

The first X-ray structure of a hexaamminecobalt(III) salt with two different complex chlorocadmium anions: synthesis, characterization and crystal structure of $[\text{Co}(\text{NH}_3)_6]_4-[\text{CdCl}_6][\text{CdCl}_4(\text{SCN})(\text{H}_2\text{O})]_2\text{Cl}_2 \cdot 2\text{H}_2\text{O}$

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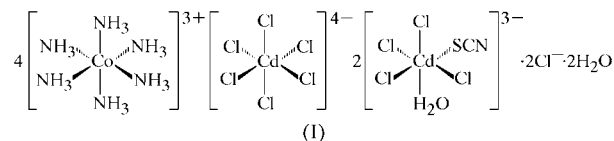
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In the title complex salt, tetrakis[hexaamminecobalt(III)] hexachlorocadmiate(II) bis[aquatetrachlorothiocyanatocadmiate(II)] dichloride dihydrate, the discrete ions, *i.e.* $[\text{Co}(\text{NH}_3)_6]^{3+}$, Cl^- , $[\text{CdCl}_6]^{4-}$ (located on an inversion centre) and $[\text{CdCl}_4(\text{SCN})(\text{H}_2\text{O})]^{3-}$, together with cocrystallized water molecules, are assembled by means of a network of hydrogen-bonding interactions. This is the first X-ray structure determination of a hexaamminecobalt(III) salt with two different complex chlorocadmium anions.

Comment

The octahedral hexaamminecobalt(III) cation serves as a powerful structure-determining component in a number of networks (Basolo, 1968; Reddy *et al.*, 2003; Dalrymple *et al.*, 2002; Takusagawa *et al.*, 1988; Gorol *et al.*, 2000; Sobolev & Figgis, 1997; Figgis *et al.*, 1979; Dahm & Adam, 2001; Baldwin & Kastner, 2002; Dalrymple & Shimizu, 2006), both metal/ligand-bonded and hydrogen-bonded. This cation should provide two triangular triamine faces with nine N—H bonds each on the sides that can act as potential hydrogen-bond donors. These H atoms are almost spread around the central metal atom in a spherical manner and, therefore, it should be an ideal choice as a receptor if the counter-ion is equipped with multiple hydrogen acceptors for second-sphere coordination. In continuation of our interest in hexaamminecobalt(III) complex salts (Bala *et al.*, 2006; Sharma, Bala, Sharma & Bond, 2005; Sharma, Bala, Sharma & Ferretti, 2005; Sharma *et al.*, 2004, 2004a,b; Sharma, Bala, Sharma, Rychlewska & Warzajtisc, 2005; Sharma, Bala, Sharma & Venugopalan, 2005, 2006; Sharma, Bala, Sharma, Venugopalan, Salas & Quiros,

2005; Sharma, Bala, Sharma, Kariuki *et al.*, 2005; Sharma, Bala, Sharma, Singh & Ferretti, 2006; Sharma, Bala, Sharma, Perez & Miguel, 2006), the present work reports the synthesis, spectroscopic characterization and crystal structure determination of a new complex salt, $[\text{Co}(\text{NH}_3)_6]_4[\text{CdCl}_6][\text{CdCl}_4(\text{SCN})(\text{H}_2\text{O})]_2\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ (I).



The vibrational spectrum shows that the stretching vibrations of the coordinated NH_3 molecule are lower than those of the free NH_3 molecule for two reasons, *viz.* the effect of coordination and the effect of the counter-ion. This is attributed to the weakening of the N—H bond owing to the formation of N—H...O and N—H... Cl^- hydrogen bonds. It is known that the antisymmetric stretch and symmetric NH_3 stretch, NH_3 degenerate deformation, NH_3 symmetric deformation, and NH_3 vibrations appear in the regions 3400–3000, 1650–1550, 1370–1200 and 800–900 cm^{-1} , respectively, for $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$ (Nakamoto, 1997); these values are comparable to those obtained for (I). The absorption band observed at 2096 cm^{-1} was assigned to the S-bonded SCN^- group, because CN stretching frequencies are generally lower in N-bonded SCN complexes (2050 cm^{-1}) than in S-coordinated (2100 cm^{-1}) complexes (Addison *et al.*, 2005). In (I), two types of water molecules are present, *viz.* one free and one coordinated. In general, the unligated water molecule absorbs in the ranges 3500–3300 (antisymmetric and symmetric stretch) and 1630–1600 cm^{-1} (OHO bending). Here, the stretching absorption frequency is observed at 3403 cm^{-1} (broad) and the bending absorption band perhaps is overlapped with NH_3

