1.415 (3)	C34—H34A	0.9700
C5—C6	1.346 (3)	C34—H34B	0.9700
C5—H5	0.81 (2)	C35—C36	1.348 (7)
C6—C7	1.534 (3)	C35—H35A	0.9700
C7—O8	1.237 (3)	C35—H35B	0.9700
C7—O7	1.267 (3)	C36—H36A	0.9600
N11—C12	1.351 (3)	C36—H36B	0.9600
N11—C16	1.358 (3)	C36—H36C	0.9600
C12—O12	1.250 (3)	N22—C49	1.513 (3)
C12—N13	1.372 (3)	N22—C41	1.519 (3)
N13—C14	1.373 (3)	N22—C37	1.520 (3)
N13—H13	0.76 (2)	N22—C45	1.525 (3)
C14—O14	1.247 (2)	C37—C38	1.510 (3)
C14—C15	1.424 (3)	C37—H37A	0.9700
C15—C16	1.349 (3)	C37—H37B	0.9700
C15—H15	0.92 (2)	C38—C39	1.511 (4)
C16—C17	1.539 (3)	C38—H38A	0.9700
C17—O18	1.230 (3)	C38—H38B	0.9700
C17—O17	1.277 (3)	C39—C40	1.421 (5)
O1W—H1A	0.86 (3)	C39—H39A	0.9700
O1W—H1B	0.74 (2)	C39—H39B	0.9700
O2W—H2A	0.83 (3)	C40—H40A	0.9600
O2W—H2B	0.74 (3)	C40—H40B	0.9600
N21—C21	1.496 (3)	C40—H40C	0.9600
N21—C29	1.513 (3)	C41—C42	1.509 (4)
N21—C33	1.521 (3)	C41—H41A	0.9700
N21—C25	1.522 (3)	C41—H41B	0.9700
C21—C22	1.497 (4)	C42—C43	1.524 (4)
C21—H21A	0.9700	C42—H42A	0.9700
C21—H21B	0.9700	C42—H42B	0.9700
C22—C23B	1.536 (17)	C43—C44	1.454 (5)
C22—C23A	1.537 (5)	C43—H43A	0.9700
C22—H22A	0.9700	C43—H43B	0.9700

C22—H22B	0.9700	C44—H44A	0.9600
C22—H22C	0.9700	C44—H44B	0.9600
C22—H22D	0.9700	C44—H44C	0.9600
C23A—C24	1.429 (7)	C45—C46	1.499 (4)
C23A—H23A	0.9700	C45—H45A	0.9700
C23A—H23B	0.9700	C45—H45B	0.9700
C23B—C24	1.374 (17)	C46—C47	1.515 (4)
C23B—H23C	0.9700	C46—H46A	0.9700
C23B—H23D	0.9700	C46—H46B	0.9700
C24—H24A	0.9600	C47—C48	1.432 (4)
C24—H24B	0.9600	C47—H47A	0.9700
C24—H24C	0.9600	C47—H47B	0.9700
C24—H24D	0.9600	C48—H48A	0.9600
C24—H24E	0.9600	C48—H48B	0.9600
C24—H24F	0.9600	C48—H48C	0.9600
C25—C26	1.505 (4)	C49—C50	1.498 (4)
C25—H25A	0.9700	C49—H49A	0.9700
C25—H25B	0.9700	C49—H49B	0.9700
C26—C27	1.502 (4)	C50—C51	1.512 (4)
C26—H26A	0.9700	C50—H50A	0.9700
C26—H26B	0.9700	C50—H50B	0.9700
C27—C28	1.498 (4)	C51—C52	1.506 (4)
C27—H27A	0.9700	C51—H51A	0.9700
C27—H27B	0.9700	C51—H51B	0.9700
C28—H28A	0.9600	C52—H52A	0.9600
C28—H28B	0.9600	C52—H52B	0.9600
C28—H28C	0.9600	C52—H52C	0.9600
C29—C30	1.508 (4)	O4WA—O4WB	1.593 (18)
C29—H29A	0.9700	O4WB—O4WB^{i}	1.63 (2)
C29—H29B	0.9700		

O7—Co1—O17	92.63 (6)	C31—C30—H30A	109.4
O7—Co1—N1	79.53 (7)	C29—C30—H30A	109.4
O17—Co1—N1	96.51 (6)	C31—C30—H30B	109.4
O7—Co1—N11	96.69 (7)	C29—C30—H30B	109.4
O17—Co1—N11	78.84 (6)	H30A—C30—H30B	108.0
N1—Co1—N11	173.94 (7)	C32—C31—C30	113.4 (4)
O7—Co1—O2W	90.75 (8)	C32—C31—H31A	108.9
O17—Co1—O2W	171.23 (7)	C30—C31—H31A	108.9
N1—Co1—O2W	92.06 (8)	C32—C31—H31B	108.9
N11—Co1—O2W	92.73 (8)	C30—C31—H31B	108.9
O7—Co1—O1W	170.24 (7)	H31A—C31—H31B	107.7
O17—Co1—O1W	87.84 (8)	C31—C32—H32A	109.5
N1—Co1—O1W	90.73 (8)	C31—C32—H32B	109.5
N11—Co1—O1W	92.97 (8)	H32A—C32—H32B	109.5
O2W—Co1—O1W	90.20 (9)	C31—C32—H32C	109.5
C2—N1—C6	117.41 (19)	H32A—C32—H32C	109.5
C2—N1—Co1	130.09 (16)	H32B—C32—H32C	109.5

C6—N1—Co1	111.94 (14)	C34—C33—N21	116.5 (2)
O2—C2—N1	122.3 (2)	C34—C33—H33A	108.2
O2—C2—N3	118.9 (2)	N21—C33—H33A	108.2
N1—C2—N3	118.8 (2)	C34—C33—H33B	108.2
C4—N3—C2	125.2 (2)	N21—C33—H33B	108.2
C4—N3—H3	120.4 (17)	H33A—C33—H33B	107.3
C2—N3—H3	114.2 (17)	C33—C34—C35	111.4 (3)
O4—C4—N3	119.5 (2)	C33—C34—H34A	109.4
O4—C4—C5	126.5 (3)	C35—C34—H34A	109.4
N3—C4—C5	114.0 (2)	C33—C34—H34B	109.4
C6—C5—C4	119.6 (2)	C35—C34—H34B	109.4
C6—C5—H5	122.4 (18)	H34A—C34—H34B	108.0
C4—C5—H5	117.9 (18)	C36—C35—C34	110.1 (5)
C5—C6—N1	124.8 (2)	C36—C35—H35A	109.6
C5—C6—C7	120.6 (2)	C34—C35—H35A	109.6
N1—C6—C7	114.6 (2)	C36—C35—H35B	109.6
O8—C7—O7	125.8 (2)	C34—C35—H35B	109.6
O8—C7—C6	117.9 (2)	H35A—C35—H35B	108.2
O7—C7—C6	116.3 (2)	C35—C36—H36A	109.5
C7—O7—Co1	116.98 (15)	C35—C36—H36B	109.5
C12—N11—C16	117.90 (19)	H36A—C36—H36B	109.5
C12—N11—Co1	128.63 (15)	C35—C36—H36C	109.5
C16—N11—Co1	112.75 (13)	H36A—C36—H36C	109.5
O12—C12—N11	123.0 (2)	H36B—C36—H36C	109.5
O12—C12—N13	118.7 (2)	C49—N22—C41	105.60 (19)
N11—C12—N13	118.3 (2)	C49—N22—C37	112.37 (19)
C12—N13—C14	125.92 (19)	C41—N22—C37	111.1 (2)
C12—N13—H13	111.8 (19)	C49—N22—C45	110.9 (2)
C14—N13—H13	121.7 (19)	C41—N22—C45	111.22 (19)
O14—C14—N13	120.7 (2)	C37—N22—C45	105.77 (18)
O14—C14—C15	125.7 (2)	C38—C37—N22	115.7 (2)
N13—C14—C15	113.6 (2)	C38—C37—H37A	108.3
C16—C15—C14	119.3 (2)	N22—C37—H37A	108.3
C16—C15—H15	121.8 (14)	C38—C37—H37B	108.3
C14—C15—H15	118.8 (14)	N22—C37—H37B	108.3
C15—C16—N11	124.5 (2)	H37A—C37—H37B	107.4
C15—C16—C17	120.6 (2)	C37—C38—C39	111.8 (3)
N11—C16—C17	114.87 (19)	C37—C38—H38A	109.2
O18—C17—O17	126.5 (2)	C39—C38—H38A	109.2
O18—C17—C16	117.9 (2)	C37—C38—H38B	109.2
O17—C17—C16	115.7 (2)	C39—C38—H38B	109.2
C17—O17—Co1	116.84 (14)	H38A—C38—H38B	107.9
Co1—O1W—H1A	108.7 (18)	C40—C39—C38	115.4 (4)
Co1—O1W—H1B	101 (2)	C40—C39—H39A	108.4
H1A—O1W—H1B	101 (3)	C38—C39—H39A	108.4
Co1—O2W—H2A	100.7 (19)	C40—C39—H39B	108.4
Co1—O2W—H2B	113 (3)	C38—C39—H39B	108.4
H2A—O2W—H2B	113 (3)	H39A—C39—H39B	107.5

C21—N21—C29	107.8 (2)	C39—C40—H40A	109.5
C21—N21—C33	111.8 (2)	C39—C40—H40B	109.5
C29—N21—C33	108.5 (2)	H40A—C40—H40B	109.5
C21—N21—C25	109.2 (2)	C39—C40—H40C	109.5
C29—N21—C25	111.7 (2)	H40A—C40—H40C	109.5
C33—N21—C25	107.8 (2)	H40B—C40—H40C	109.5
N21—C21—C22	116.7 (3)	C42—C41—N22	115.5 (2)
N21—C21—H21A	108.1	C42—C41—H41A	108.4
C22—C21—H21A	108.1	N22—C41—H41A	108.4
N21—C21—H21B	108.1	C42—C41—H41B	108.4
C22—C21—H21B	108.1	N22—C41—H41B	108.4
H21A—C21—H21B	107.3	H41A—C41—H41B	107.5
C21—C22—C23B	116.0 (11)	C41—C42—C43	111.3 (3)
C21—C22—C23A	108.3 (3)	C41—C42—H42A	109.4
C21—C22—H22A	110.0	C43—C42—H42A	109.4
C23A—C22—H22A	110.0	C41—C42—H42B	109.4
C21—C22—H22B	110.0	C43—C42—H42B	109.4
C23A—C22—H22B	110.0	H42A—C42—H42B	108.0
H22A—C22—H22B	108.4	C44—C43—C42	114.1 (3)
C21—C22—H22C	108.3	C44—C43—H43A	108.7
C23B—C22—H22C	108.3	C42—C43—H43A	108.7
C21—C22—H22D	108.3	C44—C43—H43B	108.7
C23B—C22—H22D	108.3	C42—C43—H43B	108.7
H22C—C22—H22D	107.4	H43A—C43—H43B	107.6
C24—C23A—C22	113.3 (5)	C43—C44—H44A	109.5
C24—C23A—H23A	108.9	C43—C44—H44B	109.5
C22—C23A—H23A	108.9	H44A—C44—H44B	109.5
C24—C23A—H23B	108.9	C43—C44—H44C	109.5
C22—C23A—H23B	108.9	H44A—C44—H44C	109.5
H23A—C23A—H23B	107.7	H44B—C44—H44C	109.5
C24—C23B—C22	116.6 (15)	C46—C45—N22	116.5 (2)
C24—C23B—H23C	108.1	C46—C45—H45A	108.2
C22—C23B—H23C	108.1	N22—C45—H45A	108.2
C24—C23B—H23D	108.1	C46—C45—H45B	108.2
C22—C23B—H23D	108.1	N22—C45—H45B	108.2
H23C—C23B—H23D	107.3	H45A—C45—H45B	107.3
C23A—C24—H24A	109.5	C45—C46—C47	111.2 (3)
C23A—C24—H24B	109.5	C45—C46—H46A	109.4
H24A—C24—H24B	109.5	C47—C46—H46A	109.4
C23A—C24—H24C	109.5	C45—C46—H46B	109.4
H24A—C24—H24C	109.5	C47—C46—H46B	109.4
H24B—C24—H24C	109.5	H46A—C46—H46B	108.0
C23B—C24—H24D	109.5	C48—C47—C46	116.6 (3)
C23B—C24—H24E	109.5	C48—C47—H47A	108.1
H24D—C24—H24E	109.5	C46—C47—H47A	108.1
C23B—C24—H24F	109.5	C48—C47—H47B	108.1
H24D—C24—H24F	109.5	C46—C47—H47B	108.1
H24E—C24—H24F	109.5	H47A—C47—H47B	107.3

C26—C25—N21	116.4 (2)	C47—C48—H48A	109.5
C26—C25—H25A	108.2	C47—C48—H48B	109.5
N21—C25—H25A	108.2	H48A—C48—H48B	109.5
C26—C25—H25B	108.2	C47—C48—H48C	109.5
N21—C25—H25B	108.2	H48A—C48—H48C	109.5
H25A—C25—H25B	107.3	H48B—C48—H48C	109.5
C27—C26—C25	112.4 (2)	C50—C49—N22	116.0 (2)
C27—C26—H26A	109.1	C50—C49—H49A	108.3
C25—C26—H26A	109.1	N22—C49—H49A	108.3
C27—C26—H26B	109.1	C50—C49—H49B	108.3
C25—C26—H26B	109.1	N22—C49—H49B	108.3
H26A—C26—H26B	107.9	H49A—C49—H49B	107.4
C28—C27—C26	113.2 (3)	C49—C50—C51	110.4 (3)
C28—C27—H27A	108.9	C49—C50—H50A	109.6
C26—C27—H27A	108.9	C51—C50—H50A	109.6
C28—C27—H27B	108.9	C49—C50—H50B	109.6
C26—C27—H27B	108.9	C51—C50—H50B	109.6
H27A—C27—H27B	107.8	H50A—C50—H50B	108.1
C27—C28—H28A	109.5	C52—C51—C50	111.3 (3)
C27—C28—H28B	109.5	C52—C51—H51A	109.4
H28A—C28—H28B	109.5	C50—C51—H51A	109.4
C27—C28—H28C	109.5	C52—C51—H51B	109.4
H28A—C28—H28C	109.5	C50—C51—H51B	109.4
H28B—C28—H28C	109.5	H51A—C51—H51B	108.0
C30—C29—N21	115.2 (2)	C51—C52—H52A	109.5
C30—C29—H29A	108.5	C51—C52—H52B	109.5
N21—C29—H29A	108.5	H52A—C52—H52B	109.5
C30—C29—H29B	108.5	C51—C52—H52C	109.5
N21—C29—H29B	108.5	H52A—C52—H52C	109.5
H29A—C29—H29B	107.5	H52B—C52—H52C	109.5
C31—C30—C29	111.3 (3)	O4WA—O4WB—O4WB^{i}	124.1 (18)

C6—N1—C2—O2	−176.5 (2)	C16—C17—O17—Co1	−3.3 (3)
Co1—N1—C2—O2	12.8 (3)	C29—N21—C21—C22	−176.9 (3)
C6—N1—C2—N3	2.7 (3)	C33—N21—C21—C22	−57.7 (3)
Co1—N1—C2—N3	−168.01 (15)	C25—N21—C21—C22	61.6 (3)
O2—C2—N3—C4	178.6 (2)	N21—C21—C22—C23B	158.3 (14)
N1—C2—N3—C4	−0.7 (4)	N21—C21—C22—C23A	−176.9 (3)
C2—N3—C4—O4	175.9 (2)	C21—C22—C23A—C24	−177.1 (4)
C2—N3—C4—C5	−2.5 (4)	C21—C22—C23B—C24	150 (2)
O4—C4—C5—C6	−174.7 (2)	C21—N21—C25—C26	169.6 (3)
N3—C4—C5—C6	3.5 (4)	C29—N21—C25—C26	50.4 (3)
C4—C5—C6—N1	−1.7 (4)	C33—N21—C25—C26	−68.7 (3)
C4—C5—C6—C7	178.0 (2)	N21—C25—C26—C27	172.9 (2)
C2—N1—C6—C5	−1.6 (3)	C25—C26—C27—C28	−180.0 (3)
Co1—N1—C6—C5	170.8 (2)	C21—N21—C29—C30	−65.0 (3)
C2—N1—C6—C7	178.69 (19)	C33—N21—C29—C30	173.6 (3)
Co1—N1—C6—C7	−9.0 (2)	C25—N21—C29—C30	54.9 (3)

C5—C6—C7—O8	5.7 (4)	N21—C29—C30—C31	−173.3 (3)
N1—C6—C7—O8	−174.6 (2)	C29—C30—C31—C32	179.1 (3)
C5—C6—C7—O7	−173.4 (2)	C21—N21—C33—C34	−59.8 (4)
N1—C6—C7—O7	6.4 (3)	C29—N21—C33—C34	59.0 (4)
O8—C7—O7—Co1	−179.1 (2)	C25—N21—C33—C34	−179.9 (3)
C6—C7—O7—Co1	−0.1 (3)	N21—C33—C34—C35	−177.2 (3)
C16—N11—C12—O12	−175.5 (2)	C33—C34—C35—C36	−71.4 (6)
Co1—N11—C12—O12	15.0 (3)	C49—N22—C37—C38	−58.9 (3)
C16—N11—C12—N13	4.8 (3)	C41—N22—C37—C38	59.2 (3)
Co1—N11—C12—N13	−164.78 (16)	C45—N22—C37—C38	−180.0 (2)
O12—C12—N13—C14	179.3 (2)	N22—C37—C38—C39	−166.1 (3)
N11—C12—N13—C14	−0.9 (4)	C37—C38—C39—C40	79.9 (5)
C12—N13—C14—O14	175.5 (2)	C49—N22—C41—C42	−177.0 (2)
C12—N13—C14—C15	−4.4 (4)	C37—N22—C41—C42	60.9 (3)
O14—C14—C15—C16	−174.0 (2)	C45—N22—C41—C42	−56.7 (3)
N13—C14—C15—C16	5.8 (4)	N22—C41—C42—C43	−178.8 (2)
C14—C15—C16—N11	−2.3 (4)	C41—C42—C43—C44	−69.2 (4)
C14—C15—C16—C17	176.8 (2)	C49—N22—C45—C46	58.9 (3)
C12—N11—C16—C15	−3.3 (3)	C41—N22—C45—C46	−58.3 (3)
Co1—N11—C16—C15	167.9 (2)	C37—N22—C45—C46	−179.1 (2)
C12—N11—C16—C17	177.53 (19)	N22—C45—C46—C47	163.2 (3)
Co1—N11—C16—C17	−11.3 (2)	C45—C46—C47—C48	176.5 (3)
C15—C16—C17—O18	10.8 (4)	C41—N22—C49—C50	−178.8 (2)
N11—C16—C17—O18	−169.9 (2)	C37—N22—C49—C50	−57.4 (3)
C15—C16—C17—O17	−169.2 (2)	C45—N22—C49—C50	60.7 (3)
N11—C16—C17—O17	10.0 (3)	N22—C49—C50—C51	−176.8 (2)
O18—C17—O17—Co1	176.7 (2)	C49—C50—C51—C52	−176.5 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O17^{ii}	0.81 (2)	2.17 (2)	2.928 (2)	157 (2)
N13—H13···O14^{iii}	0.76 (2)	2.15 (2)	2.903 (2)	169 (3)
O1*W*—H1*A*···O4^{ii}	0.86 (3)	1.96 (3)	2.786 (3)	161 (2)
O1*W*—H1*B*···O2	0.74 (2)	1.93 (3)	2.660 (3)	165 (3)
O1*W*—H1*B*···O4*WB*^{i}	0.74 (2)	2.89 (3)	2.937 (12)	86 (2)
O2*W*—H2*A*···O12	0.83 (3)	1.90 (3)	2.703 (3)	163 (3)
O2*W*—H2*B*···O4*WA*^{i}	0.74 (3)	2.05 (3)	2.751 (4)	158 (3)
O2*W*—H2*B*···O4*WB*^{i}	0.74 (3)	2.29 (3)	2.970 (11)	154 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*, −*y*+1, −*z*; (iii) −*x*+1, −*y*+1, −*z*+1.

Tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(III) trihydrate (2a)

Crystal data

$(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$
$M_r = 819.79$
Monoclinic, $P2/n$
$a = 13.1679$ (12) Å
$b = 9.3413$ (9) Å
$c = 16.3388$ (14) Å
$\beta = 102.669$ (9)°
$V = 1960.8$ (3) Å^3
$Z = 2$
$F(000) = 868$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2532 reflections
$\theta = 4.2$–27.5°
$\mu = 0.51$ mm^{-1}
$T = 277$ K
Plate, red
0.18 × 0.17 × 0.06 mm

Data collection

Oxford Diffraction KM-4/Xcalibur with a Sapphire3 detector diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0655 pixels mm^{-1}
ω–scans
Absorption correction: multi-scan CrysAlis RED (Oxford Diffraction, 2009)
$T_{min} = 0.823$, $T_{max} = 1.000$
10924 measured reflections
4564 independent reflections
2511 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 28.9°$, $\theta_{min} = 3.8°$
$h = -14\rightarrow17$
$k = -12\rightarrow11$
$l = -17\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.110$
$S = 1.02$
4564 reflections
264 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.2500	0.33659 (6)	0.7500	0.03719 (18)
N1	0.24864 (16)	0.3156 (3)	0.86825 (12)	0.0380 (6)
C2	0.1864 (2)	0.3821 (3)	0.91432 (17)	0.0413 (7)
O2	0.12172 (16)	0.4736 (3)	0.88636 (12)	0.0612 (7)
N3	0.19950 (17)	0.3371 (3)	0.99698 (12)	0.0421 (6)
H3	0.1595	0.3767	1.0256	0.051*
C4	0.2682 (2)	0.2375 (3)	1.03861 (17)	0.0427 (7)

O4	0.27290 (14)	0.2103 (2)	1.11331 (11)	0.0511 (6)
C5	0.3298 (2)	0.1719 (3)	0.98680 (15)	0.0414 (7)
H5	0.3784	0.1018	1.0086	0.050*
C6	0.3167 (2)	0.2128 (3)	0.90651 (16)	0.0365 (7)
C7	0.3769 (2)	0.1442 (3)	0.84799 (17)	0.0400 (7)
O7	0.35450 (14)	0.1945 (2)	0.77301 (10)	0.0434 (5)
O8	0.44135 (17)	0.0516 (2)	0.87219 (12)	0.0561 (6)
N9	0.34807 (17)	0.4915 (3)	0.77553 (12)	0.0366 (6)
C10	0.4511 (2)	0.4772 (4)	0.80157 (17)	0.0489 (8)
H10	0.4798	0.3858	0.8076	0.059*
C11	0.5161 (2)	0.5946 (4)	0.81986 (18)	0.0528 (9)
H11	0.5875	0.5823	0.8386	0.063*
C12	0.4739 (3)	0.7293 (4)	0.8099 (2)	0.0601 (9)
H12	0.5162	0.8097	0.8221	0.072*
C13	0.3682 (3)	0.7438 (4)	0.78174 (19)	0.0563 (9)
H13	0.3383	0.8344	0.7742	0.068*
C14	0.3064 (2)	0.6228 (3)	0.76465 (16)	0.0409 (7)
N15	0.7500	0.1433 (4)	0.7500	0.0547 (10)
C16	0.8409 (2)	0.0458 (3)	0.78462 (18)	0.0544 (9)
H16A	0.8591	−0.0049	0.7380	0.065*
H16B	0.8182	−0.0250	0.8202	0.065*
C17	0.9390 (3)	0.1172 (4)	0.8349 (2)	0.0698 (10)
H17A	0.9239	0.1588	0.8853	0.084*
H17B	0.9590	0.1943	0.8018	0.084*
C18	1.0314 (3)	0.0119 (4)	0.8599 (2)	0.0846 (12)
H18A	1.0553	−0.0142	0.8098	0.102*
H18B	1.0881	0.0607	0.8973	0.102*
C19	1.0072 (3)	−0.1188 (5)	0.9008 (3)	0.1034 (15)
H19A	0.9792	−0.0943	0.9485	0.155*
H19B	1.0695	−0.1741	0.9189	0.155*
H19C	0.9570	−0.1739	0.8619	0.155*
C20	0.7270 (3)	0.2411 (4)	0.8175 (2)	0.0766 (12)
H20A	0.7868	0.3027	0.8366	0.092*
H20B	0.6687	0.3019	0.7927	0.092*
C21	0.7021 (3)	0.1679 (5)	0.8927 (2)	0.0844 (13)
H21A	0.7531	0.0939	0.9129	0.101*
H21B	0.6339	0.1236	0.8777	0.101*
C22	0.7039 (4)	0.2822 (7)	0.9619 (4)	0.162 (3)
H22A	0.7758	0.2897	0.9922	0.194*
H22B	0.6869	0.3726	0.9329	0.194*
C23	0.6534 (6)	0.2771 (9)	1.0134 (4)	0.242 (5)
H23A	0.5815	0.2923	0.9874	0.363*
H23B	0.6763	0.3500	1.0547	0.363*
H23C	0.6616	0.1848	1.0399	0.363*
O1W	0.5898 (3)	0.5125 (3)	0.6088 (2)	0.0689 (8)
H1WA	0.529 (4)	0.515 (6)	0.597 (3)	0.14 (2)*
H1WB	0.613 (3)	0.558 (5)	0.653 (3)	0.12 (2)*
O2W	0.7500	0.6271 (4)	0.7500	0.0640 (11)

H2WA	0.743 (3)	0.674 (4)	0.7910 (19)	0.089 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0458 (3)	0.0359 (4)	0.0372 (3)	0.000	0.0251 (2)	0.000
N1	0.0439 (13)	0.0409 (16)	0.0355 (12)	0.0029 (12)	0.0225 (10)	−0.0015 (11)
C2	0.0460 (17)	0.046 (2)	0.0388 (16)	−0.0021 (16)	0.0240 (13)	−0.0026 (14)
O2	0.0692 (15)	0.0749 (18)	0.0497 (12)	0.0324 (14)	0.0353 (11)	0.0122 (12)
N3	0.0500 (14)	0.0476 (16)	0.0366 (12)	0.0024 (14)	0.0265 (11)	−0.0018 (13)
C4	0.0473 (17)	0.046 (2)	0.0394 (16)	−0.0092 (16)	0.0198 (14)	−0.0042 (15)
O4	0.0620 (13)	0.0636 (17)	0.0331 (10)	−0.0025 (11)	0.0222 (9)	0.0022 (10)
C5	0.0492 (16)	0.0424 (19)	0.0372 (14)	0.0027 (16)	0.0194 (13)	0.0033 (15)
C6	0.0434 (16)	0.0331 (19)	0.0386 (15)	−0.0010 (14)	0.0210 (13)	−0.0006 (13)
C7	0.0499 (17)	0.034 (2)	0.0425 (16)	−0.0041 (16)	0.0237 (13)	−0.0013 (15)
O7	0.0555 (12)	0.0439 (15)	0.0388 (11)	0.0110 (10)	0.0274 (9)	0.0043 (9)
O8	0.0711 (14)	0.0495 (16)	0.0548 (12)	0.0211 (13)	0.0293 (11)	0.0062 (11)
N9	0.0385 (14)	0.0427 (18)	0.0329 (12)	−0.0017 (12)	0.0172 (10)	−0.0012 (11)
C10	0.050 (2)	0.048 (2)	0.0543 (18)	0.0025 (17)	0.0234 (15)	0.0001 (16)
C11	0.0406 (18)	0.059 (3)	0.061 (2)	−0.0080 (18)	0.0157 (15)	−0.0015 (18)
C12	0.059 (2)	0.047 (3)	0.076 (2)	−0.0151 (19)	0.0196 (18)	−0.0037 (19)
C13	0.063 (2)	0.039 (2)	0.068 (2)	0.0011 (18)	0.0186 (17)	0.0005 (18)
C14	0.0512 (16)	0.034 (2)	0.0421 (16)	0.0001 (15)	0.0202 (14)	0.0005 (14)
N15	0.059 (2)	0.035 (2)	0.075 (2)	0.000	0.0238 (19)	0.000
C16	0.069 (2)	0.042 (2)	0.060 (2)	0.0074 (18)	0.0306 (17)	0.0015 (16)
C17	0.061 (2)	0.067 (3)	0.086 (2)	−0.001 (2)	0.0241 (19)	0.011 (2)
C18	0.095 (3)	0.082 (3)	0.086 (3)	0.002 (3)	0.039 (2)	0.008 (2)
C19	0.092 (3)	0.105 (4)	0.118 (4)	−0.002 (3)	0.031 (3)	0.017 (3)
C20	0.066 (2)	0.050 (3)	0.113 (3)	0.0059 (19)	0.019 (2)	−0.038 (2)
C21	0.076 (3)	0.094 (3)	0.091 (3)	−0.005 (2)	0.037 (2)	−0.039 (3)
C22	0.103 (4)	0.203 (7)	0.189 (6)	−0.016 (4)	0.052 (4)	−0.133 (5)
C23	0.229 (7)	0.322 (11)	0.232 (8)	−0.143 (7)	0.174 (7)	−0.199 (8)
O1W	0.072 (2)	0.075 (2)	0.0710 (19)	−0.0110 (16)	0.0410 (16)	−0.0046 (15)
O2W	0.080 (2)	0.073 (3)	0.049 (2)	0.000	0.0341 (18)	0.000

Geometric parameters (Å, º)

Co1—O7^{i}	1.8891 (19)	N15—C16	1.512 (4)
Co1—O7	1.8891 (18)	N15—C20	1.513 (4)
Co1—N9	1.923 (2)	N15—C20^{ii}	1.513 (4)
Co1—N9^{i}	1.923 (2)	C16—C17	1.524 (4)
Co1—N1^{i}	1.9459 (19)	C16—H16A	0.9700
Co1—N1	1.9459 (19)	C16—H16B	0.9700
N1—C6	1.368 (3)	C17—C18	1.548 (5)
N1—C2	1.376 (3)	C17—H17A	0.9700
C2—O2	1.222 (3)	C17—H17B	0.9700
C2—N3	1.389 (3)	C18—C19	1.460 (5)
N3—C4	1.370 (4)	C18—H18A	0.9700

N3—H3	0.8600	C18—H18B	0.9700
C4—O4	1.235 (3)	C19—H19A	0.9600
C4—C5	1.432 (4)	C19—H19B	0.9600
C5—C6	1.340 (3)	C19—H19C	0.9600
C5—H5	0.9300	C20—C21	1.503 (5)
C6—C7	1.512 (3)	C20—H20A	0.9700
C7—O8	1.216 (3)	C20—H20B	0.9700
C7—O7	1.284 (3)	C21—C22	1.552 (5)
N9—C10	1.336 (3)	C21—H21A	0.9700
N9—C14	1.340 (3)	C21—H21B	0.9700
C10—C11	1.383 (4)	C22—C23	1.182 (6)
C10—H10	0.9300	C22—H22A	0.9700
C11—C12	1.371 (4)	C22—H22B	0.9700
C11—H11	0.9300	C23—H23A	0.9600
C12—C13	1.373 (4)	C23—H23B	0.9600
C12—H12	0.9300	C23—H23C	0.9600
C13—C14	1.385 (4)	O1W—H1WA	0.78 (5)
C13—H13	0.9300	O1W—H1WB	0.84 (5)
C14—C14^{i}	1.457 (6)	O2W—H2WA	0.83 (3)
N15—C16^{ii}	1.512 (4)		

O7^{i}—Co1—O7	90.76 (12)	C16^{ii}—N15—C16	105.9 (3)
O7^{i}—Co1—N9	175.38 (9)	C16^{ii}—N15—C20	111.55 (18)
O7—Co1—N9	93.45 (9)	C16—N15—C20	111.06 (18)
O7^{i}—Co1—N9^{i}	93.45 (9)	C16^{ii}—N15—C20^{ii}	111.06 (18)
O7—Co1—N9^{i}	175.38 (9)	C16—N15—C20^{ii}	111.55 (18)
N9—Co1—N9^{i}	82.42 (14)	C20—N15—C20^{ii}	105.8 (4)
O7^{i}—Co1—N1^{i}	84.07 (8)	N15—C16—C17	116.5 (3)
O7—Co1—N1^{i}	87.83 (8)	N15—C16—H16A	108.2
N9—Co1—N1^{i}	97.99 (9)	C17—C16—H16A	108.2
N9^{i}—Co1—N1^{i}	90.70 (9)	N15—C16—H16B	108.2
O7^{i}—Co1—N1	87.83 (8)	C17—C16—H16B	108.2
O7—Co1—N1	84.07 (8)	H16A—C16—H16B	107.3
N9—Co1—N1	90.70 (9)	C16—C17—C18	112.8 (3)
N9^{i}—Co1—N1	97.99 (9)	C16—C17—H17A	109.0
N1^{i}—Co1—N1	168.46 (15)	C18—C17—H17A	109.0
C6—N1—C2	118.4 (2)	C16—C17—H17B	109.0
C6—N1—Co1	112.13 (16)	C18—C17—H17B	109.0
C2—N1—Co1	129.3 (2)	H17A—C17—H17B	107.8
O2—C2—N1	124.1 (2)	C19—C18—C17	114.5 (3)
O2—C2—N3	120.2 (2)	C19—C18—H18A	108.6
N1—C2—N3	115.7 (3)	C17—C18—H18A	108.6
C4—N3—C2	128.0 (2)	C19—C18—H18B	108.6
C4—N3—H3	116.0	C17—C18—H18B	108.6
C2—N3—H3	116.0	H18A—C18—H18B	107.6
O4—C4—N3	121.0 (2)	C18—C19—H19A	109.5
O4—C4—C5	125.8 (3)	C18—C19—H19B	109.5
N3—C4—C5	113.3 (2)	H19A—C19—H19B	109.5

C6—C5—C4	119.2 (3)	C18—C19—H19C	109.5
C6—C5—H5	120.4	H19A—C19—H19C	109.5
C4—C5—H5	120.4	H19B—C19—H19C	109.5
C5—C6—N1	125.3 (2)	C21—C20—N15	115.8 (3)
C5—C6—C7	121.7 (3)	C21—C20—H20A	108.3
N1—C6—C7	113.0 (2)	N15—C20—H20A	108.3
O8—C7—O7	124.7 (2)	C21—C20—H20B	108.3
O8—C7—C6	121.4 (2)	N15—C20—H20B	108.3
O7—C7—C6	113.9 (3)	H20A—C20—H20B	107.4
C7—O7—Co1	116.87 (17)	C20—C21—C22	108.1 (4)
C10—N9—C14	119.4 (3)	C20—C21—H21A	110.1
C10—N9—Co1	125.5 (2)	C22—C21—H21A	110.1
C14—N9—Co1	115.09 (19)	C20—C21—H21B	110.1
N9—C10—C11	121.8 (3)	C22—C21—H21B	110.1
N9—C10—H10	119.1	H21A—C21—H21B	108.4
C11—C10—H10	119.1	C23—C22—C21	124.7 (6)
C12—C11—C10	119.1 (3)	C23—C22—H22A	106.1
C12—C11—H11	120.4	C21—C22—H22A	106.1
C10—C11—H11	120.4	C23—C22—H22B	106.1
C11—C12—C13	119.0 (3)	C21—C22—H22B	106.1
C11—C12—H12	120.5	H22A—C22—H22B	106.3
C13—C12—H12	120.5	C22—C23—H23A	109.5
C12—C13—C14	119.7 (3)	C22—C23—H23B	109.5
C12—C13—H13	120.2	H23A—C23—H23B	109.5
C14—C13—H13	120.2	C22—C23—H23C	109.5
N9—C14—C13	121.0 (3)	H23A—C23—H23C	109.5
N9—C14—C14^{i}	113.70 (16)	H23B—C23—H23C	109.5
C13—C14—C14^{i}	125.31 (19)	H1WA—O1W—H1WB	110 (4)

C6—N1—C2—O2	−178.7 (3)	N1^{i}—Co1—O7—C7	−169.7 (2)
Co1—N1—C2—O2	−3.1 (4)	N1—Co1—O7—C7	2.1 (2)
C6—N1—C2—N3	0.1 (4)	C14—N9—C10—C11	1.7 (4)
Co1—N1—C2—N3	175.78 (18)	Co1—N9—C10—C11	−178.97 (19)
O2—C2—N3—C4	−179.4 (3)	N9—C10—C11—C12	−0.9 (4)
N1—C2—N3—C4	1.7 (4)	C10—C11—C12—C13	−0.3 (5)
C2—N3—C4—O4	178.4 (3)	C11—C12—C13—C14	0.6 (5)
C2—N3—C4—C5	−2.1 (4)	C10—N9—C14—C13	−1.4 (4)
O4—C4—C5—C6	−179.7 (3)	Co1—N9—C14—C13	179.2 (2)
N3—C4—C5—C6	0.9 (4)	C10—N9—C14—C14^{i}	179.4 (2)
C4—C5—C6—N1	0.8 (4)	Co1—N9—C14—C14^{i}	0.1 (4)
C4—C5—C6—C7	−178.0 (2)	C12—C13—C14—N9	0.3 (4)
C2—N1—C6—C5	−1.3 (4)	C12—C13—C14—C14^{i}	179.3 (3)
Co1—N1—C6—C5	−177.7 (2)	C16^{ii}—N15—C16—C17	−171.4 (3)
C2—N1—C6—C7	177.6 (2)	C20—N15—C16—C17	−50.1 (3)
Co1—N1—C6—C7	1.2 (3)	C20^{ii}—N15—C16—C17	67.6 (3)
C5—C6—C7—O8	−1.7 (4)	N15—C16—C17—C18	−174.0 (2)
N1—C6—C7—O8	179.4 (3)	C16—C17—C18—C19	−51.3 (4)
C5—C6—C7—O7	179.3 (2)	C16^{ii}—N15—C20—C21	59.5 (4)

