



Reactions of 2-pyridinethiolate cobalt(III) complexes with pyridinethiolate or benzenethiolate ligands

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ABSTRACT

Four half-sandwich cobalt complexes, $\text{Cp}^*\text{Co}(\text{2-PyS})_2$ (**2**), $\text{Cp}^*\text{Co}(\text{2-PyS})_2 \cdot \text{HI}$ (**3**), $\text{Cp}^*\text{Co}(\text{2-PyS})(\text{4-PyS})$ (**4**), $(\text{Cp}^*\text{Co})_2(\mu\text{-PhS})_2(\mu\text{-2-PyS})\text{I}$ (**5**) [Cp^* = pentamethylcyclopentadienyl, 2-PyS = 2-pyridinethiolate, 4-PyS = 4-pyridinethiolate, PhS = benzenethiolate] were successfully synthesized by the reactions of 2-pyridinethione, lithium 4-pyridinethiolate and lithium benzenethiolate with $\text{Cp}^*\text{Co}(\text{2-PyS})\text{I}$ (**1**), respectively. Complexes **2** and **3** have the structures with two 2-pyridinethiolates ligands coordinated to the cobalt atom. Two different pyridinethiolates ligands can be identified in complex **4**. The molecular structure of **5** consists of two $\text{Cp}^*\text{-Co}$ fragments, which are triply bridged by three sulfur atoms from different ligands. The molecular structures of **3** and **5** were determined by X-ray crystallographic analysis. All the complexes have been well characterized by elemental analysis, NMR and IR spectra.

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1. Introduction

The coordination chemistry of 2-pyridinethiolates ligand is of great importance in a variety of reactions [1], due to their unusual coordination geometry, variety of bonding, and interesting spectral and electrochemical behavior of these complexes [2–4]. There are two donor atoms in these types of ligands, a sulfur atom and a nitrogen atom, which are considered as a soft center and a hard center, respectively [5,6], enabling these ligands to coordinate to both soft and hard metals. 2-Pyridinethiolates ligands interact with metal atoms in several ways, which may be coordinated as a monodentate group through the sulfur or nitrogen atom, as a bidentate chelating moiety through the sulfur and nitrogen donors, or as a bridging ligand through both donor groups [7–13].

The products of such ligands with metal atoms are found to have special properties for many applications, such as, antitumor potential with gold and platinum [14], dimerization catalyst of alkynes with iridium [15], vitro potency against trypanozoma cultures with tin [16], and control of the behavior of nucleic acids as tautomeric systems [17]. The half-sandwich complexes, which have cobalt, rhodium, and iridium centers with the 2-pyridinethiolate ligand, are still under comprehensive investigation. In our previous work, we synthesized half-sandwich Cp^*Co and Cp^*Rh complexes with 2-pyridinethiolate [$\text{Cp}^*\text{M}(\text{PyS})\text{X}$] ($\text{M} = \text{Co}, \text{Rh}$; $\text{X} = \text{I}, \text{Cl}$), which were used as precursors to study the complexes with the 1,2-dicarba-closo-dodecaborane (12)-1-thiolate ligand and 1,2-dicarba-closo-dodecaborane (12)-1,2-dithiolate ligand

[18]. Herein, we report the synthesis and structure of half-sandwich cobalt(III) complexes containing the 2-pyridinethiolate ligands with 2-pyridinethione, 4-pyridinethiolate and benzenethiolate ligand. In particular, the unusual reactions and stereochemistry of those organometallic compounds are described.

