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2-*N*-Tosylaminobenzaldehyde Ferrocenoylhydrazone and Its Nickel(II) Complex: Molecular and Crystal Structures

L. D. Popov[a], E. A. Raspopova[a], A. N. Morozov[a], S. I. Levchenkov[b, *], G. G. Aleksandrov[c], A. S. Burlov[a], I. N. Shcherbakov[a], and V. A. Kogan[b]

[a] *Southern Federal University, Rostov-on-Don, 344104 Russia*
[b] *Southern Scientific Center, Russian Academy of Sciences, Rostov-on-Don, Russia*
[c] *Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii pr. 31, Moscow, 119991 Russia*
**e-mail: physchem@yandex.ru*

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Abstract—2-*N*-Tosylaminobenzaldehyde ferrocenoylhydrazone (H_2L) and the octahedral nickel(II) complex $[Ni(HL)_2] \cdot 2CH_3OH$ (**I**) are synthesized and structurally characterized (CIF files CCDC 981876 (H_2L) and 981877 (**I**)). The crystal structures of both compounds include two independent molecules with different mutual orientations of the tosyl and ferrocene fragments. In a single crystal of H_2L, the independent molecules are joined by intermolecular hydrogen bonds into infinite linear chains extended along the crystallographic axis *x*. The π-stacking interaction between the cyclopentadienyl rings is observed along with hydrogen bonds in a single crystal of complex **I**.

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INTRODUCTION

Hydrazones of polyfunctional carbonyl compounds are among the most studied types of ligand systems in modern coordination and supramolecular chemistry [1–5]. Among compounds of this type, hydrazones containing the ferrocene fragment are of a special interest, because some of them exhibit nonlinear optical properties of the second order [6–8] or biological activity [9, 10]. Numerous complexes based on hydrazones of the carbonyl ferrocene derivatives were described [11–19], whereas ferrocenoylhydrazones of carbonyl compounds are studied to a significantly lower extent [20–26]. In particular, none of ferrocenoylhydrazones and their complexes was studied by X-ray diffraction analysis. The X-ray diffraction results for 2-*N*-tosylaminobenzaldehyde ferrocenoylhydrazone (H_2L) and the nickel(II) complex based on H_2L (**I**) are presented.

EXPERIMENTAL

Ferrocenecarboxylic acid hydrazide and 2-*N*-tosylaminobenzaldehyde obtained by described procedures [26, 27] were used for the synthesis of H_2L.

Synthesis of H_2L. A hot solution of ferrocenecarboxylic acid hydrazide (2 mmol) in ethanol (10 mL) was poured to a hot solution of 2-*N*-tosylaminobenzaldehyde (2 mmol) in ethanol (10 mL). The reaction mixture was refluxed for 4 h and left to stay overnight. A precipitate was filtered off, washed with ethanol, and dried in vacuo. The product was recrystallized from an ethanol–dimethylformamide (1 : 1) mixture. The yield was 0.50 g (50%); mp > 250°C.

For $C_{25}H_{23}N_3O_3SFe$			
anal. calcd., %:	C, 59.9;	H, 4.62;	N, 8.38.
Found, %:	C, 60.3;	H, 4.49;	N, 8.61.

IR, ν, cm^{-1}: 3400, 3209 ν(NH), 1637 ν(C=O), 1608 ν(C=N), 1167 $\nu_{as}(SO_2)$, 1091 $\nu_s(SO_2)$, 515, 496 π(Cp–Fe). ^1H NMR (DMSO-d_6), δ, ppm: 11.42 s (1H, NH), 11.16 s (1H, NH), 8.46 s (1H, CH=N), 7.65 s (2H, J = 7.8 Hz, CH_{arom}), 7.55 s (2H, J = 6.9 Hz, CH_{arom}), 7.30 s (2H, J = 7.8 Hz, CH_{arom}), 7.16 m (2H, CH_{arom}), 4.98 s (2 H, CH_{Fc}), 4.50 s (2H, CH_{Fc}), 4.24 s (5H, CH_{Fc}), 2.30 s (3H, CH_3).