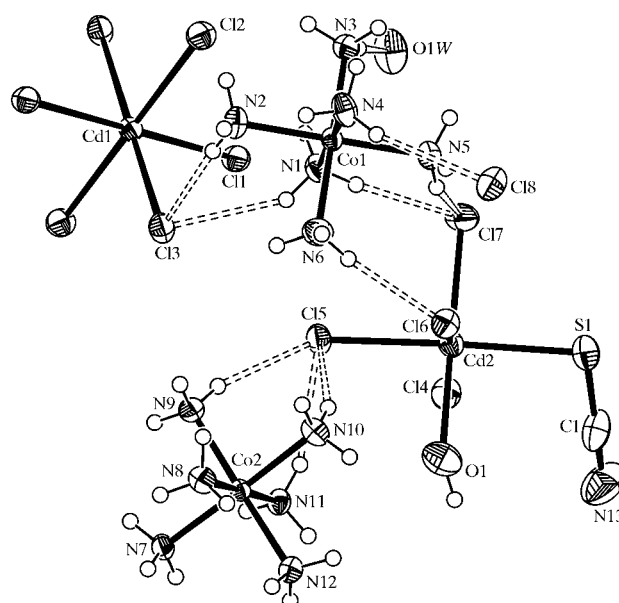


Figure 1
An ORTEP (Burnett & Johnson, 1996) view of the asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 40% probability level. Hydrogen bonds are drawn as broken lines.

degenerate deformation. Owing to instrumental limitations, the absorption bands in the lower region (*i.e.* less than 600 cm^{-1}) could not be observed.

The two electronic transitions $^1A_{1g} \rightarrow T_{1g}$ and $^1A_{1g} \rightarrow ^1T_{2g}$ for hexaamminecobalt(III) complexes are observed around 470 and 340 nm, respectively, producing the orange–yellow colour usual for a number of classical coordination compounds containing cobalt(III), as reported by Hendry & Ludi (1990). λ_{max} for lower energy was observed at 474 nm and for higher energy at 338 nm (showing *d–d* transitions typical for a low-spin Co^{III} d^6 octahedral complex).

The ^{13}C NMR spectrum shows a chemical shift value of 119 p.p.m., which is characteristic of the coordinated thiocyanate ion.

An *ORTEP* view (Burnett & Johnson, 1996) of the title compound is shown in Fig. 1. The asymmetric unit is quite complex, consisting of two hexaamminecobalt(III) cations, one-half of a hexachlorocadmate(II) ion, with the Cd atom located on an inversion centre, one aquatetrachlorothiocyano-cadmate(II) ion and one chloride counter-ion, together with a solvent water molecule. The coordination around the two Co and the two Cd atoms is slightly distorted octahedral, as shown by the values of the bond angles reported in Table 1. The Co–N distances, ranging from 1.956 (3) to 1.981 (3) Å, are in good agreement with those usually found in hexaamminecobalt(III) complexes. The Cd–ligand bond distances can be compared with those found in Cd complexes retrieved from the Cambridge Structural Database (CSD; Allen, 2002) and Inorganic Crystal Structure Database (ICSD, 2005). Out of a total of 23 structures containing $[\text{CdCl}_6]^{4-}$ complexes, only three show the presence of isolated anions (Beck & Milius, 1986; Veal & Hodgson 1972; Wagner *et al.*, 1996); the Cd–Cl distances in these compounds agree well with those reported in Table 1 for (I), ranging from 2.53 to 2.68 Å. The mean value of 2.72 (3) Å for the Cd–SCN bond length, calculated for a sample containing 68 entries (from the CSD), compares well with the length of 2.696 (1) Å reported in Table 1. The thiocyanate group is essentially linear, the S–C–N angle being $179.4 (6)^\circ$. Finally, a bibliographic search has been performed looking only for Cd complexes in which the water ligand is *trans* to chlorine to avoid bond-length variations due to the

trans influence. In the 24 structures retrieved, the Cd–O(water) bond distances vary in the quite large interval of 2.27–2.45 Å, the shortest distances being associated with water molecules *trans* to a Cl atom bridging two metal atoms. Accordingly, in the present compound, a relatively long value of 2.441 (4) Å is found.