N1—C6—C7—O7	0.4 (3)	C16—N15—C20—C21	−58.5 (4)
O8—C7—O7—Co1	179.1 (2)	C20^{ii}—N15—C20—C21	−179.7 (4)
C6—C7—O7—Co1	−1.9 (3)	N15—C20—C21—C22	168.3 (3)
O7^{i}—Co1—O7—C7	−85.63 (19)	C20—C21—C22—C23	150.6 (8)
N9—Co1—O7—C7	92.5 (2)		

Symmetry codes: (i) −*x*+1/2, *y*, −*z*+3/2; (ii) −*x*+3/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1*W*^{iii}	0.86	2.08	2.925 (3)	169
O1*W*—H1*WA*···O2^{i}	0.78 (5)	2.10 (5)	2.828 (4)	156 (5)
O1*W*—H1*WB*···O2*W*	0.84 (5)	2.22 (5)	2.964 (4)	147 (4)
O2*W*—H2*WA*···O4^{iv}	0.83 (3)	1.95 (3)	2.772 (3)	179 (4)
C10—H10···O7	0.93	2.41	2.923 (4)	114
C16—H16*B*···O4^{v}	0.97	2.49	3.442 (4)	168
C20—H20*A*···O1*W*^{ii}	0.97	2.58	3.525 (5)	166
C21—H21*B*···O8	0.97	2.61	3.546 (4)	163

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

Tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(III) trihydrate (2b)

Crystal data

$(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$
M_r = 819.79
Triclinic, $P\overline{1}$
a = 12.9054 (8) Å
b = 9.3791 (8) Å
c = 16.1290 (12) Å
α = 88.724 (6)°
β = 102.898 (6)°
γ = 88.528 (6)°
V = 1901.6 (2) Å^3
Z = 2
F(000) = 868
D_x = 1.432 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5379 reflections
θ = 4.4–27.9°
μ = 0.52 mm^{-1}
T = 100 K
Plate, red
0.18 × 0.17 × 0.06 mm

Data collection

Agilent Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0655 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2011)
T_{min} = 0.865, T_{max} = 1.000
15136 measured reflections
15136 independent reflections
10265 reflections with $I > 2\sigma(I)$
θ_{max} = 27.0°, θ_{min} = 4.3°
h = −16→16
k = −11→11
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.095
$wR(F^2)$ = 0.248
S = 1.44
15136 reflections
501 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

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

$w = 1/[\sigma^2(F_o^2) + (0.144P)^2 + 1.0673P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.88$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}

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. Refined as a 2-component twin. Full details can be found in the embedded Shelx .res and .hkl files.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.2603 (2)	0.3424 (3)	0.75555 (17)	0.0141 (8)
N1	0.2604 (13)	0.320 (2)	0.8756 (10)	0.016 (4)
C2	0.1953 (16)	0.385 (2)	0.9200 (12)	0.016 (4)
O2	0.1324 (11)	0.4849 (18)	0.8917 (9)	0.021 (3)
N3	0.2034 (12)	0.334 (2)	1.0035 (10)	0.016 (4)
H3	0.1614	0.3752	1.0325	0.020*
C4	0.2705 (15)	0.225 (2)	1.0447 (13)	0.016 (4)
O4	0.2714 (11)	0.1911 (17)	1.1198 (9)	0.020 (3)
C5	0.3337 (15)	0.158 (2)	0.9940 (12)	0.015 (4)
H5	0.3808	0.0803	1.0162	0.018*
C6	0.3255 (15)	0.209 (2)	0.9134 (12)	0.014 (4)
C7	0.3891 (16)	0.142 (2)	0.8560 (12)	0.015 (4)
O7	0.3700 (11)	0.2001 (17)	0.7796 (8)	0.017 (3)
O8	0.4539 (12)	0.0445 (18)	0.8799 (9)	0.023 (4)
N1A	0.2591 (13)	0.321 (2)	0.6350 (10)	0.016 (4)
C2A	0.3191 (16)	0.387 (2)	0.5860 (13)	0.016 (4)
O2A	0.3818 (11)	0.4819 (18)	0.6116 (9)	0.022 (4)
N3A	0.3057 (13)	0.342 (2)	0.5030 (10)	0.018 (4)
H3A	0.3446	0.3832	0.4715	0.022*
C4A	0.2380 (16)	0.240 (2)	0.4649 (13)	0.017 (4)
O4A	0.2312 (11)	0.2124 (17)	0.3885 (8)	0.019 (3)
C5A	0.1790 (16)	0.176 (3)	0.5197 (12)	0.018 (5)
H5A	0.1302	0.1033	0.4996	0.021*
C6A	0.1930 (15)	0.219 (2)	0.6008 (12)	0.016 (4)
C7A	0.1333 (16)	0.152 (2)	0.6607 (12)	0.016 (4)
O7A	0.1565 (12)	0.2018 (17)	0.7362 (9)	0.019 (3)
O8A	0.0688 (11)	0.0584 (18)	0.6381 (9)	0.022 (3)
N9	0.3584 (13)	0.495 (2)	0.7781 (10)	0.014 (4)
N9A	0.1573 (13)	0.498 (2)	0.7271 (10)	0.015 (4)
C10	0.4623 (16)	0.479 (3)	0.8086 (12)	0.018 (5)
H10	0.4935	0.3856	0.8205	0.022*
C11	0.5270 (18)	0.598 (3)	0.8236 (13)	0.022 (5)
H11	0.6015	0.5848	0.8453	0.027*

C12	0.4822 (17)	0.733 (3)	0.8068 (13)	0.021 (5)
H12	0.5251	0.8139	0.8169	0.025*
C13	0.3727 (17)	0.748 (3)	0.7747 (13)	0.020 (5)
H13	0.3398	0.8403	0.7620	0.024*
C14	0.3133 (16)	0.627 (3)	0.7619 (12)	0.017 (4)
C14A	0.1979 (16)	0.627 (2)	0.7306 (12)	0.016 (4)
C13A	0.1329 (17)	0.749 (3)	0.7095 (13)	0.021 (5)
H13A	0.1630	0.8412	0.7127	0.025*
C12A	0.0243 (17)	0.735 (3)	0.6838 (13)	0.022 (5)
H12A	−0.0213	0.8171	0.6672	0.026*
C11A	−0.0172 (17)	0.601 (3)	0.6826 (13)	0.023 (5)
H11A	−0.0919	0.5890	0.6675	0.027*
C10A	0.0525 (16)	0.483 (3)	0.7039 (12)	0.019 (5)
H10A	0.0247	0.3899	0.7018	0.023*
N15	0.7579 (12)	0.1479 (19)	0.7378 (10)	0.016 (4)
C16	0.8515 (16)	0.047 (3)	0.7778 (13)	0.019 (5)
H16A	0.8262	−0.0247	0.8139	0.023*
H16B	0.8742	−0.0041	0.7316	0.023*
C17	0.9477 (16)	0.116 (3)	0.8312 (14)	0.022 (5)
H17A	0.9288	0.1572	0.8821	0.027*
H17B	0.9704	0.1943	0.7978	0.027*
C18	1.0396 (18)	0.006 (3)	0.8593 (14)	0.024 (5)
H18A	1.0993	0.0525	0.8975	0.028*
H18B	1.0646	−0.0241	0.8085	0.028*
C19	1.0105 (19)	−0.127 (3)	0.9055 (15)	0.029 (6)
H19A	0.9565	−0.1793	0.8663	0.044*
H19B	0.9824	−0.0980	0.9544	0.044*
H19C	1.0742	−0.1886	0.9252	0.044*
C20	0.7347 (17)	0.254 (3)	0.8016 (13)	0.019 (5)
H20A	0.6748	0.3186	0.7727	0.023*
H20B	0.7977	0.3135	0.8197	0.023*
C21	0.7069 (17)	0.188 (3)	0.8805 (13)	0.020 (5)
H21A	0.6343	0.1503	0.8658	0.024*
H21B	0.7575	0.1078	0.9031	0.024*
C22	0.7127 (18)	0.301 (3)	0.9474 (13)	0.024 (5)
H22A	0.6675	0.3848	0.9221	0.028*
H22B	0.7869	0.3324	0.9648	0.028*
C23	0.676 (2)	0.247 (3)	1.0258 (15)	0.035 (6)
H23A	0.6025	0.2173	1.0090	0.053*
H23B	0.6812	0.3234	1.0668	0.053*
H23C	0.7219	0.1654	1.0519	0.053*
C16A	0.6635 (16)	0.053 (2)	0.7081 (14)	0.018 (5)
H16C	0.6860	−0.0250	0.6747	0.022*
H16D	0.6452	0.0078	0.7590	0.022*
C17A	0.5639 (17)	0.126 (3)	0.6547 (14)	0.022 (5)
H17C	0.5775	0.1588	0.5995	0.026*
H17D	0.5443	0.2101	0.6844	0.026*
C18A	0.4724 (16)	0.022 (3)	0.6393 (13)	0.020 (5)

H18C	0.4102	0.0687	0.6001	0.025*
H18D	0.4521	0.0020	0.6940	0.025*
C19A	0.4976 (18)	−0.120 (3)	0.6019 (14)	0.025 (5)
H19D	0.4335	−0.1770	0.5896	0.038*
H19E	0.5538	−0.1728	0.6430	0.038*
H19F	0.5215	−0.1019	0.5492	0.038*
C20A	0.7804 (17)	0.234 (2)	0.6633 (13)	0.018 (4)
H20C	0.8432	0.2926	0.6847	0.022*
H20D	0.7191	0.3012	0.6418	0.022*
C21A	0.8007 (17)	0.150 (3)	0.5891 (13)	0.020 (5)
H21C	0.7486	0.0728	0.5769	0.024*
H21D	0.8727	0.1040	0.6050	0.024*
C22A	0.7918 (18)	0.244 (3)	0.5085 (13)	0.025 (5)
H22C	0.7933	0.1813	0.4600	0.030*
H22D	0.7221	0.2957	0.4962	0.030*
C23A	0.8772 (18)	0.350 (3)	0.5139 (14)	0.026 (5)
H23D	0.8748	0.3908	0.4571	0.039*
H23E	0.9467	0.3019	0.5360	0.039*
H23F	0.8662	0.4255	0.5521	0.039*
O1W	0.5969 (12)	0.5096 (18)	0.6152 (9)	0.023 (4)
O1WA	0.9134 (12)	0.5143 (19)	0.8898 (9)	0.025 (4)
O2W	0.7507 (12)	0.6326 (17)	0.7510 (9)	0.024 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0140 (14)	0.0189 (15)	0.0115 (13)	0.0005 (12)	0.0072 (10)	−0.0002 (11)
N1	0.013 (8)	0.023 (11)	0.013 (8)	−0.001 (7)	0.004 (7)	0.001 (7)
C2	0.017 (10)	0.023 (13)	0.012 (9)	−0.004 (9)	0.011 (9)	−0.002 (8)
O2	0.020 (7)	0.030 (10)	0.014 (7)	0.008 (7)	0.009 (6)	0.000 (6)
N3	0.011 (8)	0.024 (11)	0.016 (8)	−0.002 (7)	0.006 (7)	0.000 (7)
C4	0.013 (9)	0.021 (12)	0.017 (10)	−0.005 (8)	0.005 (9)	−0.003 (9)
O4	0.020 (7)	0.026 (9)	0.015 (7)	0.000 (6)	0.008 (6)	0.002 (6)
C5	0.013 (9)	0.018 (11)	0.017 (9)	−0.001 (8)	0.010 (9)	0.000 (8)
C6	0.014 (9)	0.014 (11)	0.016 (9)	0.001 (8)	0.007 (9)	−0.001 (8)
C7	0.017 (10)	0.015 (11)	0.012 (9)	0.000 (9)	0.003 (8)	−0.003 (8)
O7	0.017 (7)	0.020 (9)	0.016 (7)	0.001 (6)	0.008 (6)	0.003 (6)
O8	0.024 (8)	0.026 (10)	0.022 (7)	0.005 (7)	0.011 (7)	−0.001 (7)
N1A	0.015 (8)	0.028 (11)	0.008 (7)	−0.001 (8)	0.006 (7)	−0.001 (7)
C2A	0.016 (10)	0.016 (12)	0.017 (10)	0.001 (9)	0.006 (9)	0.003 (8)
O2A	0.021 (8)	0.030 (10)	0.017 (7)	−0.007 (7)	0.009 (7)	−0.002 (7)
N3A	0.013 (8)	0.030 (12)	0.014 (8)	−0.003 (8)	0.007 (7)	0.001 (8)
C4A	0.016 (10)	0.018 (12)	0.017 (10)	0.005 (8)	0.004 (9)	−0.002 (9)
O4A	0.021 (7)	0.022 (9)	0.014 (7)	0.004 (6)	0.007 (6)	−0.001 (6)
C5A	0.014 (10)	0.026 (13)	0.015 (10)	0.000 (9)	0.006 (9)	−0.001 (9)
C6A	0.012 (9)	0.021 (12)	0.014 (9)	0.003 (8)	0.003 (8)	0.001 (8)
C7A	0.016 (10)	0.016 (11)	0.015 (10)	0.001 (9)	0.004 (9)	0.002 (8)
O7A	0.022 (7)	0.021 (9)	0.016 (7)	−0.003 (6)	0.005 (6)	−0.001 (6)

O8A	0.022 (8)	0.025 (10)	0.020 (7)	−0.005 (7)	0.008 (7)	−0.002 (7)
N9	0.013 (8)	0.020 (10)	0.011 (8)	0.000 (7)	0.005 (7)	0.000 (7)
N9A	0.014 (8)	0.024 (11)	0.007 (7)	0.001 (7)	0.003 (7)	0.000 (7)
C10	0.017 (10)	0.023 (13)	0.015 (10)	0.002 (9)	0.004 (9)	0.000 (9)
C11	0.018 (10)	0.031 (14)	0.017 (10)	−0.006 (10)	0.004 (9)	0.000 (9)
C12	0.023 (11)	0.024 (13)	0.016 (10)	−0.007 (10)	0.006 (9)	−0.001 (9)
C13	0.021 (11)	0.026 (13)	0.014 (10)	−0.001 (9)	0.005 (9)	−0.001 (9)
C14	0.016 (10)	0.025 (13)	0.012 (9)	0.000 (9)	0.008 (9)	−0.003 (8)
C14A	0.019 (10)	0.024 (12)	0.009 (9)	−0.002 (9)	0.009 (9)	−0.001 (8)
C13A	0.026 (11)	0.017 (12)	0.019 (10)	0.002 (9)	0.004 (10)	−0.003 (9)
C12A	0.020 (11)	0.025 (13)	0.021 (10)	0.002 (9)	0.004 (10)	0.000 (9)
C11A	0.013 (10)	0.035 (15)	0.020 (10)	0.005 (9)	0.002 (9)	−0.006 (10)
C10A	0.018 (10)	0.027 (14)	0.014 (10)	−0.001 (9)	0.007 (9)	0.000 (9)
N15	0.018 (9)	0.017 (9)	0.016 (8)	0.002 (7)	0.009 (8)	−0.003 (7)
C16	0.017 (10)	0.020 (13)	0.022 (10)	0.003 (9)	0.008 (9)	−0.002 (9)
C17	0.018 (10)	0.029 (14)	0.021 (11)	−0.003 (9)	0.008 (10)	−0.004 (10)
C18	0.024 (11)	0.024 (13)	0.023 (11)	−0.001 (10)	0.005 (10)	−0.003 (10)
C19	0.021 (11)	0.031 (15)	0.033 (13)	0.001 (10)	0.002 (11)	0.009 (11)
C20	0.018 (10)	0.020 (12)	0.022 (10)	0.001 (9)	0.008 (9)	−0.001 (9)
C21	0.015 (10)	0.027 (14)	0.021 (10)	−0.003 (9)	0.010 (9)	−0.002 (9)
C22	0.025 (11)	0.028 (14)	0.019 (10)	0.000 (10)	0.005 (10)	−0.003 (9)
C23	0.044 (15)	0.040 (17)	0.027 (12)	−0.012 (13)	0.018 (12)	−0.012 (11)
C16A	0.017 (10)	0.017 (12)	0.022 (10)	−0.005 (9)	0.005 (9)	−0.001 (9)
C17A	0.022 (11)	0.023 (13)	0.021 (11)	0.000 (9)	0.007 (10)	0.000 (9)
C18A	0.014 (10)	0.027 (14)	0.018 (10)	0.002 (9)	0.002 (9)	0.002 (9)
C19A	0.020 (11)	0.032 (15)	0.024 (11)	−0.004 (10)	0.005 (10)	−0.006 (10)
C20A	0.018 (10)	0.019 (12)	0.019 (10)	−0.003 (9)	0.007 (9)	0.003 (9)
C21A	0.020 (10)	0.022 (13)	0.021 (10)	−0.001 (9)	0.012 (9)	0.001 (9)
C22A	0.026 (11)	0.031 (14)	0.020 (11)	−0.004 (10)	0.010 (10)	−0.005 (10)
C23A	0.025 (11)	0.030 (14)	0.024 (11)	−0.004 (10)	0.008 (10)	0.004 (10)
O1W	0.023 (8)	0.026 (10)	0.024 (8)	−0.001 (7)	0.011 (7)	−0.001 (7)
O1WA	0.026 (8)	0.026 (10)	0.025 (8)	0.002 (7)	0.011 (7)	0.000 (7)
O2W	0.021 (7)	0.031 (9)	0.023 (7)	0.001 (7)	0.008 (6)	−0.002 (7)

Geometric parameters (Å, º)

Co1—O7A	1.885 (15)	N15—C20A	1.52 (3)
Co1—O7	1.889 (15)	N15—C20	1.52 (3)
Co1—N9	1.920 (18)	N15—C16A	1.53 (3)
Co1—N9A	1.924 (18)	N15—C16	1.53 (3)
Co1—N1	1.943 (16)	C16—C17	1.51 (3)
Co1—N1A	1.957 (16)	C16—H16A	0.9900
N1—C2	1.36 (3)	C16—H16B	0.9900
N1—C6	1.37 (3)	C17—C18	1.53 (3)
C2—O2	1.23 (3)	C17—H17A	0.9900
C2—N3	1.40 (3)	C17—H17B	0.9900
N3—C4	1.38 (3)	C18—C19	1.54 (3)
N3—H3	0.8800	C18—H18A	0.9900