Synthesis of I. A hot solution of nickel(II) acetate (0.5 mmol) in methanol (10 mL) was poured to a hot solution of H_2L (1 mmol) in methanol (10 mL). The reaction mixture was refluxed for 1 h. A precipitate was filtered off, washed with methanol, dried in vacuo, and recrystallized from methanol. The yield was 0.17 g (30%); mp > 250°C.

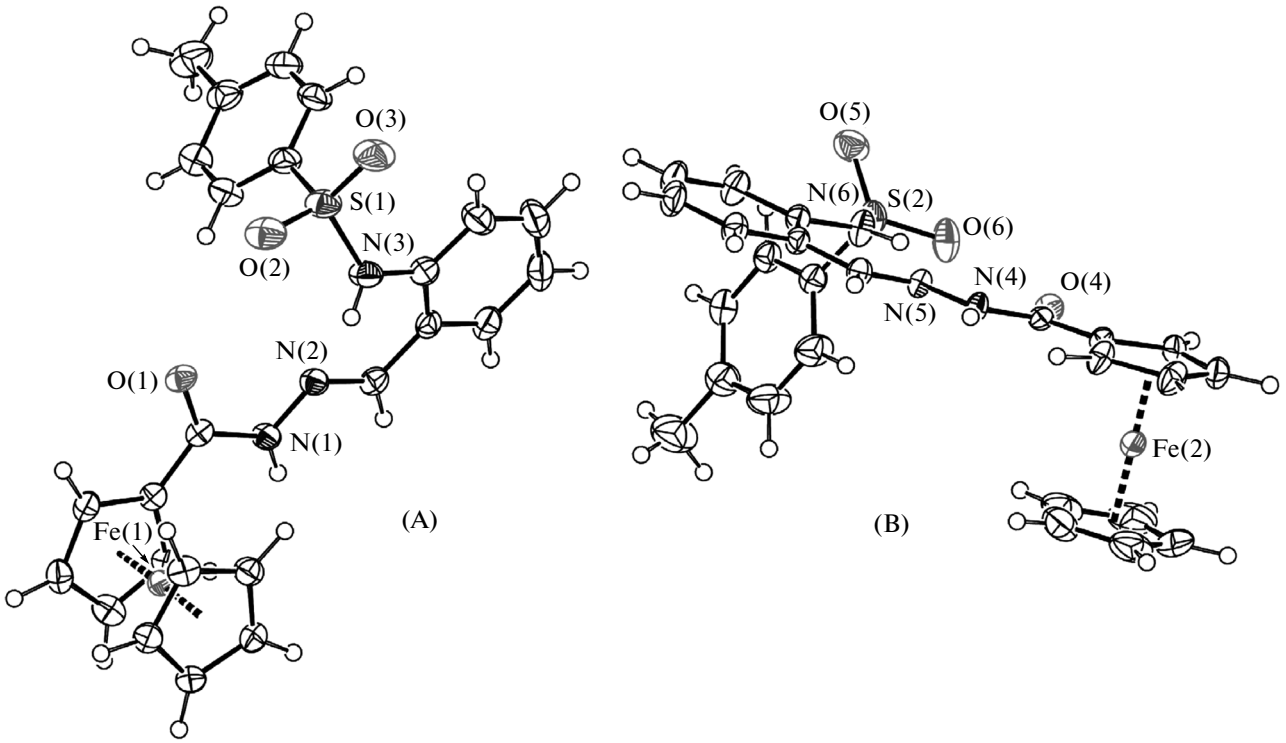

Fig. 1. Structures of two (A, B) independent H_2L molecules in the representation of atoms by thermal shift ellipsoids with 50% probability.

For $C_{51}H_{47}N_6O_7S_2Fe_2Ni$			
anal. calcd., %:	C, 56.2;	H, 4.34;	N, 7.71.
Found, %:	C, 56.5;	H, 4.45;	N, 7.92.

