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Investigation tetranuclear complexes of iron (II) by Mössbauer spectroscopy

A series of Fe (II) complexes, $\{[(\text{tpma})\text{Fe}(\mu\text{-CN})_4]\}_4\text{X}_4$ ($\text{X} = \text{ClO}_4^-$, BF_4^- , and PF_6^- ; tpma = tris(2-pyridylmethyl)amine) were prepared by reacting the $\{\text{Fe}(\text{tpma})\}^{2+}$ building block with $(\text{Bu}_4\text{N})\text{CN}$. All three complexes exhibit gradual temperature-driven spin crossover (SCO) associated with the high-spin (HS) \leftrightarrow low-spin (LS) transition at two out of four metal centers. The evolution of HS and LS Fe (II) ions with temperature was followed by a combination of Mössbauer spectroscopy.

Key words: spin-crossover, Fe (II) complexes, Mössbauer spectroscopy.

Introduction

Spin crossover (SCO) is a magnetic property of molecular and extended solids that can be observed for some compounds of d^4 - d^7 transition metal ions. These ions can adopt electron configurations with different numbers of unpaired electrons; hence their compounds can undergo SCO between a low spin (LS) and a high spin (HS) state under external stimuli such as a change in temperature or pressure or photoexcitation. Cooperativity may lead to bistability in SCO materials, which is especially appealing for applications in information storage. The quest for materials useful for such applications explains the extensive research carried out on SCO complexes over the last half a century. These research efforts have been dominated by studies of Fe (II) complexes, which account for ~ 90 % of known SCO compounds [1].

It was only in the early 1960s, about three decades after Cambi's discovery of the first spin state interconversion in iron (III) complexes, that the SCO phenomenon was reported to occur also in iron (II) compounds. The first SCO compounds of iron (II) described in the literature are $[\text{Fe}(\text{phen})_2\text{X}_2]$ (phen = 1,10-phenanthroline; $\text{X} = \text{NCS}$, NCSe), frequently called the «classical» iron (II) SCO complexes. Extensive research activities began thereafter in this area, particularly favored by the discovery of the Mössbauer effect (recoilless nuclear resonance absorption) by Rudolf L. Mössbauer at nearly the same time. This nuclear resonance effect formed the basis for the development of a new spectroscopic technique, Mössbauer spectroscopy, for the detection of hyperfine interactions, which turned out to be extremely powerful for the characterization of iron-containing substances [2, 3].

SCO occurs mainly for six-coordinate iron (II) compounds involving the change of electron configuration $t_{2g}^6 e_g^0$ ($^1A_{1g}$, LS) \leftrightarrow $t_{2g}^4 e_g^2$ ($^5T_{2g}$, HS). Many review articles describing the preparation, structure, chemical, and physical properties of SCO systems of iron (II) have appeared in the literature. Most iron (II) SCO systems possess an $[\text{FeN}_6]$ coordination center [4, 5].

The synthesis of the SCO heteroleptic tetranuclear complexes $\{[\text{LFe}(\text{CN})_2]_2[\text{Fe}(\text{L}')_2]\}_4\text{X}_4$ ($\text{L} \neq \text{L}'$) listed above required the separate preparation of the $[\text{FeL}(\text{CN})_2]$ and $[\text{FeL}']^{2+}$ building blocks. Ligand redistribution side-reactions may occur during the synthesis and limit the yield of the desired tetranuclear complex. This synthetic disadvantage is eliminated in the synthesis of homoleptic complexes ($\text{L} = \text{L}'$), which can be performed directly by reacting $\{\text{FeL}\}^{2+}$ (or $\{\text{FeL}_2\}^{2+}$) fragments with CN^- ligands. The cyanide ion represents an excellent bridging ligand for the assembly of polynuclear transition metal complexes. When coordinated

through the carbon end, the CN^- behaves as a strong-field ligand that usually results in LS configuration of a metal ion. The CN^- coordinated through the nitrogen end behaves as an intermediate strength ligand, and Fe (II) ions with such CN^- ligands might show SCO, provided that appropriate co-ligands are employed, such as N-imine donors. Unfortunately, as mentioned above, such a reaction using $[\text{Fe}(\text{bpy})_2]^{2+}$ led to a complex that did not exhibit SCO. We reasoned that the use of tpma as sole capping ligand could lead to a homoleptically capped SCO complex [6].