C4—O4	1.24 (2)	C18—H18B	0.9900
C4—C5	1.42 (3)	C19—H19A	0.9800
C5—C6	1.35 (3)	C19—H19B	0.9800
C5—H5	0.9500	C19—H19C	0.9800
C6—C7	1.51 (3)	C20—C21	1.52 (3)
C7—O8	1.22 (3)	C20—H20A	0.9900
C7—O7	1.31 (2)	C20—H20B	0.9900
N1A—C6A	1.34 (3)	C21—C22	1.52 (3)
N1A—C2A	1.37 (3)	C21—H21A	0.9900
C2A—O2A	1.23 (3)	C21—H21B	0.9900
C2A—N3A	1.39 (3)	C22—C23	1.52 (3)
N3A—C4A	1.38 (3)	C22—H22A	0.9900
N3A—H3A	0.8800	C22—H22B	0.9900
C4A—O4A	1.25 (2)	C23—H23A	0.9800
C4A—C5A	1.42 (3)	C23—H23B	0.9800
C5A—C6A	1.35 (3)	C23—H23C	0.9800
C5A—H5A	0.9500	C16A—C17A	1.52 (3)
C6A—C7A	1.50 (3)	C16A—H16C	0.9900
C7A—O8A	1.23 (3)	C16A—H16D	0.9900
C7A—O7A	1.29 (2)	C17A—C18A	1.53 (3)
N9—C10	1.33 (3)	C17A—H17C	0.9900
N9—C14	1.35 (3)	C17A—H17D	0.9900
N9A—C14A	1.33 (3)	C18A—C19A	1.53 (3)
N9A—C10A	1.33 (3)	C18A—H18C	0.9900
C10—C11	1.40 (3)	C18A—H18D	0.9900
C10—H10	0.9500	C19A—H19D	0.9800
C11—C12	1.37 (3)	C19A—H19E	0.9800
C11—H11	0.9500	C19A—H19F	0.9800
C12—C13	1.39 (3)	C20A—C21A	1.52 (3)
C12—H12	0.9500	C20A—H20C	0.9900
C13—C14	1.38 (3)	C20A—H20D	0.9900
C13—H13	0.9500	C21A—C22A	1.54 (3)
C14—C14A	1.46 (3)	C21A—H21C	0.9900
C14A—C13A	1.40 (3)	C21A—H21D	0.9900
C13A—C12A	1.38 (3)	C22A—C23A	1.49 (3)
C13A—H13A	0.9500	C22A—H22C	0.9900
C12A—C11A	1.38 (4)	C22A—H22D	0.9900
C12A—H12A	0.9500	C23A—H23D	0.9800
C11A—C10A	1.40 (3)	C23A—H23E	0.9800
C11A—H11A	0.9500	C23A—H23F	0.9800
C10A—H10A	0.9500		

O7A—Co1—O7	90.8 (6)	C20—N15—C16	111.3 (15)
O7A—Co1—N9	175.6 (7)	C16A—N15—C16	105.7 (16)
O7—Co1—N9	93.0 (7)	C17—C16—N15	116 (2)
O7A—Co1—N9A	93.6 (7)	C17—C16—H16A	108.3
O7—Co1—N9A	174.9 (7)	N15—C16—H16A	108.3
N9—Co1—N9A	82.7 (7)	C17—C16—H16B	108.3

O7A—Co1—N1	86.9 (7)	N15—C16—H16B	108.3
O7—Co1—N1	84.6 (7)	H16A—C16—H16B	107.4
N9—Co1—N1	91.3 (7)	C16—C17—C18	111 (2)
N9A—Co1—N1	98.4 (7)	C16—C17—H17A	109.5
O7A—Co1—N1A	84.1 (7)	C18—C17—H17A	109.5
O7—Co1—N1A	87.5 (7)	C16—C17—H17B	109.5
N9—Co1—N1A	98.3 (7)	C18—C17—H17B	109.5
N9A—Co1—N1A	90.2 (7)	H17A—C17—H17B	108.1
N1—Co1—N1A	167.9 (8)	C17—C18—C19	114.1 (19)
C2—N1—C6	118.9 (17)	C17—C18—H18A	108.7
C2—N1—Co1	128.7 (14)	C19—C18—H18A	108.7
C6—N1—Co1	111.7 (13)	C17—C18—H18B	108.7
O2—C2—N1	124.3 (18)	C19—C18—H18B	108.7
O2—C2—N3	119.5 (18)	H18A—C18—H18B	107.6
N1—C2—N3	116.2 (19)	C18—C19—H19A	109.5
C4—N3—C2	126.6 (18)	C18—C19—H19B	109.5
C4—N3—H3	116.7	H19A—C19—H19B	109.5
C2—N3—H3	116.7	C18—C19—H19C	109.5
O4—C4—N3	120.3 (19)	H19A—C19—H19C	109.5
O4—C4—C5	125 (2)	H19B—C19—H19C	109.5
N3—C4—C5	114.4 (18)	N15—C20—C21	114.9 (19)
C6—C5—C4	119 (2)	N15—C20—H20A	108.5
C6—C5—H5	120.7	C21—C20—H20A	108.5
C4—C5—H5	120.7	N15—C20—H20B	108.5
C5—C6—N1	125.3 (19)	C21—C20—H20B	108.5
C5—C6—C7	121.2 (19)	H20A—C20—H20B	107.5
N1—C6—C7	113.5 (17)	C22—C21—C20	109.1 (19)
O8—C7—O7	123.7 (19)	C22—C21—H21A	109.9
O8—C7—C6	122.6 (18)	C20—C21—H21A	109.9
O7—C7—C6	113.8 (18)	C22—C21—H21B	109.9
C7—O7—Co1	115.8 (13)	C20—C21—H21B	109.9
C6A—N1A—C2A	119.3 (17)	H21A—C21—H21B	108.3
C6A—N1A—Co1	111.3 (14)	C21—C22—C23	112 (2)
C2A—N1A—Co1	129.2 (14)	C21—C22—H22A	109.1
O2A—C2A—N1A	124.0 (19)	C23—C22—H22A	109.1
O2A—C2A—N3A	120.0 (19)	C21—C22—H22B	109.1
N1A—C2A—N3A	116.0 (18)	C23—C22—H22B	109.1
C4A—N3A—C2A	126.8 (18)	H22A—C22—H22B	107.8
C4A—N3A—H3A	116.6	C22—C23—H23A	109.5
C2A—N3A—H3A	116.6	C22—C23—H23B	109.5
O4A—C4A—N3A	120 (2)	H23A—C23—H23B	109.5
O4A—C4A—C5A	126 (2)	C22—C23—H23C	109.5
N3A—C4A—C5A	113.8 (18)	H23A—C23—H23C	109.5
C6A—C5A—C4A	119 (2)	H23B—C23—H23C	109.5
C6A—C5A—H5A	120.3	C17A—C16A—N15	115.9 (19)
C4A—C5A—H5A	120.3	C17A—C16A—H16C	108.3
N1A—C6A—C5A	125 (2)	N15—C16A—H16C	108.3
N1A—C6A—C7A	114.5 (17)	C17A—C16A—H16D	108.3

C5A—C6A—C7A	121 (2)	N15—C16A—H16D	108.3
O8A—C7A—O7A	125 (2)	H16C—C16A—H16D	107.4
O8A—C7A—C6A	121.8 (18)	C16A—C17A—C18A	109.9 (19)
O7A—C7A—C6A	113.6 (18)	C16A—C17A—H17C	109.7
C7A—O7A—Co1	116.4 (14)	C18A—C17A—H17C	109.7
C10—N9—C14	119.9 (19)	C16A—C17A—H17D	109.7
C10—N9—Co1	125.4 (16)	C18A—C17A—H17D	109.7
C14—N9—Co1	114.7 (13)	H17C—C17A—H17D	108.2
C14A—N9A—C10A	120 (2)	C17A—C18A—C19A	114.6 (18)
C14A—N9A—Co1	114.9 (14)	C17A—C18A—H18C	108.6
C10A—N9A—Co1	124.8 (17)	C19A—C18A—H18C	108.6
N9—C10—C11	121 (2)	C17A—C18A—H18D	108.6
N9—C10—H10	119.5	C19A—C18A—H18D	108.6
C11—C10—H10	119.5	H18C—C18A—H18D	107.6
C12—C11—C10	120 (2)	C18A—C19A—H19D	109.5
C12—C11—H11	120.2	C18A—C19A—H19E	109.5
C10—C11—H11	120.2	H19D—C19A—H19E	109.5
C11—C12—C13	119 (2)	C18A—C19A—H19F	109.5
C11—C12—H12	120.5	H19D—C19A—H19F	109.5
C13—C12—H12	120.5	H19E—C19A—H19F	109.5
C14—C13—C12	119 (2)	N15—C20A—C21A	116.1 (19)
C14—C13—H13	120.7	N15—C20A—H20C	108.3
C12—C13—H13	120.7	C21A—C20A—H20C	108.3
N9—C14—C13	122.0 (19)	N15—C20A—H20D	108.3
N9—C14—C14A	113 (2)	C21A—C20A—H20D	108.3
C13—C14—C14A	125 (2)	H20C—C20A—H20D	107.4
N9A—C14A—C13A	121.4 (19)	C20A—C21A—C22A	112 (2)
N9A—C14A—C14	114 (2)	C20A—C21A—H21C	109.2
C13A—C14A—C14	124 (2)	C22A—C21A—H21C	109.2
C12A—C13A—C14A	119 (2)	C20A—C21A—H21D	109.2
C12A—C13A—H13A	120.5	C22A—C21A—H21D	109.2
C14A—C13A—H13A	120.5	H21C—C21A—H21D	107.9
C13A—C12A—C11A	119 (2)	C23A—C22A—C21A	114.6 (18)
C13A—C12A—H12A	120.4	C23A—C22A—H22C	108.6
C11A—C12A—H12A	120.4	C21A—C22A—H22C	108.6
C12A—C11A—C10A	119 (2)	C23A—C22A—H22D	108.6
C12A—C11A—H11A	120.6	C21A—C22A—H22D	108.6
C10A—C11A—H11A	120.6	H22C—C22A—H22D	107.6
N9A—C10A—C11A	121 (2)	C22A—C23A—H23D	109.5
N9A—C10A—H10A	119.3	C22A—C23A—H23E	109.5
C11A—C10A—H10A	119.3	H23D—C23A—H23E	109.5
C20A—N15—C20	106.8 (17)	C22A—C23A—H23F	109.5
C20A—N15—C16A	110.6 (15)	H23D—C23A—H23F	109.5
C20—N15—C16A	110.9 (15)	H23E—C23A—H23F	109.5
C20A—N15—C16	111.6 (15)		

C6—N1—C2—O2	−179 (2)	N9A—Co1—O7A—C7A	92.1 (16)
Co1—N1—C2—O2	−10 (3)	N1—Co1—O7A—C7A	−169.7 (16)

C6—N1—C2—N3	2 (3)	N1A—Co1—O7A—C7A	2.2 (16)
Co1—N1—C2—N3	171.0 (14)	C14—N9—C10—C11	−1 (3)
O2—C2—N3—C4	−179.1 (19)	Co1—N9—C10—C11	−179.4 (15)
N1—C2—N3—C4	0 (3)	N9—C10—C11—C12	0 (3)
C2—N3—C4—O4	178.6 (19)	C10—C11—C12—C13	0 (3)
C2—N3—C4—C5	−2 (3)	C11—C12—C13—C14	1 (3)
O4—C4—C5—C6	−179 (2)	C10—N9—C14—C13	1 (3)
N3—C4—C5—C6	2 (3)	Co1—N9—C14—C13	−180.0 (15)
C4—C5—C6—N1	0 (3)	C10—N9—C14—C14A	−178.6 (18)
C4—C5—C6—C7	−179.1 (18)	Co1—N9—C14—C14A	0 (2)
C2—N1—C6—C5	−2 (3)	C12—C13—C14—N9	−1 (3)
Co1—N1—C6—C5	−173.0 (18)	C12—C13—C14—C14A	178.6 (19)
C2—N1—C6—C7	177.0 (18)	C10A—N9A—C14A—C13A	−1 (3)
Co1—N1—C6—C7	6 (2)	Co1—N9A—C14A—C13A	178.3 (15)
C5—C6—C7—O8	−3 (3)	C10A—N9A—C14A—C14	176.5 (17)
N1—C6—C7—O8	177 (2)	Co1—N9A—C14A—C14	−4 (2)
C5—C6—C7—O7	178.2 (19)	N9—C14—C14A—N9A	3 (2)
N1—C6—C7—O7	−1 (3)	C13—C14—C14A—N9A	−177.0 (19)
O8—C7—O7—Co1	176.6 (16)	N9—C14—C14A—C13A	179.8 (18)
C6—C7—O7—Co1	−5 (2)	C13—C14—C14A—C13A	0 (3)
O7A—Co1—O7—C7	−80.0 (15)	N9A—C14A—C13A—C12A	−1 (3)
N9—Co1—O7—C7	97.8 (15)	C14—C14A—C13A—C12A	−177.4 (19)
N1—Co1—O7—C7	6.8 (15)	C14A—C13A—C12A—C11A	2 (3)
N1A—Co1—O7—C7	−164.0 (15)	C13A—C12A—C11A—C10A	−3 (3)
C6A—N1A—C2A—O2A	−180 (2)	C14A—N9A—C10A—C11A	0 (3)
Co1—N1A—C2A—O2A	−4 (3)	Co1—N9A—C10A—C11A	−178.8 (15)
C6A—N1A—C2A—N3A	0 (3)	C12A—C11A—C10A—N9A	2 (3)
Co1—N1A—C2A—N3A	175.5 (14)	C20A—N15—C16—C17	74 (2)
O2A—C2A—N3A—C4A	−179 (2)	C20—N15—C16—C17	−45 (2)
N1A—C2A—N3A—C4A	1 (3)	C16A—N15—C16—C17	−165.6 (18)
C2A—N3A—C4A—O4A	178 (2)	N15—C16—C17—C18	−173.8 (18)
C2A—N3A—C4A—C5A	−1 (3)	C16—C17—C18—C19	−54 (3)
O4A—C4A—C5A—C6A	−178 (2)	C20A—N15—C20—C21	178.4 (16)
N3A—C4A—C5A—C6A	0 (3)	C16A—N15—C20—C21	58 (2)
C2A—N1A—C6A—C5A	−1 (3)	C16—N15—C20—C21	−60 (2)
Co1—N1A—C6A—C5A	−177.1 (18)	N15—C20—C21—C22	166.1 (17)
C2A—N1A—C6A—C7A	178.9 (18)	C20—C21—C22—C23	174.9 (19)
Co1—N1A—C6A—C7A	3 (2)	C20A—N15—C16A—C17A	−51 (2)
C4A—C5A—C6A—N1A	1 (3)	C20—N15—C16A—C17A	68 (2)
C4A—C5A—C6A—C7A	−179.0 (19)	C16—N15—C16A—C17A	−171.6 (18)
N1A—C6A—C7A—O8A	179 (2)	N15—C16A—C17A—C18A	−172.8 (17)
C5A—C6A—C7A—O8A	−1 (3)	C16A—C17A—C18A—C19A	−53 (2)
N1A—C6A—C7A—O7A	−1 (3)	C20—N15—C20A—C21A	−177.1 (17)
C5A—C6A—C7A—O7A	179 (2)	C16A—N15—C20A—C21A	−56 (2)
O8A—C7A—O7A—Co1	178.9 (17)	C16—N15—C20A—C21A	61 (2)
C6A—C7A—O7A—Co1	−1 (2)	N15—C20A—C21A—C22A	164.2 (17)
O7—Co1—O7A—C7A	−85.2 (15)	C20A—C21A—C22A—C23A	68 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O7	0.95	2.39	2.90 (3)	114
C10*A*—H10*A*···O7*A*	0.95	2.39	2.91 (3)	114
C11—H11···O4^{i}	0.95	2.69	3.29 (3)	122
C12—H12···O8^{ii}	0.95	2.64	3.22 (3)	120
C12—H12···O4^{i}	0.95	2.60	3.24 (3)	126
C18—H18*B*···O7*A*^{iii}	0.99	2.81	3.30 (3)	111
C11*A*—H11*A*···O4*A*^{iv}	0.95	2.56	3.20 (3)	125
C12*A*—H12*A*···O4*A*^{iv}	0.95	2.68	3.26 (3)	120
C12*A*—H12*A*···O8*A*^{ii}	0.95	2.65	3.20 (3)	117
C13*A*—H13*A*···O8*A*^{ii}	0.95	2.49	3.12 (3)	124
C16—H16*A*···O4^{v}	0.99	2.41	3.38 (3)	168
C16*A*—H16*C*···O4*A*^{vi}	0.99	2.40	3.38 (3)	170
C18*A*—H18*D*···O7	0.99	2.67	3.32 (3)	124
C20*A*—H20*D*···O1*W*	0.99	2.45	3.41 (3)	165
C21*A*—H21*D*···O8*A*^{iii}	0.99	2.49	3.45 (3)	164
N3—H3···O1*WA*^{i}	0.88	2.03	2.90 (2)	171
N3*A*—H3*A*···O1*W*^{vii}	0.88	2.00	2.86 (2)	168

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

Tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(III) trihydrate (2c)

Crystal data

$(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$
M_r = 819.79
Monoclinic, *P*2/*n*
a = 13.0259 (4) Å
b = 9.3504 (3) Å
c = 16.3308 (5) Å
β = 103.847 (3)°
V = 1931.24 (11) Å^3
Z = 2
F(000) = 868
D_x = 1.410 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 12294 reflections
θ = 4.2–28.7°
μ = 0.51 mm^{-1}
T = 220 K
Rhomb, red
0.67 × 0.39 × 0.30 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0655 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.912, T_{max} = 1.000
21720 measured reflections
4673 independent reflections
3836 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.8°, θ_{min} = 4.2°
h = −17→17
k = −11→12
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.129
S = 1.07
4673 reflections

264 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.9763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.04$ e Å^{-3}
$\Delta\rho_{min} = -0.92$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.2500	0.33599 (4)	0.7500	0.02272 (13)
N1	0.25009 (14)	0.31516 (18)	0.86903 (10)	0.0242 (4)
C2	0.18681 (17)	0.3800 (2)	0.91408 (13)	0.0268 (4)
O2	0.12250 (14)	0.47428 (19)	0.88633 (10)	0.0394 (4)
N3	0.19918 (14)	0.33411 (19)	0.99698 (11)	0.0276 (4)
H3	0.1594	0.3753	1.0259	0.033*
C4	0.26756 (16)	0.2305 (2)	1.03818 (13)	0.0260 (4)
O4	0.27188 (13)	0.20141 (18)	1.11312 (9)	0.0331 (4)
C5	0.33075 (17)	0.1657 (2)	0.98759 (14)	0.0287 (4)
H5	0.3802	0.0941	1.0099	0.034*
C6	0.31732 (16)	0.2102 (2)	0.90692 (13)	0.0243 (4)
C7	0.37898 (17)	0.1424 (2)	0.84921 (13)	0.0259 (4)
O7	0.35619 (12)	0.19438 (15)	0.77356 (9)	0.0281 (3)
O8	0.44460 (13)	0.04958 (17)	0.87394 (11)	0.0373 (4)
N9	0.35004 (13)	0.49043 (18)	0.77636 (11)	0.0250 (4)
C10	0.45493 (17)	0.4766 (2)	0.80301 (14)	0.0315 (5)
H10	0.4844	0.3844	0.8106	0.038*
C11	0.52116 (19)	0.5935 (3)	0.81962 (16)	0.0374 (5)
H11	0.5946	0.5811	0.8387	0.045*
C12	0.4781 (2)	0.7294 (3)	0.80788 (17)	0.0420 (6)
H12	0.5217	0.8106	0.8190	0.050*
C13	0.3702 (2)	0.7435 (3)	0.77965 (17)	0.0407 (6)
H13	0.3392	0.8347	0.7707	0.049*
C14	0.30789 (17)	0.6223 (2)	0.76457 (14)	0.0292 (5)
N15	0.7500	0.1495 (3)	0.7500	0.0362 (6)
C16	0.84331 (17)	0.0515 (2)	0.78540 (15)	0.0318 (5)
H16A	0.8620	0.0018	0.7381	0.038*
H16B	0.8204	−0.0212	0.8204	0.038*
C17	0.94257 (19)	0.1229 (3)	0.83773 (17)	0.0399 (6)
H17A	0.9284	0.1610	0.8898	0.048*
H17B	0.9626	0.2029	0.8060	0.048*
C18	1.03332 (19)	0.0155 (3)	0.85930 (18)	0.0421 (6)

