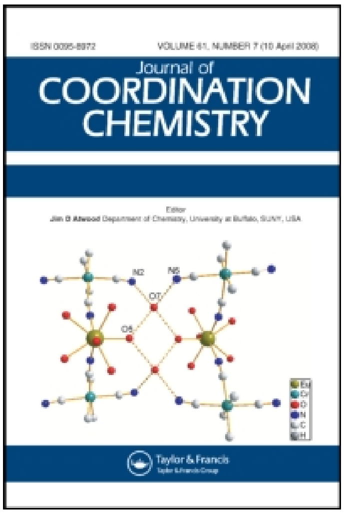

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Cationic cobaltammines as anion receptors. Part V. Synthesis, characterization and X-ray structure of $[CO(NH_3)_6]ClMoO_4 \cdot 3H_2O$

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CATIONIC COBALTAMMINES AS ANION RECEPTORS. PART V. SYNTHESIS, CHARACTERIZATION AND X-RAY STRUCTURE OF $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$

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Orange, single crystals of the bimetallic salt hexaamminecobalt(III) chloride molybdate trihydrate, $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$, have been prepared by reacting hot aqueous solutions of hexaamminecobalt(III) chloride and sodium molybdate dihydrate in a 1:1 molar ratio. The salt was characterized by elemental analyses and spectroscopic studies (IR and electronic). The title complex salt crystallizes in orthorhombic, space group *Pnma*, with $a = 18.408(2)$, $b = 8.672(1)$, $c = 8.661(1)$ Å, $V = 1382.6(3)$ Å^3, $Z = 4$ and $R = 0.0255$. A single-crystal X-ray structure determination revealed the presence of discrete ions $[Co(NH_3)_6]^{3+}$, Cl^- and MoO_4^{2-} and three lattice water molecule in the solid state. The formation of this salt suggests that $[Co(NH_3)_6]^{3+}$ may be used as an anion receptor for the molybdate ion.

Keywords: Anion receptor; Cobalt(III); Coordination chemistry; Inorganic synthesis; Spectroscopy; X-ray Crystallography

INTRODUCTION

Anions play an important role in chemistry, biology and the environment. It is believed that they participate in 70% of enzymatic reactions [1]. Anion coordination chemistry, the binding of anions by receptor molecules, has been recognized and developed as a new area of chemistry [2,3]. The design and synthesis of smart molecules that are able to function as sensors of charged species are of immense interest [4–6]. While cation receptors have been studied extensively, the design of anion receptors has been taken up only recently [7,8]. Because of their varied shapes and sizes [9], anions pose greater challenges than cations. The capture of oxoanions (with, for instance, molybdenum, antimony, chromium, selenium) in particular is difficult because of their large ionic radii, high energy of solvation and low Lewis basicities [10]. Such receptors could be valuable if they were capable of sequestering anionic wastes or sensing

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their presence. Traditional design has relied on the use of hydrogen donors and cationic Lewis acid site-attracting anions. More recently, the value of weak interactions, such as C–H…π interactions, has been recognized [11] and the use of selective anion receptors for oxoanions [12,13], such as sapphyrins [14], cationic cyclic polyamines, calixarenes, azacages or open-chain counterparts have been reported; however, these anion receptors are difficult to synthesize or are expensive. Therefore, the search for new anion receptors that may find potential applications [15] in the areas of analytical chemistry, biology, catalysis, coordination equilibria and waste management is important.