IR, ν, cm^{-1}: 3320 ν(OH), 3193 ν(NH), 1608 ν(C=O), 1598 ν(C=N), 1128 $\nu_{as}(SO_2)$, 1086 $\nu_s(SO_2)$, 508, 496 π(Cp–Fe). μ_{eff} 2.94 μ_B (298 K), 2.91 μ_B (77.4 K).

IR spectra were recorded on a Varian Scimitar 1000 FT-IR instrument in the range 400–4000 cm^{-1}. The samples were prepared as suspensions in Nujol. ^1H NMR spectra were detected in DMSO-d_6 in a Varian Unity 300 spectrometer (300 MHz) in the Fourier impulse mode. Elemental analyses were carried out on a PerkinElmer 240C instrument at the Laboratory of Microanalysis of the Southern Federal University. The magnetic susceptibility was determined by the relative Faraday method in the temperature range from 77.4 to 298 K.

X-ray diffraction analyses of compounds H_2L and **I** were carried out on a Bruker APEX II diffractometer (MoK_α, λ = 0.71073 Å, graphite monochromator) at 150(2) K. The initial array of measured intensities was processed using the SAINT [28] and SADABS programs [29]. The structures were solved by a direct method and refined by full-matrix least squares in the anisotropic approximation for non-hydrogen atoms for F^2_{hkl}. Hydrogen atoms were placed in the geometrically calculated positions and refined by the riding model ($U_{iso}(H) = nU_{iso}(C)$, where $n = 1.5$ for the carbon atoms of the methyl groups, and $n = 1.2$ for other C atoms). All calculations were performed using the SHELXTL program package [30]. The PLATON program was used for the analysis of the molecular and crystal structures [31]. The experimental characteristics of the crystallographic data for compounds H_2L and **I** are presented in Table 1. Selected interatomic distances and bond angles are listed in Table 2. The coordinates of atoms and temperature factors were deposited with the Cambridge Crystallographic Data Centre (CCDC 981876 (H_2L) and 981877 (**I**); http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

2-*N*-Tosylaminobenzaldehyde ferrocenoylhydrazone H_2L was synthesized by the condensation of 2-*N*-tosylaminobenzaldehyde with ferrocenecarboxylic acid hydrazide in ethanol. A single crystal of H_2L was obtained by the slow crystallization from a dimethylformamide solution. The single crystal contains two independent molecules (A and B) with similar

Table 1. Crystallographic data and experimental and refinement characteristics for compounds H_2L and **I**

Parameter	Value	
	H_2L	**I**
FW	501.37	1090.48
Crystal size, mm	0.33 × 0.11 × 0.09	0.23 × 0.11 × 0.05
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$
a, Å	9.4453(4)	18.6857(11)
b, Å	11.6977(5)	21.0992(13)
c, Å	23.4047(11)	24.2412(15)
α, deg	77.4728(7)	90
β, deg	82.1968(7)	93.5870(10)
γ, deg	66.5253(7)	90
V, Å^3	2311.93(18)	9538.5(10)
Z	4	8
ρ_{calcd}, g/cm^3	1.440	1.519
μ, mm^{-1}	0.775	1.139
F(000)	1040	4504
$2\theta_{max}$, deg	61.0	55.4
Ranges of reflection indices	$-13 < h < 13, -16 < k < 16, -32 < l < 33$	$-22 < h < 22, -25 < k < 25, -28 < l < 24$
Number of measured reflections	28053	34863
Number of independent reflections	13899	16580
Number of reflections with $I > 2\sigma(I)$	10491	7088
Number of refined parameters	595	1238
Goodness-of-fit (all reflections)	1.001	1.031
R_1 ($I > 2\sigma(I)$)	0.0408	0.0970
wR_2 (all reflections)	0.1362	0.2367
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.591/–0.640	1.916/–0.678

geometric parameters (Fig. 1). In the crystalline state H_2L exists in the hydrazone tautometric form [1, 32], and the oxygen–carbon interatomic distances in the carbonyl group correspond to the double bond (C(11)–O(1) 1.237(2), C(36)–O(4) 1.233(2) Å). The main distinction between molecules A and B is the different mutual orientations of the tosyl group and ferrocene fragment: at different sides from the hydrazone group C(11)N(1)N(2)C(12) in molecule A and at one side from the same-type group C(36)N(4)N(5)C(37) in molecule B.

