

Rotation of the iron triangle in  $\text{Fe}_3(\text{CO})_{12}$  confirms, in part, the ideas originally proposed by Johnson<sup>8</sup> to explain the fluxional behavior of this molecule.

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**Registry No.**  $\text{Fe}_3(\text{CO})_{12}$ , 17685-52-8.

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### Linkage Isomers of the Pentaammine(selenocyanato)ruthenium(III) Cation: Synthesis and Characterization

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In continuation of our earlier investigations on the linkage isomerism of the thiocyanate ligand,<sup>1-5</sup> the possibility of selenocyanate ion exhibiting linkage isomerism in similar systems was speculated. The simple and classical substitution reactions of  $[(\text{NH}_3)_5\text{RuX}]X_2$  ( $X = \text{Cl}, \text{Br}, \text{I}$ ) with thiocyanate ion simultaneously generate two linkage isomers, viz.,  $[(\text{NH}_3)_5\text{RuNCS}]^{2+}$  and  $[(\text{NH}_3)_5\text{RuSCN}]^{2+}$  ions, with the yield of S-isomer approximately one-third of that of N-isomer.<sup>1b,2b</sup> Furthermore, the literature survey reveals practically no study on the ruthenium complexes having selenocyanate ion as coligand.<sup>6</sup>

During the course of our studies on the linkage isomerism of selenocyanate we encountered some problems in the published procedure for the synthesis.<sup>7</sup> We have, therefore, made a thorough reexamination of the syntheses and purification of the complexes, the results of which are reported herein, with a method to separate the Se- and N-bonded isomers of the selenocyanate ligand.

### Experimental Section

**Materials.** All chemicals used were of chemically pure or of AnalaR grade. Doubly distilled water was used throughout.  $[(\text{NH}_3)_5\text{RuX}]X_2$  ( $X = \text{Cl}, \text{Br}$ ) were prepared by the published procedures.<sup>8</sup> Potassium selenocyanate was obtained from Fluke AG and was used without further purification. Sephadex G-10 (40-120 mesh), Sephadex LH-20 (25-100 mesh), Dowex 1-X8 (100-200 mesh) in  $\text{Cl}^-$  form, and Dowex 50W-X8 (200-400 mesh) in  $\text{Na}^+$  form were used for chromatographic studies.

**Preparations.** (a)  $[(\text{NH}_3)_5\text{RuSeCN}]I_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}]I_2 \cdot 2\text{H}_2\text{O}$ . A typical reaction carried out is as follows. An aqueous solution (8-10 mL) of approximately 10 times the stoichiometric excess of potassium selenocyanate was added to an aqueous solution (15-20 mL) of  $[(\text{NH}_3)_5\text{RuX}]X_2$  ( $X = \text{Cl}$  or  $\text{Br}$ ) (0.2 g) maintained at 50-60 °C. The solution was stirred vigorously until the reaction mixture turned bright blue (~10 min; stirring for longer periods deposited selenium powder). The resulting solution was cooled to room temperature and was filtered from the selenium powder into a saturated solution of potassium iodide.

The solution, when cooled at 0 °C, yielded a greenish blue precipitate, which was filtered and washed several times with ethanol and with ether.

The greenish blue compound was dissolved in a minimum quantity of water and was sorbed onto a column of cation exchanger in  $\text{Na}^+$  form (20 x 2 cm). It was eluted with potassium iodide solution of different molarities. The elution with 1 and 4 M potassium iodide solution gave minor amounts of monovalent and tetravalent species, which are being studied presently.<sup>5</sup> The eluate from 2 M potassium iodide solution elution was collected and concentrated at room temperature, which yielded a violet-green compound, after the addition of saturated potassium iodide solution (yield 70-80%). From the IR and electronic spectra, it was found to be a mixture of both N- and Se-bonded isomers. This was further corroborated later, by recording the IR and electronic spectra of the mixture containing authentic samples of both isomers.

