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Synthesis and Structure of Co(III) Coordination Compounds $[Co(DH)_2(Anil)_2][BF_4]$ and $[Co(DH)_2(Py)_2][BF_4]$

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Abstract—The complex compounds $[Co(DH)_2(Anil)_2][BF_4]$ and $[Co(DH)_2(Py)_2][BF_4]$ were synthesized from $Co(BF_4)_2 \cdot 6H_2O$–DH_2–A–alcohol–water systems (DH_2 is dimethylglyoxim and A is pyridine (Py) or aniline (Anil)), and their crystal structures were determined using X-ray diffraction analysis. In octahedral Co(III) complexes, two dimethylglyoxime radicals lie in the equatorial plane and are joined via the intramolecular hydrogen bond O–H··O. The complexes with pyridine and aniline have similar configurations but different crystal structures.

Co(III) is known to form stable complexes with chelating α-dioxime ligands. These complexes can be applied as industrial catalysts [1, 2], stimulators of hemogenesis [3], antihypoxic medicines [4], and as a model of vitamin B_{12} [5, 6].

Coordination compounds of Co(III) with α-dioximes have different structures depending on their synthesis conditions (pH of solutions), composition, and nature of the coordinated ligands.

In this work, the results of synthesis and X-ray diffraction analysis of two Co(III) coordination compounds—$[Co(DH)_2(Anil)_2][BF_4]$ (**I**) and $[Co(DH)_2(Py)_2][BF_4]$ (**II**), where DH is dimethylglyoxime radical[1], Anil is aniline, and Py is pyridine—are reported.

EXPERIMENTAL

Synthesis of $[Co(DH)_2(Anil)_2][BF_4]$ (I). A solution of 0.23 g (0.002 mol) of dimethylglyoxime in 30 ml of methanol and 0.23 ml (0.0025 mol) of aniline were added to a solution of 0.34 g (0.001 mol) of $Co(BF_4)_2 \cdot 6H_2O$ in 30 ml of water. The reaction mixture was placed into a graphite crucible and heated on a water bath at 50°C. Crystalline needles precipitated from a dark brown solution upon slow cooling (yield, 40%). Compound **I** is soluble in alcohol and slightly soluble in water.

For $C_{20}H_{28}CoF_4N_6O_4B$
anal. calcd. (%): Co, 10.48; C, 42.73; H, 5.02; N, 14.95.
Found (%): Co, 10.24; C, 41.86; H, 5.50; N, 14.55.

Synthesis of $[Co(DH)_2(Py)_2][BF_4]$ (II) was carried out using a similar procedure (yield, 45%).

For $C_{18}H_{24}CoF_4N_6O_4B$
anal. calcd. (%): Co, 11.03; C, 40.47; H, 4.53; N, 15.73.
Found (%): Co, 10.86; C, 40.20; H, 5.75; N, 15.63.

UV spectra of solutions **I** and **II** were recorded on a Specord M-40 spectrometer in the 200–400 nm range using a methanol/water mixture as a solvent; the solution concentration was 10^{-4} mol/l.

X-ray diffraction analysis. The single crystals of compound **I** were obtained in the form of dark brown needles (0.54 × 0.12 × 0.10 mm). Their diffraction patterns were taken on a CAD-4 diffractometer (MoK_α radiation, graphite monochromator, $\theta/2\theta$ scan mode).

Compound **II** crystallized in the form of dark brown prisms (0.42 × 0.22 × 0.18 mm); their diffraction patterns were taken on a SYNTEX P1 diffractometer (CuK_α radiation, graphite monochromator, $\theta/2\theta$ scan mode).

Crystals **I** are orthorhombic: $a = 23.828(3)$ Å, $b = 17.035(3)$ Å, $c = 12.623$ Å, space group *Pbca*, $Z = 8$, $\rho(\text{calcd}) = 1.455$ g/cm^3.

Crystals **II** are monoclinic: $a = 12.034(2)$ Å, $b = 7.549(2)$ Å, $c = 13.506(3)$ Å, $\beta = 104.74(3)°$, space group *Pn*, $Z = 2$, $\rho(\text{calcd}) = 1.495$ g/cm^3.

Structures **I** and **II** were solved by direct methods (SHELX86 program [7]) and refined using 2320 (for **I**) and 1356 (for **II**) independent reflections with $I > 3\sigma$.