The crystal architecture is built up by a complex three-dimensional hydrogen-bonding network. Hydrogen-bond parameters are reported in Table 2, and the unit-cell content, projected along the *a* axis, is shown in Fig. 2. All N atoms bound to the Co cations and the O atom of the coordinated water molecule $[\text{O}1 \cdots \text{Cl}2(x+1, y, z+1) = 3.138 (4) \text{ Å}$ and $\text{O}1 \cdots \text{Cl}8(x+\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 3.230 (5) \text{ Å}]$ act as donors in hydrogen-bond interactions, mainly towards Cl-atom acceptors. In only two interactions are different atoms, *i.e.* the O atom of the solvent molecule and atom N13 of the thiocyanate group, involved as hydrogen-bond acceptors. Both the coordinated and the non-coordinated Cl atoms interact with at least two *D–H* groups, this fact being attributable to the excess of donors with respect to acceptors. The nitrogen–halide distances can be compared with those recently reviewed by Steiner (1998), where mean values of 3.18 and 3.30 Å for $\text{Nsp}^2 \cdots \text{Cl}$ distances are reported. In the present structure, longer distances are found in the range 3.190 (4)–3.593 (4) Å, possibly because $\text{Nsp}^3\text{–H}$ amines are weaker donors than $\text{Nsp}^2\text{–H}$ and, moreover, on account of the bifurcated nature of the hydrogen-bond interactions. As an example, the distances between N-donors and the isolated anion Cl8, involved in six hydrogen bonds, turn out to be very long, in the range 3.298 (3)–3.525 (3) Å. The uncoordinated water molecule is also involved in the three-dimensional network of intermolecular interactions through its O atom, acting both as a hydrogen-bond acceptor ($\text{N}3\text{–H}33 \cdots \text{O}1\text{W}$; see Table 2) and as a hydrogen-bond donor $[\text{O}1\text{W} \cdots \text{N}13(x-1, y, z-1) = 2.868 (7) \text{ Å}$ and $\text{O}1\text{W} \cdots \text{N}12(x, y, z-1) = 3.177 (5) \text{ Å}]$, thus bridging both the cations and the aquatetrachlorothiocyano-cadmate complex. The $\text{N} \cdots \text{O}$ distances are typical of isolated hydrogen bonds, not assisted either by charge or by resonance (Gilli & Gilli, 2000).

Experimental

Hexaamminecobalt(III) chloride (1.00 g, 0.0037 mol) was dissolved in hot water (30 ml) in a beaker by mechanical stirring. In another beaker, cadmium(II) chloride monohydrate (0.68 g, 0.0037 mol) and ammonium thiocyanate (1.14 g, 0.0150 mol) were dissolved in hot water (20 ml). The two solutions were mixed and allowed to cool slowly. Shiny orange crystals appeared within 2 h, and these were filtered off and dried in air. The red–orange clear supernatant solution gave a second crop of crystals. The overall yield was nearly quantitative, and the salt obtained decomposes at 408 K. Solubility (298 K) in water: 0.76 g/100 ml. Elemental analysis calculated for $\text{C}_2\text{H}_{80}\text{Cd}_3\text{Cl}_{16}\text{Co}_4\text{N}_{26}\text{O}_4\text{S}_2$: C 1.38, H 4.60, N 20.95, Co 13.56%; found: C 1.32, H 4.56, N 20.89, Co 13.35%. IR: ν 3403 (*s*, –OH), ν_{as} 3248 (*b*, –NH₃), ν_s 3178 (*b*, –NH₃), ν 2096 (*s*, –SCN), δ_d 1607 (*b*, –NH₃), δ_s 1341 (*s*, –NH₃), ρ_r 846 (*s*, –NH₃) cm^{-1} . UV–vis (solution): λ_{max} = 474 (ϵ = $106.07\text{ M}^{-1}\text{L}^{-1}\text{cm}^{-1}$), 338 (ϵ = $86.87\text{ M}^{-1}\text{L}^{-1}\text{cm}^{-1}$). ^{13}C NMR (D_2O , 300 MHz): 119 (C, SCN) p.p.m.

