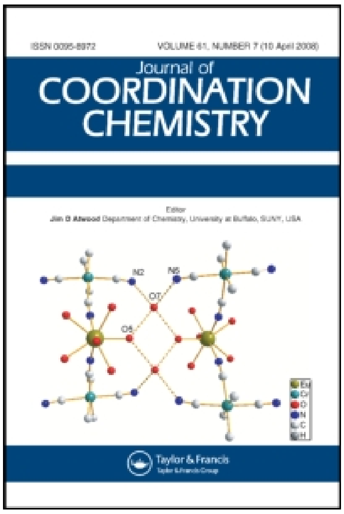

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Cationic Cobaltammines as anion receptors: a convenient synthesis, characterization and X-ray structure of $[CO(NH_3)_6]ClSeO_4 \cdot 3H_2O$

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CATIONIC COBALTAMMINES AS ANION RECEPTORS: A CONVENIENT SYNTHESIS, CHARACTERIZATION AND X-RAY STRUCTURE OF $[CO(NH_3)_6]ClSeO_4 \cdot 3H_2O$

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Reddish-orange single crystals of hexaamminecobalt(III) chloride selenate trihydrate, $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$, have been prepared by reacting aqueous solutions of hexaamminecobalt(III) chloride and potassium selenate in a 1 : 1 molar ratio. The salt was characterized by elemental analyses, thermogravimetry, magnetic moment and spectroscopic studies (IR, electronic). A single crystal X-ray structure determination revealed that the title complex salt crystallizes in the orthorhombic space group *Pnma* with $a = 18.230(2)$, $b = 8.591(2)$, $c = 8.593(2)$ Å, $V = 1345.9(4)$ Å^3, $Z = 4$, $R = 0.0400$ and $R_w = 0.0980$. In the salt the network of hydrogen bonds between the hexaamminecobalt(III) cation, water molecule, chloride and oxygen atoms of the selenate ion represents the second sphere coordination and also suggests that $[Co(NH_3)_6]^{3+}$ may be used as an anion receptor for the selenate ion.

Keywords: Cobalt; Coordination chemistry; Anion receptor; Synthesis; IR spectroscopy; Electronic spectroscopy; X-ray crystallography

INTRODUCTION

Anions play an important role in chemistry and biology. Anion coordination chemistry, the binding of anions by receptor molecules, has been recognized and developed as a new area of chemistry [1,2]. The design and synthesis of smart molecules that are able to function as sensors of charged species are of immense interest [3–5]. Although cation receptors have been studied extensively, the design of anion receptors has only recently been investigated [6,7]. Because of their varied shapes and sizes [8], anions pose a greater challenge than cations. The capture of oxoanions (antimony, chromium, selenium, etc.), in particular, is difficult because of their large ionic radii, high energy of solvation and lower Lewis basicities [9]. Such receptors could be valuable

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if they are capable of sequestering anionic wastes or sensing 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$\cdots\pi$ interactions has been recognized [10] and the use of selective extractants to extract oxoanions [11,12] such as cationic polyamines, calixarenes, azacages or open chain counterparts has been investigated. Recovery and recycling of oxoanions is necessary, as these species are problematic contaminants in soil and water [11] because they are either toxic [13], environmentally undesirable or radioactive. Therefore, the search for new anion receptors that may find potential applications [14] 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 [15] unusual chloro anions that were hitherto unknown, e.g. $[Co(NH_3)_6][MCl_6]$ (M = Fe, Bi, In) and $[Co(NH_3)_6][MCl_5]$ (M = Cd, Cu, Hg). Few studies, however, are reported in the literature regarding complex formulation of the hexaamminecobalt(III) cation with oxoanions [16,17], e.g. $[Co(NH_3)_6]Cl\cdot S_2O_3\cdot H_2O$ and $[Co(NH_3)_6]Cl\cdot CrO_4\cdot 3H_2O$. We have undertaken a research program to exploit the hexaamminecobalt(III) cation as an anion receptor. This paper reports its use as an anion receptor for the selenate ion and involves the synthesis, characterization and X-ray structure determination of hexaamminecobalt(III) chloride selenate trihydrate, $[Co(NH_3)_6]ClSeO_4\cdot 3H_2O$, in the continuation [18–24] of our interest in cobalt(III) complex salts.

