Synthesis, structure, thermal decomposition and spectral properties of neutral $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$

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Abstract: The new iron(II) complex $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ (bpta = 3,3'-(1,2,4-thiadiazole-3,5-diyl)dipyridine; EtOH = ethanol) has been prepared by refluxing $\text{Fe}(\text{NCS})_3$ or $\text{Fe}(\text{NCS})_2$ with thionicotinamide in an ethanol solution and characterized by elemental analysis, infrared spectroscopy, thermal decomposition and X-ray structural analysis. The characterized compound is a mixture of three regioisomers resulting from differential coordination by the 3- and 5-position 2-pyridyl groups. The bpta ligand was generated *in situ* by the oxidation dimerization of thionicotinamide. X-ray single-crystal diffraction revealed that the crystal structure is centrosymmetric and the complex crystallizes in the triclinic space group $P\bar{1}$. The iron(II) atom lies in the centre of symmetry and it is octahedrally coordinated by two nitrogen atoms from two thiocyanate anions and two oxygen atoms from ethanol molecules in the equatorial plane and two nitrogen atoms of pyridine rings from two bpta ligands in the axial positions. This complex represents the second example of metal complex with bpta .

Keywords: 3,3'-(1,2,4-Thiadiazole-3,5-diyl)dipyridine – Thiocyanate complex – Iron(II) complex – Crystal structure

Introduction

Compounds containing a thiadiazole moiety possess interesting biological activity due to the strong aromaticity of this ring system (Siddiqui et al., 2009; Semwal et al., 2013). In recent years, thiadiazoles and their metal complexes have been extensively studied because of their potential applications in pharmaceutical, agriculture, industrial, polymer

and coordination chemistry (Richardson et al., 2002; Leung-Toung et al., 2003; Bentiss et al., 2004; Niu et al., 2008; Boeini, 2009; Klingele et al., 2010; Bhuvaa et al., 2011; Klingele et al., 2012; Liu et al., 2012).

Thiadiazoles with two substituted pyridyl groups, i.e. N,N' -(thiadiazole-diyl)dipyridine ($\text{N} = \text{N}' = 2, 3$ or 4), occur in 12 different isomeric forms such as N,N' -(1,2,3-thiadiazole-4,5-diyl)dipyridine, N,N' -(1,2,4-

thiadiazole-3,5-diyl)dipyridine, N,N'-(1,3,4-thiadiazole-2,5-diyl)dipyridine and N,N'-(1,2,5-thiadiazole-3,4-diyl)dipyridine. In literature, almost all information concerning N,N'-(1,3,4-thiadiazole-2,5-diyl)dipyridine and its coordination compounds (84 examples) (Allen, 2002) two complexes with 2,2'-(1,2,5-thiadiazole-3,4-diyl)dipyridine (Richardson et al., 2002) and one complex with 3,3'-(1,2,4-thiadiazole-3,5-diyl)dipyridine (Uhrecký et al., 2014) can be found. In addition, six crystal structures of N-[5-(pyridine-N-yl)-1,2,4-thiadiazole-3-yl]pyridinium salts are known; two with 4-[5-(pyridine-4-yl)-1,2,4-thiadiazole-3-yl]pyridinium and four with 3-[5-(pyridine-3-yl)-1,2,4-thiadiazole-3-yl]pyridinium (Aragoni et al., 2012). Thiadiazoles have four potential donor sites, *viz.* two N atoms of the pyridine rings and two N atoms of the thiadiazole ring. Only seven iron complexes with other isomers such as 2,2'-(1,3,4-thiadiazole-2,5-diyl)dipyridine and 2,2'-(1,2,5-thiadiazole-3,4-diyl)dipyridine which are coordinated to the Fe(II) atom forming five membered chelate rings [4–6]. Only one example with monodentate N,N'-(1,2,4-thiadiazole-3,5-diyl)dipyridine (the first complex contains coordinated methanol molecules instead ethanol) has been presented (Uhrecký et al., 2014).

