Inorganica Chimica Acta, *187* (1991) 67–72

Synthesis and magnetic properties of Cu(II), Ni(II) and Co(II) complexes with nitroenamine — a stable nitroxide imidazolidine radical. Crystal structure of $CoL_2(CH_3OH)_2$

V. I. Ovcharenko*, V. N. Ikorskii, G. V. Romanenko

Institute of Inorganic Chemistry, Siberian Branch of the Academy of Sciences of the USSR, Lavrentyev prospekt 3, Novosibirsk 630090 (U.S.S.R.)

V. A. Reznikov and L. B. Volodarskii

Institute of Organic Chemistry, Siberian Branch of the Academy of Sciences of the USSR, Lavrentyev prospekt 9, Novosibirsk 630090 (U.S.S.R.)

(Received January 3, 1991; revised April 17, 1991)

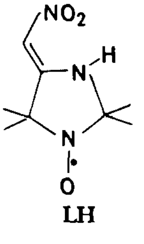
Abstract

The $ML_2(CH_3OH)_2$ complexes have been obtained by the reaction of Cu(II), Ni(II) and Co(II) acetates with 2,2,5,5-tetramethyl-4-nitromethylidene-3-imidazolidine-1-oxyl (LH) in methanol and the CuL_2 complex by the reaction of $Cu(CH_3COO)_2 \cdot H_2O$ with LH in ethanol. Investigation of the magnetic properties of the complexes indicates ferromagnetic intramolecular and antiferromagnetic intermolecular exchange interactions in $ML_2(CH_3OH)_2$. For CuL_2 at $T < 10$ K there is a ferromagnetic ordering. An X-ray structural investigation of $CoL_2(CH_3OH)_2$ reveals that the compound has a layered structure.

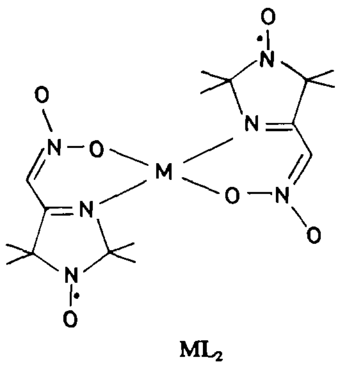
Introduction

At the present time studies are being carried out on the synthesis of transition metal complexes with stable nitroxide radicals which are capable of undergoing magnetic phase transitions to a ferro- or ferrimagnetic state, that may be a basis for creation of new magnetic materials [1–4]. Earlier in the course of our investigations in this direction we have synthesized mixed ligand complexes $ML_2^{CF_3}(R'OH)_2$ (M = Co(II), Ni(II), R' = CH_3, C_2H_5) and inner complex compounds $ML_2^{COOC_2H_5}$ (M = Co(II), Ni(II), Cu(II)) with deprotonated enaminoketone derivatives of the 3-imidazoline nitroxide radical L^R. These complexes exhibited magnetic phase transitions [5–8]. The crystal structures of these complexes show that all of them have layered structures. In a continuation of these investigations CuL_2 and $ML_2(CH_3OH)_2$ complexes, where M = Co(II), Ni(II), Cu(II); L is a deprotonated aci-nitroenamine form of 2,2,5,5-tetramethyl-4-nitromethylidine-3-imidazolidine-1-oxyl (LH), have been synthesized and their solid-state magnetic properties studied. Molecular and crystal structures of $CoL_2(CH_3OH)_2$ have been determined.

LH was chosen as a ligand because its deprotonated aci-form may be treated as an analog of the deprotonated enaminoketones L^R. The LH ability to form complexes has been noted earlier by its reaction with Cu(II) salts [9]. Besides the presence of the NO_2 group in the LH molecule suggested sufficiently strong acceptor properties for the ML_2 molecule central atom. It could create favorable conditions for polymerization of the complexes in the solid state.



LH

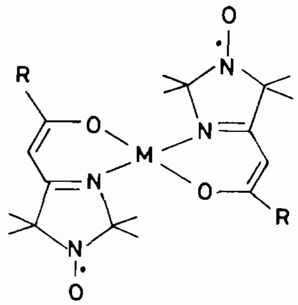


ML_2

---

*Author to whom correspondence should be addressed.