Synthesis

All reactions were performed in an inert N_2 atmosphere using standard Schlenk techniques. All reagents were purchased from Aldrich and used as received. Tris(2-pyridylmethyl)amine (tpma) was prepared according to the published procedure [7]. Anhydrous commercial solvents were additionally purified by passing through a double-stage drying/purification system (Glass Contour Inc.).

Warning! Metal perchlorate salts used in this study are potentially explosive and should only be used on a small scale. Appropriate protective measures should always be taken when handling these compounds. Tetrabutylammonium cyanide, $(\text{Bu}_4\text{N})\text{CN}$, is poisonous and should be handled with extreme precautions.

$\{[\text{Fe}(\text{tpma})(\mu\text{-CN})]_4\}(\text{ClO}_4)_4$ (**1a**).

Elem. analysis: calcd. (found) for $\text{Fe}_4\text{Cl}_4\text{O}_{18}\text{N}_{20}\text{C}_{76}\text{H}_{76}$ (**1a**·2 H_2O), %: C, 47.48 (47.44); H, 3.98 (4.13); N, 14.57 (14.28). *IR*, $\nu(\text{C}\equiv\text{N})$, cm^{-1} : 2081, 2072.

$\{[\text{Fe}(\text{tpma})(\mu\text{-CN})]_4\}(\text{BF}_4)_4$ (**1b**).

Elem. analysis: calcd. (found) for $\text{Fe}_4\text{F}_{16}\text{O}_5\text{N}_{20}\text{C}_{76}\text{B}_4\text{H}_{82}$ (**1b**·5 H_2O), %: C, 47.39 (47.45); H, 4.29 (4.13); N, 14.54 (14.50). *IR*, $\nu(\text{C}\equiv\text{N})$, cm^{-1} : 2081, 2072.

$\{[\text{Fe}(\text{tpma})(\mu\text{-CN})]_4\}(\text{PF}_6)_4$ (**1c**).

Elem. analysis: calcd. (found) for $\text{Fe}_4\text{P}_4\text{F}_{24}\text{N}_{20.65}\text{C}_{77.3}\text{H}_{73.95}$ (**1c**·0.65 CH_3CN), %: C, 44.31 (44.11); H, 3.56 (3.74); N, 13.80 (14.01). *IR*, $\nu(\text{C}\equiv\text{N})$, cm^{-1} : 2080, 2073.

Mössbauer spectroscopy. ^{57}Fe Mössbauer spectra (WissEl, CF-506) were collected on a constant acceleration instrument at 80 K, 90 K and 180 K. Samples were prepared by placing crystalline solids in Mössbauer cups closed with Teflon lids. The samples were cooled fast by immersion into liquid N_2 . They were placed in the cryostat at 77 K and then warmed up from 77 K to 80 K or 90 K at a rate of ~ 1 K/min. After obtaining Mössbauer spectra at 80 or 90 K, the samples were warmed up to 180 K at a rate of ~ 3 K/min. We have examined two different batches of **1a** and **1b** and obtained similar spectra at 80 K, 90 K and 180 K. Spectral simulations were generated using WMOSS (WEB Research, Edina, MN). Isomer shifts are reported relative to Fe metal foil at room temperature.

Results and discussion

Synthesis. The use of only one capping ligand, namely tpma, allowed the assembly of the Fe (II) tetranuclear complex, $\{[\text{Fe}(\text{tpma})(\mu\text{-CN})]_4\}\text{X}_4$, by addition of the bridging CN^- ligands to the pre-formed $[\text{Fe}(\text{tpma})]^{2+}$ building block. IR spectra of **1a–1c** revealed two $\text{C}\equiv\text{N}$ stretches at 2170 and 2180 cm^{-1} , which are typical of bridging CN^- ligands in the Fe(II)– $\text{C}\equiv\text{N}$ –Fe(II) unit [6;155]. The complexes have been synthesized in methanol and recrystallized from $\text{CH}_3\text{CN}/\text{Et}_2\text{O}$. The compounds are air-sensitive in solution, but stable when exposed to air for several hours in solid form. Upon exposure to air, they readily exchange any interstitial solvent for water, as shown by the elemental analysis. Thermogravimetric analysis performed under Ar atmosphere revealed that complexes **1a–1c** gradually lose interstitial solvent upon heating to ~ 150 °C (Fig. 1). Complexes **1b** and **1c** decompose above 300 °C, while complex **1a** decomposes abruptly at ~ 180 °C, which is explained by the explosive nature of dry perchlorate salts.