This paper is a continuation of our previously reported studies on iron complexes with pyridine derivatives (Ondrejčovičová et al., 2008; Štefániková et al., 2008; Ondrejčovičová et al., 2009; Fargašová et al., 2010). The synthesis, crystal and molecular structure and properties of an Fe(II) complex, $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ where *bpta* is 3,3'-(1,2,4-thiadiazole-3,5-diyl)dipyridine and EtOH is ethanol, are reported on.

Experimental

Materials and physical measurements

All chemicals were purchased commercially and used without further purification. A 96 % vol. ethanol was used as the solvent. Elemental analyses (C, H, N and S) were accomplished by means of a Flash EA 1112 analyzer. The presence of gaseous hydrogen sulfide, released during the reaction, was proven with $\text{Pb}(\text{NO}_3)_2$. Infrared spectra (4000–100 cm^{-1}) were measured in solid state using the KBr technique and recorded on a Nicolet 5700 FTIR spectrophotometer. The thermal decomposition study was carried out on a Derivatographe MOM Budapest instrument in air atmosphere using a ceramic crucible with the sample mass of 150 mg from room temperature up to 1000 °C. The rate of temperature increase of 10 °C min^{-1} was chosen for the measurement.

Synthesis of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$

A solution of $\text{Fe}(\text{NCS})_3$ prepared from KSCN (0.145 g, 1.5 mmol) and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.135 g, 0.5 mmol) in EtOH (25 mL) was added to a solution of thionicotinamide (0.276 g, 2.0 mmol) in EtOH (25 mL) under continuous stirring. The reaction mixture was refluxed for 2 h. The insoluble KCl formed during the reaction was filtered out after cooling. The dark red filtrate was allowed to stand at room temperature for seven days. The resulting well-formed orange crystals of the title compound were suitable for X-ray structure analysis. Anal. Calc. for $\text{C}_{30}\text{H}_{28}\text{N}_{10}\text{O}_2\text{S}_4\text{Fe}$: C, 48.38; H, 3.79; N, 18.81; S, 17.22. Found: C, 48.19; H, 3.82; N, 19.02; S, 17.18. The same compound was similarly prepared from $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, KSCN and thionicotinamide in the molar ratio of 1:2:4. Found for $\text{C}_{30}\text{H}_{28}\text{N}_{10}\text{O}_2\text{S}_4\text{Fe}$ prepared from $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$: C, 48.22; H, 3.47; N, 19.12; S, 17.54.

X-ray crystallographic data collection and refinement

Intensity data for $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ were obtained on a Bruker-Nonius KappaCCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) at 150 K using an Oxford Cryostream low-temperature device. Absorption correction was applied using the program SADABS (Sheldrick, 2003). Data reduction was performed employing EvalCCD (Duisenberg et al., 2003). The structure was solved by direct methods using the program SIR-2011 (Burla et al., 2012) and refined by the full-matrix least-squares method on all F^2 data using the program SHELXL-2013 (Sheldrick, 2015). Geometrical analysis was performed using SHELXL-2013. The structure was drawn by OLEX2 (Dolomanov et al., 2009) and MERCURY (Macrae et al., 2006) softwares. The single crystal suite WINGX was used as an integrated system for all crystallographic programs and software when preparing the material for publication (Farrugia, 2012).

All non-hydrogen atoms of the title compound were refined anisotropically as independent atoms. Hydrogen atoms were mostly localized on a difference Fourier map; however, to ensure uniformity of the crystal treatment, all hydrogen atoms were recalculated into idealized positions and assigned temperature factors $U_{\text{iso}}(\text{H}) = 1.2 \text{ Ueq}$ for aromatic and methylene carbon atoms or of 1.5 Ueq for the methyl group and O—H group with $d(\text{C—H}) = 0.95 \text{ \AA}$ for hydrogen atoms in aromatic rings, $d(\text{C—H}) = 0.98$ and $0.99 \text{ \AA}$ for hydrogen atoms in the ethyl group ($-\text{CH}_2-$ and $-\text{CH}_3$, respectively); and $d(\text{O—H}) = 0.82 \text{ \AA}$ for hydrogen atoms in the hydroxyl group. Crystal data and conditions of data collection and refinement are

reported in Table 1, bond lengths and bond angles are reported in Table 2, and hydrogen bond parameters are reported in Table 3. Discrete positional disorder of the thiadiazole ring [C9A, C10A, N3A, N4A, S2A] and [C9B, C10B, N3B, N4B, S2B] was observed in the title compound with site occupancy factors of 0.84 and 0.16. Both disordered groups were restrained using SADI and EADP commands in the SHELXL-2013 program.