EXPERIMENTAL

Analytical grade reagents were used without any further purification. $[Co(NH_3)_6]Cl_3$ was prepared according to the method described by Bjerrum and McReynold [25].

Instrumentation

Cobalt was determined by a standard method and C, H and N were estimated microanalytically using a Perkin-Elmer 2400CHN instrument. Electronic spectra were recorded using a Hitachi 330 spectrophotometer with water as solvent. Infrared spectra were recorded using a Perkin–Elmer Spectrum RX FT-IR system using Nujol mull in KBr plates. Thermogravimetric analysis was performed on a MOM (Budapest) instrument. Magnetic moments were determined at room temperature by Gouy's method.

Synthesis of $[Co(NH_3)_6]ClSeO_4\cdot 3H_2O$

One gram of hexaamminecobalt(III) chloride was dissolved in 30 cm^3 of hot water in a beaker with mechanical stirring. In another beaker, 0.83 g of potassium selenate was dissolved in 10 cm^3 of hot water. Both solutions were mixed and allowed to cool slowly, orange, shining crystals appearing within 30 min; these were filtered and dried in air. The reddish-orange coloured, clear supernatant solution gave a second crop of crystals. The overall yield was nearly quantitative. The advantages of this method are a one-pot synthesis, pure products and direct formation of single crystals suitable for X-ray structure determination. Elemental analyses were consistent with the composition $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$. Found (%): H, 6.10; N, 21.30; Cl, 8.90; Co, 14.90. Calcd.: H, 6.15; N, 21.35; Cl, 9.02; Co, 14.97.

Crystallography

Crystal data of a paralleloepiped reddish-orange crystal ($0.16 \times 0.18 \times 0.33$ mm) were collected with a Rigaku AFC6S diffractometer equipped with a sealed molybdenum tube and a highly oriented graphite monochromator. A constant scan speed of 8° per min in ω was used and weak reflections $[I < 5\sigma(I)]$ were rescanned to a maximum of six times and counts accumulated to assure good counting statistics. Intensities of monitor reflections measured after every 150 reflections did not change significantly during 12 h of X-ray exposure. Unit cell dimensions were obtained by least-squares fit to 25 reflections ($15° < 2\theta < 30°$). The data were corrected for Lorentz and polarization effects and an absorption correction was also applied using a psi-scan method. Cell parameters and other relevant data are given in Table I. The systematic absences ($0kl$, $k+l=2n+1$; and $hk0$, $h=2n+1$) indicated a choice between the space groups *Pnma* and $Pna2_1$. The former space group was chosen, as confirmed by successful refinement of the structure. The number of molecules per unit cell, $Z=4$, in the space group *Pnma* suggested that the ion possessed local mirror symmetry. The structure was solved by the Patterson method using SHELXS-86 [26]. All non-hydrogen atoms were refined anisotropically by the full-matrix least-squares method. The function minimized was

TABLE I Crystal data and structure refinement details

Empirical formula	$H_{24}ClCoN_6O_7Se$
Formula weight	393.59
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	*Pnma*
Unit cell dimensions	$a = 18.230(2)$ Å $b = 8.591(2)$ Å $c = 8.593(2)$ Å
Volume	1345.9(4) Å^3
Z and $F(000)$	4 and 800
Density (calculated)	1.942 Mg m^{-3}
Absorption coefficient	4.208 mm^{-1}
Crystal size	$0.33 \times 0.18 \times 0.16$ mm
Θ range for data collection	2.23–25.08°
Scan type	$2\Theta - \Theta$
Scan width	$1.260 + 0.350 \tan\Theta$
Scan time/background time	2 : 3
Index ranges	$0 \le h \le 21$, $0 \le k \le 10$, $0 \le l \le 10$
Reflections collected	1277
Independent reflections	1277 ($R_{int} = 0.0000$)
Absorption correction	Semi-empirical from psi-scans
Max. and min. transmission	1.00 and 0.75
Refinement method	Full-matrix least-square on F^2
Data/restraints/parameters	1276/0/98
Goodness-of-fit on F^2, (S)	1.124
Final R indices $[I > 2\sigma(I)]$	$R1 = 0.0400$, $wR2 = 0.0980$
R indices (all data)	$R1 = 0.0702$, $wR2 = 0.1339$
Extinction coefficient	0.022(2)
Largest diff. peak and hole	0.739 and -1.080 e Å^{-3}