Mössbauer spectroscopy. We have collected ^{57}Fe Mössbauer spectra, for **1a–1c** at 90 K and 180 K, and also at room temperature for **1a** and **1b**. Analysis of the spectra provides insight into which of the four Fe (II) ions present in the molecular tetranuclear complexes undergo SCO.

The 90 K Mössbauer spectrum of **1a** (Fig. 2b, A) shows three quadrupole doublets. Two of these doublets have parameters characteristic of LS Fe (II), namely isomer shift/quadrupole splitting $\delta/\Delta E_Q = 0.23/0.72$ mm s^{-1} and $0.49/0.48$ mm s^{-1} , respectively, and represent ~ 50 % and ~ 35 % of the Fe ions present in the sample, respectively (Table 1). The third doublet has Mössbauer parameters characteristic of HS Fe (II) ($\delta/\Delta E_Q = 1.06/2.50$ mm/s) and represents ~ 15 % of the Fe ions in the sample. The spectrum obtained for **1a** at 180 K (Fig. 2b, B) shows an increase in the amount of HS Fe (II) in the sample and a de-

crease in the amount of the LS Fe (II) that has higher isomer shift (i.e. $\delta = 0.45 \text{ mm s}^{-1}$ at 180 K). The LS Fe (II) ions with lower isomer shift (i.e., $\delta = 0.22 \text{ mm s}^{-1}$ at 180 K) continue to represent 50 % of Fe in the sample (which corresponds to two Fe ions/cluster) at 180 K. We associate the low-isomer shift, LS Fe (II) ions with the Fe sites that coordinate two bridging CN^- ligands through the C atoms. As mentioned above, the CN^- creates a strong ligand field when coordinated through the C end; consequently, these two Fe ions are unlikely to undergo SCO, which is indeed what our Mössbauer studies showed. The LS Fe (II) ions that have higher isomer shift coordinate two CN^- through the N-end and consequently experience a weaker ligand field and undergo SCO. These conclusions about the relationship between the C/N coordination of the Fe sites, their low/high isomer shift, and their in/ability to undergo SCO is in agreement with the results we have obtained in the study of CN^- bridged trigonal bipyramid, pentanuclear clusters [8, 9].

A behavior similar to that observed for **1a**, specifically the manifestation of a gradual and incomplete SCO at the $\{\text{N}_6\}$ -coordinated Fe sites, was also observed for **1b** and **1c** (Table and Fig. 1b and 2b).

The χT values at 90 K and 180 K for **1a** are 2.5 and 3.9 emu K mol^{-1} , respectively. Assuming a χT value of 3.5 emu K mol^{-1} for HS Fe (II), we calculate that there would be about 0.7 HS Fe (II) per cluster at 90 K, which is slightly larger than the value of 0.6 HS Fe (II) per cluster determined by Mössbauer spectroscopy. The amount of HS Fe (II) estimated from the χT value at 180 K corresponds to ~ 1.1 HS Fe (II) ion per cluster, while the Mössbauer data indicate ~ 0.9 HS Fe (II) per cluster at that temperature. Thus, in both cases the amount of the HS Fe (II) increases at higher temperature, but the increase derived from magnetic susceptibility is slightly larger than that established from Mössbauer spectroscopy. A reason for the difference between the estimate for the number of HS Fe (II) ions based on magnetic susceptibility and that based on Mössbauer spectroscopy may be differences in the exact solvent content of the samples and/or in the different thermal histories of the samples examined by magnetic susceptibility and Mössbauer spectroscopy.

The complexes $\{[(\text{L})\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{L})]_2\}\text{X}_4$ **1a–1c** are the first homoleptically-capped square clusters to exhibit temperature-driven SCO. In contrast, the Fe (II) ions in the previously reported, homoleptic complex $\{[(\text{bpy})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{bpy})_2]_2\}(\text{PF}_6)_4$ existed in the LS state up to room temperature. Judged, by magnetic susceptibility, the counter anion X^- in **1a–1c** has a relatively small influence on the character of the SCO in these compounds, which is gradual and occurs between 80 K and room temperature, although the relative amounts of the HS and LS fractions observed at specific temperature are slightly different for various X^- . The loose packing of tetranuclear cations in the crystal structure leads to weak intermolecular interactions, which are only slightly impacted by the change in the nature of X^- . Hence, the SCO occurs gradually due to the low cooperativity. In contrast, the related heteroleptic complex $\{[(\text{bpy})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{tpma})]_2\}(\text{PF}_6)_4$ undergoes a cooperative, two-step SCO. Thus, one could expect that the modification of crystal packing might have a more pronounced impact on intermolecular interactions and thus on the character of the SCO. In this vein, we note the existence of an analogous complex, $\{[(\text{bpy})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{tpma})]_2\}(\text{BF}_4)_4$, the magnetic properties of which have not been reported. It would be interesting to investigate the magnetic behavior of this compound [10].

