Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

μ_2-Aqua-bis(μ_2-trifluoroaceto-κ^2O,O')bis[bis(pyridine-κN)(trifluoroacetato-κO)cobalt(**II**)]

Jonathan C. Burley[a]* and Timothy J. Prior[b]

[a]University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England, and [b]Synchrotron Radiation Department, CCLRC Daresbury Laboratories, Warrington, Cheshire WA4 4AD, England

Correspondence e-mail: jb442@cam.ac.uk

Received 17 June 2005
Accepted 21 June 2005
Online 30 June 2005

Key indicators

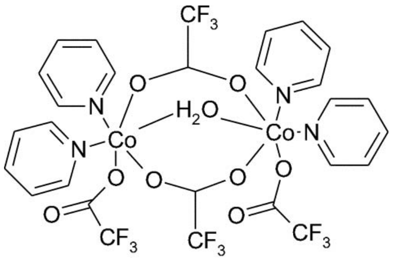
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.079
wR factor = 0.240
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, $[Co_2(C_2F_3O_2)_4(C_5H_5N)_4(H_2O)]$, crystallizes as a neutral dinuclear molecule with two crystallographically distinct octahedrally coordinated Co^{II} ions in the asymmetric unit. The metal ions are connected by two μ_2-bridging trifluoroacetate ions and a single μ_2-bridging water molecule. Each cobalt(II) coordination sphere is completed by a further trifluoroacetate ion, which coordinates in a monodentate manner, and also by two pyridine molecules, resulting in local *cis*-CoN_2O_4 coordination. The water molecule H atoms participate in intramolecular O—H···O hydrogen bonds to the pendant O atoms of the monodentate trifluoroacetate ligands.

Comment

Complexes of divalent transition metals with mixed N-donor and acetate-related ligands have been studied widely due to their close structural analogy with reduced non-heme iron(II) proteins (Hagen *et al.*, 1993). During the course of investigations into possible complexes formed by cobalt(II), pyridine and acetate derivatives, the title compound, (I) (Fig. 1), was isolated. The red crystals consist of neutral dinuclear cobalt(II) molecules in which each cobalt ion is coordinated (Table 1) by two pyridine N atoms and one monodentate trifluoroacetate ion. Two further trifluoroacetate ions bridge the metal nuclei in a μ_2-manner, and the coordination shell is completed by a single μ_2-bridging water molecule. The water molecule H atoms make intramolecular hydrogen bonds (Table 2) to the uncoordinated O atoms of the non-bridging trifluoroacetate ions.



(I)

The overall molecular architecture of (I) is similar to that of related compounds (Corkery & Hockless, 1997; Turpeinen *et al.*, 1987; Hagen *et al.*, 1993). The Co1—O*W* and Co2—O*W* distances of 2.190 (3) and 2.196 (3) Å, respectively, are the same within experimental uncertainty. The Co—N distances for the pyridine molecules (weak π acceptors) *trans* to the water O atom (π neutral) are significantly shorter than those *trans* to the fluoroacetate O atoms (weak π donor) as a result

© 2005 International Union of Crystallography
Printed in Great Britain – all rights reserved

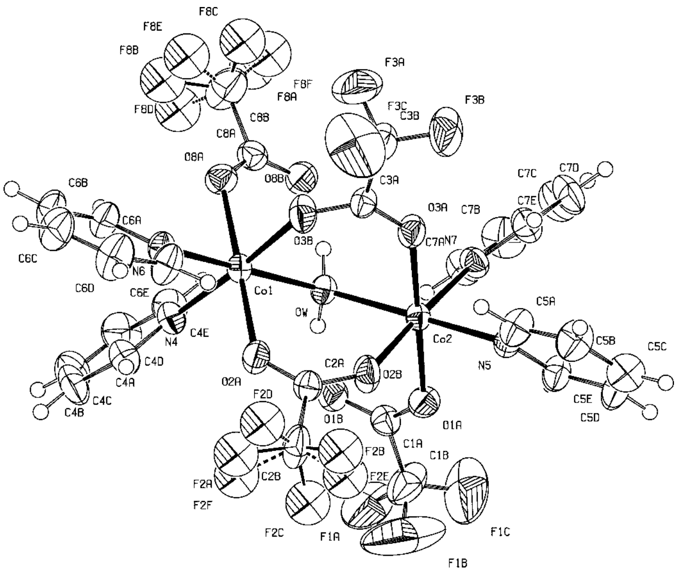


Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids for the non-H atoms. The minor disorder components are indicated by dashed C—F bonds.