2. Results and discussion

2.1. Reaction with 2-pyridinethiolate

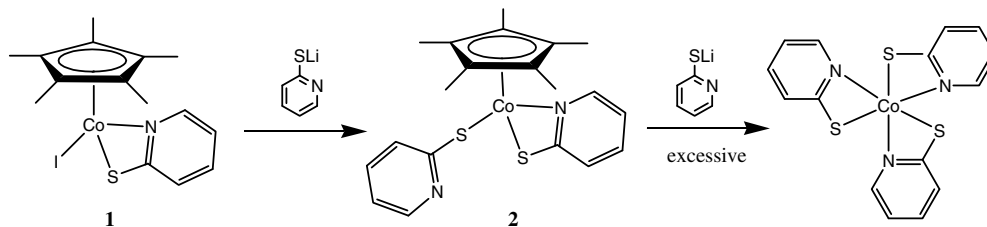
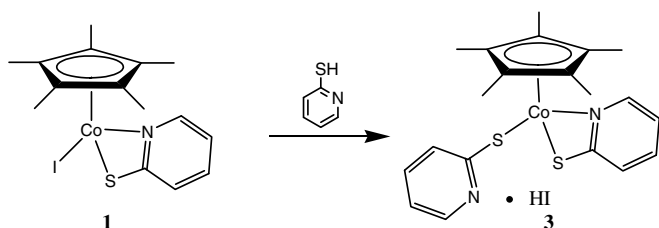
The complex **2** was prepared as shown in Scheme 1. The starting material, $\text{Cp}^*\text{Co}(\text{2-PyS})\text{I}$ **1** was easily prepared by the literature procedures [18].

Complex **1** was reacted with lithium 2-pyridinethiolate in the THF solvent, giving the product $\text{Cp}^*\text{Co}(\text{2-PyS})_2$ (**2**) as brown crystals in 83% yield. Eight broad peaks in the ^1H NMR spectrum of the PyS region, together with other detailed analysis of the spectroscopic data (^{13}C NMR and IR spectra), show that there are two different 2-PyS ligands in one molecule for complex **2**. There is no special product besides the known compound tris(2-pyridinethiolato)cobalt(III), when **2** was reacted with excessive lithium 2-pyridinethiolate in the above reaction.

2.2. Reaction with 2-pyridinethione

When complex **1** was reacted with 2-pyridinethione in the same molar ratio in THF, the product $\text{Cp}^*\text{Co}(\text{2-PyS})_2 \cdot \text{HI}$ (**3**) was obtained as dark green crystals in 73% yield (Scheme 2). The IR spectrum of **3** shows a typical N–H absorption at 3141 cm^{-1} , which is consistent with the previously reported analogous complexes.

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Scheme 1. Preparation of $\text{Cp}^*\text{Co}(2\text{-PyS})_2$ (**2**).Scheme 2. Preparation of $\text{Cp}^*\text{Co}(2\text{-PyS})_2 \cdot \text{HI}$ (**3**).

Similar peaks can be identified in the ^1H NMR spectrum of the PyS region as complex **2**. And in particular, we found a peak appeared at 13.82 ppm, which could be assigned as N–H proton. All these data were consistent with the structure established by X-ray diffraction.

Suitable single crystal of **3** was obtained by slowly diffusion of hexane into a dichloromethane solution. The molecular structure has been established by single crystal X-ray structure analysis (Fig. 1). The crystal structure of **3** consists of a cobalt atom, a $\text{Cp}^*\text{-Co}$ half-sandwich tripod structure and two PyS ligands, which is similar to the rhodium complex $\text{Cp}^*\text{Rh}(\text{PyS})_2$ [19] and $\text{Cp}^*\text{Rh}(2\text{-SC}_5\text{H}_3\text{N-3-SiMe}_3)_2$ [20]. One 2-PyS ligand chelates to the cobalt center in a N,S-bidentate mode (Co(1)–S(1) 2.3080(2) Å, Co(1)–N(1) 1.970(4) Å), while the other bonds to the metal atom in a S-monodentate mode (Co(1)–S(2) 2.2656(2) Å). The distance of Co(1)–S(2) is a little shorter than that of Co(1)–S(1) (2.3080(2) Å), because of the different coordination modes of the 2-PyS ligands.