The hexaamminecobalt(III) cation has proved very useful as a large counter ion in stabilizing [16] unusual chloro anions that were hitherto unknown; for example $[Co(NH_3)_6][MCl_6]$ (M = Fe, Bi, In) and $[Co(NH_3)_6][MCl_5]$ (M = Cd, Cu, Hg). However, few studies are reported in the literature regarding salt formation of the hexaamminecobalt(III) cation with oxoanions [17,18], e.g., $[Co(NH_3)_6]ClS_2O_3 \cdot H_2O$ and $[Co(NH_3)_6]ClCrO_4 \cdot 3H_2O$. A low value of the ion pairing constant, K_o, for molybdate(VI) as compared to sulfate [19] (K_o for SO_4^{2-} is six times that for MoO_4^{2-}) suggests that the hexaamminecobalt(III) salt of molybdate [20] may be difficult to isolate. We have undertaken an extensive research program to explore hexaamminecobalt(III) chloride as an anion receptor because it is relatively easy to synthesize from readily available materials. This article reports the synthesis, characterization and X-ray structure determination of hexaamminecobalt(III) chloride molybdate trihydrate, $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$, in continuation of our interest in cobalt(III) salts [21–30].

EXPERIMENTAL

Materials

Analytical grade reagents were used without further purification. $[Co(NH_3)_6]Cl_3$ was prepared by air oxidation of a Co(II) salt in ammoniacal solution in the presence of activated charcoal as catalyst, according to the method described by Bjerrum and McReynold [31].

Instrumentation

Cobalt and chloride were determined by standard methods [32]. C, H and N were estimated with a Perkin Elmer 2400CHN instrument. Electronic spectrum was recorded using a Hitachi 330 spectrophotometer with water as solvent. Infrared spectra of the complex salt and $Na_2MoO_4 \cdot 3H_2O$ were recorded using a Perkin Elmer spectrum RX FTIR system using Nujol mull between KBr plates.

Crystal Structure Determination

Good single crystals of $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$ suitable for X-ray diffraction studies were grown from an aqueous solution (obtained by mixing the reactants in equimolar quantities) by slow evaporation. A single crystal of dimension 0.29 × 0.21 × 0.18 mm was mounted along the largest dimension and used for data collection. Intensity data were collected on a Siemens P4 single-crystal diffractometer equipped

TABLE I Crystal data and structure refinement details for the salt

Empirical formula	$H_{24}ClCoMoN_6O_7$
Formula weight	410.57
Temperature	293(2) *K*
Diffractometer used	Siemens P4
Radiation used, Wavelength	Mo Kα, 0.71073 Å
Crystal system, Space group	Orthorhombic, *Pnma*
Unit cell dimensions	$a = 18.408(2)$ Å $b = 8.672(1)$ Å $c = 8.661(1)$ Å
Volume	1382.6(3) $Å^3$
Z, Calculated density	4, 1.972 mg m^{-3}
Absorption coefficient	2.328 mm^{-1}
F(000)	832
Crystal size	0.29 × 0.21 × 0.18 mm
Max. and min. transmission	0.963, 0.849
Theta range for data collection	2.21 to 24.99°
Index ranges	$-21 \leq h \leq 0$, $0 \leq k \leq 10$, $0 \leq l \leq 10$
Reflections collected	1309
Independent reflections	1309
Completeness to theta = 24.99°	99.9%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	1309/0/88
Goodness-of-fit on F^2	1.111
Weighting scheme	$1/[\sigma^2(F_o^2) + (0.0253P)^2 + 1.63P]$ $P = (\max(F_o^2, 0) + 2^*F_c^2)/3$
Final *R* indices, 1212 reflections $[I > 2\sigma(I)]$	$R1 = 0.0255$, $wR2 = 0.0681$
R indices (all data)	$R1 = 0.0282$, $wR2 = 0.0694$
Extinction coefficient	0.0019(3)
Largest diff. peak and hole	0.576 and –0.410 e $Å^{-3}$

with a sealed molybdenum tube ($\lambda = 0.71073$ Å) and a highly oriented graphite monochromator. Data were collected by the 2θ–θ scan mode with variable scan speed ranging from 2.0 to a maximum of 60.0°/min. Data were corrected for Lorentz and polarization effects and an absorption correction based on psi-scans was also applied. The structure was solved by direct methods using the SHELX-97 package [33] and refined using the same source. Refinement converged to a final *R* value of 0.0255 and $wR = 0.0681$. All other information regarding the refinement is presented in Table I.