In principle, one should expect the $\text{LS} \leftrightarrow \text{HS}$ interconversion at two of the four Fe (II) ions — those with the FeN_6 coordination environment — in the $\{[(\text{L})\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{L}')]_2\}\text{X}_4$ complexes. Even in $\{[(\text{bpy})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{tpma})]_2\}(\text{PF}_6)_4$, however, the SCO is rather gradual and not complete at 300 K. More gradual, and also incomplete, transitions were observed in $\{[(\text{phen})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{tpma})]_2\}(\text{PF}_6)_4$ [11] and $\{[(\text{bpy})_2\text{Fe}(\text{CN})_2]_2[\text{Fe}(\text{bpym})]_2\}(\text{PF}_6)_4$ [12]. All these observations suggest that crystal packing of the tetranuclear complexes is rather loose and not conducive of the cooperative SCO behavior that would lead to a more abrupt spin transition.

The loose nature of crystal packing in **1a**, **1b** and **1c** could also be conjectured from significant disorder of the X^- counter anions and interstitial solvent molecules: while the disorder in the latter two structures (with $\text{X}^- = \text{BF}_4^-$ and PF_6^- , respectively) resulted in rather high R -factors (8–10 %), the disorder in **1a** ($\text{X}^- = \text{ClO}_4^-$) was so severe that we could not attain a well-converged refinement ($R \sim 20\%$).