0020-1693/91/$3.50



ML$_2^R$ (R = CF$_3$, COOC$_2$H$_5$)

Experimental

Syntheses

The ligand was prepared by a literature procedure [10].

CuL$_2$

To a solution of LH (3 g, 15 mmol) in ethanol (240 ml), Cu(CH$_3$COO)$_2$·H$_2$O (1.5 g, 7.5 mmol) was added, the reaction mixture stirred and heated to boiling. Solid Cu(CH$_3$COO)$_2$·H$_2$O reacts with the LH solution slowly and it should be ground to a fine powder to accelerate the reaction. After all the Cu(CH$_3$COO)$_2$·H$_2$O dissolved the hot reaction mixture was filtered and allowed to stay for 1 or 2 days at room temperature. Dark red well-shaped crystals separated from the green solution. The CuL$_2$ crystals were filtered off, washed with ethanol and air-dried. Yield 35%. *Anal.* Calc. for $C_{16}H_{26}N_6O_6Cu$:C, 41.4; H, 5.7; N, 18.3; Cu, 13.7. Found: C, 41.6; H, 5.7; N, 18.2; Cu, 13.8%.

CuL$_2$(CH$_3$OH)$_2$

The same procedure in methanol results in crystallization of well-shaped snuff-colored CuL$_2$-(CH$_3$OH)$_2$ crystals. The crystals were filtered off, washed with methanol and air-dried. It is necessary to use 140 ml of methanol per 1 g of LH in the reaction. Yield 60–70%. *Anal.* Calc. for $C_{18}H_{34}N_6O_8Cu$: C, 40.7; H, 6.5; N, 16.0; Cu, 12.0. Found: C, 41.4; H, 6.5; N, 16.0; Cu, 12.1%.

CuL$_2$(CH$_3$OH)$_2$ is rather unstable under normal conditions. After a day microscopic observation of the crystals reveals the presence of brown spots on the crystal corners and edges resulting from gradual transformation of CuL$_2$(CH$_3$OH)$_2$ to CuL$_2$. Within 10–15 days the desolvation process is practically complete. CuL$_2$(CH$_3$OH)$_2$ can be kept for a longer time in a desiccator saturated with methanol vapour or under a layer of the saturated methanol solution of the compound.

CoL$_2$(CH$_3$OH)$_2$, NiL$_2$(CH$_3$OH)$_2$

A solution of LH (0.5 g, 2.5 mmol) and Co(CH$_3$COO)$_2$·4H$_2$O or Ni(CH$_3$COO)$_2$·4H$_2$O (0.3 g, 1.25 mmol) in 20 ml of methanol was boiled for 5 min, filtered and allowed to stay at room temperature for 3–5 days. Methanol evaporated gradually from the reaction mixture producing a red precipitate of CoL$_2$(CH$_3$OH)$_2$ or a green one of NiL$_2$(CH$_3$OH)$_2$. After the solution volume had reduced to about one half, the well-shaped crystals of the complexes were filtered off, washed with cold methanol and air-dried. The yield of CoL$_2$(CH$_3$OH)$_2$ and NiL$_2$(CH$_3$OH)$_2$ was 30–40%. *Anal.* Calc. for $C_{18}H_{34}N_6O_8Co$: C, 41.5; H, 6.6; N, 16.1; Co, 11.3. Found: C, 40.5; H, 6.7; N, 15.8; Co, 11.3%. Calc. for $C_{18}H_{34}N_6O_8Ni$: C, 41.5; H, 6.6; N, 16.1; Ni, 11.3. Found: C, 40.8; H, 6.6; N, 15.7; Ni, 11.4%. The mixed ligand complexes CoL$_2$(CH$_3$OH)$_2$ and NiL$_2$(CH$_3$OH)$_2$ are considerably more stable than CuL$_2$(CH$_3$OH)$_2$. They did not reveal any changes in composition and properties after having been kept for weeks under normal conditions.