Synthesis of $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$

One gram of hexaamminecobalt(III) chloride (1 g in 30 cm^3 of hot water) and sodium molybdate dihydrate (0.909 g in 10 cm^3 of hot water) were mixed together and then allowed to cool slowly to give an orange microcrystalline solid, within half an hour, which was filtered, washed with ice-cold water and dried in air. Reddish-orange single crystals were obtained by dissolving the product in hot water and allowing the solution to cool slowly. The melting point of the salt was > 200°C. Elemental analyses are consistent with the composition $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$ (Found: H, 5.82; N, 20.40, Cl, 8.61; Co, 14.10%. Calculated: H, 5.84; N, 20.45; Cl, 8.64; Co, 14.34%).

RESULTS AND DISCUSSION

Hexaamminecobalt(III)chloride and sodium molybdate could react in a number of ways depending upon the stoichiometric ratios of the reactants. Reddish-orange crystals obtained by mixing 1:1 stoichiometric quantities of hexaamminecobalt(III) chloride and sodium molybdate dihydrate gave satisfactory elemental analyses corresponding to $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$. The salt is soluble in hot water and DMSO but insoluble in ethanol and acetone.

Electronic Spectra

As reported in the literature [34] the two 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 familiar orange yellow color for a number of classical coordination compounds containing cobalt(III). The electronic spectrum of the title complex salt was recorded in water. Strong absorption maxima were observed at 470 and 340 nm, in agreement with those of related salts such as hexaamminecobalt(III) chloride selenate trihydrate ($\lambda_{max} = 470$; 340 nm) [28] and hexaamminecobalt(III) chloride dichromate monohydrate ($\lambda_{max} = 360$; 260 nm) [29]. However, in the case of hexaamminecobalt(III) chloride dimethanesulfonate [30] there are appreciable shifts of absorption bands ($\lambda_{max} = 430$; 373 nm).

Infrared Spectra

Vibrational spectra of metal ammine complexes have been studied extensively and were reviewed by Schmidt and Müller [35,36]. NH_3 stretching frequencies of the complexes are lower (by 40–200 cm^{-1}) than those of free NH_3 molecules for two reasons, reflecting coordination and effects of the counter ion. This is attributed to weakening of the N–H bond due to the formation of, for example, $NH \cdots Cl^-$ and $N\text{–}H \cdots O$ hydrogen bonds. The antisymmetric and symmetric NH_3 stretch, NH_3 degenerate deformation, NH_3 symmetric deformation and other NH_3 vibrations appear in the regions 3400–3000, 1650–1550, 1370–1200 and 800–900 cm^{-1}, respectively, for $[Co(NH_3)_6]Cl_3$. These are comparable with those in $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$ (Table II). Free MoO_4^{2-} ions have tetrahedral symmetry, lowered to C_{3v} local symmetry on coordination as a monodentate ligand and C_{2v} on coordination as a bidentate. As the Co–O–Mo bond is likely to be bent, the overall molecular symmetry will be C_s whether the ligand is monodentate or bidentate. Spectra of coordination isomers $[Co(NH_3)_5MoO_4]Cl$ and $[Co(NH_3)_5Cl]MoO_4$ are quite different and suggest that the molybdate group function as a ligand in the former but free MoO_4^{2-} ion is present in the latter [37]. A similar situation is observed for $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$ in that a sharp

TABLE II Infrared data (cm^{-1}) and peak assignments for $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$, and related species

Comples salts *$[Co(NH_3)_6]Cl_3$*	*$[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$*	*$Na_2MoO_4 \cdot 2H_2O$*	*Assignment*
	3392[a]	3296[a]	(O–H)ν
3240	3242[a]		(N–H)ν_{as}
3160	3139[a]		(N–H)ν_s
1619	1648		(N–H)δ_d
1326	1330		(N–H)δ_s
	814	830	(Mo=O)ν
831	[b]		(N–H)ρ_r

[a]Broad bands. [b]Hidden by the absorption of the molybdate ion.

peak is observed at 814 cm^{-1} for ionic MoO_4^{2-}. The peak assignments [38,39] were made in consultation with literature values (Table II).