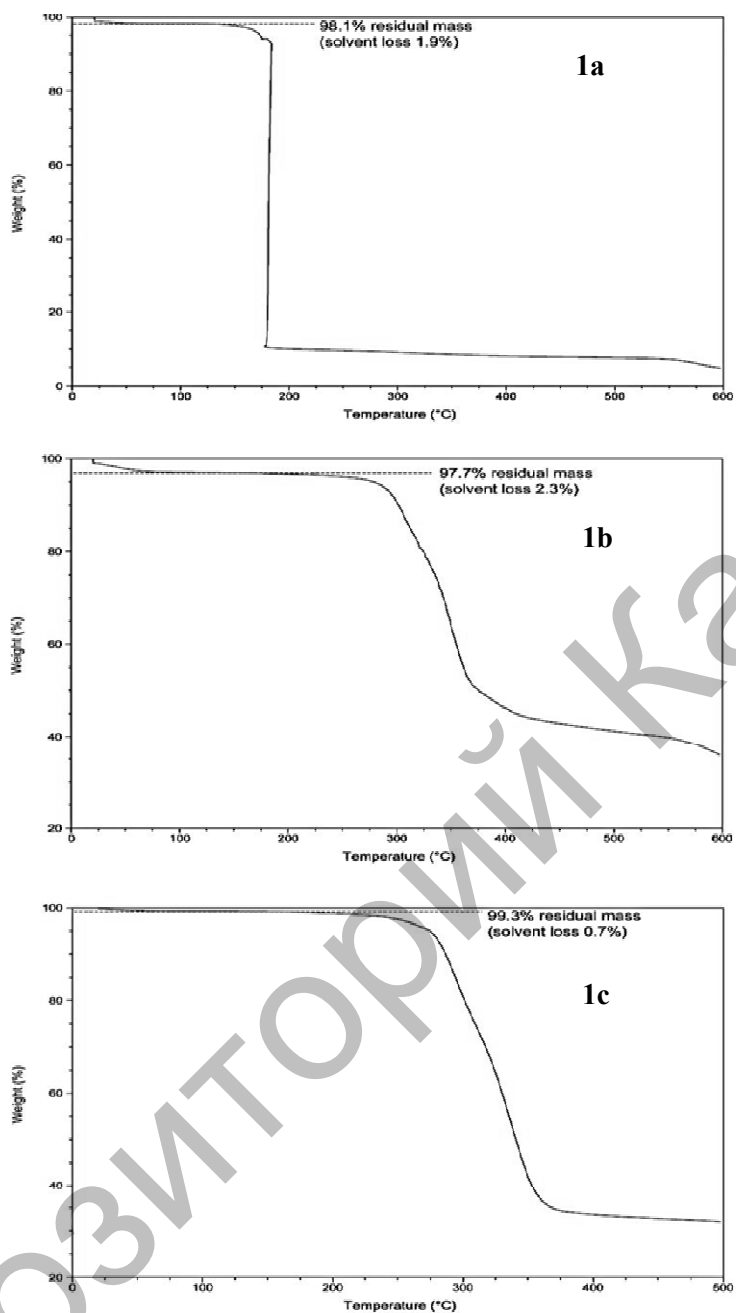


Figure 1. Thermogravimetric curves for **1a–1c** measured under continuous flow of Ar gas

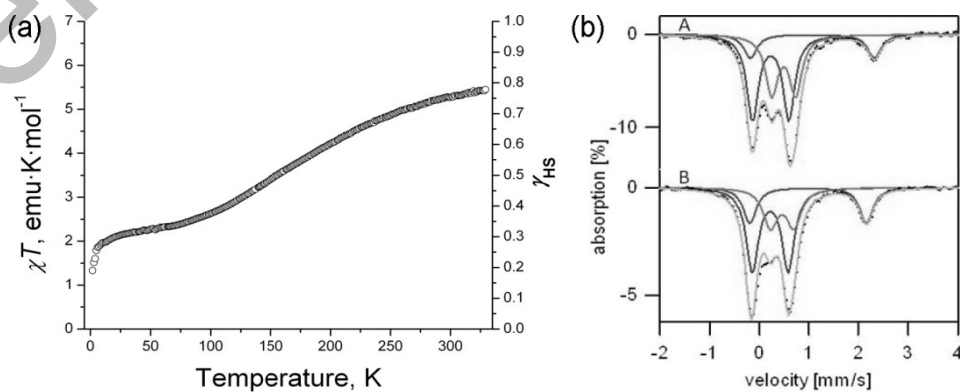


Figure 2. (b) Mössbauer spectra of **1a**, recorded at 90 K (A) and 180 K (B)

Table

Mössbauer parameters for the Fe ions in samples of $\{[\text{Fe}(\text{tpma})(\mu\text{-CN})]_4\}\text{X}_4$ (1a-1c)

Compound	T (K)	δ (mm/s)	ΔE_Q (mm/s)	Fe type	%	$\gamma_{\text{HS}}(\text{FeN}_6 \text{ only})^a$	
						Mössbauer	curve χT
1a X=ClO ₄ ⁻	90	1.06(2)	2.50(1)	BC-Fe(II)N ₄ N ₂	15(1)	0.29(2)	0.36(3)
		0.49(1)	0.48(1)	HC-Fe(II)N ₄ N ₂	36(1)		
		0.23(1)	0.72(1)	HC-Fe(II)N ₄ C ₂	51(1)		
	180	0.98(2)	2.36(1)	BC-Fe(II)N ₄ N ₂	22(2)	0.44(4)	0.56(5)
		0.45(1)	0.47(1)	HC-Fe(II)N ₄ N ₂	28(2)		
		0.22(1)	0.72(1)	HC-Fe(II)N ₄ C ₂	51(1)		
	300	0.90	2.15	BC-Fe(II)N ₄ N ₂	28	0.56	0.75(7)
		0.31	0.45	HC-Fe(II)N ₄ N ₂	22		
		0.20	0.72	HC-Fe(II)N ₄ C ₂	50		
1b X=BF ₄ ⁻	90	1.07(3)	2.47(7)	BC-Fe(II)N ₄ N ₂	8(1)	0.16(3)	0.39(3)
		0.47(1)	0.44(1)	HC-Fe(II)N ₄ N ₂	43(2)		
		0.23(1)	0.71(2)	HC-Fe(II)N ₄ C ₂	51(1)		
	180	0.98(1)	2.35(1)	BC-Fe(II)N ₄ N ₂	23(1)	0.48(3)	0.59(5)
		0.46(1)	0.43(1)	HC-Fe(II)N ₄ N ₂	25(2)		
		0.22(1)	0.71(1)	HC-Fe(II)N ₄ C ₂	51(1)		
	300	0.83	2.15	BC-Fe(II)N ₄ N ₂	35	0.70	0.83(7)
		0.30	0.43	HC-Fe(II)N ₄ N ₂	15		
		0.22	0.71	HC-Fe(II)N ₄ C ₂	50		
1c X=PF ₆ ⁻	90	1.10(3)	2.34(6)	BC-Fe(II)N ₄ N ₂	20(1)	0.41(2)	0.43(4)
		0.48(1)	0.47(1)	HC-Fe(II)N ₄ N ₂	29(1)		
		0.23(1)	0.75(1)	HC-Fe(II)N ₄ C ₂	51(1)		
	180	1.01(6)	2.24(11)	BC-Fe(II)N ₄ N ₂	26(2)	0.50(4)	0.62(6)
		0.44(1)	0.46(1)	HC-Fe(II)N ₄ N ₂	26(2)		
		0.21(1)	0.73(3)	HC-Fe(II)N ₄ C ₂	51(1)		