Physical measurements

IR spectra of the complexes in the range 400–4000 cm^{-1} were recorded on a SPECORD-75-IR spectrophotometer as KBr pellets. The TG and DTG curves were obtained on a PAULIK, PAULIK, ERDEY derivatograph in air on a sample of 50 mg, heating rate 2.5 °/min. The powder diffraction patterns of the compounds were obtained on a DRON-2.0 diffractometer (Cu Kα radiation, Ni-filter). Magnetic measurements were performed on polycrystalline samples by the Faraday method in the temperature range 4.2–300 K in magnetic fields up to 10.9 kOe. In the studies of magnetic properties the samples of the complexes were sealed into quartz ampoules. Molar magnetic susceptibility calculations were performed taking into account atomic diamagnetism and temperature-independent paramagnetism of metal ions.

X-ray structural analysis

Dark red crystals of CoL$_2$(CH$_3$OH)$_3$ are monoclinic: $a = 10.883(2)$, $b = 11.987(1)$, $c = 9.447(1)$ Å, $\beta = 101.57(1)°$, $V = 1207.5(3)$ Å^3, space group $P2_1/c$, $Z = 2$, $D_c = 1.43$ g/cm^3, $D_m = 1.43 \pm 0.03$ g/cm^3 (chloroform/hexane), μ(Cu Kα) = 58.3 cm^{-1}. The experimental data were collected on a SYNTEX $P2_1$ four-circle automated diffractometer by standard procedures (Cu Kα radiation, graphite monochromator, θ–2θ scan technique with a variable velocity, $V_{min} = 5$ °/min, $2\theta_{max} = 115°$, 1841 I_{hkl} observed from 1895 measured). Corrections for absorption were made experimentally using a transmission curve. The structure was solved by direct methods using SHELX86

TABLE 1. Final atomic coordinates ($\times 10^4$, $\times 10^3$ for H) and equivalent isotropic temperature factors (Å^2) ($\times 10^2$) for $CoL_2(CH_3OH)_2$ complex

Atom	x	y	z	U
Co	5000	0000	0000	1.9(1)
O011	5282(5)	1578(5)	−743(7)	2.9(3)
N01	6092(7)	2318(6)	−268(8)	3.0(4)
O012	5926(6)	3302(5)	−761(8)	4.4(4)
C1	7170(8)	2093(7)	683(10)	2.3(5)
C2	7506(7)	1013(7)	1229(9)	1.8(5)
N1	6851(6)	97(6)	1135(7)	2.0(4)
C3	8874(7)	898(7)	2113(9)	1.9(4)
C31	9084(11)	1468(11)	3559(12)	3.5(6)
C32	9845(9)	1257(9)	1249(11)	3.3(6)
N11	8877(7)	−318(6)	2311(9)	2.5(4)
O11	9849(6)	−853(6)	2894(9)	5.0(5)
C4	7598(7)	−816(7)	1910(10)	2.1(5)
C41	7655(10)	−1807(8)	928(14)	3.3(6)
C42	7131(10)	−1090(9)	3238(11)	3.3(6)
Os	4440(6)	687(6)	1887(7)	3.4(4)
Cs	3594(12)	165(14)	2648(14)	5.9(8)
HC1	766(6)	273(5)	94(7)	1(3)
H311	867(11)	122(9)	419(12)	5(4)
H312	1014(16)	137(12)	430(16)	7(6)
H313	925(15)	220(13)	355(16)	12(7)
H321	1001(8)	209(7)	118(8)	7(3)
H322	965(7)	94(6)	12(8)	2(2)
H323	1055(9)	88(8)	165(10)	4(3)
H411	808(11)	−154(9)	4(11)	7(4)
H412	816(11)	−243(9)	149(11)	7(3)
H413	701(11)	−211(8)	82(11)	5(4)
H421	707(8)	−40(7)	373(9)	2(2)
H422	762(9)	−161(8)	374(10)	4(3)
H423	635(11)	−155(9)	306(11)	7(3)
HOs	458(8)	111(7)	265(10)	3(3)
HCs1	431(20)	−45(14)	315(21)	19(9)
HCs2	288(15)	−37(12)	206(17)	12(6)
HCs3	315(11)	60(10)	327(12)	8(4)

$U = 1/3(U_{11} + U_{22} + U_{33})$.