Non-hydrogen atoms were refined in the full-matrix anisotropic approximation on F^2; hydrogen atoms were isotropically refined. The hydrogen atoms were refined using the rigid group model. The calculations were performed using the SHELX93 program [8].

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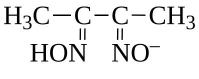
[1] 
$H_3C-\underset{HON}{\overset{}{C}}-\underset{NO^-}{C}-CH_3$

Table 1. Coordinates of non-hydrogen atoms ($\times 10^4$) and their isotropic thermal parameters U_{eq} ($\times 10^3$) in structures **I** and **II**

	I				**II**			
Atom	x	y	z	U_{eq}, Å^2	x	y	z	U_{eq}, Å^2
Co	6344(1)	7412(1)	6201(1)	30(1)	6418	7505(3)	6331	41(1)
O(1)	6355(2)	8775(2)	7556(3)	51(1)	5129(11)	4842(13)	5024(10)	71(3)
O(2)	5701(2)	7206(2)	4316(3)	41(1)	5711(9)	10955(12)	6746(9)	57(3)
O(3)	6965(2)	7627(2)	8085(3)	40(1)	7726(10)	10162(12)	7653(8)	54(3)
O(4)	6351(2)	6098(2)	4785(3)	46(1)	7197(11)	4049(11)	5916(8)	63(3)
N(1)	6124(2)	8408(3)	6709(3)	35(1)	5129(11)	6490(15)	5402(10)	54(4)
N(2)	5803(2)	7664(3)	5143(3)	33(1)	5373(12)	9435(13)	6224(9)	43(3)
N(3)	6876(2)	7160(2)	7259(3)	31(1)	7714(10)	8496(12)	7245(10)	43(3)
N(4)	6567(2)	6427(2)	5681(3)	32(1)	7466(12)	5602(13)	6423(11)	52(3)
N(5)	5786(2)	6906(4)	7171(4)	36(1)	5870(10)	6515(15)	7459(8)	39(3)
N(6)	6920(2)	7912(3)	5262(4)	35(1)	6948(11)	8436(15)	5164(10)	50(3)
B	6393(3)	9983(4)	3834(7)	60(2)	5602(18)	1130(30)	10708(18)	78(5)
F(1)	6225(3)	9556(4)	3018(6)	196(4)	6547(15)	2023(19)	11322(13)	123(5)
F(2)	6574(2)	9516(3)	4630(6)	159(3)	4688(10)	2168(14)	10725(13)	92(3)
F(3)	5976(2)	10437(3)	4180(4)	110(2)	5732(10)	861(13)	9800(8)	86(3)
F(4)	6842(2)	10405(3)	3519(4)	102(2)	5551(15)	–455(13)	11215(9)	121(5)
C(1)	5726(2)	8758(3)	6212(5)	44(1)	4182(14)	7471(14)	5131(10)	40(4)
C(2)	5533(2)	8316(3)	5288(4)	39(1)	4413(14)	9247(18)	5630(12)	47(4)
C(3)	5495(5)	9533(6)	6527(9)	73(3)	3478(13)	10600(20)	5476(13)	61(4)
C(4)	5066(3)	8585(5)	4586(6)	58(2)	3132(13)	6750(20)	4454(12)	57(4)
C(5)	7158(2)	6517(3)	7127(4)	36(1)	8514(15)	5822(18)	7001(14)	60(5)
C(6)	6964(2)	6080(3)	6181(5)	38(1)	8607(13)	7570(20)	7466(14)	59(5)
C(7)	7614(4)	6250(6)	7816(7)	56(2)	9800(20)	8140(30)	8144(14)	85(7)
C(8)	7202(6)	5314(6)	5835(8)	66(2)	9440(19)	4470(20)	7189(16)	79(6)
C(9)	5242(2)	6680(4)	6774(4)	43(2)	5820(20)	7550(16)	8301(16)	80(8)
C(10)	5162(3)	5951(4)	6339(6)	58(2)	5470(20)	6840(20)	9076(15)	84(6)
C(11)	4636(5)	5748(7)	5961(6)	86(3)	5066(19)	5160(30)	9056(16)	76(5)
C(12)	4206(4)	6268(9)	6043(7)	103(4)	5098(16)	4060(20)	8201(16)	70(5)
C(13)	4283(3)	6976(8)	6475(7)	93(3)	5531(14)	4816(19)	7474(12)	54(4)
C(14)	4801(3)	7200(5)	6856(5)	62(2)	7344(16)	10110(18)	5211(13)	57(4)
C(15)	7506(2)	7813(3)	5525(4)	33(1)	7680(16)	10850(20)	4355(13)	64(5)
C(16)	7743(3)	8291(3)	6283(5)	44(1)	7655(17)	9910(30)	3545(15)	75(5)
C(17)	8296(3)	8168(5)	6552(5)	59(2)	7357(17)	8100(30)	3529(13)	72(5)
C(18)	8600(3)	7575(5)	7575(5)	61(2)	6997(14)	7503(18)	4309(13)	53(5)
C(19)	8360(3)	7095(4)	7095(4)	53(2)				
C(20)	7808(3)	7220(3)	7220(3)	41(1)				

