

$[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)$, a binuclear complex containing a nitrosyl bridge without a metal–metal bond

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Abstract

The compounds $(C_5(CH_3)_5)Co(NO)X$ ($X = Cl, Br, I$) have been prepared by reaction of $[(C_5(CH_3)_5)Co]_2(\mu-NO)_2$ with the appropriate dihalogen X_2 . Either by spontaneous decomposition or by addition of half an equivalent of $[(C_5(CH_3)_5)CoCl]_2(\mu-Cl)_2$, the chloro derivative forms the binuclear complex $[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)$, the crystal structure of which indicates the presence of a nitrosyl bridge without a metal–metal bond.

Introduction

While there are vast numbers of carbonyl and nitrosyl complexes with CO or NO ligands bridging two metal centres connected by a single or multiple metal–metal bond ***, CO or NO bridges without an underlying metal–metal bond are exceedingly rare ****. We present here structural data for such a nitrosyl-bridged binuclear complex without a metal–metal bond, the pentamethylcyclopentadienylcobalt compound $[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)$ (**1**).

Results and discussion

The nitrosyl halide derivatives $(C_5(CH_3)_5)Co(NO)X$ ($X = Cl, Br, I$), were prepared by the reaction of $[(C_5(CH_3)_5)Co]_2(\mu-NO)_2$ [6] with a stoichiometric amount

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*** See e.g. ref. 1 and literature cited therein.

**** CO- and NO-bridged complexes without metal–metal bonds have been claimed, without structural characterization, in ref. 2 and in refs. 3 and 4, respectively. NO bridges without an underlying metal–metal bond have been shown by an X-ray study to be present in the trinuclear ruthenium cluster $Ru_3(CO)_{10}(\mu-NO)_2$ (ref. 5).

Table 1

Spectral data for $(C_5(CH_3)_5)Co(NO)X$ ($X = Cl, Br, I$)

| Compound | $\nu(NO)$ (cm^{-1}) in CH_2Cl_2 | 1H NMR (δ in ppm) in C_6D_6 |
|-------------------------|--|---|
| $(C_5(CH_3)_5)Co(NO)Cl$ | 1784 | 0.91 (s) |
| $(C_5(CH_3)_5)Co(NO)Br$ | 1785 | 0.98 (s) |
| $(C_5(CH_3)_5)Co(NO)I$ | 1795 | 1.53 (s) |

of the appropriate dihalogen X_2 . The IR spectra of these compounds in CH_2Cl_2 solution show $\nu(NO)$ bands between 1780 and 1795 cm^{-1} (Table 1). As expected, these frequencies are lower by about 40 cm^{-1} than those reported by Brunner and coworkers [4] for the corresponding species $(C_5H_5)Co(NO)X$. In addition to these terminal NO bands, typical μ -NO bands at 1530 cm^{-1} were present with varying intensities in CH_2Cl_2 solutions of the chloro and bromo (but not the iodo) derivatives. Similar μ -NO bands at 1548 and 1538 cm^{-1} were observed in KBr spectra of $(C_5H_5)Co(NO)Br$ and $(C_5H_5)Co(NO)I$ [4], and were interpreted in terms of an association of the compounds to linear or cyclic polymers.

In trying to crystallize from solutions of $(C_5(CH_3)_5)Co(NO)Cl$ an NO-bridged polymer of this species, we obtained instead a compound of composition $[(C_5(CH_3)_5)CoCl]_2(NO)Cl$ (**1**) with $\nu(NO)$ at 1533 cm^{-1} . This compound is formally an adduct of $(C_5(CH_3)_5)Co(NO)Cl$ with the dichloro derivative $(C_5(CH_3)_5)CoCl_2$. In accord with this picture, we observed that an IR band at 1533 cm^{-1} grew progressively when increasing amounts, up to 0.5 equivalents, of $[(C_5(CH_3)_5)CoCl_2]_2$ [7,8] were added to a CH_2Cl_2 solution of $(C_5(CH_3)_5)Co(NO)Cl$. The mixed dimer **1** was also obtained by treatment of the dimeric cobalt(II) compound $[(C_5(CH_3)_5)CoCl]_2$ [7] with stoichiometric amounts of NO in pentane.