[11] and refined by full-matrix least-squares methods. In the last stage of the refinement all non-hydrogen atoms (in the anisotropic approximation) and all hydrogen atoms were located in the $\Delta\rho(xyz)$ synthesis. The refinement of the hydrogen atoms (in isotropic approximation) together with the non-hydrogen atoms gave a final value of the R factor of 0.072. The positional and thermal parameters for the atoms are listed in Table 1. The calculations of the structure refinement were performed using YANX [12] on an EC-1037 computer.

Results and discussion

Syntheses

The reaction of Cu(II), Ni(II) and Co(II) acetates with LH in methanol results in the formation of $ML_2(CH_3OH)_2$. Co(II) and Ni(II) complexes are considerably more soluble in methanol than the Cu(II) complex and their crystallization occurs from more concentrated solutions as compared with Cu(II). In the synthesis of the Co(II) and Ni(II) complexes cooling of the reaction mixture is undesirable since LH coprecipitates with $CoL_2(CH_3OH)_2$ or $NiL_2(CH_3OH)_2$. The precipitated LH can be easily washed out with methanol but this procedure reduces the yield of the complex. The use of ethanol as a solvent in the synthesis of Co(II) and Ni(II) complexes is also unfavourable since mainly the starting LH precipitates upon slow removal of this solvent from the reaction mixture at room temperature or upon keeping the solution at a low temperature. The reaction of $Cu(CH_3COO)_2 \cdot H_2O$ with LH in ethanol unlike Co(II) and Ni(II) results in CuL_2 crystallization. No LH crystallization along with the complex was observed in the synthesis of either $CuL_2(CH_3OH)_2$ or CuL_2. As mentioned above, $CuL_2(CH_3OH)_2$ is unstable under normal conditions and gradually loses alcohol forming CuL_2. This process can be accelerated by heating $CuL_2(CH_3OH)_2$ in vacuum at 50–60 °C. CuL_2 thus formed is stable upon heating up to 170 °C. At higher temperatures it decomposes with explosion. We failed to obtain CoL_2 or NiL_2 by heating $CoL_2(CH_3OH)_2$ or $NiL_2(CH_3OH)_2$ since methanol removal from the compounds is accompanied by decomposition.

IR spectroscopy

IR spectra of the discussed compounds are of interest mainly because they allow an easy detection of the OH groups of the methanol molecules in the complexes. In the IR spectra of $CuL_2(CH_3OH)_2$, $CoL_2(CH_3OH)_2$ and $NiL_2(CH_3OH)_2$ there are broad bands at 3410, 3300 and 3300 cm^{-1}, respectively, due to ν(OH) of the methanol molecules. In the spectra of $CuL_2(CH_3OD)_2$, $CoL_2(CH_3OD)_2$ and $NiL_2(CH_3OD)_2$ obtained in deuterated methanol bands due to ν(OD) at 2520, 2440 and 2440 cm^{-1}, respectively, are registered instead of the above bands confirming the presence of solvent molecules in the complexes. In the IR spectra of CuL_2 synthesized in C_2H_5OH or C_2H_5OD there are no bands in the range 3200–3600 or 2300–2700 cm^{-1}.