The violet-green compound (mixture of Se- and N-isomers) was dissolved in a minimum amount of water and was sorbed into a Sephadex G-10 column (50 x 2 cm). It was eluted with a  $10^{-5}$  M solution of potassium iodide. With a very slow elution rate (3-4 mL/h), clear separation of two bands (band I, pink; band II, blue) was observed. The bands were eluted separately, and the eluates were concentrated at room temperature; a saturated solution of potassium iodide was added separately to the concentrates of pink and blue solutions to precipitate  $[(\text{NH}_3)_5\text{RuSeCN}]I_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}]I_2 \cdot 2\text{H}_2\text{O}$ , respectively (yields 8-12% and 45-50% based on  $[(\text{NH}_3)_5\text{RuX}]X_2$ ).

(b)  $[(\text{NH}_3)_5\text{RuSeCN}](\text{ClO}_4)_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}](\text{ClO}_4)_2 \cdot 2\text{H}_2\text{O}$ . The procedure used to prepare these complexes was the same as that described in (a) except that sodium perchlorate was used in place of potassium iodide. Purple  $[(\text{NH}_3)_5\text{RuSeCN}](\text{ClO}_4)_2 \cdot 2\text{H}_2\text{O}$  and Prussian blue colored  $[(\text{NH}_3)_5\text{RuNCSe}](\text{ClO}_4)_2 \cdot 2\text{H}_2\text{O}$  were precipitated from the concentrates of first and second band eluates, respectively.

(c)  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ . As the direct precipitation of chloride salts was not possible, anion-exchange resin in  $\text{Cl}^-$  form was used. The iodide salts of the complexes were sorbed into the anion-exchange column (50 x 1 cm), and the compounds were eluted with water. The eluates were evaporated partially at room temperature. The chloride salts, viz.,  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ , were precipitated and were centrifuged and washed with ethanol and ether.

(d)  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Br}_2 \cdot 2\text{H}_2\text{O}$  and  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Br}_2 \cdot 2\text{H}_2\text{O}$ . The procedure for the syntheses of these complexes was the same as described in (c) except that the anion-exchange resin used was in bromide form instead of chloride form. After concentration of the eluates at room temperature, bluish purple  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Br}_2 \cdot 2\text{H}_2\text{O}$  and blue  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Br}_2 \cdot 2\text{H}_2\text{O}$  were precipitated.

(e)  $[(\text{NH}_3)_5\text{RuNCSe}](\text{BPh}_4)_2$  and  $[(\text{NH}_3)_5\text{RuSeCN}](\text{BPh}_4)_2$ . A concentrated solution of the bromide or chloride salt of the N- or Se-isomer was added to a solution of  $\text{NaBPh}_4$  in acetone. The solution was left at 0 °C for several hours, and subsequently the cooled solution was concentrated at reduced pressure, whereupon green or dark green water-insoluble  $[(\text{NH}_3)_5\text{RuNCSe}](\text{BPh}_4)_2$  or  $[(\text{NH}_3)_5\text{RuSeCN}](\text{BPh}_4)_2$  precipitated. The product was centrifuged and washed with a little ethanol and ether. Both isomers are soluble in ethanol and acetone.

(f)  $[(\text{NH}_3)_5\text{RuNCSe}](\text{C}_7\text{H}_7\text{SO}_3)_2$  and  $[(\text{NH}_3)_5\text{RuSeCN}](\text{C}_7\text{H}_7\text{SO}_3)_2$ . The procedure used to synthesize the complexes was the same as that given in (e) except that a saturated solution of sodium *p*-toluenesulfonate in ethanol was used instead of sodium tetraphenylborate in acetone. The precipitated greenish blue compound  $[(\text{NH}_3)_5\text{RuNCSe}](\text{C}_7\text{H}_7\text{SO}_3)_2$  or green  $[(\text{NH}_3)_5\text{RuSeCN}](\text{C}_7\text{H}_7\text{SO}_3)_2$  was centrifuged and washed with ethanol and ether.

All the experiments were carried out under acid-free atmosphere, as even traces of acid decomposed selenocyanate, depositing selenium powder.

In all of the above procedures, the compounds, either as solids or in solution, were preserved at 0 °C, as at room temperature they have a tendency to decompose to an insoluble polymeric form along with the deposition of some selenium powder. The purity of the compounds was checked at intervals by passing the solutions through Sephadex G-10. In the case of the  $\text{BPh}_4^-$  salt, Sephadex LH-20 was used instead of Sephadex G-10.