Tab. 1. Crystal data, X-ray measurements and structure determination summary for $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

Chemical formula	$\text{C}_{30}\text{H}_{28}\text{FeN}_{10}\text{OS}_4$
Formula weight	744.72
Temperature (K)	150
Crystal color, habit	Orange, needle
Crystal system, space group	Triclinic, $P-1$
a (Å)	7.413 (2)
b (Å)	9.157 (2)
c (Å)	12.804 (3)
α (°)	80.61 (3)
β (°)	77.69 (3)
γ (°)	81.42 (3)
V (Å ³)	831.8 (3)
Z	2
D_{calc} (g.cm ⁻³)	1.487
Radiation type	Mo $K\alpha$
α (mm ⁻¹)	0.75
Crystal size (mm)	$0.23 \times 0.23 \times 0.17$
Diffractionmeter	Bruker-Nonius KappaCCD
Abs. correction	Multi-scan SADABS
$T_{\text{min}}, T_{\text{max}}$	0.842, 0.880
S	1.068
R_{int}	0.040
$R_1[F^2 > 2\sigma(F^2)], wR_2(F^2)$	0.0306, 0.0699
No. of reflections	3337
No. of parameters	234
No. of restraints	19
$\Delta_{\text{max}}, \Delta_{\text{min}}$ (e Å ⁻³)	0.47, -0.46
CCDC no.	889109

Tab. 3. Selected hydrogen bond parameters for $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

$D-H \cdots A$	$D-H$ (Å)	$H \cdots A$ (Å)	$D \cdots A$ (Å)	$D-H \cdots A$ (°)
$\text{O1S}-\text{H1S} \cdots \text{N5}^{\text{ii}}$	0.82	1.91	2.727(2)	178

Symmetry code: (ii) $x - 1, y, z + 1$.

Tab. 2. Selected geometric parameters (Å, °) for $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

Bonds			
$\text{Fe1}-\text{N1}$	2.107(2)	$\text{Fe1}-\text{N2}$	2.271(2)
$\text{Fe1}-\text{O1S}$	2.108(1)		
Angles			
$\text{N1}-\text{Fe1}-\text{N2}^{\text{i}}$	88.97(6)	$\text{N2}-\text{Fe1}-\text{O1S}^{\text{i}}$	88.29(6)
$\text{N1}-\text{Fe1}-\text{N2}$	91.03(6)	$\text{N2}-\text{Fe1}-\text{O1S}$	91.71(6)
$\text{N1}-\text{Fe1}-\text{O1S}^{\text{i}}$	91.90(6)	$\text{N1}-\text{Fe1}-\text{O1S}$	88.10(6)

Symmetry code: (i) $-x + 1, -y, -z + 1$.

Results and discussion

Synthesis and structure

$[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ was prepared by refluxing the reaction mixtures containing thionicotinamide (*tnia*), KSCN and FeCl_3 or FeCl_2 in an EtOH solution (Scheme 1). It is believed that the *bpta* ligand is generated *in situ* by the oxidation dimerization of thionicotinamide (Scheme 2). The formed product is a mixture of three regioisomers resulting from differential coordination by the 3- and 5-position 2-pyridyl groups (Scheme 3). In case of $\text{Fe}(\text{NCS})_3$ as the starting compound, the iron(III) atom was reduced to iron(II) at first and then formed *bpta* molecules coordinated to the Fe atom. Released gaseous hydrogen sulfide was detected by smell and also by a reaction with $\text{Pb}(\text{NO}_3)_2$. An excess of *tnia* with respect to the reaction stoichiometry reaction remained unreacted (Scheme 1).