Crystal structure

The projection of the $CoL_2(CH_3OH)_2$ structure onto the (100) plane is shown in Fig. 1. The complex has a layered structure. The centrosymmetric octahedral environment of the Co atom is formed in the equatorial plane by one of the oxygens of the NO_2 group and by the imine nitrogen of the imidazoline heterocycle of the ligand. The axial positions

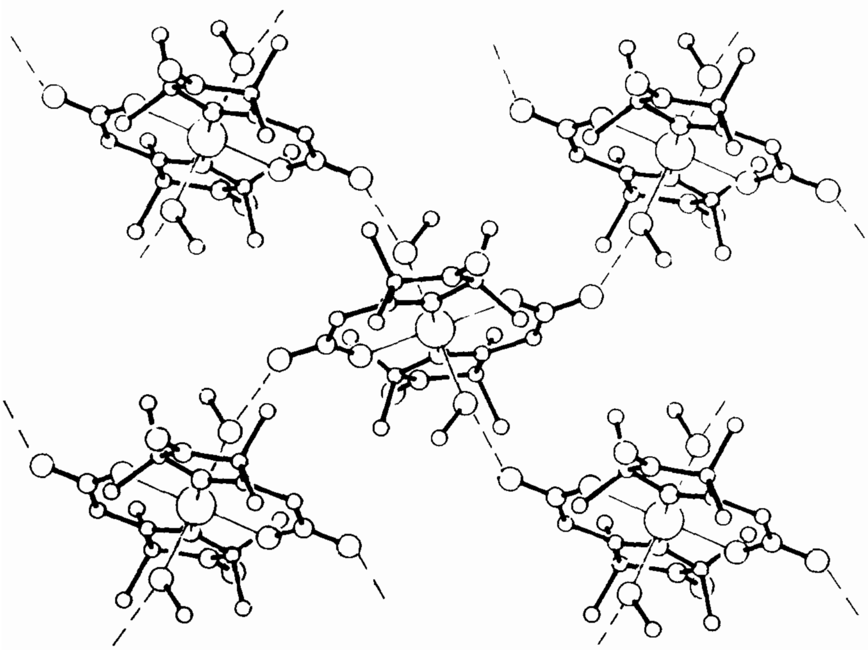


Fig. 1. The projection of $CoL_2(CH_3OH)_2$ onto the (100) plane.

are occupied by the oxygens of the methanol molecules ($Co–O_{NO_2}$ 2.062(6), Co–N 2.087(6), $Co–O_{CH_3OH}$ 2.159(7) Å) (Fig. 2). Thus L forms with the central atom a six-membered chelate ring with an $NCoO_{NO_2}$ angle of 86.0(2)°. The chelate ring with the second oxygen of the NO_2 group as well as the imidazoline heterocycle are practically planar. The angle between their planes is 11.6°. The oxygen of the $N\dot{-}O$ group lies in the plane of the imidazoline heterocycle. Selected intramolecular bond distances and angles of the complex are listed in Table 2. The C–H and O–H bond distances vary within 0.79(11)–1.12(8) Å (the average C–H bond distance is 0.99(10) Å; O–H 0.87(9) Å); the average angle value in the CH_3 groups is 106°. The OH groups of the coordinated alcohols form hydrogen bonds O–H...O with the NO_2 group oxygens of the neighbouring molecules (Fig. 1) with O...O distances of 2.753(9) Å. These hydrogen bonds ensure the existence in the $CoL_2(CH_3OH)_2$ structure of polymeric layers running parallel to the (100) plane.

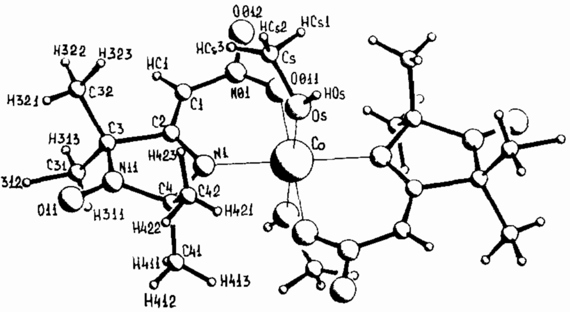


Fig. 2. Perspective view of the $CoL_2(CH_3OH)_2$ molecule.