**Physical Measurements.** Analyses for carbon, hydrogen, and nitrogen were done by the Microanalytical Section, Indian Institute of Technology, Kanpur. Selenium was estimated by oxidizing to selenate, followed by reducing it to selenite by boiling with concentrated HCl and finally to selenium powder by hydroxylammonium chloride.<sup>9,10</sup> Analyses for sulfur and halogen were carried out by known procedures. IR spectra were

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Table I. Microanalytical Data<sup>a</sup>

no.	compd	%				
		C	H	N	halogen	Se
1	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe]I <sub>2</sub> ·2H <sub>2</sub> O	1.87 (2.06)	2.84 (3.27)	13.80 (14.45)	43.74 (43.72)	13.41 (13.60)
2	[(NH <sub>3</sub> ) <sub>5</sub> RuSeCN]I <sub>2</sub> ·2H <sub>2</sub> O	1.93 (2.06)	2.95 (3.27)	13.95 (14.45)	43.44 (43.72)	13.55 (13.60)
3	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe](ClO <sub>4</sub> ) <sub>2</sub> ·2H <sub>2</sub> O	2.59 (2.30)	4.19 (3.62)	15.85 (16.00)		14.86 (15.11)
4	[(NH <sub>3</sub> ) <sub>5</sub> RuSeCN](ClO <sub>4</sub> ) <sub>2</sub> ·2H <sub>2</sub> O	2.26 (2.30)	4.10 (4.12)	16.10 (16.00)		14.75 (15.11)
5	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe]Cl <sub>2</sub> ·2H <sub>2</sub> O	2.90 (3.02)	4.40 (4.77)	20.95 (21.11)	17.90 (17.84)	20.05 (19.85)
6	[(NH <sub>3</sub> ) <sub>5</sub> RuSeCN]Cl <sub>2</sub> ·2H <sub>2</sub> O	2.74 (3.02)	4.50 (4.77)	20.83 (21.11)	18.00 (17.84)	19.63 (19.85)
7	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe]Br <sub>2</sub> ·2H <sub>2</sub> O	2.70 (2.46)	2.13 (3.90)	17.44 (17.25)	32.80 (32.85)	15.90 (16.22)
8	[(NH <sub>3</sub> ) <sub>5</sub> RuSeCN]Br <sub>2</sub> ·2H <sub>2</sub> O	2.65 (2.46)	2.54 (3.90)	17.15 (17.25)	32.95 (32.85)	15.85 (16.22)
9	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe](BPh <sub>4</sub> ) <sub>2</sub>	63.10 (63.30)	5.00 (5.90)	9.88 (9.00)		
10	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe](C <sub>7</sub> H <sub>7</sub> SO <sub>3</sub> ) <sub>2</sub>	28.03 (28.44)	4.00 (4.58)	13.10 (13.28)		

<sup>a</sup>For no. 10, sulfur estimation gave 9.60%, while theoretically it is 10.10%. Values in parentheses are the theoretically calculated ones.

Table II. Spectral Data

no.	ion	infrared spectral data, <sup>a</sup> cm <sup>-1</sup>			electronic spectral data, nm (M <sup>-1</sup> cm <sup>-1</sup> )
		$\nu(\text{CN})$	$\nu(\text{CX})$ (X = S or Se)	$\delta(\text{NCX})$ (X = S or Se)	
1	[(NH <sub>3</sub> ) <sub>5</sub> RuSeCN] <sup>2+</sup>	2100 w	535-540	375-3800	827 (154), 555 (2200), 570 sh, 395 w, 327 (3900), 255 (12 575), 223 s
2	[(NH <sub>3</sub> ) <sub>5</sub> RuNCSe] <sup>2+</sup>	2060 s 2035 sh	640		535 (1450), 382 (121), 322 (3086), 269 (9010), 215 s
3	[(NH <sub>3</sub> ) <sub>5</sub> RuSCN] <sup>2+</sup> <sup>b</sup>	2073	790	420	515 (2450)
4	[(NH <sub>3</sub> ) <sub>5</sub> RuNCS] <sup>2+</sup> <sup>b</sup>	2057	783	460	495 (3000)