The ferrocene fragment in molecule B exists in the screened conformation. The conformation of molecule A is also close to the screened one, but the cyclopentadienyl rings are turned at an angle of ~7°. The angles between the root-mean-square planes of the cyclopentadienyl rings slightly differ from zero, being 1.94(12)° and 1.72(15)° for molecules A and B, respectively.

The hydrazone fragment in molecule A is somewhat distorted because of the turn over the N(1)–N(2) bond, and the dihedral angle between the mean planes of the benzene and substituted cyclopentadienyl ring is 13.56(11)°. The hydrazone fragment in molecule B is nearly planar.

In addition to the mentioned above distortion, a distinction in the conformation of the tosylamine group is observed, resulting in a significant difference in geometric characteristics of the intramolecular hydrogen bonds formed between the tosylamine NH groups and azomethine N(2) and N(5) atoms. In molecule B, the six-membered cycle closed by the hydrogen bond is nearly planar (the deviation of the H(6B) and S(2) atoms from the root-mean-square plane N(5), C(37)–C(43), N(6) is 0.13 and 0.1616(5) Å, respectively), which favors the optimal characteristics of the hydrogen bonds (N(6)–H(6B) 0.88, H(6B)···N(5) 1.92, and N(6)···N(5) 2.637(2) Å, angle N(6)H(6B)N(5) 137°). In molecule A, the sulfur and hydrogen atoms of the tosylamine fragment considerably shift from a similar plane due to the turn around the C(18)–N(3) bond (the deviation of the H(3B) and S(1) atoms from the root-mean-square plane N(2), C(12)–C(18), N(3) is 0.57 and 0.758(6) Å, respectively), which noticeably worsens the conditions for hydrogen bond formation (N(3)–H(3B) 0.88, H(3B)···N(2) 2.22, and N(3)···N(2) 2.657(2) Å; angle N(3)H(3B)N(2) 110°). The tosyl groups are turned relatively to the S(1)–N(3) and S(2)–N(6) bonds in such a way that the S(1)=O(2) and N(3)–H(3B), S(2)=O(6) and N(6)–H(6B) bonds are coplanar in pairs.

The hydrazone NH group of each molecule (A and B) forms intermolecular bonds with the carbonyl O

Table 2. Selected interatomic distances and bond angles in structures H_2L and **I**