TABLE 2. Bond distances (Å) and angles (°) in the $CoL_2(CH_3OH)_2$ complex

Distances			
Co–O011	2.062(6)	C3–C31	1.50(1)
Co–N1	2.087(6)	C3–C32	1.52(1)
Co–Os	2.159(7)	C3–N11	1.47(1)
O011–N01	1.267(9)	N11–O11	1.26(1)
O012–N01	1.268(9)	N11–C4	1.49(1)
N01–C1	1.35(1)	C4–C41	1.52(1)
C1–C2	1.41(1)	C4–C42	1.48(1)
C2–C3	1.56(1)	C4–N1	1.49(1)
		N1–C2	1.30(1)
Angles			
N1–Co–O011	86.0(2)	N11–C3–C32	111.4(7)
O011–N01–O012	118.9(6)	C31–C3–C32	111.3(8)
O011–N01–C1	123.0(6)	C3–N11–C4	112.7(6)
N01–C1–C2	123.4(6)	C3–N11–O11	122.6(6)
C1–C2–N1	130.5(7)	C4–N11–O11	124.3(6)
Co–O011–N01	131.4(5)	N1–C4–N11	102.9(6)
Co–N1–C2	122.7(5)	C2–N1–C4	110.7(7)
Co–N1–C4	126.6(5)	N1–C4–C41	111.4(7)
C2–C3–N11	97.7(6)	N1–C4–C42	109.7(7)
C2–C3–C31	113.6(7)	N11–C4–C41	108.6(7)
C2–C3–C32	112.3(6)	N11–C4–C42	109.5(7)
N11–C3–C31	109.8(7)	C41–C4–C42	114.0(8)
C1–C2–C3	115.2(6)	Co–Os–Cs	124.6(8)

The distances between the paramagnetic centres $Co...\dot{-}O$ are 5.530(6) Å within the molecule, the intermolecular $Co...O\dot{-}$ contacts are not less than

6.789(8) Å, and the shortest ∸O...O∸ distance is 4.427(12) Å.

X-ray powder diffraction patterns for $CoL_2(CH_3OH)_2$ and $NiL_2(CH_3OH)_2$ are practically identical, indicating that these complexes are isostructural. The powder diffraction pattern of $CuL_2(CH_3OH)_2$ is also in many respects similar to that for $CoL_2(CH_3OH)_2$ while the diffraction pattern of CuL_2 is essentially different. It should be noted that the described procedure for CuL_2 synthesis allows one to obtain well-shaped crystals of this complex. However all of them were twins. We failed to obtain CuL_2 single crystals suitable for X-ray structural analysis.

The data obtained allow $CoL_2(CH_3OH)_2$ and $NiL_2(CH_3OH)_2$ to be regarded as new representatives of layered polymers based on mixed ligand metal complexes with imidazoline nitroxide derivatives and alcohols. But in contrast to the earlier described layered polymers $ML_2^{CF_3}(R'OH)_2$ (M = Co(II), Ni(II); R' = CH_3, C_2H_5) which are polymerized through hydrogen bond formation between the OH groups of the coordinated alcohols and the oxygen of the N∸O group of the neighbouring molecules [5, 7], in $ML_2(CH_3OH)_2$ the N∸O group oxygens do not participate in hydrogen bond formation. In our opinion this circumstance is the main reason for the substantial difference in magnetic properties of $CoL_2(CH_3OH)_2$ and $NiL_2(CH_3OH)_2$ from $CoL_2^{CF_3}(R'OH)_2$ and $NiL_2^{CF_3}(R'OH)_2$.