Note. ^aThe fraction of HS Fe(II) centers was calculated as $\gamma_{\text{HS}} = \frac{[\text{Fe}^{\text{HS}}]}{[\text{Fe}^{\text{HS}}] + [\text{Fe}^{\text{LS}}]}$, where the relative concentrations of the HS and LS fractions, $[\text{Fe}^{\text{HS}}]$ and $[\text{Fe}^{\text{LS}}]$, were taken only over the sites with the FeN₆ coordination, assuming that $\chi T(\text{Fe}^{\text{HS}}) \approx 3.5 \text{ emu K mol}^{-1}$ and $\chi T(\text{Fe}^{\text{LS}}) \approx 0 \text{ emu K mol}^{-1}$.

Conclusions

The $\{[(\text{tpma})\text{Fe}(\mu\text{-CN})]_4\}\text{X}_4$ (X = ClO₄⁻, BF₄⁻, PF₆⁻) complexes are the first example of homoleptic tetranuclear CN — bridged Fe (II) complexes that exhibit SCO. The temperature-driven LS \leftrightarrow HS interconversion is very gradual, which is associated with loose packing of the $\{[(\text{tpma})\text{Fe}(\mu\text{-CN})]_4\}^{4+}$ cations in the crystal structure and consequently low cooperativity of the spin transition. Increasing the ability of the capping ligands to foster intermolecular interactions (e.g., π - π interactions, hydrogen bonding) should promote stronger elastic interactions between the SCO molecules and thus make the spin transition more cooperative/abrupt.

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Н.Б.Қасенова, Р.Ш.Ерқасов, Ғ.Ж.Байсалова

Мёссбауэр спектроскопия әдісімен төртядролы темір (II) комплекстерін зерттеу

Темірдің (II) моноядролы прекурсоры $[\text{Fe}(\text{tpma})]^{2+}$ (tpma = трис-(2-пиридилметил)амин) тетрабутиламмоний цианидмен $(\text{Bu}_4\text{N})\text{CN}$ әрекеттесу нәтижесінде $\{[(\text{tpma})\text{Fe}(\mu\text{-CN})]_4\}\text{X}_4$, бұл жерде $\text{X} = \text{ClO}_4^-$, BF_4^- және PF_6^- , комплекстер қатары синтезделді. Температураны төмендеткен кезде барлық үш комплексті қосылыс бірқалыпты температура-индуцирленген спин-кроссоверге ұшырайды. Спин-кроссовер ауысуы Мёссбауэр спектроскопия әдісімен зерттелді.

Н.Б.Касенова, Р.Ш.Ерқасов, Ғ.Ж.Байсалова

Исследование тетраядерных комплексов железа (II) методом Мёссбауэровской спектроскопии

Синтезирована серия комплексов $\{[(\text{tpma})\text{Fe}(\mu\text{-CN})]_4\}\text{X}_4$, где $\text{X} = \text{ClO}_4^-$, BF_4^- и PF_6^- при взаимодействии моноядерного прекурсора железа (II), $[\text{Fe}(\text{tpma})]^{2+}$ (tpma = трис-(2- пиридилметил)амин) с цианидом тетрабутиламмония $(\text{Bu}_4\text{N})\text{CN}$. При понижении температуры все три комплекса демонстрируют плавный температурно-индуцированный переход из высокоспинового в низкоспиновое состояние, так называемый спин-кроссовер. Переход был изучен комбинацией Мёссбауэровской спектроскопии.

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