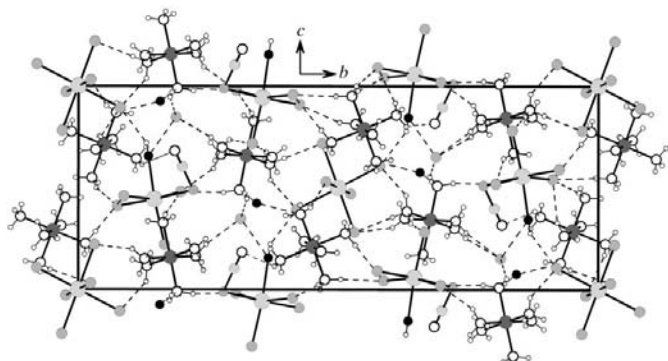


Figure 2

The packing of the title compound (viewed along *a*). N, O and Cl atoms are drawn as white, black and light-grey spheres, respectively.

Crystal data

[Co(NH₃)₆]₄[CdCl₆][CdCl₄(SCN)-
(H₂O)₂Cl₂·2H₂O
M_r = 1735.18
Monoclinic, *P*₂₁/*n*
a = 8.8511 (1) Å
b = 28.6847 (4) Å
c = 11.9607 (2) Å
β = 109.9580 (6)°
V = 2854.34 (7) Å³
Z = 2
D_x = 2.021 Mg m⁻³
Mo *Kα* radiation
μ = 3.10 mm⁻¹
T = 295 K
Prism, orange
0.29 × 0.22 × 0.11 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and *ω* scans
Absorption correction: empirical
(using intensity measurements)
(*SORTAV*; Blessing, 1995)
T_{min} = 0.486, *T_{max}* = 0.703
17048 measured reflections
6689 independent reflections
5417 reflections with *I* > 2σ(*I*)
R_{int} = 0.047
θ_{max} = 28.0°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.038
wR (*F*²) = 0.083
S = 1.05
6689 reflections
273 parameters
H atoms treated by a mixture of
independent and constrained
refinement
 $w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 5.6025P]$
where $P = (F_o^2 + 2F_c^2)/3$
(Δ/σ)_{max} = 0.003
Δρ_{max} = 1.45 e Å⁻³
Δρ_{min} = -0.64 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cd1—Cl1	2.5937 (9)	Co1—N1	1.965 (3)
Cd1—Cl2	2.631 (1)	Co1—N6	1.966 (4)
Cd1—Cl3	2.691 (1)	Co1—N5	1.972 (3)
Cd2—O1	2.441 (4)	Co1—N4	1.973 (3)
Cd2—Cl7	2.581 (1)	Co2—N10	1.962 (3)
Cd2—Cl4	2.593 (1)	Co2—N12	1.964 (3)
Cd2—Cl6	2.607 (1)	Co2—N11	1.965 (3)
Cd2—Cl5	2.633 (1)	Co2—N8	1.967 (3)
Cd2—S1	2.696 (1)	Co2—N7	1.974 (3)
Co1—N3	1.956 (3)	Co2—N9	1.981 (3)
Co1—N2	1.958 (3)		
Cl1—Cd1—Cl2	89.23 (3)	N3—Co1—N5	89.0 (1)
Cl1—Cd1—Cl3	90.26 (3)	N1—Co1—N5	90.7 (1)
Cl2—Cd1—Cl3	95.50 (3)	N6—Co1—N5	90.8 (1)
O1—Cd2—Cl4	87.10 (9)	N3—Co1—N4	91.0 (1)
Cl7—Cd2—Cl4	100.07 (4)	N2—Co1—N4	88.3 (1)
O1—Cd2—Cl6	78.91 (9)	N6—Co1—N4	90.3 (2)
Cl7—Cd2—Cl6	93.96 (3)	N5—Co1—N4	90.4 (1)
O1—Cd2—Cl5	87.62 (9)	N10—Co2—N12	90.4 (1)
Cl7—Cd2—Cl5	93.01 (4)	N10—Co2—N11	90.7 (1)
Cl4—Cd2—Cl5	88.64 (3)	N12—Co2—N11	88.6 (1)
Cl6—Cd2—Cl5	87.37 (3)	N10—Co2—N8	89.3 (1)
O1—Cd2—S1	89.4 (1)	N12—Co2—N8	90.3 (1)
Cl7—Cd2—S1	89.68 (4)	N12—Co2—N7	90.9 (1)
Cl4—Cd2—S1	93.47 (3)	N11—Co2—N7	90.3 (1)
Cl6—Cd2—S1	89.84 (3)	N8—Co2—N7	89.6 (1)
N3—Co1—N2	91.2 (1)	N10—Co2—N9	88.8 (1)
N3—Co1—N1	89.1 (1)	N11—Co2—N9	89.7 (1)
N2—Co1—N1	90.6 (1)	N8—Co2—N9	91.3 (1)
N2—Co1—N6	89.0 (1)	N7—Co2—N9	89.9 (1)
N1—Co1—N6	89.6 (2)		