Magnetic properties

All $ML_2(CH_3OH)_2$ are paramagnetic. Their $\mu_{eff}(T)$ dependences are shown in Figs. 3 and 4. The theoretical $\mu_{eff}(T)$ curves for $NiL_2(CH_3OH)_2$ and $CuL_2(CH_3OH)_2$ have been obtained due to the (O∸N...M(II)...N∸O) exchange cluster model with the intramolecular exchange interaction energy J and the intermolecular exchange interaction energy $J'z$ [13, 14]. The optimum parameters obtained with this model for the $NiL_2(CH_3OH)_2$ and $CuL_2(CH_3OH)_2$ complexes are, respectively, equal to: $g = 2.19$, $J = 13.2$ cm^{-1}, D (zero field splitting parameter) = -8.9 cm^{-1}, $J'z = -1.2$ cm^{-1} and $g = 1.83$ cm^{-1}, $J = 32.9$ cm^{-1}, $J'z = -1.0$ cm^{-1}. The value of the g factor for the Cu(II) complex indicates an inadequate description of its magnetic properties by the molecular model. Analysis of the structural data suggest, for example, that the exchange interactions within the polymeric layers and between them may be different. However, on the basis of the performed calculations it is possible to point out a property that is common to all complexes which consists in the fact that the intramolecular parameter $J > 0$, i.e. the intramolecular exchange interactions are of ferromagnetic character. For the heterospin system (O∸...Co(II)...∸O) there is no analytical expression for theoretical calculation of the $\mu_{eff}(T)$ dependences. The experimental data (Fig. 3) indicate only the presence in $CoL_2(CH_3OH)_2$, and also in $NiL_2(CH_3OH)_2$ and $CuL_2(CH_3OH)_2$, of exchange clusters with a ferromagnetic character of the exchange interactions. Thus for the solid phases of $ML_2(CH_3OH)_2$, unlike those of $ML_2^{CF_3}(R'OH)_2$, no magnetic ordering has been found down to 4.2 K. Besides, in $ML_2^{CF_3}(R'OH)_2$ there are no exchange clusters with a ferromagnetic character of the exchange interactions. As has already been pointed out, this may be a result of the N∸O group oxygens not taking part in the polymerization of the $ML_2(CH_3OH)_2$ molecules.

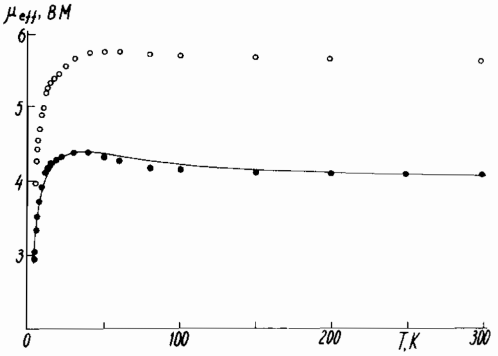


Fig. 3. Experimental μ_{eff} vs. T dependence for $CoL_2(CH_3OH)_2$ (○). Experimental (●) and theoretical (curve) μ_{eff} vs. T dependence for $NiL_2(CH_3OH)_2$.

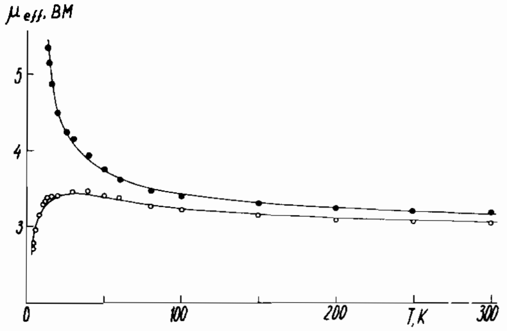


Fig. 4. Experimental and theoretical (curves) μ_{eff} vs. T dependences for CuL_2 (●) and $CuL_2(CH_3OH)_2$ (○).

Magnetic properties of CuL_2 differ from those described above for $ML_2(CH_3OH)_2$. The experimental and theoretical (within the molecular cluster model) $\mu_{eff}(T)$ curves for this complex for $T > 13$ K are shown in Fig. 4. The optimum theoretical curves parameters are $g = 2.0$, $J = 27.4$ cm^{-1}, $J'z = 1.8$ cm^{-1}. It is evident from these data that both J and $J'z$ have positive values, i.e. both the intra- and intermolecular exchange interactions in CuL_2 are of ferromagnetic character. Below 10 K for CuL_2 there is a trend to a ferromagnetic ordering. However, the magnetization curves σ(H) (Fig. 5) have no saturation

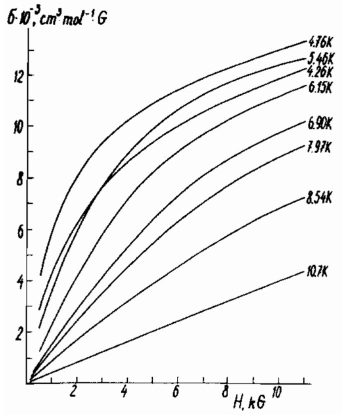


Fig. 5. Field dependences of the magnetization σ for a polycrystalline sample of CuL_2 at different temperatures.

in the studied magnetic field range which does not allow the spontaneous part of the magnetization to be resolved.