$R1 = (\sum \|\|F_o\| - \|F_c\|\|/\sum \|F_o\|)$, $wR2 = \sum w(F_o^2 - F_c^2)^2/\sum w[(F_o^2)^2]^{1/2}$, $s = [\sum w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$.

$\sum w(\|F_o\| - \|F_c\|)^2$. Hydrogen atoms were included in the ideal positions with fixed isotropic U values of 0.08 Å^2. A weighing scheme of the form $w = 1/[\sigma^2(F_o)^2 + (aP)^2 + bP]$ with $a = 0.05$, $b = 9.00$ was used; P is defined as $[\max(F_o^2, 0) + 2F_c^2]/3$. An extinction correction was also applied to the data. The final difference map was devoid of significant features. All calculations were performed on Silicon Graphics Personal Iris UD/35 and an IBM compatible PC using programs TEXSAN (data reduction) [27], SHELXL-93 (refinement) and SHELXL-PC (plots) [28].

RESULTS AND DISCUSSIONS

Hexaamminecobalt(III) chloride and potassium selenate could react in a number of ways depending upon the stoichiometric ratio of the reactants given in the following equations.

1. $[Co(NH_3)_6]Cl_3 + K_2SeO_4 \xrightarrow{H_2O} [Co(NH_3)_6]ClSeO_4 + 2KCl$
2. $[Co(NH_3)_6]Cl_3 + 2K_2SeO_4 \xrightarrow{H_2O} K[Co(NH_3)_6](SeO_4)_2 + 3KCl$
3. $2[Co(NH_3)_6]Cl_3 + 3K_2SeO_4 \xrightarrow{H_2O} [Co(NH_3)_6]_2(SeO_4)_3 + 6KCl$

Reddish-orange coloured crystals obtained by mixing 1 : 1 stoichiometric quantities of hexaamminecobalt(III) chloride and potassium selenate gave satisfactory elemental analyses corresponding to the formula $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$. This clearly indicated that only two of the three chloride ions present in $[Co(NH_3)_6]Cl_3$ were replaced by SeO_4^{2-}.

Thermogravimetric analysis (rate of heating = 10°C per min) showed the loss of three water molecules at 80–130°C. The anhydrous $[Co(NH_3)_6]ClSeO_4$ salt formed is stable up to 240°C. Therefore, this study not only confirmed the composition but also provided useful information about the wide range of temperature at which the anhydrous salt is stable, thereby providing a convenient and simple method for its synthesis. A second step corresponds to the evolution of coordinated ammonia molecules.

At room temperature, the effective magnetic moment (μ_{eff}) of the title complex salt was found to be 0.70 BM. This value is consistent with results for many low-spin Co(III) compounds having magnetic moments in the range 0–1 BM [29,30].

Vibrational spectra of metal ammine complexes have been studied extensively, and reviewed by Schmidt and Muller [31,32]. NH_3 stretching of the complexes is at frequencies lower than free NH_3 molecules because of coordination and the effect of the counter ion, i.e. Cl^- and SeO_4^{2-}. This is attributed to weakening of the N–H bond due to the formation of NH···Cl and N–H···O hydrogen bonds. The characterization of $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$ was further substantiated by characteristic IR bands at 3400sh, 3240br, 3150br, 1620m and 1328s cm^{-1} corresponding to υ(O–H), υ_a(N–H), υ_s(N–H), δ_d(N–H) and δ_{as}(N–H), respectively, for water and coordinated ammonia molecules in the $[Co(NH_3)_6]^{3+}$ cation. For ionic SeO_4^{2-} there is a sharp peak at 876 (Se–O) cm^{-1}. All IR bands are in the agreement with values reported in the literature [33].