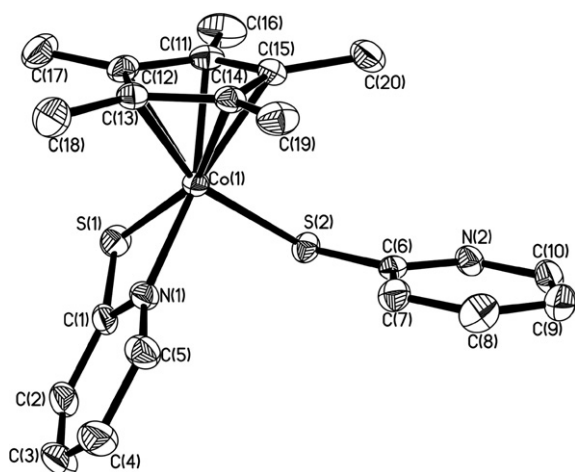


Fig. 1. Molecular structure of **3**, ellipsoids at the 30% probability level. H atoms are omitted for clarity. Selected bond lengths (Å) and angles ($^\circ$): Co(1)–C(11) 2.054(5), Co(1)–C(12) 2.075(5), Co(1)–C(13) 2.068(5), Co(1)–C(14) 2.075(4), Co(1)–C(15) 2.090(4), Co(1)–N(1) 1.970(4), Co(1)–S(1) 2.3080(2), Co(1)–S(2) 2.2656(2), N(1)–C(1) 1.348(6), N(1)–C(5) 1.324(7), N(2)–C(10) 1.343(6), N(2)–C(6) 1.347(6), S(1)–C(1) 1.723(6), S(2)–C(6) 1.721(5); N(1)–Co(1)–S(2) 94.18(2), N(1)–Co(1)–S(1) 71.67(2), S(2)–Co(1)–S(1) 86.52(5), C(1)–S(1)–Co(1) 78.06(2), C(6)–S(2)–Co(1) 110.45(2), C(1)–N(1)–Co(1) 100.3(3), N(1)–C(1)–S(1) 109.7(3), N(2)–C(6)–S(2) 117.2(4).

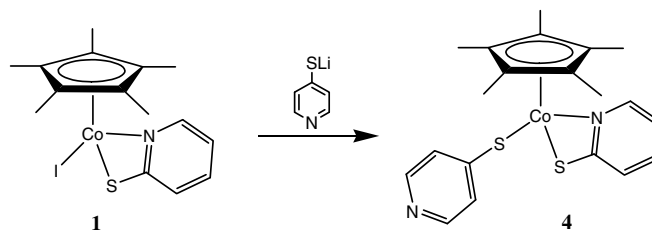
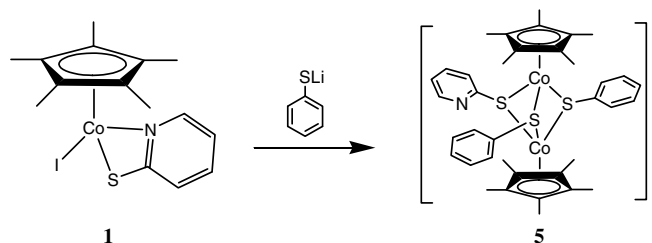
The local geometry around the cobalt atom is distorted due to the ‘typical four-membered bite angle’ with the bite angle of S(1)–Co–N(1) (71.67°). The distance of iodine anion to the N(2) atom is 3.3505(4) Å, suggesting that they are bonded by one hydrogen atom, which has been identified in the ^1H NMR spectrum.

2.3. Reaction with lithium 4-pyridinethiolate

The direct reaction of complex **1** with 4-pyridinethione in THF solution, does not afford any products as the reaction with 2-pyridinethione. Then lithium salt of 4-pyridinethione was considered. When the complex **1** reacted with 1 equiv of the lithium salt in the THF at -78°C , 4-PyS ligand replaced the iodine anion to give the brown product **4** in high yield (Scheme 3). Four different signals at 8.47, 7.97, 6.71, 6.43 ppm ^1H NMR spectrum of **4** are very close to those of complex **1**, showing that the metal center of **4** is coordinated by one 2-PyS ligand in bidentate mode. Another two signals at 8.51 and 7.38 ppm, which are identified as the hydrogen atoms of the 4-PyS ligand, were also detected obviously. Each of them follows the integral ratio at 2:1 to other PyS–H peaks, reveals that the 4-PyS ligand coordinates to the cobalt atom in a S-monodentate mode.