Acknowledgements

The authors are thankful to V. I. Lisoivan and S. A. Gromilov for recording the powder diffraction patterns of the complexes.

References

1 A. Caneschi, D. Gatteschi, R. Sessoli and P. Rey, *Acc. Chem. Res., 22* (1989) 392.

2 P. Rey, J. Laugier, A. Caneschi and D. Gatteschi, *Int. Conf. on Nitroxide Radicals, Novosibirsk, U.S.S.R., Sept. 18–23, 1989,* 2C.

3 V. I. Ovcharenko, V. N. Ikorskii, K. E. Vostrikova, A. B. Burdukov, G. V. Romanenko, N. V. Pervukhina and N. V. Podberezskaya, *Int. Conf. on Nitroxide Radicals, Novosibirsk, U.S.S.R., Sept. 18–23, 1989,* 18C.

4 V. I. Ovcharenko, V. N. Ikorskii, K. E. Vostrikova, A. B. Burdukov, G. V. Romanenko and N. V. Podberezskaya, *Izv. Sib. Otd. Akad. Nauk SSSR, Ser. Khim., 5* (1990) 100.

5 V. I. Ovcharenko, K. E. Vostrikova, G. V. Romanenko, V. N. Ikorskii, N. V. Podberezskaya and S. V. Larionov, *Dokl. Akad. Nauk SSSR, 306* (1989) 115.

6 V. I. Ovcharenko, K. E. Vostrikova, V. N. Ikorskii, S. V. Larionov and R. Z. Sagdeev, *Dokl. Akad. Nauk SSSR, 306* (1989) 660.

7 V. I. Ovcharenko, K. E. Vostrikova, V. N. Ikorskii, G. V. Romanenko, N. V. Podberezskaya, S. V. Larionov and R. Z. Sagdeev, *Sintez,* structura i magnitnie svojstva nizkotemperatupnih ferromagnetikov na osnove kompleksov Ni(II) i Co(II) s imidazolinovim nitroksil'nim radikalom, Preprint 89-2, Novosibirsk, 1989, 59P.

8 V. I. Ovcharenko, K. E. Vostrikova, G. V. Romanenko, N. V. Podberezskaya, V. A. Reznikov and L. B. Volodarskii, *Thesisi XYII Vsesoyuznogo Chugaevskogo Sovetcshaniya po khimii kompleksnih soedinenii, chast 3, Maya 29–31, 1990,* Minsk, 1990, p. 415.

9 L. B. Volodarskii, I. A. Grigor'ev, S. A. Dikanov, V. A. Reznikov and G. I. Tcshukin, *Imidazolinovie Nitroksil'nie Radicali,* Nauka, Novosibirsk, 1988, p. 52.

10 V. A. Reznikov and L. B. Volodarskii, *Zh. Org. Khim., 23* (1987) 214.

11 G. M. Sheldrick, SHELX86, in G. M. Sheldrick, C. Kruger and R. Goddard (eds.), *Crystallographic Computing 3,* Oxford University Press, London, 1987, p. 175.

12 R. G. Gerr, A. I. Yanowskii and Yu. T. Struchkov, *Kristallografiya, 28* (1983) 1029.

13 N. V. Pervukhina, V. N. Ikorskii, N. V. Podberezskaya, P. S. Nikitin, A. B. Gel'man, V. I. Ovcharenko, S. V. Larionov and V. V. Bakakin, *Zh. Strukt. Khim., 27* (1986) 61.

14 A. B. Gel'man and V. N. Ikorskii, *Koord. Khim., 14* (1988) 491.