H18A	1.0539	−0.0102	0.8073	0.051*
H18B	1.0942	0.0621	0.8967	0.051*
C19	1.0087 (2)	−0.1208 (3)	0.9014 (2)	0.0542 (7)
H19A	0.9809	−0.0967	0.9497	0.081*
H19B	1.0728	−0.1767	0.9198	0.081*
H19C	0.9566	−0.1761	0.8615	0.081*
C20	0.7278 (2)	0.2471 (3)	0.8177 (2)	0.0516 (7)
H20A	0.7895	0.3090	0.8375	0.062*
H20B	0.6681	0.3089	0.7920	0.062*
C21	0.7033 (2)	0.1756 (3)	0.8928 (2)	0.0579 (8)
H21A	0.7558	0.1013	0.9145	0.069*
H21B	0.6333	0.1309	0.8772	0.069*
C22	0.7059 (3)	0.2906 (5)	0.9601 (3)	0.0931 (15)
H22A	0.7800	0.3163	0.9837	0.112*
H22B	0.6705	0.3758	0.9318	0.112*
C23	0.6631 (5)	0.2607 (9)	1.0237 (4)	0.177 (4)
H23A	0.6013	0.2009	1.0039	0.266*
H23B	0.6425	0.3488	1.0468	0.266*
H23C	0.7142	0.2103	1.0671	0.266*
O1W	0.59064 (17)	0.5169 (2)	0.60831 (13)	0.0419 (4)
H1WA	0.528 (4)	0.511 (5)	0.604 (3)	0.102 (16)*
H1WB	0.617 (3)	0.558 (4)	0.654 (3)	0.078 (13)*
O2W	0.7500	0.6371 (3)	0.7500	0.0408 (6)
H2W	0.742 (3)	0.685 (4)	0.788 (2)	0.066 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0278 (2)	0.0220 (2)	0.0220 (2)	0.000	0.01305 (15)	0.000
N1	0.0288 (8)	0.0259 (8)	0.0207 (8)	0.0015 (7)	0.0118 (7)	−0.0003 (6)
C2	0.0297 (10)	0.0285 (10)	0.0254 (10)	0.0013 (8)	0.0131 (8)	−0.0004 (8)
O2	0.0454 (10)	0.0467 (10)	0.0313 (9)	0.0202 (8)	0.0195 (7)	0.0068 (7)
N3	0.0315 (9)	0.0316 (9)	0.0241 (8)	0.0023 (7)	0.0150 (7)	−0.0017 (7)
C4	0.0288 (10)	0.0276 (10)	0.0236 (10)	−0.0053 (8)	0.0101 (8)	−0.0014 (8)
O4	0.0401 (9)	0.0397 (9)	0.0225 (7)	−0.0017 (7)	0.0133 (6)	0.0014 (6)
C5	0.0347 (11)	0.0264 (10)	0.0278 (10)	0.0018 (9)	0.0127 (9)	0.0014 (8)
C6	0.0286 (10)	0.0205 (9)	0.0269 (10)	−0.0024 (7)	0.0128 (8)	−0.0031 (8)
C7	0.0332 (10)	0.0209 (9)	0.0273 (10)	−0.0018 (8)	0.0145 (8)	−0.0031 (8)
O7	0.0361 (8)	0.0263 (7)	0.0266 (7)	0.0061 (6)	0.0169 (6)	0.0013 (6)
O8	0.0464 (10)	0.0333 (8)	0.0362 (9)	0.0143 (7)	0.0175 (7)	0.0039 (7)
N9	0.0273 (9)	0.0268 (8)	0.0236 (9)	−0.0009 (7)	0.0117 (7)	−0.0012 (7)
C10	0.0303 (11)	0.0329 (11)	0.0339 (11)	0.0004 (9)	0.0126 (9)	−0.0001 (9)
C11	0.0297 (11)	0.0399 (13)	0.0428 (13)	−0.0056 (10)	0.0094 (10)	−0.0010 (10)
C12	0.0379 (13)	0.0350 (13)	0.0521 (15)	−0.0121 (10)	0.0091 (11)	−0.0042 (11)
C13	0.0432 (14)	0.0256 (11)	0.0527 (15)	−0.0034 (10)	0.0103 (11)	−0.0009 (10)
C14	0.0322 (11)	0.0259 (10)	0.0316 (11)	−0.0004 (8)	0.0115 (9)	−0.0016 (8)
N15	0.0333 (14)	0.0236 (13)	0.0511 (17)	0.000	0.0090 (12)	0.000
C16	0.0342 (11)	0.0250 (10)	0.0376 (12)	0.0012 (8)	0.0114 (9)	0.0013 (9)

C17	0.0362 (12)	0.0337 (12)	0.0493 (15)	−0.0052 (9)	0.0091 (11)	0.0017 (11)
C18	0.0332 (12)	0.0432 (14)	0.0497 (15)	−0.0037 (10)	0.0096 (11)	0.0024 (11)
C19	0.0497 (16)	0.0526 (16)	0.0579 (18)	0.0012 (13)	0.0083 (14)	0.0148 (14)
C20	0.0399 (14)	0.0324 (13)	0.080 (2)	0.0027 (10)	0.0096 (14)	−0.0212 (13)
C21	0.0481 (16)	0.0605 (18)	0.070 (2)	−0.0095 (14)	0.0232 (15)	−0.0366 (16)
C22	0.065 (2)	0.108 (3)	0.109 (3)	−0.012 (2)	0.025 (2)	−0.078 (3)
C23	0.161 (6)	0.255 (8)	0.154 (6)	−0.125 (6)	0.114 (5)	−0.158 (6)
O1W	0.0426 (11)	0.0451 (10)	0.0448 (11)	−0.0054 (8)	0.0236 (9)	−0.0041 (8)
O2W	0.0505 (15)	0.0456 (15)	0.0316 (13)	0.000	0.0201 (12)	0.000

Geometric parameters (Å, º)

Co1—O7^{i}	1.8867 (15)	N15—C20^{ii}	1.514 (3)
Co1—O7	1.8868 (14)	N15—C16	1.521 (3)
Co1—N9^{i}	1.9236 (17)	N15—C16^{ii}	1.521 (3)
Co1—N9	1.9236 (17)	C16—C17	1.523 (3)
Co1—N1	1.9534 (16)	C16—H16A	0.9800
Co1—N1^{i}	1.9534 (16)	C16—H16B	0.9800
N1—C6	1.362 (3)	C17—C18	1.527 (3)
N1—C2	1.371 (2)	C17—H17A	0.9800
C2—O2	1.225 (3)	C17—H17B	0.9800
C2—N3	1.392 (3)	C18—C19	1.518 (4)
N3—C4	1.377 (3)	C18—H18A	0.9800
N3—H3	0.8700	C18—H18B	0.9800
C4—O4	1.242 (2)	C19—H19A	0.9700
C4—C5	1.433 (3)	C19—H19B	0.9700
C5—C6	1.352 (3)	C19—H19C	0.9700
C5—H5	0.9400	C20—C21	1.497 (5)
C6—C7	1.516 (3)	C20—H20A	0.9800
C7—O8	1.217 (3)	C20—H20B	0.9800
C7—O7	1.294 (3)	C21—C22	1.532 (4)
N9—C10	1.337 (3)	C21—H21A	0.9800
N9—C14	1.345 (3)	C21—H21B	0.9800
C10—C11	1.379 (3)	C22—C23	1.321 (7)
C10—H10	0.9400	C22—H22A	0.9800
C11—C12	1.383 (4)	C22—H22B	0.9800
C11—H11	0.9400	C23—H23A	0.9700
C12—C13	1.377 (4)	C23—H23B	0.9700
C12—H12	0.9400	C23—H23C	0.9700
C13—C14	1.381 (3)	O1W—H1WA	0.81 (5)
C13—H13	0.9400	O1W—H1WB	0.84 (4)
C14—C14^{i}	1.469 (4)	O2W—H2W	0.79 (3)
N15—C20	1.514 (3)		

O7^{i}—Co1—O7	90.86 (9)	C20—N15—C20^{ii}	105.9 (3)
O7^{i}—Co1—N9^{i}	93.26 (7)	C20—N15—C16	111.10 (15)
O7—Co1—N9^{i}	175.41 (7)	C20^{ii}—N15—C16	111.46 (13)
O7^{i}—Co1—N9	175.41 (7)	C20—N15—C16^{ii}	111.46 (14)

O7—Co1—N9	93.26 (7)	C20^{ii}—N15—C16^{ii}	111.10 (15)
N9^{i}—Co1—N9	82.69 (10)	C16—N15—C16^{ii}	106.0 (2)
O7^{i}—Co1—N1	87.64 (7)	N15—C16—C17	116.32 (18)
O7—Co1—N1	84.33 (7)	N15—C16—H16A	108.2
N9^{i}—Co1—N1	97.85 (7)	C17—C16—H16A	108.2
N9—Co1—N1	90.75 (7)	N15—C16—H16B	108.2
O7^{i}—Co1—N1^{i}	84.33 (7)	C17—C16—H16B	108.2
O7—Co1—N1^{i}	87.64 (7)	H16A—C16—H16B	107.4
N9^{i}—Co1—N1^{i}	90.75 (7)	C16—C17—C18	110.4 (2)
N9—Co1—N1^{i}	97.85 (7)	C16—C17—H17A	109.6
N1—Co1—N1^{i}	168.56 (10)	C18—C17—H17A	109.6
C6—N1—C2	118.63 (17)	C16—C17—H17B	109.6
C6—N1—Co1	111.79 (12)	C18—C17—H17B	109.6
C2—N1—Co1	129.26 (14)	H17A—C17—H17B	108.1
O2—C2—N1	124.42 (19)	C19—C18—C17	114.9 (2)
O2—C2—N3	119.42 (18)	C19—C18—H18A	108.5
N1—C2—N3	116.15 (18)	C17—C18—H18A	108.5
C4—N3—C2	127.06 (17)	C19—C18—H18B	108.5
C4—N3—H3	116.5	C17—C18—H18B	108.5
C2—N3—H3	116.5	H18A—C18—H18B	107.5
O4—C4—N3	120.46 (18)	C18—C19—H19A	109.5
O4—C4—C5	125.4 (2)	C18—C19—H19B	109.5
N3—C4—C5	114.17 (17)	H19A—C19—H19B	109.5
C6—C5—C4	118.25 (19)	C18—C19—H19C	109.5
C6—C5—H5	120.9	H19A—C19—H19C	109.5
C4—C5—H5	120.9	H19B—C19—H19C	109.5
C5—C6—N1	125.70 (18)	C21—C20—N15	116.4 (2)
C5—C6—C7	120.87 (19)	C21—C20—H20A	108.2
N1—C6—C7	113.43 (17)	N15—C20—H20A	108.2
O8—C7—O7	124.53 (18)	C21—C20—H20B	108.2
O8—C7—C6	121.75 (19)	N15—C20—H20B	108.2
O7—C7—C6	113.71 (18)	H20A—C20—H20B	107.3
C7—O7—Co1	116.59 (12)	C20—C21—C22	107.6 (3)
C10—N9—C14	119.06 (19)	C20—C21—H21A	110.2
C10—N9—Co1	125.78 (15)	C22—C21—H21A	110.2
C14—N9—Co1	115.15 (14)	C20—C21—H21B	110.2
N9—C10—C11	122.0 (2)	C22—C21—H21B	110.2
N9—C10—H10	119.0	H21A—C21—H21B	108.5
C11—C10—H10	119.0	C23—C22—C21	118.3 (4)
C10—C11—C12	119.2 (2)	C23—C22—H22A	107.7
C10—C11—H11	120.4	C21—C22—H22A	107.7
C12—C11—H11	120.4	C23—C22—H22B	107.7
C13—C12—C11	118.8 (2)	C21—C22—H22B	107.7
C13—C12—H12	120.6	H22A—C22—H22B	107.1
C11—C12—H12	120.6	C22—C23—H23A	109.5
C12—C13—C14	119.4 (2)	C22—C23—H23B	109.5
C12—C13—H13	120.3	H23A—C23—H23B	109.5
C14—C13—H13	120.3	C22—C23—H23C	109.5

N9—C14—C13	121.6 (2)	H23A—C23—H23C	109.5
N9—C14—C14^{i}	113.49 (12)	H23B—C23—H23C	109.5
C13—C14—C14^{i}	124.89 (14)	H1WA—O1W—H1WB	107 (4)
C6—N1—C2—O2	−179.1 (2)	N1—Co1—O7—C7	3.05 (15)
Co1—N1—C2—O2	−6.2 (3)	N1^{i}—Co1—O7—C7	−168.78 (15)
C6—N1—C2—N3	1.5 (3)	C14—N9—C10—C11	0.7 (3)
Co1—N1—C2—N3	174.43 (14)	Co1—N9—C10—C11	179.27 (17)
O2—C2—N3—C4	−179.7 (2)	N9—C10—C11—C12	−0.5 (4)
N1—C2—N3—C4	−0.3 (3)	C10—C11—C12—C13	−0.2 (4)
C2—N3—C4—O4	178.8 (2)	C11—C12—C13—C14	0.6 (4)
C2—N3—C4—C5	−0.3 (3)	C10—N9—C14—C13	−0.3 (3)
O4—C4—C5—C6	−179.4 (2)	Co1—N9—C14—C13	−179.00 (18)
N3—C4—C5—C6	−0.4 (3)	C10—N9—C14—C14^{i}	−179.9 (2)
C4—C5—C6—N1	1.8 (3)	Co1—N9—C14—C14^{i}	1.4 (3)
C4—C5—C6—C7	−177.90 (18)	C12—C13—C14—N9	−0.4 (4)
C2—N1—C6—C5	−2.4 (3)	C12—C13—C14—C14^{i}	179.2 (3)
Co1—N1—C6—C5	−176.47 (18)	C20—N15—C16—C17	−49.0 (3)
C2—N1—C6—C7	177.27 (18)	C20^{ii}—N15—C16—C17	68.8 (3)
Co1—N1—C6—C7	3.2 (2)	C16^{ii}—N15—C16—C17	−170.2 (2)
C5—C6—C7—O8	−2.3 (3)	N15—C16—C17—C18	−172.76 (18)
N1—C6—C7—O8	177.96 (19)	C16—C17—C18—C19	−53.7 (3)
C5—C6—C7—O7	178.77 (19)	C20^{ii}—N15—C20—C21	−180.0 (3)
N1—C6—C7—O7	−0.9 (3)	C16—N15—C20—C21	−58.8 (3)
O8—C7—O7—Co1	179.15 (17)	C16^{ii}—N15—C20—C21	59.1 (3)
C6—C7—O7—Co1	−2.0 (2)	N15—C20—C21—C22	168.7 (2)
O7^{i}—Co1—O7—C7	−84.49 (14)	C20—C21—C22—C23	164.5 (6)
N9—Co1—O7—C7	93.48 (15)		

Symmetry codes: (i) −*x*+1/2, *y*, −*z*+3/2; (ii) −*x*+3/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1*W*^{iii}	0.87	2.05	2.911 (2)	170
O1*W*—H1*WA*···O2^{i}	0.81 (5)	2.03 (5)	2.827 (3)	169 (5)
O1*W*—H1*WB*···O2*W*	0.84 (4)	2.17 (4)	2.937 (3)	152 (4)
O1*W*—H1*WA*···O2^{i}	0.81 (5)	2.03 (5)	2.827 (3)	169 (5)
O2*W*—H2*W*···O4^{iv}	0.79 (3)	1.98 (3)	2.768 (2)	177 (4)
C10—H10···O7	0.94	2.41	2.924 (3)	114
C16—H16*B*···O4^{v}	0.98	2.47	3.433 (3)	168
C20—H20*A*···O1*W*^{iii}	0.98	2.52	3.471 (3)	164
C21—H21*B*···O8	0.98	2.56	3.513 (3)	164

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

Tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(III) trihydrate (2d)

Crystal data

$(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$
$M_r = 819.79$
Monoclinic, $P2/n$
$a = 13.0080\ (8)$ Å
$b = 9.3320\ (6)$ Å
$c = 16.3753\ (12)$ Å
$\beta = 104.364\ (7)°$
$V = 1925.7\ (2)$ Å^3
$Z = 2$
$F(000) = 868$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7757 reflections
$\theta = 4.1–28.3°$
$\mu = 0.51$ mm^{-1}
$T = 170$ K
Block, red
0.67 × 0.39 × 0.30 mm

Data collection

Agilent Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0655 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2011)
$T_{min} = 0.680$, $T_{max} = 1.000$
22527 measured reflections
4643 independent reflections
3676 reflections with $I > 2\sigma(I)$
$R_{int} = 0.165$
$\theta_{max} = 28.8°$, $\theta_{min} = 4.2°$
$h = -17\rightarrow17$
$k = -12\rightarrow11$
$l = -21\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.165$
$S = 1.08$
4643 reflections
282 parameters
39 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 1.2057P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.74$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}

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. This was a two-domain crystal with monoclinic and triclinic phases present. The structures of the two phases were analyzed independently of each other.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co1	0.2500	0.33524 (5)	0.7500	0.01891 (16)	
N1	0.25035 (17)	0.3147 (2)	0.86903 (13)	0.0208 (4)	
C2	0.18683 (19)	0.3799 (3)	0.91394 (16)	0.0218 (5)	
O2	0.12219 (16)	0.4741 (2)	0.88602 (13)	0.0318 (5)	
N3	0.19927 (17)	0.3329 (2)	0.99702 (14)	0.0231 (4)	