2.4. Reaction with lithium benzenethiolate

The reaction of **1** with the same molar quantity of lithium benzenethiolate gave a gradual color change from green to dark brown, as shown in Scheme 4. Brown crystals of the product iodide salt **5** could be isolated in a moderate yield. Seven kinds of signals are shown in aromatic hydrogen region of the ^1H NMR spectrum, indicating both the PhS ligand and 2-PyS ligands coordinated to

Scheme 3. Synthesis of $\text{Cp}^*\text{Co}(2\text{-PyS})(4\text{-PyS})$ (**4**).Scheme 4. Preparation of $(\text{Cp}^*\text{Co})_2(\mu\text{-PhS})_2(\mu\text{-2-PyS})\text{I}$ (**5**).

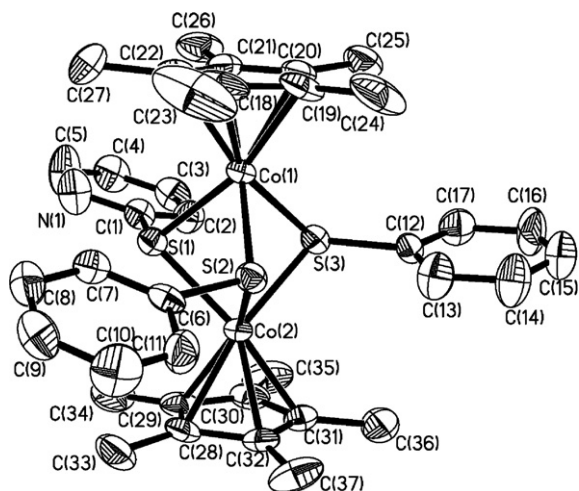


Fig. 2. Molecular structure of **5**, ellipsoids at the 30% probability level. Anion and H atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Co(1)–S(1) 2.2733(5), Co(1)–S(2) 2.2669(5), Co(1)–S(3) 2.2783(5), Co(2)–S(1) 2.2579(4), Co(2)–S(2) 2.2879(5), Co(2)–S(3) 2.2596(5), S(1)–C(1) 1.8108(5), S(2)–C(6) 1.8068(6), S(3)–C(12) 1.8131(5), N(1)–C(1) 1.3512(3), N(1)–C(5) 1.3871(4); S(1)–Co(1)–S(3) 82.779(2), S(1)–Co(2)–S(3) 83.54(2), S(1)–Co(2)–S(2) 81.90(2), S(2)–Co(1)–S(1) 82.03(2), S(2)–Co(1)–S(3) 82.291(2), S(2)–Co(2)–S(3) 82.236(2), Co(2)–S(1)–Co(1) 81.10(2), Co(1)–S(2)–Co(2) 80.59(2), Co(2)–S(3)–Co(1) 80.95(3).

the cobalt center. Recrystallization of **5** was processed in order to confirm the detail information of its structure. Unexpectedly, the molecular structure of **5** is different from **2**, **3** and **4**, through the single crystal X-ray diffraction.