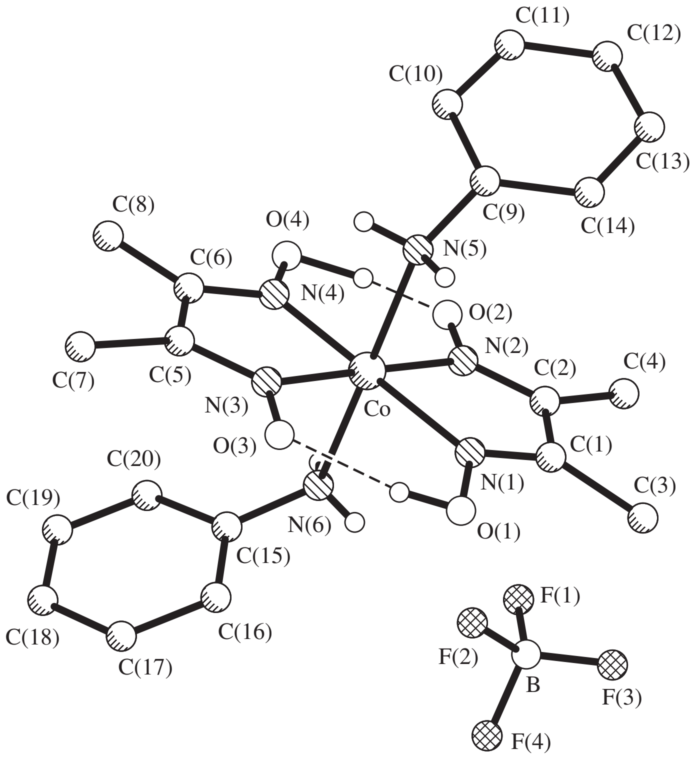


Fig. 1. The structure of the molecule of complex **I**.

The final *R*-factor was equal 0.023 for **I** and 0.084 for **II**.

Coordinates and thermal parameters of the basis atoms in structures **I** and **II** are presented in Table 1.

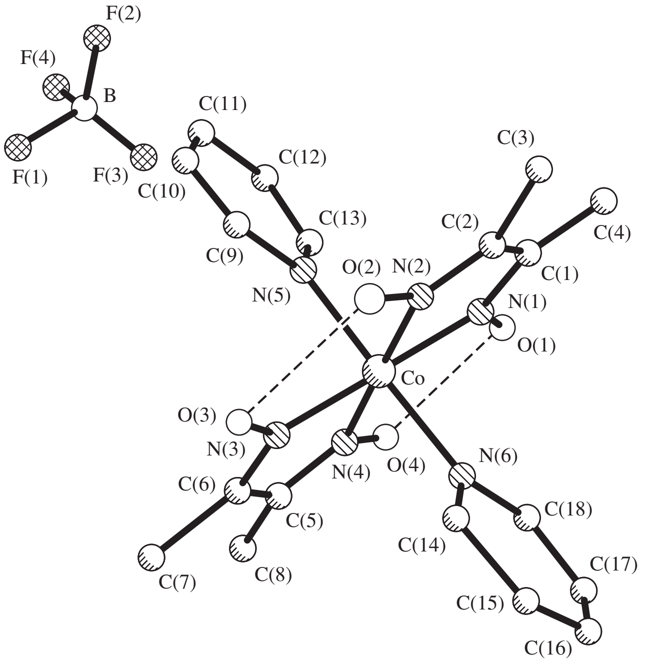


Fig. 2. The structure of the molecule of complex **II**.