Only one H atom (H100 bound to O1) was found in a difference Fourier map; its coordinates were refined, keeping its displacement parameter fixed. H atoms belonging to ammine groups were included in calculated positions, riding on their carrier atoms [*U*_{iso}(H) = 1.2*U*_{eq}(N)]. The H atoms of the solvent water molecule (bound to O1W) were not found in a difference Fourier map and were not included in the refinement. The highest difference peak is not close to any atom (within 2.76 Å).

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H11...Cl7	0.89	2.54	3.424 (3)	170
N1—H13...Cl3	0.89	2.54	3.327 (4)	148
N2—H22...Cl3	0.89	2.42	3.302 (3)	173
N2—H23...Cl2	0.89	2.56	3.190 (4)	128
N3—H33...O1W	0.89	2.16	3.052 (6)	179
N4—H41...Cl8	0.89	2.59	3.455 (3)	163
N5—H51...Cl8	0.89	2.52	3.403 (4)	170
N5—H53...Cl7	0.89	2.78	3.562 (4)	147
N6—H61...Cl6	0.89	2.39	3.263 (4)	166
N9—H91...Cl5	0.89	2.51	3.371 (4)	163
N10—H101...Cl5	0.89	2.57	3.421 (4)	159
N11—H111...Cl5	0.89	2.54	3.381 (4)	158
N2—H21...Cl7 ⁱ	0.89	2.41	3.294 (4)	176
N4—H42...Cl7 ⁱ	0.89	2.57	3.460 (4)	174
N8—H81...Cl4 ⁱ	0.89	2.55	3.397 (3)	160
N9—H92...Cl4 ⁱ	0.89	2.46	3.339 (3)	168
N10—H103...N13 ⁱ	0.89	2.48	3.147 (7)	131
N3—H31...Cl6 ⁱⁱ	0.89	2.39	3.264 (4)	168
N3—H32...Cl8 ⁱⁱ	0.89	2.55	3.298 (3)	142
N7—H73...Cl1 ⁱⁱⁱ	0.89	2.41	3.230 (3)	152
N7—H71...Cl4 ⁱⁱⁱ	0.89	2.71	3.593 (4)	173
N9—H93...Cl4 ⁱⁱⁱ	0.89	2.63	3.468 (4)	158
N11—H112...Cl5 ⁱⁱⁱ	0.89	2.44	3.291 (3)	161
N12—H122...Cl1 ⁱⁱⁱ	0.89	2.54	3.370 (3)	156
N7—H72...Cl1 ^{iv}	0.89	2.64	3.424 (4)	147
N12—H123...Cl1 ^{iv}	0.89	2.50	3.264 (4)	144
N8—H83...Cl3 ^v	0.89	2.49	3.376 (4)	175
N10—H102...Cl8 ^{vi}	0.89	2.57	3.455 (3)	175
N12—H121...Cl8 ^{vi}	0.89	2.64	3.525 (3)	177
N11—H113...Cl2 ^{vii}	0.89	2.57	3.436 (3)	166
O1—H100...Cl2 ^{vii}	0.88	2.40	3.138 (4)	141

Symmetry codes: (i) *x* − 1, *y*, *z*; (ii) *x* − ½, −*y* + ½, *z* − ½; (iii) −*x* + 1, −*y*, −*z* + 1; (iv) *x*, *y*, *z* + 1; (v) −*x*, −*y*, −*z* + 1; (vi) *x* + ½, −*y* + ½, *z* + ½; (vii) *x* + 1, *y*, *z* + 1.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*, *PARST* (Nardelli, 1995) and *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA3042). Services for accessing these data are described at the back of the journal.