Generally, oxidation dimerization of thioamides is a method for the preparation of 3,5-disubstituted 1,2,4-thiadiazoles (Takikawa et al., 1985; Boeini, 2009; Patil et al., 2009).

The centrosymmetric complex $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ crystallizes in the triclinic system with the space group $P-1$. Molecular structure of the main part of the neutral mononuclear complex disorder is displayed in Fig. 1. The other two parts of disorders are drawn in Fig. 2.

The iron(II) atom lies in the centre of symmetry. It is octahedrally coordinated by two nitrogen atoms from two thiocyanate anions and two oxygen atoms from ethanol molecules in the equatorial plane and two nitrogen atoms of pyridine rings from two *bpta*

Scheme 1. Preparation of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

Scheme 2. Formation of *bpta* by oxidation dimerization of thionicotinamide.

Scheme 3. Regioisomers of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

ligands in the axial positions. The data indicate that all Fe—N and Fe—O distances are in the range of 2.107(2)–2.271(2) Å, typical for high-spin iron(II) compounds. However, Fe—N(*bpta*) distances are longer than the Fe—N(NCS) and Fe—O(EtOH) ones, where Fe—N(NCS) = 2.107(2) Å and Fe—O(EtOH) = 2.108(1) Å; Fe—N(*bpta*) = 2.271(2) Å. These bond distances are comparable with those reported for $[\text{Fe}(\text{tpa})(\text{NCS})_2]$ (*tpa* = tris(pyridin-2-yl)amine) (Vei et al., 2011), $[\text{Fe}(\text{NCS})_4(\mathbf{1})_5]$ ($\mathbf{1}$ = 4 phenylamino-1,2,4-triazole) (Roubeau et al., 2013) and

$[\text{Fe}(\text{NCS})_2(3\text{-bpo})_2(\text{H}_2\text{O})_2]$ (3-*bpo* = 2,5-di-3-pyridyl-1,3,4-oxadiazole) (Zhao et al., 2005). Thiocyanate anions, *bpta* and EtOH molecules are coordinated to iron(II) in a monodentate terminal mode in mutual *trans*-positions.

In the title complex, the uncoordinated nitrogen atom of the pyridine ring from the *bpta* ligand and the hydrogen atom from the ethanol molecule are joined by an O1S—H1S...N5 hydrogen bond (Table 3) into a 1D polymeric chain (Fig. 3). Moreover, the thiadiazole rings of the *bpta* ligands [N3, N4,

C9, C10, S2] are involved in face-to-face aromatic π - π stacking interactions with the centroid to centroid distance of 3.627 Å (Fig. 4a). These 1D chains are stacked in parallel to form 2D sheet through the inter-chain aromatic interaction between the thiadiazole rings. The hydrogen bonds and π - π stacking interactions play a significant role in the expanding of the mononuclear units to the 2D supramolecular network in the complex (Fig. 4b).

Fig. 1. Molecular structure of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.
Symmetry code: (i) $-x + 1, -y, -z + 1$.

Fig. 2. Molecular structure of the other two isomers of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.
Symmetry code: (i) $-x + 1, -y, -z + 1$.

Fig. 3. View of the 1D chain of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ formed by $\text{O}—\text{H} \cdots \text{N}$ hydrogen bonds.

Fig. 4. Schematic representation of π - π stacking interactions in $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ (a) and packing diagram along the b axis in $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$ showing the 2D supramolecular network (b).