X-ray Crystallography

The structure of the title salt has been unambiguously determined by single-crystal X-ray crystallography. Discrete $[Co(NH_3)_6]^{3+}$, Cl^- and MoO_4^{2-} ions and three molecules of water are present in the solid state as shown in Figure 1. A mirror plane passes through the Mo atom, reducing the possible T_d symmetry to C_s. Nevertheless, the molybdate ion is very nearly tetrahedral and the maximum deviation in Mo–O distances is 0.007 Å and in O–Mo–O angles is 3.9° (Table IV). Atomic coordinates and equivalent isotropic displacement parameters are given in Table III and selected bond lengths and angles are listed in Table IV. Hydrogen bonding parameters are given in Table V. Co–N bond distances are in the range 1.965(2)–1.973(4) Å, while *cis* N–Co–N bond angles are in the range 88.95(11)–90.64(11)°. In the hexaamminecobalt(III) chloride [40], the average Co–N distance is 1.963 Å and the maximum deviation of *cis* N–Co–N bond angles from ideal values is 2.1°. Values for the title complex salt are also in agreement with the salts hexaamminecobalt(III) chloride selenate trihydrate [28], hexaamminecobalt(III)

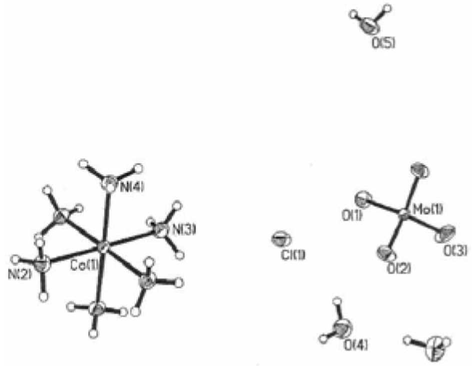

FIGURE 1 ORTEP Diagram of $[Co(NH_3)_6]Cl\ MoO_4.3H_2O$ (Labels are not shown for symmetry related atoms)

TABLE III Atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for the complex. U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor

	x/a	*y/b*	*z/c*	*U*(eq)
Mo(1)	974(1)	2500	6132(1)	21(1)
Co(1)	3324(1)	2500	18242(1)	17(1)
Cl(1)	1877(1)	2500	11889(2)	36(1)
O(1)	1526(1)	4139(3)	5889(3)	37(1)
O(3)	240(2)	2500	4842(5)	55(1)
N(4)	4059(1)	888(3)	17886(3)	27(1)
O(2)	614(2)	2500	8014(4)	37(1)
N(3)	3073(2)	2500	16028(4)	29(1)
N(2)	3589(2)	2500	20443(4)	27(1)
N(1)	2591(1)	904(3)	18643(3)	26(1)
O(4)	109(2)	2500	11736(4)	43(1)
O(5)	4102(1)	404(3)	4477(3)	47(1)

TABLE IV Selected bond lengths (Å) and angles (°) for $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$