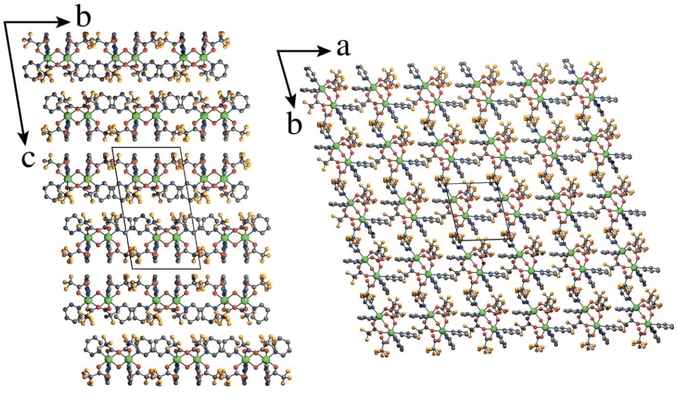


Figure 2
Packing diagrams for (I), showing the layered arrangement of molecules (left) and the plan of the layers (right).

of the well known *trans* influence [for example, Co1—N4 = 2.139 (4) Å *versus* Co1—N6 = 2.128 (4) Å]. The Co1—O*W*—Co2 angle is 116.86 (15)°, which is well within the expected range of values (*e.g.* Corkery & Hockless, 1997; Turpeinen *et al.*, 1997; Hagen *et al.*, 1993). There is no evidence of intermolecular hydrogen bonding or any other directional forces between the individual molecules. In terms of crystal packing, the molecules are arranged in layers in the *ab* plane (Fig. 2).

Experimental

$CoCl_2 \cdot 4H_2O$ (0.502 g) was mixed with Na_2CO_3 (0.154 g) and distilled water (approximately 10 ml) was added with stirring. Following this, trifluoroacetic acid (1 ml) was added dropwise. Pyridine (approximately 1 ml) was added and the mixture was reduced to dryness at 343 K on a rotary evaporator. Further pyridine (5 ml) was added and a pink precipitate was formed by addition of hexane (30 ml). The precipitate was dissolved in chloroform and mixed crystals (blue and red) were grown by vapour transport of diethyl ether. The blue crystals were shown to be pyridinium trichloropyridinecobalt(II) (Hahn *et al.*, 1997) by X-ray single-crystal analysis and the red crystals the title compound, (I).

Crystal data

$[Co_2(C_2F_3O_2)_4(C_5H_5N)_4(H_2O)]$
M_r = 903.86
Triclinic, $P\bar{1}$
a = 9.4211 (19) Å
b = 10.741 (2) Å
c = 19.185 (4) Å
α = 78.17 (3)°
β = 79.00 (3)°
γ = 76.00 (3)°
V = 1823.2 (6) Å^3
Z = 2
D_x = 1.647 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13594 reflections
θ = 1–27.5°
μ = 1.02 mm^{-1}
T = 180 (2) K
Block, red
0.28 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Thin–slice ω and φ scans
Absorption correction: multi-scan (SORTAV; Blessing 1995)
$T_{\min}$ = 0.775, $T_{\max}$ = 0.890
19699 measured reflections
8305 independent reflections
6603 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044
$\theta_{\max}$ = 27.5°
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.240
S = 1.05
8305 reflections
483 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.128P)^2 + 5.9158P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.007
$\Delta\rho_{\max}$ = 1.69 e Å^{-3}
$\Delta\rho_{\min}$ = −1.14 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O*W*	2.190 (3)	Co2—O*W*	2.196 (3)
Co1—O2*A*	2.070 (4)	Co2—O1*A*	2.095 (4)
Co1—O8*A*	2.092 (4)	Co2—O3*A*	2.074 (4)
Co1—O3*B*	2.098 (4)	Co2—O2*B*	2.095 (4)
Co1—N6	2.128 (4)	Co2—N5	2.124 (4)
Co1—N4	2.139 (4)	Co2—N7	2.138 (4)
Co1—O*W*—Co2	116.86 (15)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*—H1···O8*B*	0.96 (5)	1.67 (5)	2.616 (5)	165 (5)
O*W*—H2···O1*B*	0.96 (5)	1.68 (5)	2.610 (5)	161 (4)

Difference maps indicated that the F atoms attached to C2*B* and C8*B* were disordered over two sets of positions. Refined occupancies (sum constrained to unity) of 0.667 (6):0.333 (6) and 0.601 (6):0.399 (6) resulted for the major and minor components of C2*B* and C8*B*, respectively. The disordered F atoms were modelled with isotropic displacement parameters. The F atoms around C1*B* and C3*B* may also be slightly disordered but this was not resolved in the present data. The water molecule H atoms were located in a difference map and refined with distance restraints (O—H = 0.96 Å). Pyridine H atoms were placed in idealized locations (C—H = 0.93 Å) and refined as riding with the constraint $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier) applied. The higest peak and depest hole in are located 1.51 Å from atom F1*C* and 0.49 Å from F2*A*, respectively.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

JB is grateful to Jesus College, Cambridge for the award of a Junior Research Fellowship.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Corkery, R. W. & Hockless, D. C. R. (1997). *Acta Cryst.* C**53**, 840–843.
Hagen, K. S, Lachicotte, R., Kitaygorodskiy, A. & Elbouadili, A. (1993). *Angew. Chem. Int. Ed. Engl.* **32**, 1321–1324.
Hahn, F. E., Scharn, D. & Lugger, T. (1997). *Z. Kristallogr. New Cryst. Struct.* **212**, 472.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, Macromolecular Crystallography, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307-326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Turpeinen, U., Hamalainen, R. & Reedijk, J. (1987). *Polyhedron*, **6**, 1603–1610.