Bond	*d*, Å	Bond	*d*, Å
	H_2L		
S(1)–O(3)	1.4296(17)	S(2)–O(5)	1.4312(16)
S(1)–O(2)	1.4351(17)	S(2)–O(6)	1.4317(15)
S(1)–N(3)	1.6387(17)	S(2)–N(6)	1.6304(17)
O(1)–C(11)	1.237(2)	O(4)–C(36)	1.233(2)
N(1)–C(11)	1.360(2)	N(4)–C(36)	1.349(2)
N(1)–N(2)	1.364(2)	N(4)–N(5)	1.373(2)
N(2)–C(12)	1.281(2)	N(5)–C(37)	1.283(2)
N(3)–C(18)	1.419(3)	N(6)–C(43)	1.398(2)
	I		
Ni(1)–N(2)	2.017(12)	Ni(1)–N(10)	2.010(11)
Ni(1)–N(5)	2.021(12)	Ni(1)–N(7)	2.027(12)
Ni(1)–N(3)	2.072(10)	Ni(1)–N(9)	2.084(10)
Ni(1)–N(6)	2.076(11)	Ni(1)–N(12)	2.111(10)
Ni(1)–O(1)	2.096(9)	Ni(1)–O(10)	2.133(8)
Ni(1)–O(4)	2.133(9)	Ni(1)–O(7)	2.159(9)
Angle	ω, deg	Angle	ω, deg
	H_2L		
O(3)S(1)O(2)	119.70(11)	O(5)S(2)O(6)	119.56(10)
O(3)S(1)N(3)	109.96(10)	O(5)S(2)N(6)	110.51(10)
O(2)S(1)N(3)	104.62(9)	O(6)S(2)N(6)	103.97(9)
O(3)S(1)C(19)	108.37(11)	O(5)S(2)C(44)	107.63(9)
O(2)S(1)C(19)	108.19(11)	O(6)S(2)C(44)	109.27(10)
N(3)S(1)C(19)	105.04(10)	N(6)S(2)C(44)	104.97(9)
C(11)N(1)N(2)	117.93(15)	C(36)N(4)N(5)	117.79(14)
C(12)N(2)N(1)	118.39(16)	C(37)N(5)N(4)	117.01(15)
C(18)N(3)S(1)	125.02(14)	C(43)N(6)S(2)	127.89(13)
	I		
N(2)Ni(1)N(5)	161.3(4)	N(10)Ni(2)N(7)	172.8(4)
N(2)Ni(1)N(3)	89.3(5)	N(10)Ni(2)N(9)	95.6(4)
N(5)Ni(1)N(3)	103.8(4)	N(7)Ni(2)N(9)	88.6(4)
N(2)Ni(1)N(6)	103.0(5)	N(10)Ni(2)N(12)	86.3(4)
N(5)Ni(1)N(6)	89.4(5)	N(7)Ni(2)N(12)	99.7(4)
N(3)Ni(1)N(6)	94.2(4)	N(9)Ni(2)N(12)	88.4(4)
N(2)Ni(1)O(1)	79.1(4)	N(10)Ni(2)O(10)	78.9(4)
N(5)Ni(1)O(1)	86.8(4)	N(7)Ni(2)O(10)	94.8(4)
N(3)Ni(1)O(1)	168.1(5)	N(9)Ni(2)O(10)	98.4(4)
N(6)Ni(1)O(1)	91.3(4)	N(12)Ni(2)O(10)	164.2(4)
N(2)Ni(1)O(4)	87.9(4)	N(10)Ni(2)O(7)	98.9(4)
N(5)Ni(1)O(4)	77.7(4)	N(7)Ni(2)O(7)	76.3(4)
N(3)Ni(1)O(4)	95.7(4)	N(9)Ni(2)O(7)	163.6(4)
N(6)Ni(1)O(4)	165.3(4)	N(12)Ni(2)O(7)	100.3(4)
O(1)Ni(1)O(4)	81.1(3)	O(10)Ni(2)O(7)	76.9(3)