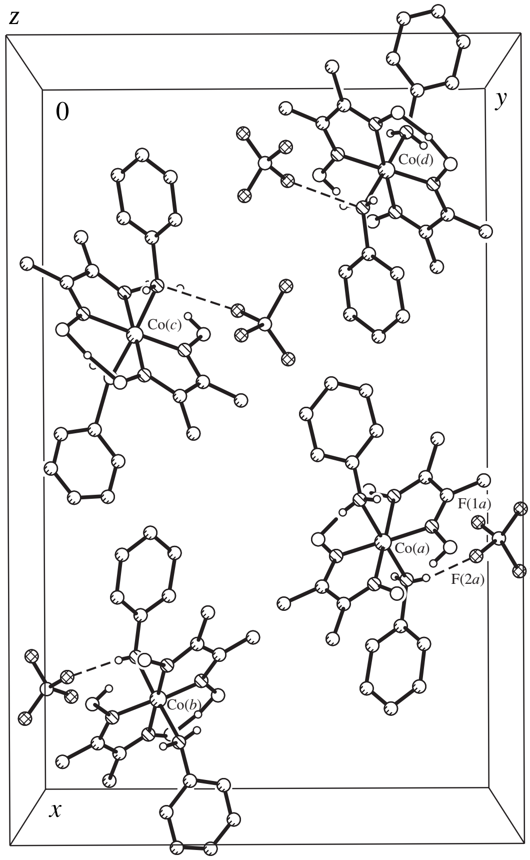


Fig. 3. A fragment of the crystal structure of complex **I**.

RESULTS AND DISCUSSION

The UV spectra of complexes **I** and **II** show two absorption bands. The band at 246 nm is typical of Co(III) dioximines with two dimethylglyoxime radicals in the equatorial plane and is due to the intraligand (DH) π–π transition [9]. The other band at ~370 nm is produced by pyridine or aniline in compounds **I** and **II**, respectively, and is due to charge transfer from the axial ligands to the cobalt ion [10].

The structures of molecules **I** and **II** with different complex cations $[Co(DH)_2(Anil)_2]^+$ and $[Co(DH)_2(Py)_2]^+$ are shown in Figs. 1 and 2, respectively. The $[BF_4]^-$ anions in both structures are in the outer sphere.

The cobalt atom in complex cations of **I** and **II** exhibits a similar *trans*-octahedral coordination (6N). The Co(III) coordination polyhedron, apart from four N atoms of two bidentate-chelate ligands DH, includes two nitrogen atoms of aniline (in the case of **I**) or of pyridine (in the case of **II**). In both cases, two dimethylglyoxime radicals lie in the equatorial plane and are joined together via intramolecular hydrogen bonds O–H…O (for **I**, O(1)··O(3) 2.528, O(1)–H(1) 0.84, and O(3)··H(1) 1.7 Å; the O(1)–H(1)··O(3) angle is 171°; for **II**, O(1)··O(4) 2.514 and O(2)··O(3) 2.481 Å). The hydrogen bond parameters are close to those of other Co(III) complexes with α-dioximes [11–13]. Location of the hydrogen atoms in structure **II** was a failure because of insufficient experimental data and the poor quality of the single crystal.

The average Co–N distance in the coordination polyhedron of the cobalt atom in compounds **I** and **II** is equal to 1.889 Å. The interatomic distances and bond angles of **I** and **II** given in Table 2 are in good agreement with the literature data [11–15] and correspond to the atomic hybridization.

The metal rings of dimethylglyoximes are planar to within 0.02 Å. The shifts of atoms from selected planar fragments of the structures are illustrated in Table 3.

In **I**, the dihedral angles formed by the equatorial plane of the central atom of the complex cation (N(1),