H3	0.157 (3)	0.371 (4)	1.021 (2)	0.028*	
C4	0.2680 (2)	0.2283 (3)	1.03858 (16)	0.0220 (5)	
O4	0.27154 (15)	0.1977 (2)	1.11326 (12)	0.0274 (4)	
C5	0.3319 (2)	0.1636 (3)	0.98850 (16)	0.0235 (5)	
H5	0.3824	0.0916	1.0115	0.028*	
C6	0.31846 (19)	0.2077 (3)	0.90772 (16)	0.0216 (5)	
C7	0.38018 (19)	0.1409 (3)	0.84996 (16)	0.0224 (5)	
O7	0.35672 (14)	0.19295 (19)	0.77379 (11)	0.0229 (4)	
O8	0.44687 (15)	0.0477 (2)	0.87518 (13)	0.0300 (4)	
N9	0.35087 (17)	0.4897 (2)	0.77653 (13)	0.0211 (4)	
C10	0.4568 (2)	0.4757 (3)	0.80340 (17)	0.0257 (5)	
H10	0.4868	0.3824	0.8114	0.031*	
C11	0.5231 (2)	0.5932 (3)	0.81961 (19)	0.0298 (6)	
H11	0.5977	0.5806	0.8392	0.036*	
C12	0.4801 (2)	0.7301 (3)	0.8071 (2)	0.0347 (6)	
H12	0.5246	0.8122	0.8181	0.042*	
C13	0.3711 (2)	0.7447 (3)	0.7784 (2)	0.0348 (6)	
H13	0.3397	0.8371	0.7686	0.042*	
C14	0.3088 (2)	0.6228 (3)	0.76410 (17)	0.0242 (5)	
N15	0.7500	0.1518 (3)	0.7500	0.0294 (7)	
C16	0.8441 (2)	0.0535 (3)	0.78585 (18)	0.0265 (5)	
H16A	0.8628	0.0033	0.7383	0.032*	
H16B	0.8211	−0.0203	0.8210	0.032*	
C17	0.9442 (2)	0.1252 (3)	0.8388 (2)	0.0310 (6)	
H17A	0.9300	0.1639	0.8913	0.037*	
H17B	0.9646	0.2059	0.8070	0.037*	
C18	1.0352 (2)	0.0163 (3)	0.8609 (2)	0.0330 (6)	
H18A	1.0563	−0.0089	0.8087	0.040*	
H18B	1.0969	0.0628	0.8995	0.040*	
C19	1.0101 (3)	−0.1217 (4)	0.9020 (2)	0.0390 (7)	
H19A	0.9799	−0.0982	0.9496	0.058*	
H19B	1.0755	−0.1772	0.9223	0.058*	
H19C	0.9589	−0.1784	0.8606	0.058*	
C20	0.7277 (2)	0.2506 (3)	0.8178 (2)	0.0402 (8)	
H20A	0.7902	0.3133	0.8380	0.048*	
H20B	0.6670	0.3130	0.7913	0.048*	
C21	0.7033 (3)	0.1795 (4)	0.8925 (2)	0.0438 (8)	
H21A	0.7576	0.1063	0.9164	0.053*	0.755 (10)
H21B	0.6328	0.1327	0.8768	0.053*	0.755 (10)
H21C	0.7465	0.0909	0.9011	0.053*	0.245 (10)
H21D	0.6286	0.1477	0.8730	0.053*	0.245 (10)
C22A	0.7046 (4)	0.3016 (5)	0.9563 (3)	0.0360 (12)	0.755 (10)
H22A	0.7772	0.3412	0.9749	0.043*	0.755 (10)
H22B	0.6568	0.3795	0.9287	0.043*	0.755 (10)
C23A	0.6692 (5)	0.2479 (6)	1.0321 (3)	0.0534 (16)	0.755 (10)
H23A	0.6706	0.3271	1.0716	0.080*	0.755 (10)
H23B	0.5968	0.2101	1.0138	0.080*	0.755 (10)
H23C	0.7172	0.1718	1.0599	0.080*	0.755 (10)

C22B	0.7136 (12)	0.2422 (17)	0.9850 (9)	0.044 (4)	0.245 (10)
H22C	0.7094	0.1628	1.0241	0.052*	0.245 (10)
H22D	0.7834	0.2896	1.0052	0.052*	0.245 (10)
C23B	0.6275 (11)	0.3471 (16)	0.9845 (9)	0.047 (4)	0.245 (10)
H23D	0.6350	0.3845	1.0416	0.071*	0.245 (10)
H23E	0.6323	0.4264	0.9463	0.071*	0.245 (10)
H23F	0.5585	0.2998	0.9653	0.071*	0.245 (10)
O1W	0.59094 (18)	0.5190 (2)	0.60803 (15)	0.0322 (5)	
H1WA	0.533 (3)	0.514 (5)	0.602 (2)	0.039*	
H1WB	0.621 (3)	0.546 (5)	0.656 (3)	0.039*	
O2W	0.7500	0.6415 (3)	0.7500	0.0331 (6)	
H2W	0.738 (3)	0.691 (5)	0.793 (2)	0.040*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0235 (3)	0.0142 (2)	0.0214 (3)	0.000	0.01007 (17)	0.000
N1	0.0246 (10)	0.0180 (10)	0.0215 (10)	0.0014 (8)	0.0087 (8)	−0.0001 (8)
C2	0.0236 (11)	0.0191 (12)	0.0244 (12)	0.0003 (10)	0.0091 (9)	0.0003 (9)
O2	0.0368 (11)	0.0315 (11)	0.0305 (10)	0.0135 (9)	0.0145 (8)	0.0046 (8)
N3	0.0267 (10)	0.0224 (11)	0.0236 (10)	0.0027 (9)	0.0126 (8)	−0.0013 (8)
C4	0.0241 (12)	0.0171 (12)	0.0254 (12)	−0.0050 (10)	0.0075 (9)	−0.0012 (9)
O4	0.0326 (10)	0.0289 (10)	0.0231 (9)	−0.0020 (8)	0.0111 (7)	0.0020 (7)
C5	0.0257 (12)	0.0186 (12)	0.0273 (12)	0.0036 (10)	0.0086 (10)	0.0008 (10)
C6	0.0233 (12)	0.0159 (11)	0.0269 (12)	−0.0010 (9)	0.0084 (9)	−0.0021 (9)
C7	0.0243 (11)	0.0206 (12)	0.0244 (12)	−0.0008 (10)	0.0101 (9)	−0.0024 (9)
O7	0.0289 (9)	0.0168 (8)	0.0254 (9)	0.0029 (7)	0.0115 (7)	0.0002 (7)
O8	0.0359 (11)	0.0216 (9)	0.0356 (11)	0.0112 (8)	0.0146 (8)	0.0019 (8)
N9	0.0260 (10)	0.0184 (10)	0.0215 (10)	0.0021 (8)	0.0106 (8)	−0.0018 (8)
C10	0.0258 (12)	0.0223 (13)	0.0311 (13)	0.0000 (10)	0.0109 (10)	−0.0020 (10)
C11	0.0257 (13)	0.0264 (14)	0.0384 (16)	−0.0015 (11)	0.0099 (11)	−0.0004 (11)
C12	0.0343 (15)	0.0218 (14)	0.0472 (17)	−0.0094 (12)	0.0086 (12)	−0.0024 (12)
C13	0.0351 (15)	0.0202 (13)	0.0478 (18)	−0.0023 (12)	0.0082 (12)	−0.0018 (12)
C14	0.0270 (13)	0.0176 (12)	0.0292 (14)	−0.0016 (10)	0.0094 (10)	0.0006 (9)
N15	0.0264 (15)	0.0170 (15)	0.044 (2)	0.000	0.0069 (13)	0.000
C16	0.0280 (13)	0.0162 (12)	0.0351 (15)	0.0012 (10)	0.0074 (11)	0.0022 (10)
C17	0.0288 (13)	0.0224 (13)	0.0408 (16)	−0.0047 (11)	0.0063 (11)	0.0011 (11)
C18	0.0266 (13)	0.0282 (15)	0.0429 (17)	−0.0038 (11)	0.0064 (11)	0.0039 (12)
C19	0.0385 (16)	0.0316 (16)	0.0453 (18)	−0.0016 (13)	0.0077 (13)	0.0096 (13)
C20	0.0322 (15)	0.0209 (14)	0.064 (2)	0.0019 (12)	0.0061 (14)	−0.0131 (13)
C21	0.0356 (16)	0.0418 (19)	0.057 (2)	−0.0036 (14)	0.0166 (14)	−0.0240 (16)
C22A	0.045 (2)	0.022 (2)	0.038 (2)	0.002 (2)	0.0029 (19)	−0.0073 (18)
C23A	0.067 (3)	0.055 (3)	0.041 (3)	−0.011 (3)	0.020 (2)	−0.020 (2)
C22B	0.049 (8)	0.022 (8)	0.063 (11)	−0.001 (7)	0.019 (8)	−0.011 (7)
C23B	0.053 (8)	0.053 (9)	0.041 (8)	−0.002 (7)	0.020 (6)	−0.006 (7)
O1W	0.0321 (10)	0.0308 (11)	0.0377 (12)	−0.0039 (9)	0.0160 (9)	−0.0028 (9)
O2W	0.0403 (16)	0.0333 (16)	0.0298 (15)	0.000	0.0163 (12)	0.000

Geometric parameters (Å, º)

Co1—O7	1.8905 (18)	C16—H16A	0.9900
Co1—O7^{i}	1.8905 (18)	C16—H16B	0.9900
Co1—N9	1.925 (2)	C17—C18	1.533 (4)
Co1—N9^{i}	1.925 (2)	C17—H17A	0.9900
Co1—N1^{i}	1.957 (2)	C17—H17B	0.9900
Co1—N1	1.957 (2)	C18—C19	1.525 (4)
N1—C2	1.377 (3)	C18—H18A	0.9900
N1—C6	1.380 (3)	C18—H18B	0.9900
C2—O2	1.225 (3)	C19—H19A	0.9800
C2—N3	1.400 (3)	C19—H19B	0.9800
N3—C4	1.383 (3)	C19—H19C	0.9800
N3—H3	0.83 (4)	C20—C21	1.494 (5)
C4—O4	1.245 (3)	C20—H20A	0.9900
C4—C5	1.437 (4)	C20—H20B	0.9900
C5—C6	1.355 (4)	C21—C22A	1.544 (5)
C5—H5	0.9500	C21—C22B	1.598 (12)
C6—C7	1.518 (3)	C21—H21A	0.9900
C7—O8	1.225 (3)	C21—H21B	0.9900
C7—O7	1.302 (3)	C21—H21C	0.9900
N9—C10	1.345 (3)	C21—H21D	0.9900
N9—C14	1.352 (3)	C22A—C23A	1.512 (7)
C10—C11	1.379 (4)	C22A—H22A	0.9900
C10—H10	0.9500	C22A—H22B	0.9900
C11—C12	1.390 (4)	C23A—H23A	0.9800
C11—H11	0.9500	C23A—H23B	0.9800
C12—C13	1.385 (4)	C23A—H23C	0.9800
C12—H12	0.9500	C22B—C23B	1.486 (13)
C13—C14	1.384 (4)	C22B—H22C	0.9900
C13—H13	0.9500	C22B—H22D	0.9900
C14—C14^{i}	1.483 (5)	C23B—H23D	0.9800
N15—C20	1.525 (4)	C23B—H23E	0.9800
N15—C20^{ii}	1.525 (4)	C23B—H23F	0.9800
N15—C16	1.525 (3)	O1W—H1WA	0.74 (4)
N15—C16^{ii}	1.525 (3)	O1W—H1WB	0.82 (4)
C16—C17	1.528 (4)	O2W—H2W	0.88 (4)
O7—Co1—O7^{i}	90.76 (11)	N15—C16—H16B	108.2
O7—Co1—N9	93.15 (8)	C17—C16—H16B	108.2
O7^{i}—Co1—N9	175.62 (8)	H16A—C16—H16B	107.3
O7—Co1—N9^{i}	175.62 (8)	C16—C17—C18	110.1 (2)
O7^{i}—Co1—N9^{i}	93.15 (8)	C16—C17—H17A	109.6
N9—Co1—N9^{i}	83.01 (13)	C18—C17—H17A	109.6
O7—Co1—N1^{i}	87.51 (8)	C16—C17—H17B	109.6
O7^{i}—Co1—N1^{i}	84.59 (8)	C18—C17—H17B	109.6
N9—Co1—N1^{i}	97.54 (9)	H17A—C17—H17B	108.2
N9^{i}—Co1—N1^{i}	90.90 (9)	C19—C18—C17	115.3 (2)

O7—Co1—N1	84.59 (8)	C19—C18—H18A	108.5
O7^{i}—Co1—N1	87.51 (9)	C17—C18—H18A	108.5
N9—Co1—N1	90.90 (9)	C19—C18—H18B	108.5
N9^{i}—Co1—N1	97.54 (9)	C17—C18—H18B	108.5
N1^{i}—Co1—N1	168.74 (13)	H18A—C18—H18B	107.5
C2—N1—C6	118.5 (2)	C18—C19—H19A	109.5
C2—N1—Co1	129.44 (17)	C18—C19—H19B	109.5
C6—N1—Co1	111.76 (16)	H19A—C19—H19B	109.5
O2—C2—N1	124.4 (2)	C18—C19—H19C	109.5
O2—C2—N3	119.4 (2)	H19A—C19—H19C	109.5
N1—C2—N3	116.2 (2)	H19B—C19—H19C	109.5
C4—N3—C2	127.1 (2)	C21—C20—N15	116.4 (3)
C4—N3—H3	120 (2)	C21—C20—H20A	108.2
C2—N3—H3	113 (2)	N15—C20—H20A	108.2
O4—C4—N3	120.4 (2)	C21—C20—H20B	108.2
O4—C4—C5	125.3 (2)	N15—C20—H20B	108.2
N3—C4—C5	114.2 (2)	H20A—C20—H20B	107.3
C6—C5—C4	118.5 (2)	C20—C21—C22A	104.9 (3)
C6—C5—H5	120.8	C20—C21—C22B	128.9 (7)
C4—C5—H5	120.8	C20—C21—H21A	110.8
C5—C6—N1	125.5 (2)	C22A—C21—H21A	110.8
C5—C6—C7	121.6 (2)	C20—C21—H21B	110.8
N1—C6—C7	112.9 (2)	C22A—C21—H21B	110.8
O8—C7—O7	124.4 (2)	H21A—C21—H21B	108.8
O8—C7—C6	121.4 (2)	C20—C21—H21C	105.1
O7—C7—C6	114.1 (2)	C22B—C21—H21C	105.1
C7—O7—Co1	116.42 (16)	C20—C21—H21D	105.1
C10—N9—C14	118.9 (2)	C22B—C21—H21D	105.1
C10—N9—Co1	125.92 (18)	H21C—C21—H21D	105.9
C14—N9—Co1	115.20 (17)	C23A—C22A—C21	110.9 (4)
N9—C10—C11	121.8 (3)	C23A—C22A—H22A	109.5
N9—C10—H10	119.1	C21—C22A—H22A	109.5
C11—C10—H10	119.1	C23A—C22A—H22B	109.5
C10—C11—C12	119.5 (3)	C21—C22A—H22B	109.5
C10—C11—H11	120.2	H22A—C22A—H22B	108.0
C12—C11—H11	120.2	C22A—C23A—H23A	109.5
C13—C12—C11	118.8 (3)	C22A—C23A—H23B	109.5
C13—C12—H12	120.6	H23A—C23A—H23B	109.5
C11—C12—H12	120.6	C22A—C23A—H23C	109.5
C14—C13—C12	118.9 (3)	H23A—C23A—H23C	109.5
C14—C13—H13	120.5	H23B—C23A—H23C	109.5
C12—C13—H13	120.5	C23B—C22B—C21	110.5 (10)
N9—C14—C13	122.1 (2)	C23B—C22B—H22C	109.5
N9—C14—C14^{i}	113.24 (14)	C21—C22B—H22C	109.5
C13—C14—C14^{i}	124.64 (17)	C23B—C22B—H22D	109.5
C20—N15—C20^{ii}	105.6 (3)	C21—C22B—H22D	109.5
C20—N15—C16	111.27 (17)	H22C—C22B—H22D	108.1
C20^{ii}—N15—C16	111.36 (16)	C22B—C23B—H23D	109.5

C20—N15—C16^{ii}	111.36 (16)	C22B—C23B—H23E	109.5
C20^{ii}—N15—C16^{ii}	111.27 (17)	H23D—C23B—H23E	109.5
C16—N15—C16^{ii}	106.1 (3)	C22B—C23B—H23F	109.5
N15—C16—C17	116.4 (2)	H23D—C23B—H23F	109.5
N15—C16—H16A	108.2	H23E—C23B—H23F	109.5
C17—C16—H16A	108.2	H1WA—O1W—H1WB	112 (4)
C6—N1—C2—O2	−178.7 (2)	N1—Co1—O7—C7	2.86 (18)
Co1—N1—C2—O2	−6.2 (4)	C14—N9—C10—C11	0.9 (4)
C6—N1—C2—N3	1.5 (3)	Co1—N9—C10—C11	179.0 (2)
Co1—N1—C2—N3	174.04 (17)	N9—C10—C11—C12	−0.7 (4)
O2—C2—N3—C4	180.0 (2)	C10—C11—C12—C13	−0.1 (5)
N1—C2—N3—C4	−0.2 (4)	C11—C12—C13—C14	0.8 (5)
C2—N3—C4—O4	179.4 (2)	C10—N9—C14—C13	−0.2 (4)
C2—N3—C4—C5	−0.1 (4)	Co1—N9—C14—C13	−178.4 (2)
O4—C4—C5—C6	179.6 (2)	C10—N9—C14—C14^{i}	−179.0 (3)
N3—C4—C5—C6	−1.0 (3)	Co1—N9—C14—C14^{i}	2.7 (4)
C4—C5—C6—N1	2.5 (4)	C12—C13—C14—N9	−0.7 (5)
C4—C5—C6—C7	−177.8 (2)	C12—C13—C14—C14^{i}	178.0 (3)
C2—N1—C6—C5	−2.7 (4)	C20—N15—C16—C17	−48.8 (3)
Co1—N1—C6—C5	−176.6 (2)	C20^{ii}—N15—C16—C17	68.7 (3)
C2—N1—C6—C7	177.5 (2)	C16^{ii}—N15—C16—C17	−170.1 (3)
Co1—N1—C6—C7	3.7 (3)	N15—C16—C17—C18	−173.0 (2)
C5—C6—C7—O8	−2.3 (4)	C16—C17—C18—C19	−53.0 (4)
N1—C6—C7—O8	177.4 (2)	C20^{ii}—N15—C20—C21	−179.9 (3)
C5—C6—C7—O7	178.7 (2)	C16—N15—C20—C21	−58.9 (3)
N1—C6—C7—O7	−1.6 (3)	C16^{ii}—N15—C20—C21	59.2 (3)
O8—C7—O7—Co1	179.5 (2)	N15—C20—C21—C22A	170.1 (3)
C6—C7—O7—Co1	−1.5 (3)	N15—C20—C21—C22B	157.9 (7)
O7^{i}—Co1—O7—C7	−84.56 (18)	C20—C21—C22A—C23A	173.8 (4)
N9—Co1—O7—C7	93.47 (18)	C20—C21—C22B—C23B	73.7 (14)
N1^{i}—Co1—O7—C7	−169.11 (19)		