<sup>a</sup>Vibrational bands for coordinated NH<sub>3</sub> and different anions are not given. <sup>b</sup>Values taken from ref 2.

obtained with a Perkin-Elmer Model 580 IR spectrophotometer. Samples were prepared as KBr pellets. Vibrational spectra in the region 600-200 cm<sup>-1</sup> were obtained with CsI pellets. Electronic spectra were obtained by using a Cary Model 17D spectrophotometer or Shimadzu Model UV-190 double-beam spectrophotometer in 1-cm matched quartz cells. Magnetic measurements were done on a parallel-field vibrating-sample magnetometer, Varian Model 150A, with [Ni(en)<sub>3</sub>]S<sub>2</sub>O<sub>3</sub> as the standard.

### Results and Discussion

The microanalytical data are presented in Table I. All the compounds showed magnetic moments around 2.0  $\mu_B$ , indicating the presence of one unpaired electron, consistent with the presence of ruthenium ion in the +3 oxidation state.

In general, the reactions between [(NH<sub>3</sub>)<sub>5</sub>MX]X<sub>2</sub> or [(NH<sub>3</sub>)<sub>5</sub>M(H<sub>2</sub>O)]X<sub>3</sub> (M = Co, Ru; X = Cl, Br, I) and SCN<sup>-</sup> yielded more of the N-isomer.<sup>11,12</sup> The ratio of the N-bonded/Se-bonded compounds (ca. 5.5) in the reaction between [(NH<sub>3</sub>)<sub>5</sub>RuX]X<sub>2</sub> (X = Cl, Br) and SeCN<sup>-</sup> is also higher than the ratio of N-bonded/S-bonded compounds (ca. 2.5)<sup>1b,2b,5</sup> in the reaction between [(NH<sub>3</sub>)<sub>5</sub>RuX]X<sub>2</sub> (X = Cl, Br, I) and NCS<sup>-</sup> ion. This fact is also supported by the relatively large yield of N-bonded complex compared to those of the S- or Se-bonded complexes, as discussed in the foregoing paragraphs.

All the compounds exhibit very similar vibrational spectra. Apart from the characteristic bands for the coordinated ammonia molecule around 3400, 3200, 1600, 1300, 800, and 450 cm<sup>-1</sup>, the bands due to  $\nu(\text{CN})$ ,  $\nu(\text{CSe})$ , and  $\delta(\text{NCSe})$  modes are also observed. The [(NH<sub>3</sub>)<sub>5</sub>RuSeCN]<sup>2+</sup> ion exhibits the  $\nu(\text{CN})$  frequency of higher energy (ca. 2100 cm<sup>-1</sup>) compared to that of the N-bonded isomer (ca. 2060 cm<sup>-1</sup>). The  $\nu(\text{CSe})$  and  $\delta(\text{NCSe})$  frequencies are also in accordance with the expected values.<sup>6</sup> The position of the  $\delta(\text{NCSe})$  mode of vibration in the [(NH<sub>3</sub>)<sub>5</sub>RuNCSe]<sup>2+</sup>

ion could not be identified with certainty as it was masked by several  $\nu(\text{Ru-N})$  modes of Ru-NH<sub>3</sub> and Ru-NCSe groups around 450 cm<sup>-1</sup>.

The intensity of the  $\nu(\text{CN})$  band has been used as a criterion for the mode of coordination of the thiocyanate ligand.<sup>13-15</sup> It has been observed that the integrated intensity for the CN stretching mode of S-bonded thiocyanate complexes is much less than that of N-bonded thiocyanate complexes. This is due to the increased contribution of the N≡C-S<sup>-</sup> form of all the resonating structures of the NCS<sup>-</sup> ion, when coordinated through the sulfur end [N≡C-S<sup>-</sup> (76%); <sup>-</sup>N=C=S (5%); <sup>2-</sup>N-C≡S<sup>+</sup> (19%)]. This results in a decrease of the dipole moment of the ion. Conversely, coordination through the nitrogen end of the NCS<sup>-</sup> ion favors an increase in the contributions of the other two forms, resulting in an increase in the dipole moment of the ion. Assuming that a change in the magnitude of the dipole moment causes a corresponding change in the rate of change of the dipole moment during vibration, the difference in the intensity of the bands is understandable.<sup>13,16</sup>