The complexes **5** can be recrystallized from CH₂Cl₂/*n*-hexane to give well-formed brown single crystals in the monoclinic space group *P*2₁/*n* with four molecules in the unit cell. The X-ray crystallographic analysis of **5** determined the triply bridged dinuclear structure of the complex cation (Fig. 2) with one μ -S-bridging 2-PyS ligands and two μ -S-bridging PhS ligand, which is found to consist of a closed trigonal bipyramid Co₂S₃ framework. Many half-sandwich complexes with Rh₂S₃ [21–30] or Ir₂S₃ framework [27,31–38] have been explored, but few cobalt compounds with such molecular structure have been reported. In complex **5**, each metal center is coordinated to a Cp* ligand, and three sulfur atoms carrying a 2-pyridyl or phenyl group. The nitrogen atom of 2-PyS ligand is uncoordinated to the cobalt atom any more in the complex **5**, which breaks the N,S-bidentate chelate ring of complex **1**. Formally, the cobalt atoms exhibit *pseudo*-octahedral coordination geometries, being coordinated to the six-electron donor Cp* ring and to the three bridging ligands. These three bridging sulfur atoms define the plane, to which two Cp* ligands are oriented so as to be nearly parallel (dihedral angles less than 3°). The Co–S bond distance (2.2733(5) Å) of the 2-PyS ligand, is shorter than that (2.303(1) Å) of complex **1**, due to the different coordination mode. Because of the relatively large separation between the two cobalt atoms, in the cation **5** the phenyl or pyridine rings are not constrained to a coplanar arrangement, rotate by 15.42° (2-PyS), 24.03° and 8.85° (PhS), with respect to the plane formed by the three bridging sulfur atoms.

3. Conclusion

In this article, three half-sandwich cobalt complexes with 2-pyridinethiolato group were synthesized from the reactions between **1** with 2-pyridinethiolate (**2**), 2-pyridinethione (**3**), 4-pyridinethiolate (**4**) and benzenethiolate ligand (**5**). Complex **2** and **3** have been established that two 2-pyridinethiolates ligands are bonded to the same cobalt center in monodentate and bidentate coordination modes, respectively. Different from the synthesis

process of **2** and **3**, complex **4** can only be obtained by the lithium salts of the 4-pyridinethione. X-ray diffraction spectrum of **5** reveals a cation of two Cp*–Co fragments connecting with two PhS ligands and one 2-PyS ligand, which would be a new method to synthesize half-sandwich complex with triply bridged dinuclear structure.

4. Experimental

4.1. General procedures

All reactions and manipulations were carried out in standard Schlenk vessels under a dry nitrogen atmosphere. Dichloromethane were distilled over CaH₂. Tetrahydrofuran and hexane was distilled over sodium/benzophenone ketyl. All the solvents were kept free of traces of water and oxygen just before use. Cp*Co(PyS)I was prepared according to the literature procedures [18]. 2-Pyridinethione, 4-pyridinethione and benzenethiol were obtained commercially and used without further purification. IR spectra were recorded on a Nicolet AVATAR-360 IR spectrometer using KBr disc in the range of 4000–400 cm⁻¹; only characteristic frequencies of each product were listed. Elemental analyses were carried out with an Elementar III Vario EI analyzer. ¹H NMR (500 MHz) and ¹³C NMR (125 MHz) spectra were obtained using Bruker DMX-500 spectrophotometer in CDCl₃ solution with TMS as the internal standard.

4.2. Preparation of Cp*Co(2-PyS)₂ (**2**)

A solution of *n*-BuLi (1.6 M, 0.14 mL, 0.22 mmol) in hexane was cooled to 0 °C, then a solution of 2-pyridinethione (22 mg, 0.2 mmol) in 10 mL THF was added dropwise. The suspension was kept at room temperature and stirred for another 1 h. The resulting solution was slowly added to a solution of Cp*Co(PyS)I (**1**) (86 mg, 0.2 mmol) in 20 mL THF at –78 °C, and the mixture was stirred for 18 hours with the color change from dark green to red brown. The volatiles were removed in vacuo and the product was extracted with CH₂Cl₂. Recrystallization of the filtrate from CH₂Cl₂/hexane afforded **2** as red brown solid (68 mg, 83%). Anal. Calc. C₂₀H₂₃CoN₂S₂ (414.47 g/mol): C, 57.96; H, 5.59; N, 6.76. Found: C, 58.02; H, 5.46; N, 6.58. IR (KBr): 3074, 1611, 1588, 1483, 1355, 1254, 1142, 1075, 1020, 763 cm⁻¹. ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.31 (d, 1H, 2-PyS), 8.09 (d, 1H, 2-PyS), 7.72 (d, 1H, 2-PyS), 7.62 (t, 1H, 2-PyS), 7.10 (t, 1H, 2-PyS), 7.08 (t, 1H, 2-PyS), 6.35 (t, 1H, 2-PyS), 6.08 (d, 1H, 2-PyS), 1.71 (s, 15H, Cp*). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 185.53, 149.62, 135.25, 124.91, 118.92 (PyS), 183.39, 148.76, 134.32, 124.44, 116.25 (PyS), 92.27, 9.67 (Cp*).