Mo(1)–O(3)	1.753(3)	Mo(1)–O(2)	1.759(3)
Mo(1)–O(1)	1.760(2)	Mo(1)–O(1)#1	1.760(2)
Co(1)–N(1)#1	1.965(2)	Co(1)–N(1)	1.965(2)
Co(1)–N(2)	1.967(4)	Co(1)–N(4)#1	1.969(2)
Co(1)–N(4)	1.969(2)	Co(1)–N(3)	1.973(4)
O(3)–Mo(1)–O(2)	107.47(18)	O(3)–Mo(1)–O(1)	111.65(10)
O(2)–Mo(1)–O(1)	109.15(10)	O(3)–Mo(1)–O(1)#1	111.65(10)
O(2)–Mo(1)–O(1)#1	109.15(10)	O(1)–Mo(1)–O(1)#1	107.74(16)
N(1)#1–Co(1)–N(1)	89.60(15)	N(1)#1–Co(1)–N(2)	89.92(11)
N(1)–Co(1)–N(2)	89.92(11)	N(1)#1–Co(1)–N(4)#1	89.97(11)
N(1)–Co(1)–N(4)#1	178.79(11)	N(2)–Co(1)–N(4)#1	88.95(11)
N(1)#1–Co(1)–N(4)	178.79(11)	N(1)–Co(1)–N(4)	89.97(11)
N(2)–Co(1)–N(4)	88.95(11)	N(4)#1–Co(1)–N(4)	90.44(15)
N(1)#1–Co(1)–N(3)	90.64(10)	N(1)–Co(1)–N(3)	90.64(11)
N(2)–Co(1)–N(3)	179.21(15)	N(4)#1–Co(1)–N(3)	90.50(11)
N(4)–Co(1)–N(3)	90.50(11)		

Symmetry transformation used to generate equivalent atoms: #1 $x, -y+1/2, z$.

TABLE V Hydrogen bonding parameters (Å) and (°) for $[Co(NH_3)_6]ClMoO_4 \cdot 3H_2O$

D–H···A	*D–H*	*H···A*	*D···A*	*< DHA*	*Symmetry operations*[a]
N2–H2A···Cl1	0.890	2.528	3.391	163.34	i
N1–H1A···Cl1	0.890	2.540	3.398	162.10	i
N1–H1B···Cl1	0.890	2.831	3.461	128.97	ii
O4–H4E···Cl1	0.850	2.566	3.258	139.34	–
N4–H4C···O5	0.890	2.121	2.984	163.06	i
N3–H3B···O5	0.890	2.127	2.948	153.15	i
N2–H2B···O1	0.890	2.304	2.948	129.16	iii
N1–H1B···O1	0.890	2.268	2.961	134.50	iv
N1–H1C···O1	0.890	2.208	3.088	169.69	v
O4–H4D···O3	0.850	1.853	2.701	175.15	i
O5–H5A···O2	0.850	2.034	2.867	166.64	vi
O5–H5B···O4	0.850	1.955	2.801	172.84	vii

[a] = i: [x,y,z+1], ii: [−x+1/2,−y, z+1/2], iii: [−x+ 1/2,−y+1, z+3/2], iv:[−x+1/2,y−1/2, z+3/2], v: [x,−y+1/2, z+1], vi: [−x+1/2, −y, z−1/2], vii: [x+1/2, y,−z−1/2].

chloride dichromate monohydrate [29] and hexaamminecobalt(III) chloride dimethanesulfonate [30].

The molybdate tetrahedron is slightly elongated along the Mo(1)–O(1) bond due to hydrogen bond interactions, but Mo–O distances for the title complex are comparable with those in $(NH_4)_4[(UO_2)_5(MoO_4)_7](H_2O)_5$ in which the average distance is 1.79(1) Å [41]. The $[Co(NH_3)_6]^{3+}$, Cl^- and MoO_4^{2-} ions are held together by electrostatic forces and hydrogen bonds of the type O–H···O and N–H···Cl^- resulting in a three-dimensional hydrogen-bonded network in the solid state. The stability of the salt suggests that $[Co(NH_3)_6]^{3+}$ may find application as a receptor for the molybdate ion. Further studies to test the hexaamminecobalt(III) ion as an anion receptor for other organic and inorganic anions are in progress.

Supplementary Data

Crystallographic data have been deposited at the FIZ, Germany, CSD number 413763. The data may be accessed as follows: tel.: (49) 7247 808 205; fax: (49) 7247 808 666; e-mail: crysdata@fiz-karlsruhe.de.

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