The selenocyanato complexes under study also show a similar behavior. The [(NH<sub>3</sub>)<sub>5</sub>RuSeCN]<sup>2+</sup> ion exhibits a much weaker  $\nu(\text{CN})$  band compared to that of the [(NH<sub>3</sub>)<sub>5</sub>RuNCSe]<sup>2+</sup> ion, consistent with the analogous thiocyanate complexes.<sup>5</sup> Further, it was also observed that the intensity of the  $\nu(\text{CN})$  band for the [(NH<sub>3</sub>)<sub>5</sub>RuSeCN]<sup>2+</sup> ion is lower than that of the [(NH<sub>3</sub>)<sub>5</sub>RuSCN]<sup>2+</sup> ion. This could be due to the fact that the probability of the N≡C-X<sup>-</sup> (X = S or Se) form among all the resonating forms of the chalcogenocyanate ions is greater for selenocyanate than for thiocyanate [N≡C-Se<sup>-</sup> (88%); <sup>2-</sup>N-C≡Se<sup>+</sup> (12%); <sup>-</sup>N=C=Se (0%)]. The electronic spectral data

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are given in Table II. For convenience, the electronic spectral data for  $[(\text{NH}_3)_5\text{RuSCN}]^{2+}$  and  $[(\text{NH}_3)_5\text{RuNCS}]^{2+}$  ions are also given therein. The spectra of the complexes show strong intra-ligand bands of the selenocyanato group around 220, 260, and 325 nm.<sup>17-19</sup> The bands having absorption maxima at 255 and 269 nm in Se- and N-bonded complexes, respectively, can be attributed to the  ${}^1\Sigma^+ \rightarrow {}^1\Sigma^-$  transition of the  $\text{SeCN}^-$  ion, which is forbidden in the free ligand but may become allowed when it is bonded to the metal ion.<sup>18</sup> The assignment of the strong highest energy band (band maximum at 215 nm) for the N-bonded isomer offers some difficulties. This may well involve some MLCT and LMCT components.<sup>7,19</sup>

Besides these bands, the  $[(\text{NH}_3)_5\text{RuSeCN}]^{2+}$  ion shows additional bands with absorption maxima at 827, 555, 570 nm, and 395 nm while the other isomer shows bands at 535 and 382 nm. The bands at 555 and 535 nm in Se- and N-bonded complexes, respectively, have been assigned to the charge-transfer transition  $[\pi(\text{NCSe}) \rightarrow \text{T}_{2g}(\text{Ru})]$ ; LMCT because of their high intensity (cf. Table II).<sup>1,4,20</sup> The red shift in the position of the LMCT band of the  $[(\text{NH}_3)_5\text{RuSeCN}]^{2+}$  ion compared to that of the  $[(\text{NH}_3)_5\text{RuSCN}]^{2+}$  ion, i.e., 555 and 515 nm, respectively (cf. Table II), could be due to the increasing ease of oxidation of the  $\text{SeCN}^-$  ion. Also, the shapes of these bands are of interest. The  $[(\text{NH}_3)_5\text{RuSeCN}]^{2+}$  ion exhibits a comparatively broad and unsymmetrical band while the N-isomer shows an almost Gaussian shape. In the case of the  $[(\text{NH}_3)_5\text{RuNCSe}]^{2+}$  ion, the micro-symmetry is closer to  $O_h$  while in the Se-isomer it is  $C_{4v}$ . Hence, the magnitude of removal of degeneracies of various energy levels in the  $[(\text{NH}_3)_5\text{RuNCSe}]^{2+}$  ion is less than that in the  $[(\text{NH}_3)_5\text{RuSeCN}]^{2+}$  ion<sup>7,21</sup> and the band for the Se-isomer might

even show a shoulder around 570 nm.