4.3. Preparation of Cp*Co(2-PyS)₂ · HI (**3**)

2-Pyridinethione (22 mg, 0.2 mmol) was added to a solution of Cp*Co(PyS)I (**1**) (86 mg, 0.2 mmol) in 20 mL THF at –78 °C, and the mixture was stirred overnight. The solvent was removed under reduced pressure and the product was extracted with CH₂Cl₂. The brown filtrate was precipitated in hexane to give **3** as dark green solid (79 mg, 73%). Anal. Calc. for C₂₀H₂₄CoN₂S₂I (542.39 g/mol): C, 44.29; H, 4.46; N, 5.17. Found: C, 44.31; H, 4.35; N, 5.24%. IR (KBr): 3141, 3079, 1601, 1571, 1485, 1437, 1361, 1257, 1130, 1077, 1021, 744 cm⁻¹. ¹H NMR (500 MHz, CDCl₃, ppm): δ 13.82 (d, 1H, H-I), 8.38(d, 1H, 2-PyS), 8.22 (d, 1H, 2-PyS), 7.39 (d, 1H, 2-PyS), 7.17 (t, 1H, 2-PyS), 7.10 (t, 1H, 2-PyS), 6.70 (t, 1H, 2-PyS), 6.60 (t, 1H, 2-PyS), 6.29 (d, 1H, 2-PyS), 1.52 (s, 15H, Cp*). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 176.27, 149.42, 135.94, 125.87, 117.05 (PyS), 173.29, 149.18, 136.00, 125.80, 118.07 (PyS), 91.13, 9.53 (Cp*).

Table 1
X-ray crystallographic data and processing parameters for **3** and **5**

Complex	3	5
Empirical formula	C ₂₀ H ₂₄ CoN ₂ S ₂ I	C ₃₈ H ₄₆ Cl ₂ Co ₂ INS ₃
Formula weight	542.39	928.65
Crystal system	Monoclinic	Monoclinic
Space group	P2 ₁ /c	P2 ₁ /n
a (Å)	18.149(6)	13.735(4)
b (Å)	7.260(3)	16.530(5)
c (Å)	18.226(6)	17.583(6)
α (°)	90	90
β (°)	115.757(4)	91.517(4)
γ (°)	90	90
Volume (Å ³)	2162.8(13)	3991(2)
Z	4	4
μ (mm ⁻¹)	2.421	1.923
Crystal size (mm)	0.30 × 0.10 × 0.10	0.20 × 0.15 × 0.10
D _{calc.} (mg/m ³)	1.666	1.546
F(000)	1080	1880
θ Range (°)	2.241–23.451	1.69–27.13
h, k, l Collected	–22, 23; –6, 9; –21, 23	±17; –15, 21; –22, 21
Reflections collected/unique (R _{int})	10419/4775 (0.0578)	19539/8667 (0.0663)
Data/restraints/parameters	4775/18/278	8667/0/434
Goodness-of-fit on F ²	0.976	0.893
Final R indices [I > 2σ(I)]	R ₁ = 0.0518, wR ₂ = 0.1136	R ₁ = 0.0647, wR ₂ = 0.1679
R indices (all data)	R ₁ = 0.0797, wR ₂ = 0.1255	R ₁ = 0.1397, wR ₂ = 0.1995
Largest difference in peak and hole (e Å ⁻³)	1.167 and –0.857	1.641 and –0.743