Electronic spectra were recorded in water. As reported in the literature [34] the two transitions $^1A_{1g} \rightarrow {}^1T_{1g}$ and $^1A_{1g} \rightarrow {}^1T_{2g}$ for hexaamminecobalt(III) complexes are observed at around 470 and 340 nm respectively, producing the familiar orange–yellow colour for a number of classical coordination compounds containing cobalt(III), as is the case in this study.

TABLE II Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$Å^2 \times 10^3$]; U(eq) is defined as $1/3\sum_i\sum_j\sum_{ij} a_i \times a_j \times a_i \cdot a_j$

	x/a	*y/b*	*z/c*	*U*(*eq*)
Se(1)	985(1)	2500	1074(1)	22(1)
O(1)	673(4)	2500	2868(8)	35(2)
O(2)	278(4)	2500	−92(9)	58(3)
O(3)	1490(3)	958(6)	817(6)	41(1)
Cl(1)	1909(1)	2500	6807(3)	34(1)
Co(1)	6661(1)	7500	6821(1)	18(1)
N(1)	6905(4)	7500	9035(9)	27(2)
N(2)	5921(3)	9120(7)	7153(7)	29(1)
N(3)	6399(6)	7500	4590(10)	32(2)
N(4)	7410(3)	9099(6)	6441(6)	25(1)
OW1	149(4)	2500	6769(9)	43(2)
OW2	4121(3)	375(7)	9378(6)	47(1)

TABLE III Selected bond lengths (Å) and bond angles (°) for the complex

Se(1)–O(2)	1.632(7)	Se(1)–O(3)	1.628(5)
Se(1)–O(3)$^{\#1}$	1.628(5)	Se(1)–O(1)	1.643(6)
Co(1)–N(4)	1.965(5)	Co(1)–N(4)$^{\#2}$	1.965(5)
Co(1)–N(1)	1.954(8)	Co(1)–N(3)	1.975(9)
Co(1)–N(2)$^{\#2}$	1.960(5)	Co(1)–N(2)	1.960(5)
O(2)–Se(1)–O(3)	111.3(2)	O(2)–Se(1)–O(3)$^{\#1}$	111.3(2)
O(3)–Se(1)–O(3)$^{\#1}$	108.9(4)	O(2)–Se(1)–O(1)	107.7(4)
O(3)–Se(1)–O(1)	108.8(2)	O(3)$^{\#1}$–Se(1)–O(1)	108.8(2)
N(4)–Co(1)–N(4)$^{\#2}$	88.7(3)	N(4)–Co(1)–N(1)	90.2(2)
N(4)$^{\#2}$–Co(1)–N(1)	90.2(2)	N(4)–Co(1)–N(3)	90.4(3)
N(4)$^{\#2}$–Co(1)–N(3)	90.4(3)	N(1)–Co(1)–N(3)	179.2(4)
N(4)–Co(1)–N(2)$^{\#2}$	178.6(2)	N(4)$^{\#2}$–Co(1)–N(2)$^{\#2}$	90.4(2)
N(1)–Co(1)–N(2)$^{\#2}$	90.9(2)	N(3)–Co(1)–N(2)$^{\#2}$	88.6(2)
N(4)–Co(1)–N(2)	90.4(2)	N(4)$^{\#2}$–Co(1)–N(2)	178.6(2)
N(1)–Co(1)–N(2)	90.9(2)	N(3)–Co(1)–N(2)	88.6(3)
N(2)$^{\#2}$–Co(1)–N(2)	90.5(3)		

Symmetry transformation to generate equivalent atoms: $^{\#1}x-1,\ y+1/2,\ z$; $^{\#2}x,\ -y+3/2,\ z$.