Table 2. Interatomic distances and bond angles in structures **I** and **II**

I				II			
Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Co–N(4)	1.879(4)	B–F(2)	1.352(9)	Co–N(4)	1.89(1)	B–F(1)	1.40(3)
Co–N(1)	1.888(4)	C(1)–C(2)	1.462(8)	Co–N(1)	1.89(1)	B–F(3)	1.29(2)
Co–N(3)	1.893(5)	C(1)–C(3)	1.484(9)	Co–N(3)	1.88(1)	B–F(4)	1.39(2)
Co–N(2)	1.906(4)	C(2)–C(4)	1.496(8)	Co–N(2)	1.91(1)	B–F(2)	1.35(2)
Co–N(5)	2.005(5)	C(5)–C(7)	1.466(9)	Co–N(5)	1.96(1)	C(1)–C(2)	1.49(2)
Co–N(6)	2.006(5)	C(5)–C(6)	1.481(7)	Co–N(6)	1.97(1)	C(1)–C(4)	1.46(2)
O(1)–N(1)	1.355(6)	C(6)–C(8)	1.490(9)	O(1)–N(1)	1.34(2)	C(2)–C(3)	1.50(2)
O(2)–N(2)	1.325(5)	C(9)–C(10)	1.371(8)	O(2)–N(2)	1.35(1)	C(5)–C(6)	1.45(2)
O(3)–N(3)	1.330(5)	C(9)–C(14)	1.379(9)	O(3)–N(3)	1.37(1)	C(5)–C(8)	1.48(3)
O(4)–N(4)	1.364(5)	C(10)–C(11)	1.39(1)	O(4)–N(4)	1.35(1)	C(6)–C(7)	1.56(2)
N(1)–C(1)	1.285(7)	C(11)–C(12)	1.36(1)	N(1)–C(1)	1.33(2)	C(9)–C(10)	1.33(4)
N(2)–C(2)	1.297(6)	C(12)–C(13)	1.34(1)	N(2)–C(2)	1.24(2)	C(10)–C(11)	1.36(3)
N(3)–C(5)	1.296(6)	C(13)–C(14)	1.38(1)	N(3)–C(6)	1.25(2)	C(11)–C(12)	1.43(3)
N(4)–C(6)	1.283(6)	C(15)–C(16)	1.376(7)	N(4)–C(5)	1.31(2)	C(12)–C(13)	1.35(2)
N(5)–C(9)	1.444(7)	C(15)–C(20)	1.378(7)	N(5)–C(13)	1.35(2)	C(14)–C(15)	1.43(2)
N(6)–C(15)	1.447(8)	C(16)–C(17)	1.379(8)	N(5)–C(9)	1.39(3)	C(15)–C(16)	1.30(3)
B–F(1)	1.323(9)	C(17)–C(18)	1.374(9)	N(6)–C(14)	1.35(2)	C(16)–C(17)	1.41(3)
B–F(3)	1.332(8)	C(18)–C(19)	1.364(9)	N(6)–C(18)	1.37(2)	C(17)–C(18)	1.32(2)
B–F(4)	1.351(9)	C(19)–C(20)	1.387(8)				

Table 2. (Contd.)