A small crystal of **1** (ca. $0.5 \times 0.4 \times 0.8$ mm) was used to determine space group, cell parameters and X-ray diffraction intensities on a Nonius-CAD 4 diffractometer at room temperature (Mo- K_α , λ 71.069 pm, graphite monochromator). The crystal was monoclinic, space group $P2_1/c$. The unit cell contains 6 molecules; since one molecule is situated on a crystallographic inversion centre, each of the four asymmetric units contains 1.5 molecules. Cell parameters: a 834.1(2), b 1524.9(3), c 2714.0(7) pm; β 94.29(2)°; V 3.4423 nm³; d_{calcd} 1.52 g/cm³. The structure was solved by the Patterson method and refined to $R = 0.078$, using 4225 independent reflections with $I \geq 2.5\sigma(I)$ (programme SHELX 76). Fractional atomic coordinates thus obtained are listed in Table 2, and relevant interatomic distances and bond angles in Table 3*. Figure 1 represents the structures of the independent molecules **1** and **2**. The quality of the structural data obtained is somewhat diminished by a disordering of both molecules. Molecule **2**, in particular, which is positioned on an inversion centre, is completely disordered with respect to the location of its μ -NO and μ -Cl ligands; the inversion-related positions of atoms Cl(33), N(33) and O(33) had to be refined with population parameters of 0.5. From space-filling models of **1**

* Supplemental structural data are available on request from Fachinformationszentrum Energie Physik Mathematik, D-7514 Eggenstein-Leopoldshafen 2, upon citation of deposit No. CSD 52645, the names of the authors and the journal reference for this article.

Table 2

Structural data for $[(C_5(CH_3)_5)CoCl]_2(\mu\text{-NO})(\mu\text{-Cl})$. Fractional atomic coordinates (with e.s.d.'s) and thermal parameters

| Atom | x | y | z | $U(\text{eq})^a$ |
|---------|------------|------------|------------|------------------|
| Co(1) | 0.3280(2) | 0.0931(1) | 0.3466(1) | 0.0350(7) |
| Co(2) | 0.5462(2) | -0.0616(1) | 0.3187(1) | 0.0387(8) |
| Cl(1) | 0.2122(5) | 0.1096(3) | 0.2697(1) | 0.084(3) |
| Cl(2) | 0.6425(4) | -0.0839(2) | 0.3975(1) | 0.059(2) |
| Cl(12) | 0.5896(7) | 0.0918(3) | 0.3189(3) | 0.051(3) a |
| N(12) | 0.357(2) | -0.0256(8) | 0.3394(5) | 0.042(8) a |
| O(12) | 0.245(2) | -0.074(1) | 0.3472(7) | 0.054(9) a |
| Cl(12') | 0.273(2) | -0.0590(8) | 0.3396(9) | 0.059(6) b |
| N(12') | 0.521(4) | 0.055(2) | 0.329(1) | 0.04 b |
| O(12') | 0.617(5) | 0.108(2) | 0.317(2) | 0.05 b |
| C(101) | 0.188(2) | 0.1996(9) | 0.3703(4) | 0.063(9) |
| C(102) | 0.145(1) | 0.1193(9) | 0.3929(4) | 0.050(7) |
| C(103) | 0.283(1) | 0.0886(7) | 0.4208(4) | 0.034(5) |
| C(104) | 0.407(1) | 0.1474(7) | 0.4137(4) | 0.034(6) |
| C(105) | 0.351(2) | 0.2170(8) | 0.3829(4) | 0.049(7) |
| C(111) | 0.063(3) | 0.255(1) | 0.3389(6) | 0.13(2) |
| C(112) | -0.018(1) | 0.072(1) | 0.3908(7) | 0.10(1) |
| C(113) | 0.294(2) | 0.0101(8) | 0.4552(5) | 0.055(8) |
| C(114) | 0.579(1) | 0.1408(9) | 0.4402(5) | 0.058(8) |
| C(115) | 0.456(2) | 0.2943(9) | 0.3682(6) | 0.10(1) |
| C(201) | 0.567(2) | -0.1902(8) | 0.2947(5) | 0.056(8) |
| C(202) | 0.461(1) | -0.137(1) | 0.2603(5) | 0.074(9) |
| C(203) | 0.568(2) | -0.0706(8) | 0.2439(4) | 0.055(8) |
| C(204) | 0.717(2) | -0.0832(9) | 0.2634(5) | 0.064(9) |
| C(205) | 0.718(1) | -0.1500(9) | 0.2934(4) | 0.051(7) |
| C(211) | 0.520(4) | -0.269(1) | 0.3261(9) | 0.17(2) |
| C(212) | 0.286(2) | -0.148(3) | 0.242(1) | 0.29(3) |
| C(213) | 0.517(4) | -0.001(1) | 0.2043(6) | 0.15(2) |
| C(214) | 0.863(2) | -0.026(1) | 0.2524(7) | 0.10(1) |
| C(215) | 0.873(2) | -0.189(2) | 0.3231(7) | 0.12(1) |
| Co(3) | 0.0219(2) | 0.0939(1) | 0.0194(7) | 0.0382(8) |
| Cl(3) | -0.1236(5) | 0.1541(2) | -0.0449(1) | 0.079(2) |
| Cl(33) | 0.183(2) | 0.0195(8) | -0.0367(4) | 0.051(5) c |
| N(33) | -0.097(2) | -0.004(2) | 0.0173(9) | 0.04(1) c |
| O(33) | -0.214(5) | 0.004(2) | 0.029(2) | 0.08(2) c |
| C(301) | 0.063(2) | 0.2232(9) | 0.0500(6) | 0.069(9) |
| C(302) | -0.019(2) | 0.168(1) | 0.0821(6) | 0.08(1) |
| C(303) | 0.077(2) | 0.097(1) | 0.0948(4) | 0.061(8) |
| C(304) | 0.222(1) | 0.1030(8) | 0.0697(4) | 0.044(6) |
| C(305) | 0.209(1) | 0.1818(8) | 0.0413(40) | 0.045(6) |
| C(311) | 0.012(3) | 0.311(1) | 0.0257(8) | 0.14(2) |
| C(312) | -0.188(2) | 0.187(2) | 0.100(1) | 0.19(2) |
| C(313) | 0.043(3) | 0.027(1) | 0.1321(5) | 0.11(1) |
| C(314) | 0.366(2) | 0.042(1) | 0.0743(6) | 0.075(9) |
| C(315) | 0.343(2) | 0.215(1) | 0.0106(7) | 0.11(1) |