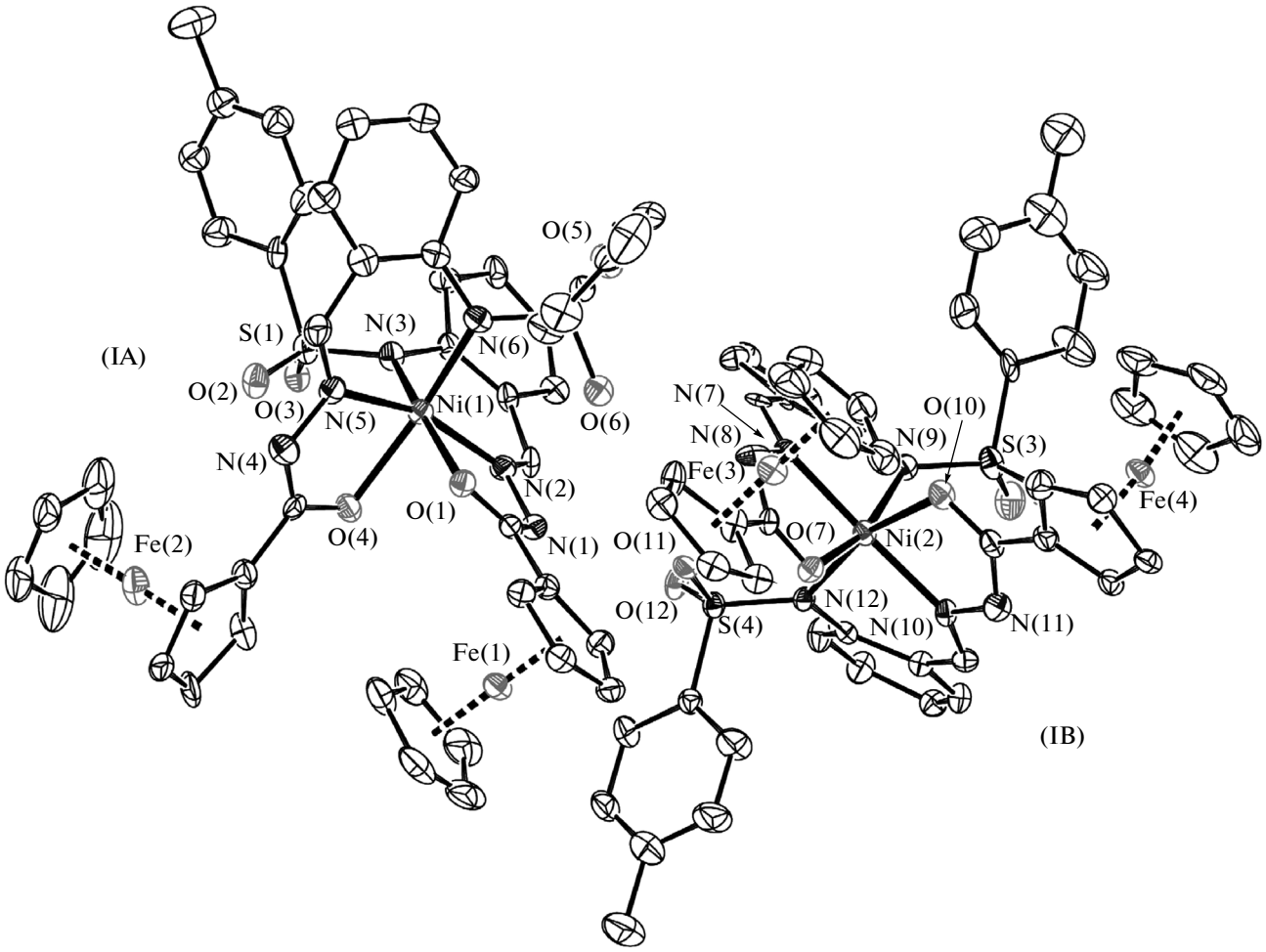

Fig. 2. Structures of two (IA, IB) independent molecules of complex **I** in the representation of atoms by thermal shift ellipsoids with 50% probability (methanol solvate molecules and hydrogen atoms are omitted).

atom of the hydrazide group in the adjacent crystallographic position (molecules A are bound to molecules B and vice versa). Molecule A forms two hydrogen bonds: N(1)–H(1B) 0.88, H(1B)···O(4)^{i} 1.94, N(1)···O(4)^{i} 2.777(2) Å, N(1)H(1B)O(4)^{i} 157° and N(4)^{ii}–H(4B)^{ii} 0.88, O(1)···H(4B)^{ii} 1.96, O(1)···N(4)^{ii} 2.786(2) Å, N(4)^{ii}H(4B)^{ii}O(1) 155°; crystallographic positions: ^{i} $2-x, -2-y, 2-z$; ^{ii} $1-x, -2-y, 2-z$). Similar hydrogen bonds are formed with the participation of molecule B, due to which independent infinite linear chains of the molecules are formed in a single crystal of compound H_2L. The chains are extended along the crystallographic axis x with the molecules alternating in conformations A and B.

The reaction of hydrazone H_2L with nickel(II) acetate in methanol gave complex **I** with the composition $[Ni(HL)_2] \cdot 2CH_3OH$, and its structure is shown in Fig. 2. As in the case of the initial hydrazone H_2L, a single crystal of complex **I** contains two independent molecules IA and IB in general positions. Organic ligands HL^- exist in the hydrazone tautomeric form (C–O in the carbonyl group 1.212(17)–1.249(18) Å) and are coordinated to the Ni^{2+} ions through the tridentate mode. Both ligands exist in different conformations in each independent molecule. In one of the ligands the tosyl group and ferrocene fragment are arranged at one side from the hydrazone group, while they are at different sides in another ligand. The conformation of all four ferrocene fragments is close to the screened one.