Symmetry codes: (i) −*x*+1/2, *y*, −*z*+3/2; (ii) −*x*+3/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1*W*^{iii}	0.83 (4)	2.11 (4)	2.910 (3)	163 (3)
O1*W*—H1*WA*···O2^{i}	0.74 (4)	2.11 (4)	2.829 (3)	166 (4)
O1*W*—H1*WB*···O2*W*	0.82 (4)	2.17 (4)	2.933 (3)	155 (4)
O2*W*—H2*W*···O4^{iv}	0.88 (4)	1.89 (4)	2.767 (3)	173 (3)
C10—H10···O7	0.95	2.42	2.929 (3)	113
C16—H16*B*···O4^{v}	0.99	2.45	3.423 (3)	168
C20—H20*A*···O1*W*^{ii}	0.99	2.49	3.449 (4)	164
C21—H21*B*···O8	0.99	2.54	3.500 (4)	164

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

Tetra-*n*-butylammonium (2,2′-bipyridine-κ^2N,N')bis(2,4-dioxo-1,2,3,4-tetrahydropyrimidin-1-ide-6-carboxylato-κN^1)cobalt(III) trihydrate (2e)

Crystal data

$(C_{16}H_{36}N)[Co(C_5H_2N_2O_4)_2(C_{10}H_8N_2)]\cdot 3H_2O$
$M_r = 819.79$
Triclinic, $P\overline{1}$
$a = 13.0155$ (15) Å
$b = 9.4028$ (14) Å
$c = 16.2640$ (17) Å
$\alpha = 88.794$ (11)°
$\beta = 103.054$ (9)°
$\gamma = 88.687$ (11)°
$V = 1937.8$ (4) Å^3
$Z = 2$
$F(000) = 868$
$D_x = 1.405$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3865 reflections
$\theta = 4.2$–28.9°
$\mu = 0.51$ mm^{-1}
$T = 170$ K
Block, red
0.67 × 0.39 × 0.30 mm

Data collection

Agilent Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0655 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2011)
$T_{min} = 0.615$, $T_{max} = 1.000$
25546 measured reflections
6796 independent reflections
5739 reflections with $I > 2\sigma(I)$
$R_{int} = 0.152$
$\theta_{max} = 25.0°$, $\theta_{min} = 4.2°$
$h = -15\rightarrow15$
$k = -11\rightarrow11$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.161$
$S = 1.05$
6796 reflections
524 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2 + 1.4515P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.63$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

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. This was a two-domain crystal with monoclinic and triclinic phases present. The structures of the two phases were analyzed independently of each other.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.25935 (3)	0.34140 (4)	0.75496 (2)	0.01676 (15)
N1	0.25887 (19)	0.3183 (3)	0.87431 (15)	0.0185 (5)
C2	0.1938 (2)	0.3841 (3)	0.91902 (18)	0.0199 (6)
O2	0.13083 (17)	0.4820 (2)	0.89068 (14)	0.0284 (5)
N3	0.2030 (2)	0.3346 (3)	1.00165 (16)	0.0206 (5)
H3	0.163 (3)	0.382 (4)	1.032 (2)	0.025*

C4	0.2703 (2)	0.2266 (3)	1.04362 (18)	0.0202 (6)
O4	0.27169 (17)	0.1942 (2)	1.11840 (13)	0.0244 (5)
C5	0.3344 (2)	0.1606 (3)	0.99350 (19)	0.0216 (6)
H5	0.3827	0.0848	1.0162	0.026*
C6	0.3245 (2)	0.2094 (3)	0.91230 (18)	0.0189 (6)
C7	0.3883 (2)	0.1421 (3)	0.85464 (18)	0.0197 (6)
O7	0.36780 (16)	0.1994 (2)	0.77924 (13)	0.0219 (5)
O8	0.45264 (18)	0.0463 (2)	0.87915 (14)	0.0287 (5)
N1A	0.25882 (19)	0.3208 (3)	0.63505 (15)	0.0186 (5)
C2A	0.3189 (2)	0.3862 (3)	0.58735 (18)	0.0204 (6)
O2A	0.38197 (18)	0.4794 (3)	0.61319 (14)	0.0292 (5)
N3A	0.3057 (2)	0.3413 (3)	0.50402 (16)	0.0215 (5)
H3A	0.343 (3)	0.391 (4)	0.478 (2)	0.026*
C4A	0.2379 (2)	0.2399 (3)	0.46525 (18)	0.0211 (6)
O4A	0.23151 (17)	0.2119 (2)	0.38960 (13)	0.0265 (5)
C5A	0.1783 (2)	0.1746 (3)	0.51854 (19)	0.0228 (6)
H5A	0.1297	0.1027	0.4978	0.027*
C6A	0.1922 (2)	0.2172 (3)	0.59974 (18)	0.0196 (6)
C7A	0.1324 (2)	0.1507 (3)	0.65968 (19)	0.0198 (6)
O7A	0.15569 (16)	0.2014 (2)	0.73528 (13)	0.0213 (5)
O8A	0.06800 (18)	0.0588 (2)	0.63668 (14)	0.0287 (5)
N9	0.35626 (19)	0.4939 (3)	0.77841 (15)	0.0180 (5)
N9A	0.15653 (19)	0.4954 (3)	0.72631 (15)	0.0183 (5)
C10	0.4616 (2)	0.4800 (3)	0.80720 (19)	0.0230 (6)
H10	0.4930	0.3872	0.8172	0.028*
C11	0.5252 (3)	0.5960 (4)	0.8225 (2)	0.0295 (7)
H11	0.5992	0.5830	0.8434	0.035*
C12	0.4805 (3)	0.7316 (4)	0.8074 (2)	0.0282 (7)
H12	0.5231	0.8128	0.8181	0.034*
C13	0.3723 (3)	0.7470 (3)	0.7763 (2)	0.0274 (7)
H13	0.3397	0.8389	0.7649	0.033*
C14	0.3125 (2)	0.6262 (3)	0.76214 (18)	0.0212 (6)
C14A	0.1969 (2)	0.6272 (3)	0.73182 (18)	0.0218 (6)
C13A	0.1327 (3)	0.7482 (4)	0.7108 (2)	0.0306 (7)
H13A	0.1626	0.8397	0.7138	0.037*
C12A	0.0245 (3)	0.7341 (4)	0.6854 (2)	0.0323 (8)
H12A	−0.0209	0.8157	0.6708	0.039*
C11A	−0.0164 (3)	0.5995 (4)	0.6817 (2)	0.0302 (7)
H11A	−0.0904	0.5874	0.6650	0.036*
C10A	0.0517 (2)	0.4827 (4)	0.70265 (19)	0.0235 (6)
H10A	0.0232	0.3903	0.7002	0.028*
N15	0.7560 (2)	0.1472 (3)	0.73949 (16)	0.0232 (6)
C16	0.8493 (2)	0.0474 (3)	0.7791 (2)	0.0246 (7)
H16A	0.8249	−0.0238	0.8156	0.030*
H16B	0.8710	−0.0047	0.7332	0.030*
C17	0.9465 (3)	0.1177 (4)	0.8316 (2)	0.0295 (7)
H17A	0.9290	0.1589	0.8824	0.035*
H17B	0.9682	0.1957	0.7980	0.035*

C18	1.0380 (3)	0.0070 (4)	0.8583 (2)	0.0333 (8)
H18A	1.0614	−0.0227	0.8073	0.040*
H18B	1.0980	0.0532	0.8956	0.040*
C19	1.0111 (3)	−0.1252 (4)	0.9040 (3)	0.0426 (9)
H19A	0.9831	−0.0969	0.9525	0.064*
H19B	1.0749	−0.1851	0.9237	0.064*
H19C	0.9581	−0.1788	0.8653	0.064*
C20	0.7335 (3)	0.2524 (3)	0.8037 (2)	0.0287 (7)
H20A	0.7963	0.3112	0.8218	0.034*
H20B	0.6743	0.3168	0.7752	0.034*
C21	0.7060 (3)	0.1869 (4)	0.8818 (2)	0.0313 (8)
H21A	0.7562	0.1074	0.9045	0.038*
H21B	0.6340	0.1488	0.8671	0.038*
C22	0.7115 (3)	0.3013 (4)	0.9485 (2)	0.0343 (8)
H22A	0.7853	0.3317	0.9667	0.041*
H22B	0.6679	0.3853	0.9226	0.041*
C23	0.6739 (4)	0.2508 (5)	1.0245 (3)	0.0561 (12)
H23A	0.6028	0.2139	1.0066	0.084*
H23B	0.6726	0.3303	1.0623	0.084*
H23C	0.7219	0.1751	1.0544	0.084*
C16A	0.6621 (2)	0.0525 (3)	0.71016 (19)	0.0230 (6)
H16C	0.6439	0.0087	0.7607	0.028*
H16D	0.6842	−0.0255	0.6774	0.028*
C17A	0.5630 (3)	0.1252 (4)	0.6564 (2)	0.0283 (7)
H17C	0.5770	0.1587	0.6020	0.034*
H17D	0.5431	0.2089	0.6859	0.034*
C18A	0.4722 (3)	0.0207 (4)	0.6401 (2)	0.0298 (7)
H18C	0.4522	−0.0007	0.6941	0.036*
H18D	0.4104	0.0677	0.6015	0.036*
C19A	0.4972 (3)	−0.1191 (4)	0.6019 (2)	0.0350 (8)
H19D	0.5191	−0.0993	0.5491	0.053*
H19E	0.4342	−0.1771	0.5904	0.053*
H19F	0.5543	−0.1708	0.6417	0.053*
C20A	0.7790 (3)	0.2356 (4)	0.6657 (2)	0.0305 (7)
H20C	0.7184	0.3023	0.6445	0.037*
H20D	0.8415	0.2934	0.6875	0.037*
C21A	0.7993 (3)	0.1511 (4)	0.5914 (2)	0.0370 (8)
H21C	0.7481	0.0738	0.5798	0.044*
H21D	0.8709	0.1066	0.6075	0.044*
C22A	0.7902 (3)	0.2426 (5)	0.5099 (3)	0.0449 (10)
H22C	0.7915	0.1787	0.4625	0.054*
H22D	0.7213	0.2947	0.4971	0.054*
C23A	0.8761 (3)	0.3477 (5)	0.5151 (3)	0.0498 (11)
H23D	0.8718	0.4164	0.5588	0.075*
H23E	0.8678	0.3980	0.4605	0.075*
H23F	0.9449	0.2975	0.5295	0.075*
O1W	0.5956 (2)	0.5118 (3)	0.61358 (17)	0.0309 (5)
H1WA	0.621 (3)	0.549 (5)	0.662 (3)	0.037*

H1WB	0.533 (4)	0.504 (5)	0.610 (3)	0.037*
O1WA	0.9128 (2)	0.5146 (3)	0.89150 (17)	0.0321 (6)
H1WC	0.889 (3)	0.541 (5)	0.844 (3)	0.038*
H1WD	0.972 (4)	0.519 (5)	0.892 (3)	0.038*
O2W	0.75028 (19)	0.6320 (3)	0.75083 (15)	0.0306 (6)
H2WA	0.758 (3)	0.683 (4)	0.707 (3)	0.037*
H2WB	0.749 (3)	0.675 (5)	0.787 (3)	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0202 (2)	0.0171 (2)	0.0140 (2)	0.00565 (15)	0.00683 (16)	0.00100 (15)
N1	0.0203 (12)	0.0200 (12)	0.0166 (12)	0.0068 (10)	0.0081 (10)	0.0024 (10)
C2	0.0211 (15)	0.0222 (15)	0.0177 (14)	0.0055 (12)	0.0076 (12)	0.0006 (12)
O2	0.0309 (12)	0.0327 (12)	0.0230 (11)	0.0189 (10)	0.0113 (9)	0.0057 (9)
N3	0.0235 (13)	0.0230 (13)	0.0170 (12)	0.0060 (11)	0.0088 (10)	−0.0005 (10)
C4	0.0225 (15)	0.0204 (15)	0.0174 (15)	−0.0013 (12)	0.0040 (12)	0.0006 (12)
O4	0.0294 (12)	0.0277 (12)	0.0170 (11)	0.0050 (9)	0.0082 (9)	0.0018 (9)
C5	0.0257 (16)	0.0187 (15)	0.0199 (15)	0.0060 (12)	0.0050 (12)	0.0018 (12)
C6	0.0213 (15)	0.0179 (14)	0.0178 (14)	0.0012 (12)	0.0052 (11)	−0.0005 (11)
C7	0.0203 (15)	0.0181 (14)	0.0210 (15)	0.0047 (12)	0.0062 (12)	−0.0001 (12)
O7	0.0259 (11)	0.0235 (11)	0.0173 (10)	0.0086 (9)	0.0081 (8)	0.0016 (9)
O8	0.0331 (12)	0.0274 (12)	0.0256 (12)	0.0170 (10)	0.0088 (10)	0.0044 (9)
N1A	0.0203 (12)	0.0186 (12)	0.0174 (12)	0.0044 (10)	0.0060 (10)	0.0006 (10)
C2A	0.0210 (15)	0.0231 (15)	0.0175 (15)	0.0072 (12)	0.0065 (12)	0.0036 (12)
O2A	0.0337 (12)	0.0350 (13)	0.0228 (11)	−0.0105 (11)	0.0129 (10)	−0.0062 (10)
N3A	0.0236 (13)	0.0247 (14)	0.0176 (13)	0.0021 (11)	0.0078 (10)	0.0007 (11)
C4A	0.0218 (15)	0.0228 (15)	0.0178 (15)	0.0110 (12)	0.0042 (12)	0.0015 (12)
O4A	0.0319 (12)	0.0316 (12)	0.0168 (11)	0.0054 (10)	0.0077 (9)	−0.0023 (9)
C5A	0.0257 (16)	0.0230 (15)	0.0196 (15)	0.0039 (13)	0.0051 (12)	−0.0024 (12)
C6A	0.0197 (14)	0.0184 (14)	0.0209 (15)	0.0086 (12)	0.0062 (12)	0.0028 (12)
C7A	0.0207 (15)	0.0177 (14)	0.0214 (15)	0.0082 (12)	0.0066 (12)	0.0047 (12)
O7A	0.0250 (11)	0.0229 (11)	0.0174 (10)	0.0029 (9)	0.0083 (8)	0.0006 (8)
O8A	0.0330 (13)	0.0265 (12)	0.0286 (12)	−0.0057 (10)	0.0103 (10)	−0.0013 (10)
N9	0.0197 (12)	0.0200 (12)	0.0153 (12)	0.0046 (10)	0.0063 (9)	0.0005 (9)
N9A	0.0217 (13)	0.0199 (12)	0.0137 (12)	0.0016 (10)	0.0049 (10)	0.0007 (10)
C10	0.0208 (15)	0.0249 (16)	0.0225 (16)	0.0039 (12)	0.0038 (12)	0.0018 (12)
C11	0.0235 (16)	0.0360 (19)	0.0281 (17)	−0.0018 (14)	0.0037 (13)	0.0011 (14)
C12	0.0305 (17)	0.0284 (17)	0.0268 (17)	−0.0041 (14)	0.0082 (13)	−0.0025 (13)
C13	0.0321 (18)	0.0213 (16)	0.0283 (17)	0.0042 (13)	0.0066 (14)	0.0009 (13)
C14	0.0257 (16)	0.0219 (15)	0.0173 (14)	0.0032 (12)	0.0083 (12)	0.0015 (12)
C14A	0.0268 (16)	0.0225 (15)	0.0164 (14)	0.0061 (13)	0.0064 (12)	0.0004 (12)
C13A	0.0341 (18)	0.0202 (16)	0.0360 (19)	0.0072 (14)	0.0060 (15)	0.0019 (14)
C12A	0.0317 (18)	0.0278 (18)	0.0355 (19)	0.0136 (14)	0.0049 (15)	0.0002 (15)
C11A	0.0224 (16)	0.0384 (19)	0.0291 (17)	0.0090 (14)	0.0053 (13)	0.0012 (14)
C10A	0.0220 (15)	0.0274 (16)	0.0210 (15)	0.0034 (13)	0.0056 (12)	0.0005 (12)
N15	0.0257 (14)	0.0181 (13)	0.0254 (14)	0.0030 (11)	0.0054 (11)	0.0022 (11)
C16	0.0231 (16)	0.0215 (15)	0.0278 (17)	0.0075 (12)	0.0040 (13)	0.0055 (13)

C17	0.0273 (17)	0.0254 (17)	0.0343 (18)	0.0024 (14)	0.0045 (14)	0.0049 (14)
C18	0.0236 (17)	0.0345 (19)	0.040 (2)	0.0026 (14)	0.0033 (14)	0.0017 (15)
C19	0.037 (2)	0.042 (2)	0.044 (2)	0.0060 (17)	0.0017 (17)	0.0139 (18)
C20	0.0291 (17)	0.0206 (16)	0.0353 (18)	0.0023 (13)	0.0050 (14)	−0.0052 (13)
C21	0.0305 (18)	0.0279 (17)	0.0372 (19)	0.0005 (14)	0.0103 (15)	−0.0082 (15)
C22	0.0382 (19)	0.0317 (18)	0.0314 (19)	0.0035 (15)	0.0048 (15)	−0.0052 (15)
C23	0.072 (3)	0.061 (3)	0.043 (2)	−0.017 (2)	0.025 (2)	−0.021 (2)
C16A	0.0261 (16)	0.0212 (15)	0.0220 (15)	0.0009 (12)	0.0063 (12)	0.0005 (12)
C17A	0.0288 (17)	0.0247 (17)	0.0293 (17)	0.0060 (13)	0.0028 (14)	0.0017 (13)
C18A	0.0239 (16)	0.0306 (18)	0.0335 (18)	0.0056 (14)	0.0038 (14)	−0.0009 (14)
C19A	0.0317 (18)	0.037 (2)	0.0346 (19)	0.0005 (15)	0.0031 (15)	−0.0083 (16)
C20A	0.0301 (18)	0.0271 (17)	0.0334 (18)	−0.0016 (14)	0.0058 (14)	0.0092 (14)
C21A	0.039 (2)	0.039 (2)	0.036 (2)	0.0022 (16)	0.0151 (16)	0.0117 (16)
C22A	0.044 (2)	0.052 (2)	0.037 (2)	−0.0039 (19)	0.0058 (17)	0.0109 (18)
C23A	0.049 (2)	0.058 (3)	0.042 (2)	−0.008 (2)	0.0083 (19)	0.011 (2)
O1W	0.0297 (13)	0.0331 (13)	0.0330 (14)	−0.0011 (11)	0.0135 (11)	−0.0044 (11)
O1WA	0.0311 (13)	0.0372 (14)	0.0312 (13)	0.0080 (11)	0.0152 (11)	0.0027 (11)
O2W	0.0386 (14)	0.0350 (14)	0.0212 (12)	0.0050 (11)	0.0135 (11)	−0.0013 (10)

Geometric parameters (Å, º)