References

- Addison, A. W., Butcher, R. J., Homonnay, Z., Pavlishchuk, V. V., Prushan, M. J. & Thompson, L. K. (2005). *Eur. J. Inorg. Chem.* pp. 2404–2408.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bala, R., Sharma, R. P., Sharma, R. & Kariuki, B. M. (2006). *Inorg. Chem. Commun.* **8**, 852–855.
- Baldwin, S. M. & Kastner, M. E. (2002). *Acta Cryst.* **C58**, m611–m612.
- Basolo, F. (1968). *Coord. Chem. Rev.* **3**, 213–223.
- Beck, H. P. & Milius, W. (1986). *Z. Anorg. Allg. Chem.* **539**, 7–17.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Dahm, M. & Adam, A. (2001). *Z. Naturforsch. Teil B*, **56**, 1117–1122.

- Dalrymple, S. A., Parvez, M. & Shimizu, G. K. H. (2002). *Inorg. Chem.* **41**, 6986–6996.
- Dalrymple, S. A. & Shimizu, G. K. H. (2006). *Chem. Commun.* pp. 956–958.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Figgis, B. N., Skelton, B. W. & White, A. H. (1979). *Aust. J. Chem.* **32**, 417–419.
- Gilli, G. & Gilli, P. (2000). *J. Mol. Struct.* **552**, 1–15.
- Gorol, M., Mosch-Zanetti, N. C., Nadia, C., Noltmeyer, M. & Roesky, H. W. (2000). *Z. Anorg. Allg. Chem.* **626**, 2318–2324.
- Hendry, P. & Ludi, A. (1990). *Adv. Inorg. Chem.* **35**, 117–198.
- ICSD (2005). Inorganic Crystal Structure Database. Version 2005–02. FIZ Karlsruhe, Germany, and The National Institute of Standards and Technology, USA.
- Nakamoto, K. (1997). *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, 5th ed. New York: John Wiley and Sons.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Nonius (1997). *KappaCCD Server Software*. 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.
- Reddy, D. S., Duncan, S. & Shimizu, G. K. H. (2003). *Angew. Chem. Int. Ed.* **42**, 1360–1364.
- Sharma, R. P., Bala, R., Sharma, R., Bhasin, K. K. & Chadha, R. K. (2004). *J. Coord. Chem.* **57**, 313–319.
- Sharma, R. P., Bala, R., Sharma, R. & Bond, A. D. (2005). *Acta Cryst.* **C61**, m356–m358.
- Sharma, R. P., Bala, R., Sharma, R. & Ferretti, V. (2005). *Inorg. Chim. Acta*, **358**, 3457–3464.
- Sharma, R. P., Bala, R., Sharma, R., Kariuki, B. M., Rychlewska, U. & Warzajtisc, B. (2005). *J. Mol. Struct.* **748**, 143–151.
- Sharma, R. P., Bala, R., Sharma, R., Perez, J. & Miguel, D. (2006). *J. Mol. Struct.* **797**, 49–55.
- Sharma, R. P., Bala, R., Sharma, R., Rychlewska, U. & Warzajtisc, B. (2005). *J. Fluorine Chem.* **126**, 967–975.
- Sharma, R. P., Bala, R., Sharma, R., Singh, K. N. & Ferretti, V. (2006). *J. Mol. Struct.* **784**, 117–123.
- Sharma, R. P., Bala, R., Sharma, R. & Venugopalan, P. (2004a). *J. Coord. Chem.* **57**, 1563–1569.
- Sharma, R. P., Bala, R., Sharma, R. & Venugopalan, P. (2004b). *J. Mol. Struct.* **694**, 229–234.
- Sharma, R. P., Bala, R., Sharma, R. & Venugopalan, P. (2005). *J. Mol. Struct.* **752**, 170–177.
- Sharma, R. P., Bala, R., Sharma, R. & Venugopalan, P. (2006). *CrystEngComm*, **8**, 215–221.
- Sharma, R. P., Bala, R., Sharma, R., Venugopalan, P., Salas, J. M. & Quiros, M. (2005). *J. Fluorine Chem.* **126**, 1543–1548.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Sobolev, A. N. & Figgis, B. N. (1997). *Acta Cryst.* **C53**, 661–663.
- Steiner, T. (1998). *Acta Cryst.* **B54**, 456–463.
- Takusagawa, F., Shaw, J. & Everett, G. W. (1988). *Inorg. Chem.* **27**, 3107–3112.
- Veal, J. T. & Hodgson, D. J. (1972). *Inorg. Chem.* **11**, 597–602.
- Wagner, B., Warda, S. A., Hitchman, M. A. & Reinen, D. (1996). *Inorg. Chem.* **35**, 3967–3974.