

The Magnetic Moment of  
 $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$   
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G. RINDORF

The Chemical Laboratory B, Technical University of Denmark, DK-2800 Lyngby, Denmark

H. MOSBÆK and K. G. POULSEN

The Chemical Laboratory A, Technical University of Denmark, DK-2800 Lyngby, Denmark

Mosbæk and Poulsen<sup>1</sup> have determined the Mössbauer parameters and have studied the infrared spectrum of  $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$ . Griffith, Lewis, and Wilkinson<sup>2</sup> have reported, that owing to the presence of a trace of a ferromagnetic

impurity, it has not been possible for them to determine the magnetic susceptibility.

We have measured the magnetic susceptibility of several samples of the compound. We have had no difficulties caused by ferromagnetic impurities, hence our samples are different from the samples prepared by Griffith, Lewis, and Wilkinson with respect to magnetic purity.

Our results are in Table 1.

In order to control the stability of the compound in the sealed-off Gouy-tube filled with  $\text{N}_2$ , the magnetic susceptibility of the samples was measured immediately after preparation and again after different time of storage in the tube as can be seen in the table. The colour of the samples A, B, C, and D was unchanged black during the period, while the colour of sample E turned brown. The tube with sample A was opened after 7 days and that with sample D after 45 days. The IR spectra of both the samples and the Mössbauer spectrum of sample D were identical with the corresponding spectra of freshly prepared compound. Only one sample, E, changed the colour to brown during one month storage and the IR

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Table 1. Magnetic measurements on  $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$ . Susceptibility in c.g.s. units.

Sample	Temp. °K	Age of sample	$\chi_g \times 10^6$				$\chi_g \times 10^6$	$\chi_M^{\text{corr}} \times 10^6$	$\mu_{\text{eff}}$
			Field strength, Ørsted						
			2640	3280	3750	4110			
A	294	2 <sup>h</sup>	44.9	44.5	45.5	45.4	45.1	11 043	5.12
		1 <sup>d</sup>	45.0	44.7	45.7	45.6	45.2	11 077	5.13
		5 <sup>d</sup>	45.1	44.8	45.8	45.6	45.3	11 101	5.13
B	297	1 <sup>d</sup>	48.5	48.7	48.4	49.7	48.8	11 953	5.37
		8 <sup>d</sup>	49.7	49.9	49.4	49.8	49.7	12 159	5.40
		25 <sup>d</sup>	50.8	50.9	50.6	50.6	50.7	12 412	5.45
	195	25 <sup>d</sup>	81.4	81.2			81.3	19 800	5.58
C	295	1 <sup>d</sup>	33.8	34.2	34.0	34.0	34.0	8 369	4.46
	195	1 <sup>d</sup>	52.0	52.2	52.0	52.7	52.2	12 767	4.48
D	297	2 <sup>h</sup>	38.9	38.9	38.8	39.0	38.9	9 550	4.78
		3 <sup>d</sup>	39.3	39.5	39.3	39.5	39.4	9 670	4.81
		12 <sup>d</sup>	39.8	39.8	39.6	39.8	39.7	9 750	4.82
		45 <sup>d</sup>	40.2	40.2	39.7	39.9	40.0	9 820	4.83
	195	11 <sup>d</sup>	61.8	62.1	61.2	61.5	61.6	15 050	4.87
E	297	2 <sup>h</sup>	37.6	37.6	37.4	37.5	37.5	9 220	4.70
	195	3 <sup>h</sup>	58.4	58.4	58.1	58.1	58.3	14.229	4.73

spectrum as well as the Mössbauer spectrum of the brown compound resembled that of  $\text{Fe}_2\text{O}_3\cdot\text{aq}$ . The destruction of this sample is probably due to untight sealing of the tube.

From the magnetic results it is seen, that the compound is reasonably stable in the Gouy-tube, since the rate of decomposition is fairly small, so it is possible to make several measurements on the same sample.

As could be expected when dealing with an unstable compound as  $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$ , the magnetic susceptibility of the freshly prepared samples has not always the same value. In order to get an impression of the effect of a beginning decomposition, we have measured the  $\chi_g$  of the brown destruction product prepared from the black compound by exposure to the air. We found  $\chi_g = 54.7 \times 10^{-8}$ , which is approx. 1.5 times the value we find for  $\chi_g$  for the best samples of the black compound. A contamination with the brown destruction product will therefore increase the measured value for  $\chi_g$  of the black compound.

As a consequence of these considerations we consider the results found for the samples C, D, and E as the most reliable. Due to a rather heavy, thickwalled Gouy-tube used for sample C, these measurements have not the same degree of accuracy as the other measurements. We therefore conclude that Fe in the compound  $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$  has a magnetic moment  $\mu_{\text{eff}}$  not exceeding 4.70 BM, which may be explained as due to three unpaired electrons and a contribution of an orbital moment. Approximately the same magnetic moment is found in the "brown-ring"-complex,  $[\text{Fe}(\text{H}_2\text{O})_5\text{NO}]^{2+}$ , by Griffith, Lewis, and Wilkinson<sup>2</sup> and in  $[\text{Fe}(\text{sal})_2(\text{py})\text{NO}]$  by Nast and Rückemann.<sup>3</sup> It is therefore probable, that the bonding of NO is similar in these compounds and in  $[\text{Fe}(\text{NH}_3)_5\text{NO}]\text{Cl}_2$ , which accordingly may be considered as an iron (I)-complex with NO coordinated as  $\text{NO}^+$ .

*Experimental.* The preparations and the analyses of the compound were all carried out as described earlier.<sup>1</sup> The compound was prepared in an  $\text{N}_2$ -chamber, packed in a Gouy-tube and sealed off without being exposed to the air. The quantitative analyses showed that the contents of Fe,  $\text{NH}_3$ , and Cl within the limits of the experimental error were equal to the theoretical values. (Found, %: Fe 23.2;  $\text{NH}_3$  35.0; Cl 28.4. Calc. Fe 22.8;  $\text{NH}_3$  35.0; Cl 29.0).

The magnetic susceptibility was measured by means of the Gouy method as described by Asmussen and Soling.<sup>4</sup>

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## Organic Selenium Compounds

### VIII. Electronic Spectra of Diselenocarbamate Complexes

K. A. JENSEN and V. KRISHNAN

*Chemical Laboratory II (General and Organic Chemistry), University of Copenhagen, The H. C. Ørsted Institute, DK-2100 Copenhagen, Denmark*

and C. KLIXBÜLL JØRGENSEN

*Laboratoire de Chimie Physique, Université de Genève, Switzerland*

Jensen and Krishnan<sup>1</sup> have described the preparation of several diselenocarbamate complexes. Some of these were independently prepared by Furlani *et al.*<sup>2</sup> who also discussed their electronic spectra. This investigation gives supplementary information on the electronic spectra of these compounds.

Sodium *N,N*-diethyldiselenocarbamate, in ethanol solution, has a broad band with maximum at 290 nm (34.4 kK), but the Zn and Cd complexes (in chloroform) show two well-developed bands in this region (see Table 1). The spectral change in the ultraviolet region on going to complexes with partly filled *d*-shells is presumably due to a change in the electron distribution in the ligand, the structure  $\text{R}_2\overset{+}{\text{N}}=\text{C}(\text{Se}^-)_2$  becoming more important. This is also