Co1—O7A	1.887 (2)	N15—C20A	1.534 (4)
Co1—O7	1.890 (2)	C16—C17	1.529 (5)
Co1—N9	1.918 (3)	C16—H16A	0.9900
Co1—N9A	1.925 (2)	C16—H16B	0.9900
Co1—N1	1.950 (2)	C17—C18	1.542 (5)
Co1—N1A	1.962 (2)	C17—H17A	0.9900
N1—C6	1.362 (4)	C17—H17B	0.9900
N1—C2	1.379 (4)	C18—C19	1.523 (5)
C2—O2	1.228 (4)	C18—H18A	0.9900
C2—N3	1.391 (4)	C18—H18B	0.9900
N3—C4	1.387 (4)	C19—H19A	0.9800
N3—H3	0.91 (4)	C19—H19B	0.9800
C4—O4	1.244 (4)	C19—H19C	0.9800
C4—C5	1.433 (4)	C20—C21	1.518 (5)
C5—C6	1.366 (4)	C20—H20A	0.9900
C5—H5	0.9500	C20—H20B	0.9900
C6—C7	1.523 (4)	C21—C22	1.535 (5)
C7—O8	1.213 (4)	C21—H21A	0.9900
C7—O7	1.297 (4)	C21—H21B	0.9900
N1A—C2A	1.367 (4)	C22—C23	1.501 (6)
N1A—C6A	1.367 (4)	C22—H22A	0.9900
C2A—O2A	1.229 (4)	C22—H22B	0.9900
C2A—N3A	1.402 (4)	C23—H23A	0.9800
N3A—C4A	1.377 (4)	C23—H23B	0.9800
N3A—H3A	0.85 (4)	C23—H23C	0.9800
C4A—O4A	1.248 (4)	C16A—C17A	1.523 (4)
C4A—C5A	1.427 (4)	C16A—H16C	0.9900

C5A—C6A	1.362 (4)	C16A—H16D	0.9900
C5A—H5A	0.9500	C17A—C18A	1.534 (5)
C6A—C7A	1.512 (4)	C17A—H17C	0.9900
C7A—O8A	1.223 (4)	C17A—H17D	0.9900
C7A—O7A	1.302 (4)	C18A—C19A	1.525 (5)
N9—C10	1.347 (4)	C18A—H18C	0.9900
N9—C14	1.352 (4)	C18A—H18D	0.9900
N9A—C10A	1.340 (4)	C19A—H19D	0.9800
N9A—C14A	1.354 (4)	C19A—H19E	0.9800
C10—C11	1.377 (5)	C19A—H19F	0.9800
C10—H10	0.9500	C20A—C21A	1.528 (5)
C11—C12	1.383 (5)	C20A—H20C	0.9900
C11—H11	0.9500	C20A—H20D	0.9900
C12—C13	1.388 (5)	C21A—C22A	1.546 (5)
C12—H12	0.9500	C21A—H21C	0.9900
C13—C14	1.384 (5)	C21A—H21D	0.9900
C13—H13	0.9500	C22A—C23A	1.498 (6)
C14—C14A	1.474 (4)	C22A—H22C	0.9900
C14A—C13A	1.386 (4)	C22A—H22D	0.9900
C13A—C12A	1.386 (5)	C23A—H23D	0.9800
C13A—H13A	0.9500	C23A—H23E	0.9800
C12A—C11A	1.381 (5)	C23A—H23F	0.9800
C12A—H12A	0.9500	O1W—H1WA	0.87 (4)
C11A—C10A	1.382 (5)	O1W—H1WB	0.80 (4)
C11A—H11A	0.9500	O1WA—H1WC	0.80 (4)
C10A—H10A	0.9500	O1WA—H1WD	0.77 (5)
N15—C16A	1.522 (4)	O2W—H2WA	0.88 (4)
N15—C20	1.524 (4)	O2W—H2WB	0.73 (5)
N15—C16	1.530 (4)		

O7A—Co1—O7	90.86 (9)	C16—N15—C20A	111.3 (2)
O7A—Co1—N9	175.09 (9)	C17—C16—N15	116.1 (3)
O7—Co1—N9	93.30 (10)	C17—C16—H16A	108.3
O7A—Co1—N9A	93.02 (10)	N15—C16—H16A	108.3
O7—Co1—N9A	175.32 (10)	C17—C16—H16B	108.3
N9—Co1—N9A	82.94 (10)	N15—C16—H16B	108.3
O7A—Co1—N1	86.59 (10)	H16A—C16—H16B	107.4
O7—Co1—N1	84.30 (9)	C16—C17—C18	110.1 (3)
N9—Co1—N1	91.24 (10)	C16—C17—H17A	109.6
N9A—Co1—N1	98.53 (10)	C18—C17—H17A	109.6
O7A—Co1—N1A	84.50 (9)	C16—C17—H17B	109.6
O7—Co1—N1A	87.71 (9)	C18—C17—H17B	109.6
N9—Co1—N1A	98.24 (10)	H17A—C17—H17B	108.2
N9A—Co1—N1A	90.06 (10)	C19—C18—C17	114.7 (3)
N1—Co1—N1A	167.93 (10)	C19—C18—H18A	108.6
C6—N1—C2	118.8 (2)	C17—C18—H18A	108.6
C6—N1—Co1	111.97 (19)	C19—C18—H18B	108.6
C2—N1—Co1	128.7 (2)	C17—C18—H18B	108.6

O2—C2—N1	124.3 (3)	H18A—C18—H18B	107.6
O2—C2—N3	119.6 (3)	C18—C19—H19A	109.5
N1—C2—N3	116.2 (2)	C18—C19—H19B	109.5
C4—N3—C2	127.1 (3)	H19A—C19—H19B	109.5
C4—N3—H3	117 (2)	C18—C19—H19C	109.5
C2—N3—H3	116 (2)	H19A—C19—H19C	109.5
O4—C4—N3	120.5 (3)	H19B—C19—H19C	109.5
O4—C4—C5	125.3 (3)	C21—C20—N15	115.6 (3)
N3—C4—C5	114.2 (3)	C21—C20—H20A	108.4
C6—C5—C4	118.3 (3)	N15—C20—H20A	108.4
C6—C5—H5	120.8	C21—C20—H20B	108.4
C4—C5—H5	120.8	N15—C20—H20B	108.4
N1—C6—C5	125.4 (3)	H20A—C20—H20B	107.4
N1—C6—C7	113.4 (2)	C20—C21—C22	109.1 (3)
C5—C6—C7	121.3 (3)	C20—C21—H21A	109.9
O8—C7—O7	124.8 (3)	C22—C21—H21A	109.9
O8—C7—C6	121.9 (3)	C20—C21—H21B	109.9
O7—C7—C6	113.3 (2)	C22—C21—H21B	109.9
C7—O7—Co1	116.67 (18)	H21A—C21—H21B	108.3
C2A—N1A—C6A	118.9 (2)	C23—C22—C21	113.0 (3)
C2A—N1A—Co1	129.5 (2)	C23—C22—H22A	109.0
C6A—N1A—Co1	111.39 (18)	C21—C22—H22A	109.0
O2A—C2A—N1A	124.1 (3)	C23—C22—H22B	109.0
O2A—C2A—N3A	119.7 (3)	C21—C22—H22B	109.0
N1A—C2A—N3A	116.2 (3)	H22A—C22—H22B	107.8
C4A—N3A—C2A	126.9 (3)	C22—C23—H23A	109.5
C4A—N3A—H3A	123 (2)	C22—C23—H23B	109.5
C2A—N3A—H3A	110 (2)	H23A—C23—H23B	109.5
O4A—C4A—N3A	120.7 (3)	C22—C23—H23C	109.5
O4A—C4A—C5A	125.0 (3)	H23A—C23—H23C	109.5
N3A—C4A—C5A	114.2 (3)	H23B—C23—H23C	109.5
C6A—C5A—C4A	118.7 (3)	N15—C16A—C17A	116.1 (2)
C6A—C5A—H5A	120.6	N15—C16A—H16C	108.3
C4A—C5A—H5A	120.6	C17A—C16A—H16C	108.3
C5A—C6A—N1A	125.0 (3)	N15—C16A—H16D	108.3
C5A—C6A—C7A	121.3 (3)	C17A—C16A—H16D	108.3
N1A—C6A—C7A	113.7 (2)	H16C—C16A—H16D	107.4
O8A—C7A—O7A	124.8 (3)	C16A—C17A—C18A	110.0 (3)
O8A—C7A—C6A	121.3 (3)	C16A—C17A—H17C	109.7
O7A—C7A—C6A	113.8 (3)	C18A—C17A—H17C	109.7
C7A—O7A—Co1	116.49 (18)	C16A—C17A—H17D	109.7
C10—N9—C14	118.5 (3)	C18A—C17A—H17D	109.7
C10—N9—Co1	126.1 (2)	H17C—C17A—H17D	108.2
C14—N9—Co1	115.3 (2)	C19A—C18A—C17A	114.6 (3)
C10A—N9A—C14A	118.9 (3)	C19A—C18A—H18C	108.6
C10A—N9A—Co1	126.1 (2)	C17A—C18A—H18C	108.6
C14A—N9A—Co1	115.0 (2)	C19A—C18A—H18D	108.6
N9—C10—C11	122.1 (3)	C17A—C18A—H18D	108.6

N9—C10—H10	118.9	H18C—C18A—H18D	107.6
C11—C10—H10	118.9	C18A—C19A—H19D	109.5
C10—C11—C12	119.5 (3)	C18A—C19A—H19E	109.5
C10—C11—H11	120.3	H19D—C19A—H19E	109.5
C12—C11—H11	120.3	C18A—C19A—H19F	109.5
C11—C12—C13	118.8 (3)	H19D—C19A—H19F	109.5
C11—C12—H12	120.6	H19E—C19A—H19F	109.5
C13—C12—H12	120.6	C21A—C20A—N15	115.8 (3)
C14—C13—C12	119.0 (3)	C21A—C20A—H20C	108.3
C14—C13—H13	120.5	N15—C20A—H20C	108.3
C12—C13—H13	120.5	C21A—C20A—H20D	108.3
N9—C14—C13	122.0 (3)	N15—C20A—H20D	108.3
N9—C14—C14A	113.3 (3)	H20C—C20A—H20D	107.4
C13—C14—C14A	124.6 (3)	C20A—C21A—C22A	113.5 (3)
N9A—C14A—C13A	121.5 (3)	C20A—C21A—H21C	108.9
N9A—C14A—C14	113.4 (3)	C22A—C21A—H21C	108.9
C13A—C14A—C14	125.1 (3)	C20A—C21A—H21D	108.9
C14A—C13A—C12A	119.2 (3)	C22A—C21A—H21D	108.9
C14A—C13A—H13A	120.4	H21C—C21A—H21D	107.7
C12A—C13A—H13A	120.4	C23A—C22A—C21A	113.8 (3)
C11A—C12A—C13A	118.9 (3)	C23A—C22A—H22C	108.8
C11A—C12A—H12A	120.5	C21A—C22A—H22C	108.8
C13A—C12A—H12A	120.5	C23A—C22A—H22D	108.8
C12A—C11A—C10A	119.2 (3)	C21A—C22A—H22D	108.8
C12A—C11A—H11A	120.4	H22C—C22A—H22D	107.7
C10A—C11A—H11A	120.4	C22A—C23A—H23D	109.5
N9A—C10A—C11A	122.2 (3)	C22A—C23A—H23E	109.5
N9A—C10A—H10A	118.9	H23D—C23A—H23E	109.5
C11A—C10A—H10A	118.9	C22A—C23A—H23F	109.5
C16A—N15—C20	110.6 (2)	H23D—C23A—H23F	109.5
C16A—N15—C16	106.2 (2)	H23E—C23A—H23F	109.5
C20—N15—C16	110.8 (2)	H1WA—O1W—H1WB	107 (4)
C16A—N15—C20A	111.3 (2)	H1WC—O1WA—H1WD	99 (4)
C20—N15—C20A	106.8 (2)	H2WA—O2W—H2WB	113 (5)

C6—N1—C2—O2	−179.1 (3)	N9A—Co1—O7A—C7A	92.2 (2)
Co1—N1—C2—O2	−8.1 (5)	N1—Co1—O7A—C7A	−169.5 (2)
C6—N1—C2—N3	1.5 (4)	N1A—Co1—O7A—C7A	2.4 (2)
Co1—N1—C2—N3	172.5 (2)	C14—N9—C10—C11	1.7 (4)
O2—C2—N3—C4	−179.4 (3)	Co1—N9—C10—C11	−179.8 (2)
N1—C2—N3—C4	0.0 (4)	N9—C10—C11—C12	−0.6 (5)
C2—N3—C4—O4	178.8 (3)	C10—C11—C12—C13	−0.6 (5)
C2—N3—C4—C5	−1.1 (4)	C11—C12—C13—C14	0.5 (5)
O4—C4—C5—C6	−179.3 (3)	C10—N9—C14—C13	−1.7 (4)
N3—C4—C5—C6	0.7 (4)	Co1—N9—C14—C13	179.6 (2)
C2—N1—C6—C5	−2.1 (4)	C10—N9—C14—C14A	−179.5 (3)
Co1—N1—C6—C5	−174.5 (3)	Co1—N9—C14—C14A	1.9 (3)
C2—N1—C6—C7	177.4 (3)	C12—C13—C14—N9	0.6 (5)

Co1—N1—C6—C7	5.0 (3)	C12—C13—C14—C14A	178.1 (3)
C4—C5—C6—N1	0.9 (5)	C10A—N9A—C14A—C13A	−2.2 (4)
C4—C5—C6—C7	−178.6 (3)	Co1—N9A—C14A—C13A	178.1 (2)
N1—C6—C7—O8	177.9 (3)	C10A—N9A—C14A—C14	177.4 (3)
C5—C6—C7—O8	−2.5 (5)	Co1—N9A—C14A—C14	−2.2 (3)
N1—C6—C7—O7	−0.8 (4)	N9—C14—C14A—N9A	0.2 (4)
C5—C6—C7—O7	178.7 (3)	C13—C14—C14A—N9A	−177.4 (3)
O8—C7—O7—Co1	177.2 (2)	N9—C14—C14A—C13A	179.8 (3)
C6—C7—O7—Co1	−4.1 (3)	C13—C14—C14A—C13A	2.2 (5)
O7A—Co1—O7—C7	−80.9 (2)	N9A—C14A—C13A—C12A	1.5 (5)
N9—Co1—O7—C7	96.5 (2)	C14—C14A—C13A—C12A	−178.1 (3)
N1—Co1—O7—C7	5.6 (2)	C14A—C13A—C12A—C11A	0.0 (5)
N1A—Co1—O7—C7	−165.4 (2)	C13A—C12A—C11A—C10A	−0.6 (5)
C6A—N1A—C2A—O2A	−179.2 (3)	C14A—N9A—C10A—C11A	1.6 (4)
Co1—N1A—C2A—O2A	−4.6 (4)	Co1—N9A—C10A—C11A	−178.8 (2)
C6A—N1A—C2A—N3A	0.8 (4)	C12A—C11A—C10A—N9A	−0.2 (5)
Co1—N1A—C2A—N3A	175.42 (19)	C16A—N15—C16—C17	−166.3 (3)
O2A—C2A—N3A—C4A	−179.2 (3)	C20—N15—C16—C17	−46.2 (4)
N1A—C2A—N3A—C4A	0.8 (4)	C20A—N15—C16—C17	72.5 (3)
C2A—N3A—C4A—O4A	178.2 (3)	N15—C16—C17—C18	−173.3 (3)
C2A—N3A—C4A—C5A	−1.4 (4)	C16—C17—C18—C19	−54.2 (4)
O4A—C4A—C5A—C6A	−179.0 (3)	C16A—N15—C20—C21	57.5 (3)
N3A—C4A—C5A—C6A	0.5 (4)	C16—N15—C20—C21	−60.0 (3)
C4A—C5A—C6A—N1A	1.0 (4)	C20A—N15—C20—C21	178.7 (3)
C4A—C5A—C6A—C7A	−179.2 (2)	N15—C20—C21—C22	166.7 (3)
C2A—N1A—C6A—C5A	−1.7 (4)	C20—C21—C22—C23	173.7 (3)
Co1—N1A—C6A—C5A	−177.3 (2)	C20—N15—C16A—C17A	68.7 (3)
C2A—N1A—C6A—C7A	178.5 (2)	C16—N15—C16A—C17A	−171.1 (3)
Co1—N1A—C6A—C7A	3.0 (3)	C20A—N15—C16A—C17A	−49.8 (3)
C5A—C6A—C7A—O8A	−1.5 (4)	N15—C16A—C17A—C18A	−173.8 (3)
N1A—C6A—C7A—O8A	178.3 (3)	C16A—C17A—C18A—C19A	−54.1 (4)
C5A—C6A—C7A—O7A	179.0 (3)	C16A—N15—C20A—C21A	−56.7 (4)
N1A—C6A—C7A—O7A	−1.2 (3)	C20—N15—C20A—C21A	−177.4 (3)
O8A—C7A—O7A—Co1	179.2 (2)	C16—N15—C20A—C21A	61.5 (4)
C6A—C7A—O7A—Co1	−1.3 (3)	N15—C20A—C21A—C22A	163.4 (3)
O7—Co1—O7A—C7A	−85.2 (2)	C20A—C21A—C22A—C23A	68.6 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O2*A*	0.80 (4)	2.00 (5)	2.803 (3)	174 (4)
O1*W*—H1*WA*···O2*W*	0.87 (4)	2.13 (4)	2.914 (4)	150 (4)
O1*WA*—H1*WD*···O2^{i}	0.77 (5)	2.09 (5)	2.851 (4)	167 (4)
O2*W*—H2*WB*···O4^{ii}	0.73 (5)	2.05 (5)	2.770 (3)	173 (5)
O2*W*—H2*WA*···O4*A*^{iii}	0.88 (4)	1.87 (4)	2.747 (3)	178 (4)
O1*WA*—H1*WD*···O2^{i}	0.77 (5)	2.09 (5)	2.851 (4)	167 (4)
O1*WA*—H1*WC*···O2*W*	0.80 (4)	2.21 (4)	2.920 (4)	147 (4)
C10—H10···O7	0.95	2.43	2.934 (4)	113

C10*A*—H10*A*···O7*A*	0.95	2.41	2.926 (4)	114
C11—H11···O4^{ii}	0.95	2.71	3.313 (4)	122
C12—H12···O8^{iv}	0.95	2.66	3.246 (4)	121
C12—H12···O4^{ii}	0.95	2.64	3.275 (4)	125
C18—H18*A*···O7*A*^{i}	0.99	2.83	3.325 (4)	112
C11*A*—H11*A*···O4*A*^{v}	0.95	2.60	3.248 (4)	126
C12*A*—H12*A*···O4*A*^{v}	0.95	2.72	3.303 (4)	121
C12*A*—H12*A*···O8*A*^{iv}	0.95	2.69	3.233 (4)	117
C13*A*—H13*A*···O8*A*^{iv}	0.95	2.53	3.158 (4)	124
C16—H16*A*···O4^{vi}	0.99	2.43	3.408 (4)	167
C16*A*—H16*D*···O4*A*^{vii}	0.99	2.45	3.429 (4)	170
C18*A*—H18*C*···O7	0.99	2.71	3.352 (4)	123
C20*A*—H20*C*···O1*W*	0.99	2.48	3.446 (4)	166
C21*A*—H21*D*···O8*A*^{i}	0.99	2.53	3.492 (4)	164

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