I				II			
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
N(4)CoN(1)	179.3(2)	F(1)BF(2)	110.5(7)	N(3)CoN(1)	179.1(7)	F(3)BF(4)	112(2)
N(4)CoN(3)	81.5(2)	F(3)BF(2)	109.6(7)	N(3)CoN(4)	80.3(5)	F(1)BF(2)	105(2)
N(1)CoN(3)	98.7(2)	F(4)BF(2)	106.1(7)	N(1)CoN(4)	99.0(5)	F(3)BF(2)	114(2)
N(4)CoN(2)	98.5(2)	N(1)C(1)C(2)	112.6(5)	N(3)CoN(2)	99.6(5)	F(4)BF(2)	111(2)
N(1)CoN(2)	81.3(2)	N(1)C(1)C(3)	123.8(6)	N(1)CoN(2)	81.1(6)	C(9)N(5)Co	121(1)
N(3)CoN(2)	179.6(2)	C(2)C(1)C(3)	123.7(6)	N(4)CoN(2)	179.2(7)	C(14)N(6)C(18)	116(1)
N(4)CoN(5)	91.0(2)	N(2)C(2)C(1)	113.4(5)	N(3)CoN(5)	91.6(5)	C(14)N(6)Co	119(1)
N(1)CoN(5)	89.7(2)	N(2)C(2)C(4)	123.2(5)	N(1)CoN(5)	88.8(6)	C(18)N(6)Co	125.7(9)
N(3)CoN(5)	85.2(2)	C(1)C(2)C(4)	123.3(5)	N(4)CoN(5)	90.6(5)	N(1)C(1)C(4)	121(1)
N(2)CoN(5)	94.3(2)	N(3)C(5)C(7)	124.8(6)	N(2)CoN(5)	90.2(5)	N(1)C(1)C(2)	109(1)
N(4)CoN(6)	88.7(2)	N(3)C(5)C(6)	111.5(5)	N(3)CoN(6)	90.0(5)	C(4)C(1)C(2)	130(1)
N(1)CoN(6)	90.6(2)	C(7)C(5)C(6)	123.7(6)	N(1)CoN(6)	89.5(5)	N(2)C(2)C(3)	124(1)
N(3)CoN(6)	93.0(2)	N(4)C(6)C(5)	113.3(5)	N(4)CoN(6)	88.7(6)	N(2)C(2)C(1)	116(1)
N(2)CoN(6)	87.4(2)	N(4)C(6)C(8)	122.8(6)	N(2)CoN(6)	90.5(5)	C(3)C(2)C(1)	120(2)
N(5)CoN(6)	178.2(2)	C(5)C(6)C(8)	123.9(6)	N(5)CoN(6)	178.1(6)	N(4)C(5)C(6)	109(2)
C(1)N(1)O(1)	118.2(5)	C(10)C(9)C(14)	120.3(7)	C(1)N(1)O(1)	120(1)	N(4)C(5)C(8)	126(1)
C(1)N(1)Co	117.1(4)	C(10)C(9)N(5)	120.4(6)	C(1)N(1)Co	117.2(9)	C(6)C(5)C(8)	125(2)
O(1)N(1)Co	124.7(4)	C(14)C(9)N(5)	119.2(6)	O(1)N(1)Co	123(1)	N(3)C(6)C(5)	116(2)
C(2)N(2)O(2)	121.6(4)	C(9)C(10)C(11)	119.4(8)	C(2)N(2)O(2)	123(1)	N(3)C(6)C(7)	126(2)
C(2)N(2)Co	115.4(4)	C(12)C(11)C(10)	119.6(9)	C(2)N(2)Co	116.8(9)	C(5)C(6)C(7)	118(2)
O(2)N(2)Co	123.0(3)	C(13)C(12)C(11)	121.0(9)	O(2)N(2)Co	120(1)	C(10)C(9)N(5)	120(1)
C(5)N(3)O(3)	121.6(5)	C(12)C(13)C(14)	121.0(1)	C(6)N(3)O(3)	120(1)	C(11)C(10)C(9)	123(2)
C(5)N(3)Co	116.8(4)	C(13)C(14)C(9)	118.7(9)	C(6)N(3)Co	117(1)	C(10)C(11)C(12)	118(2)
O(3)N(3)Co	121.6(3)	C(16)C(15)C(20)	121.4(6)	O(3)N(3)Co	123.4(9)	C(13)C(12)C(11)	116(2)
C(6)N(4)O(4)	119.8(5)	C(16)C(15)N(6)	119.1(5)	C(5)N(4)O(4)	119(1)	N(5)C(13)C(12)	126(2)
C(6)N(4)Co	116.7(4)	C(20)C(15)N(6)	119.4(5)	C(5)N(4)Co	117.7(9)	N(6)C(14)C(15)	120(2)
O(4)N(4)Co	123.4(3)	C(15)C(16)C(17)	118.3(6)	O(4)N(4)Co	124(1)	C(16)C(15)C(14)	121(2)
C(9)N(5)Co	120.0(4)	C(18)C(17)C(16)	120.8(6)	C(13)N(5)C(9)	116(1)	C(15)C(16)C(17)	120(2)
C(15)N(6)Co	118.6(4)	C(19)C(18)C(17)	120.6(6)	C(13)N(5)Co	122.6(9)	C(18)C(17)C(16)	117(2)
F(1)BF(3)	110.4(7)	C(18)C(19)C(20)	119.5(6)	F(1)BF(3)	111(2)	C(17)C(18)N(6)	126(1)
F(1)BF(4)	107.7(8)	C(15)C(20)C(19)	119.3(6)	F(1)BF(4)	105(2)		
F(3)BF(4)	112.4(6)						

Table 3. The deviations of atoms (Å) from selected mean-square planes in structures **I** and **II**