Though the d-d transitions in Ru(III) complexes are often obscured due to large ligand field parameters, the two spin-forbidden transitions  ${}^4\text{T}_{1g} \leftrightarrow {}^2\text{T}_{2g}$  and  ${}^4\text{T}_{2g} \leftrightarrow {}^2\text{T}_{2g}$  are often observed as shoulders.<sup>22</sup> Because of the low intensities of the bands at 382 and 395 nm for N- and Se-bonded isomers, these bands could tentatively be assigned to ligand field transitions.

Furthermore, we were unable to locate the bands for the  $[(\text{NH}_3)_5\text{RuSeCN}]^{2+}$  ion reported earlier<sup>7</sup> at 586 nm ( $2080 \text{ cm}^{-1} \text{ M}^{-1}$ ) and at 455 nm ( $325 \text{ cm}^{-1} \text{ M}^{-1}$ ). The compound reported is unusual in that its lowest energy feature is often a strong charge-transfer band that is followed by a very weak d-d absorption, the reverse of the more commonly occurring situation.<sup>22</sup> Also, this compound had been prepared from  $[(\text{NH}_3)_5\text{RuH}_2\text{O}]^{2+}$  and  $\text{SeCN}^-$  ions and the possibility of formation of the N-isomer had been ruled out. The claimed Se-isomer could well have been a mixture of both N- and Se-isomers, as, in general, the reaction of  $[(\text{NH}_3)_5\text{MH}_2\text{O}]^{2+}$  with  $\text{NCX}^-$  yields both isomers simultaneously; the separation of the isomers is mostly difficult.<sup>12</sup>

Further efforts to resolve the problem by other physical techniques such as EPR spectroscopy, multinuclear ( $^{13}\text{C}$  and  $^{15}\text{N}$ ) NMR spectroscopy, resonance Raman spectroscopy, cyclic voltametry, and crystal structure studies are in progress.

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**Registry No.**  $[(\text{NH}_3)_5\text{RuSeCN}]_2$ , 104155-25-1;  $[(\text{NH}_3)_5\text{RuNCSe}]_2$ , 104155-26-2;  $[(\text{NH}_3)_5\text{RuSeCN}](\text{ClO}_4)_2$ , 33864-46-9;  $[(\text{NH}_3)_5\text{RuNCSe}](\text{ClO}_4)_2$ , 104155-28-4;  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Cl}_2$ , 104155-29-5;  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Cl}_2$ , 104155-30-8;  $[(\text{NH}_3)_5\text{RuSeCN}]\text{Br}_2$ , 104155-31-9;  $[(\text{NH}_3)_5\text{RuNCSe}]\text{Br}_2$ , 104155-32-0;  $[(\text{NH}_3)_5\text{RuNCSe}](\text{BPh}_4)_2$ , 104155-33-1;  $[(\text{NH}_3)_5\text{RuSeCN}](\text{BPh}_4)_2$ , 104155-34-2;  $[(\text{NH}_3)_5\text{RuNCSe}](\text{C}_7\text{H}_7\text{SO}_3)_2$ , 104172-19-2;  $[(\text{NH}_3)_5\text{RuSeCN}](\text{C}_7\text{H}_7\text{SO}_3)_2$ , 104155-35-3;  $[(\text{NH}_3)_5\text{RuCl}]\text{Cl}_2$ , 18532-87-1;  $[(\text{NH}_3)_5\text{RuBr}]\text{Br}_2$ , 16446-65-4.

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## Additions and Corrections

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**Thomas C. W. Mak, Khalid S. Jasim, and Chung Chieh\***: Structural and Spectroscopic Study of  $\text{Mo}_4(\mu_3\text{-S})_4(\mu\text{-S}_2\text{CN}(\text{C}_2\text{H}_5)_2)_2(\text{S}_2\text{CN}(\text{C}_2\text{H}_5)_2)_4$ , a Compound Containing a Cubane-like Cluster with Six Molybdenum-Molybdenum Bonds.

Page 1590. In the reference list, ref 25 and 26 should be reversed.—  
 Khalid S. Jasim