4.4. Preparation of Cp⁺Co(2-PyS)(4-PyS) (**4**)

To a solution of 4-pyridinethione (22 mg, 0.2 mmol) in 10 mL THF was added a solution of *n*-BuLi (1.6 M, 0.14 mL, 0.22 mmol) in hexane dropwise at 0 °C. The suspension was kept at room temperature and stirred for another 1 hour. The resulting solution was slowly added to a solution of Cp⁺Co(PyS)I (**1**) (86 mg, 0.2 mmol) in 20 mL THF at –78 °C, and the mixture was stirred for 18 hours. The solvent was removed in vacuo and the product was extracted with CH₂Cl₂. Recrystallization of the brown filtrate from CH₂Cl₂/hexane afforded **4** as brown solid (68 mg, 83%). Anal. Calc. for C₂₀H₂₃CoN₂S₂ (414.47 g/mol): C, 57.96; H, 5.59; N, 6.76. Found: C, 58.56; H, 5.63. N, 6.88%. IR (KBr): 2955, 1619, 1441, 1261, 1091, 1024, 801, 702, 640, 579, 517, 458 cm⁻¹. ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.47 (d, 1H, 2-PyS), 7.97 (t, 1H, 2-PyS), 6.71 (t, 1H, 2-PyS), 6.43 (d, 1H, 2-PyS), 8.51 (d, 2H, 4-PyS), 7.38 (d, 2H, 4-PyS), 1.71 (s, 15H, Cp⁺). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 175.60, 152.02, 135.63, 125.81, 117.33 (2-PyS), 149.32, 136.76, 129.77 (4-PyS), 93.17, 10.20 (Cp⁺).

4.5. Preparation of Cp⁺Co(μ-PhS)₂(μ-2-PyS)I (**5**)

To an Et₂O (10 mL) solution of benzenethiol (22 mg, 0.2 mmol) was added a solution of *n*-BuLi (1.6 M, 0.14 mL, 0.22 mmol) in hexane dropwise at 0 °C. A white precipitate was formed immediately. After being kept at room temperature for 1 h, the solution was slowly added to a solution of Cp⁺Co(PyS)I (**1**) (86 mg, 0.2 mmol) in 20 mL THF at –78 °C, and the mixture was stirred overnight. The solvent was removed under reduced pressure and the residue was redissolved in 10 mL CH₂Cl₂. The brown filtrate was collected. Recrystallization of the filtrate from CH₂Cl₂/hexane afforded **5** as brown solid (54 mg, 58%). Anal. Calc. for C₃₇H₄₄Co₂INS₃ · CH₂Cl₂ (928.65 g/mol): C, 49.15; H, 4.99; N, 1.51. Found: C, 49.03; H, 4.86; N, 1.52%. IR (KBr): 2970, 2907, 1633, 1576, 1440, 1415, 1376, 1255, 1134, 1087, 1023, 800, 760, 734 cm⁻¹. ¹H NMR (500 MHz, CDCl₃, ppm): δ 7.78 (d, 1H, 2-PyS), 7.20 (d, 1H, 2-PyS), 6.76 (t, 1H, 2-PyS), 6.48 (t, 1H, 2-PyS), 7.03 (t, 2H, PhS), 6.86 (t,

4H PhS), 6.23 (d, 4H PhS), 1.04 (s, 30H, Cp⁺). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 176.54, 149.57, 135.51, 126.10, 117.13 (2-PyS), 130.32–126.62 (PhS), 91.53, 10.64 (Cp⁺).

4.6. Crystal structure determinations

Crystals of **3** and **5** suitable for X-ray crystallography were grown by slow diffusion of hexane into CH₂Cl₂ solution. Diffraction data of **3** and **5** were collected on a Bruker Smart APEX CCD diffractometer (Mo Kα radiation). The structures were solved by direct methods and subsequently refined on F² by using full-matrix least-squares techniques (SHELXL-97), all non-hydrogen atoms were refined anisotropically, and hydrogen atoms were located at calculated positions (see Table 1).

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.jorganchem.2008.06.011.

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