I	II
N(1) (0.009), N(2) (–0.009), N(3) (–0.009), N(4) (0.009), Co* (–0.002)	N(1) (0.011), N(2) (–0.012), N(3) (0.012), N(4) (–0.011), Co* (–0.023)
Co (–0.023), N(1) (0.0224), N(2) (0.027), C(1) (–0.011), C(2) (–0.017), O(1)* (0.090), O(2)* (0.090)	Co (0.012), N(1) (–0.019), N(2) (0.008), C(1) (0.017), C(2) (0.002), O(1)* (0.037), O(2)* (0.011)
Co (0.025), N(3) (–0.030), N(4) (–0.025), C(5) (0.019), C(6) (0.010)	Co (0.005), N(3) (0), N(4) (–0.013), C(5) (0.014), C(6) (–0.007), O(3)* (0.010), O(4)* (0.046)
C(9) (–0.006), C(10) (0.006), C(11) (–0.003), C(12) (0), C(13) (0), C(14) (0.003), N(5)* (0)	N(6) (–0.018), C(14) (0.007), C(15) (0.001), C(16) (0), C(17) (–0.001), C(18) (0.020)
C(15) (–0.001), C(16) (0.008), C(17) (–0.005), C(18) (–0.004), C(19) (0.011), C(20) (–0.008), N(6)* (0.061)	N(5) (0.003), C(9) (0.026), C(10) (–0.048), C(11) (0.040), C(12) (–0.012), C(13) (–0.009)

* The atoms that were not used in the calculation of the mean-square planes are marked by an asterisk.

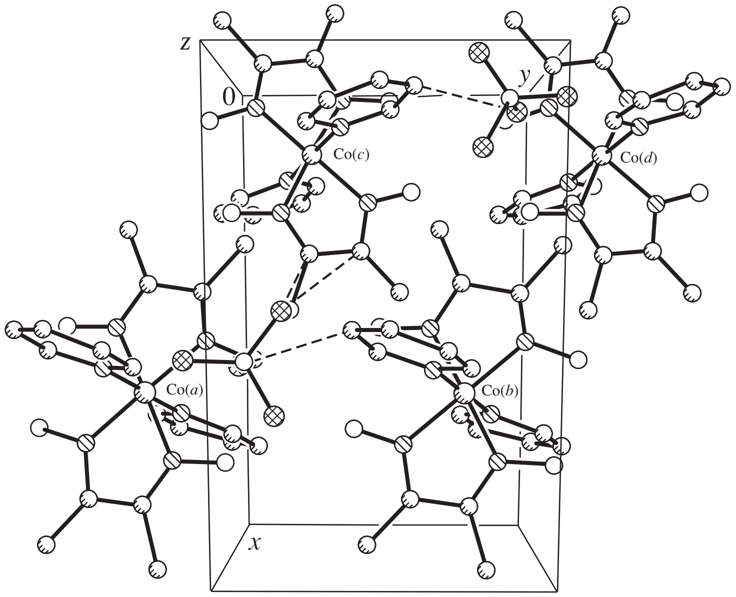


Fig. 4. A fragment of the crystal structure of complex **II**.

N(2), N(3), N(4)) and the mean-square planes of the aniline molecules (C(9), C(10), C(11), C(12), C(13), C(14) and C(15), C(16), C(17), C(18), C(19), C(20)) are equal to 145.0° and 151.4°, respectively. In **II**, the analogous angles formed by the plane of N(1)–N(9) atoms and pyridine molecules (N(5), C(9), C(10), C(11), C(12), C(13) and N(6), C(14), C(15), C(16), C(17), C(18)) are equal to 89.0° and 91.1°.

The N–H··F hydrogen bond is of crucial importance in the formation of the crystal structure of compound **I** (Fig. 3): F(2)··N(6) 2.96, F(2)··H(6) 2.29, and N(6)··H(6) 0.73 Å; the angle at the hydrogen atom is equal to 156°.

In complex **II**, the complex cation and anions are bound via electrostatic interactions F(3)··C(12), F(3)··C(10) ($0.5 + x$, $-1 - y$, $0.5 - z$), F(2)··C(5) ($-0.5 + x$; $1 - y$; $0.5 + z$), and F(2)··C(6) ($-0.5 + x$; $1 - y$, $0.5 + z$) (Fig. 4); the corresponding interatomic distances are equal to 3.16, 3.18, 2.92, and 2.97 Å. Short F(2)··C(5) and F(2)··C(6) contacts can be interpreted as the interaction of the fluorine electron cloud with the π cloud of the metal ring.

Other intermolecular contacts shorter than the sum of the van der Waals radii of the respective atoms were not observed in structures **I** and **II**.

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