The crystal structure of the title complex salts has been determined unambiguously. Final non-H coordinates are listed in Table II and selected bond distances and angles in Table III. The molecular structure of the complex salt is shown in Fig. 1. The structure reveals the existence of discrete $[Co(NH_3)_6]^{3+}$, Cl^- and SeO_4^{2-} ions in addition to three lattice water molecules in the solid state. In the $[Co(NH_3)_6]^{3+}$ ion, the cobalt(III) metal ion is surrounded by six nitrogen atoms originating from six coordinating ammonia molecules resulting in near octahedral geometry. The N–H bond distances are in the range 1.954(8)–1.975(9) Å, while cis N–Co–N bond angles are in the range 88.6(3)–90.9(2)° and trans N–Co–N bond angles are in the range 178.6(2)–179.2(4)°. In $[Co(NH_3)_6]Cl_3$, the average Co–N distance is 1.963 Å and the maximum deviation of cis N–Co–N bond angles from ideal values [35] is 2.1° with a standard deviation of 0.7°.

In the slightly distorted selenate ion, Se–O distances are nearly equal [1.632(7), 1.625(5), 1.628(5), 1.643(6) Å]. The tetrahedron is slightly elongated along the Se–O1 bond due to hydrogen bonding interactions. The interatomic distances and interbond angles (O–Se–O) for the SeO_4 group in anhydrous selenic acid [36] are 1.57, 1.57,

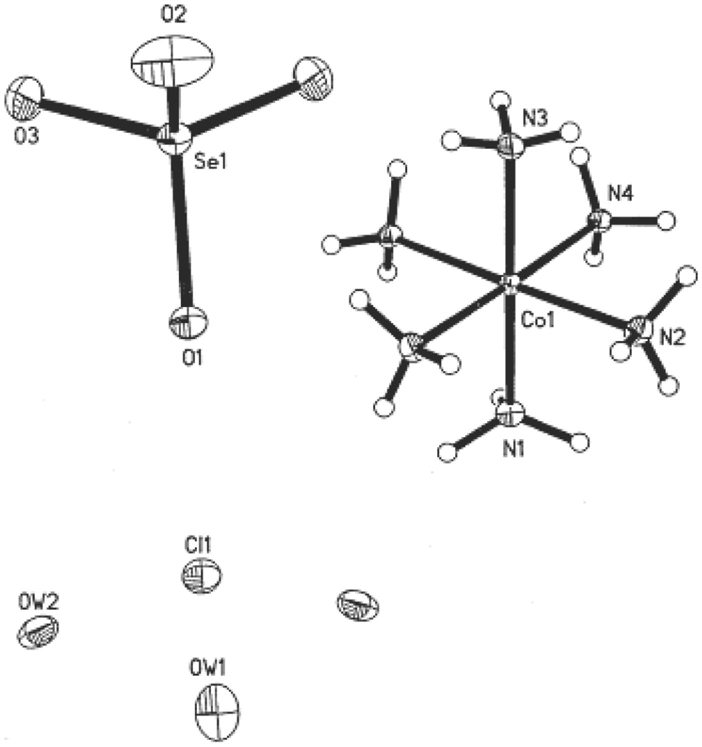

FIGURE 1 ORTEP diagram of $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$ with the asymmetric unit labelled.

1.64 and 1.66 Å (average 1.61 Å) and 116, 109, 112, 112, 106 and 106°, respectively, while in the orthorhombic form of selenious acid (H_2SeO_3) [37] Se–O distances are 1.72, 1.75 and 1.76 Å and O–Se–O bond angles 97, 112 and 93° (average 100°).

The $[Co(NH_3)_6]^{3+}$, Cl^- and SeO_4^{2-} ions are held together by electrostatic forces and hydrogen bonding of the type O–H···O, N–H···Cl and O–H···Cl, etc., resulting in a three-dimensional hydrogen-bonded network through second sphere coordination. Thus the formation of a complex salt of definite composition $[Co(NH_3)_6]ClSeO_4 \cdot 3H_2O$, when hexaamminecobalt(III) chloride and potassium selenate are reacted in a 1 : 1 molar ratio in water, indicates that $[Co(NH_3)_6]^{3+}$ may find application as a receptor for the highly toxic selenate ion.

Acknowledgments

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Supplementary Data

Full lists of crystallographic data are available from the authors upon request. Crystallographic data for the structural analysis of the title compound has also been deposited at the FIZ, 76344 Eggenstein-Leopoldshafen (Germany), having CSD number 413895 (tel.: (49) 7247-808-205; fax: (49) 7247-808-666; 3-mail: crysdata@fiz-karlsruhe.de).

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