IR spectra

Infrared spectra of the title compound prepared by two methods are practically identical. They show characteristic bands of *bpta* ($\nu_{\text{max}}/\text{cm}^{-1}$: 1598(s), 1579(sh), 1478(m), 1393(s), 1338(s), 1302(m), 1042(s) and 731(s)), which are very close to the published data for free *bpta* (Uhrecký et al., 2014). The spectra show typical bands for N-bond thiocyanate complexes (Zhao et al., 2005; Ondrejčovičová et al., 2008). It was assumed that the C—N stretch in the Fe—NCS complexes lies at 2075(s) cm^{-1} , the C—S stretch at 809(m) cm^{-1} and the NCS bend at 475(w) cm^{-1} . The presence of ethanol molecules in the complex is indicated by bands at 1097(w) and 1045(m) cm^{-1} ($\nu(\text{C—O})$), 2801(w) cm^{-1} ($\nu(\text{O—H})$), 2722(m) cm^{-1} ($\nu(\text{C—H})$), 1457(m) cm^{-1} ($\nu(\text{CH}_3)$) and 1473(m) cm^{-1} ($\nu(\text{CH}_2)$) (Patil et al., 2009). In the far infrared area, important stretches were observed: the band at 292(m) cm^{-1} can be assigned

to $\nu(\text{Fe—N})$ of the N-bond NCS group and the band at 253(w) cm^{-1} indicates that the *bpta* ligand is bond to the iron atom through the nitrogen atom of pyridine ring. The band assigned to $\nu(\text{Fe—O})$ occurred at 407(m) cm^{-1} (Ondrejčovičová et al., 2008; Štefániková et al., 2008; Nakamoto, 2009).

Thermal decomposition

TG and DTA curves of the decomposition of the title compound are shown in Fig. 5. The TG curve of this complex indicates that it is stable up to 150 °C, when the slow decomposition to Fe_2O_3 starts, as to the final product formed at 550 °C (Table 4). The first step, between 150 and 300 °C, is accompanied by a 12.40 % mass loss, in the release of two molecules of ethanol. The second step took place between 300 and 550 °C and is accompanied by a 64.56 % mass loss. It is attributed to the decomposition of two molecules of *bpta*. The third step, above 550 °C,

Scheme 4. Thermal decomposition of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.

Fig. 5. TG and DTA curves of $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$.
Sample weight: 150 mg; atmosphere: static air; heating rate: $10\text{ }^\circ\text{C min}^{-1}$.

Tab. 4. Thermal decomposition data.

[Fe(<i>bpta</i>) ₂ (EtOH) ₂ (NCS) ₂]					
<i>T</i> _{peaks} [°C]	<i>T</i> _{range} [°C]	Mass loss/%		Intermediate decomposition	Final product
		Found	Calc.		
198	150–300	12.40	12.38	Fe(<i>bpta</i>) ₂ (NCS) ₂	
462	300–550	64.56	64.53	“ <i>Fe</i> · 2 <i>NCS</i> ”	Fe ₂ O ₃
872	500–1000	15.36	15.60	“2 <i>NCS</i> ”	

is accompanied by a 15.36 % mass loss. During this step, Fe_2O_3 is formed as the final product of thermal decomposition. We assume that gaseous products: SO_2 , CO_2 and N_2 , are released similarly as in the case of the CuSCN and AgSCN decomposition (Ptaszyński et al., 1998). The most probable thermal decomposition scheme is as follows:

Conclusions

In summary, a new iron(II) complex, $[\text{Fe}(\text{bpta})_2(\text{EtOH})_2(\text{NCS})_2]$, was prepared by the reaction of $\text{Fe}(\text{NCS})_3$ or $\text{Fe}(\text{NCS})_2$ with thionicotinamide in ethanol. The *bpta* ligand was generated in situ by the oxidation dimerization of thionicotinamide. This complex represents the second example of a metal complex with N,N'-(1,2,4-thiadiazole-3,5-diyl)dipyridine. The characterized compound is a mixture of three regioisomers resulting from differential coordination by the 3- and 5-position 2-pyridyl groups. X-ray diffraction analysis of the title complex showed that the system of the $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ stacking interactions stabilize the crystal lattice to a 2D supramolecular network. Its molecular structure is in good agreement with

the observed infrared spectra and also with the data from literature. Thermal decomposition of the compound was initiated by the elimination of ethanol. The results revealed that Fe_2O_3 remains as a residue at the end of the thermal degradation process.

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