In both independent molecules, the coordination polyhedron of the nickel atoms is a strongly distorted octahedron formed by the azomethine and amine nitrogen atoms and the hydrazone oxygen atoms. The oxygen atoms of the tosyl fragments do not form coordination bonds with the nickel atoms: the Ni–O_{tos} distance is fairly long being 3.197(9), 3.252(9) Å in molecule IA and 3.216(9), 3.305(10) Å in molecule IB.

However, the small value of torsion angles Ni–N–S–O (3°–15°) indicates a certain attractive interaction.

All five-membered metallocycles have an envelope conformation, whose "valve" is formed by the Ni atom shifting from the mean plane of other four atoms by 0.1804(17) and 0.2742(17) Å in molecule IA and by 0.2727(18) and 0.2940(17) Å in molecule IB. The six-membered metallocycles are distorted to a significantly higher extent due to the inflection over the N···N and N···C lines.

In a single crystal of complex **I**, the independent molecules are joined by two intermolecular hydrogen bonds involving the NH group of the hydrazone fragment of one of the ligands and the oxygen atom of the tosyl group: N(1)–H(1B)···O(11) (N(1)–H(1B) 0.88, H(1B)···O(11) 2.15, N(1)···O(11) 2.841(15) Å, angle N(1)H(1B)O(11) 136°) and N(8)–H(8B)···O(6) (N(8)–H(8B) 0.88, H(8B)···O(6) 2.29, N(8)···O(6) 2.939(14) Å, N(8)H(8B)O(6) 130°). In addition, it can be assumed that there is a π-stacking interaction between the substituted cyclopentadienyl rings of the ferrocene fragments including the Fe(1) and Fe(3) atoms (dihedral angle between the mean planes of the carbon atoms 5.4(9)°, intercentroid distance 3.379(9) Å).

The hydrazone NH group of the second ligand of molecule IA forms the bifurcate intermolecular hydrogen bond with the oxygen atoms of two methanol molecules (N(4)–H(4B) 0.88, H(4B)···O(2S)^{i} 2.37, H(4B)···O(1S)^{ii} 2.52, N(4)···O(2S)^{i} 3.01(2), N(4)···O(1S)^{ii} 3.30(3) Å, N(4)H(4B)O(2S)^{i} 130°, N(4)H(4B)O(1S)^{ii} 148°; crystallographic positions: ^{i} $1-x, -y, -z$; ^{ii} $1/2+x, 1/2-y, -1/2+z$). The OH groups of both methanol molecules form hydrogen bonds with the O(9) atom of the tosyl group of the adjacent molecule IB (the hydrogen atoms of the methanol molecules were not localized). The O(2S)···O(9) and O(2S)···O(9) distances are 2.707(19) and 2.90(2) Å, which are shorter by 0.33 and 0.14 Å, respectively, than the doubled van der Waals radius of oxygen. In turn, the hydrazone NH group of the second ligand of molecule IB forms the intermolecular hydrogen bond with the O atom of the tosyl group of another molecule IA (N(11)–H(11A) 0.88, H(11A)···O(11)^{iii} 2.19, N(11)···O(11)^{iii} 2.929(16) Å, angle N(11)H(11A)O(11)^{iii} 141°; crystallographic position: ^{iii} $1/2-x, 1/2+y, -1/2-z$), due to which infinite layers of the molecules are formed in the single crystal of complex **I**. The molecules in the layers are bound by intermolecular hydrogen bonds parallel to the crystallographic plane *x*0*y*.

Thus, 2-*N*-tosylaminobenzaldehyde ferrocenoylhydrazone and its complex with nickel(II) were synthesized for the first time and studied by X-ray diffraction analysis. Their structures include two independent molecules with different mutual orientations of the tosyl and ferrocene fragments.

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