^a Special population parameters: a 0.72(6), b 0.28(6), c 0.5; Cl(12'), N(12') and O(12') were refined isotropically, the two latter with a fixed temperature parameter; all other U values are equivalent isotropic values calculated from the anisotropic parameters.

Table 3

Bond lengths (in pm) and bond angles (in degrees) for $[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)^a$. Data in square brackets refer to refinement without partial disordering for molecule 1 (see text)

| Molecule 1 | | | Molecule 2 | | |
|--------------------|-------------|---------|----------------------|------------|--|
| Co(1)–Co(2) | 310.8(2) | | Co(3)–Co(3') | 306.4(2) | |
| Co(1)–Cl(1) | 224.7(4) | | Co(3)–Cl(3) | 224.6(4) | |
| Co(2)–Cl(2) | 225.3(3) | | | | |
| Co(1)–Cl(12) | 236.1(7) | [236] | Co(3)–Cl(33) | 239(1) | |
| Co(2)–Cl(12) | 236.7(5) | [238] | Co(3')–Cl(33) | 250(1) | |
| Co(1)–N(12) | 184(1) | [191] | Co(3)–N(33) | 178(2) | |
| Co(2)–N(12) | 180(2) | [191] | Co(3')–N(33) | 184(2) | |
| N(12)–O(12) | 122(2) | [96] | N(3')–O(3') | 106(5) | |
| Co(1)–C(Cp1) | 201(1) ± 3 | | | | |
| Co(2)–C(Cp2) | 209(1) ± 6 | | Co(3)–C(Cp3) | 210(1) ± 5 | |
| C(Cp1)–C(Cp2) | 141(2) ± 1 | | | | |
| C(Cp2)–C(Cp2) | 140(2) ± 10 | | C(Cp3)–C(Cp3) | 142(2) ± 2 | |
| C(Cp1)–C(Me) | 153(2) ± 2 | | | | |
| C(Cp2)–C(Me) | 155(2) ± 2 | | C(Cp3)–C(Me) | 154(2) ± 2 | |
| Cl(1)–Co(1)–Cl(12) | 92.8(2) | [92.8] | Cl(3)–Co(3)–Cl(33) | 89.8(3) | |
| Cl(1)–Co(1)–N(12) | 93.6(4) | [92.5] | Cl(3)–Co(3)–N(33) | 92.8(7) | |
| Cl(12)–Co(1)–N(12) | 80.0(5) | [85.0] | Cl(33)–Co(3)–N(33) | 85.5(8) | |
| Cl(2)–Co(2)–Cl(12) | 95.9(2) | [96.0] | Cl(3')–Co(3')–Cl(33) | 95.3(3) | |
| Cl(2)–Co(2)–N(12) | 90.5(4) | [90.0] | Cl(3')–Co(3')–N(33) | 94.1(7) | |
| Cl(12)–Co(2)–N(12) | 80.5(4) | [84.6] | Cl(33)–Co(3')–N(33) | 81.3(8) | |
| Co(1)–Cl(12)–Co(2) | 82.2(2) | [81.0] | Co(3)–Cl(33)–Co(3') | 77.6(4) | |
| Co(1)–N(12)–Co(2) | 117.2(7) | [108.5] | Co(3)–N(33)–Co(3') | 116(1) | |
| Co(1)–N(12)–O(12) | 118(1) | [123] | Co(3)–N(33)–O(33) | 115(3) | |
| Co(2)–N(12)–O(12) | 125(1) | [129] | Co(3')–N(33)–O(33) | 128(3) | |

^a A prime indicates a symmetry related atom at \bar{x} , \bar{y} , \bar{z} .

it is apparent that the Van der Waals envelopes of the NO and Cl bridges are sufficiently similar to accommodate both orientations of the molecule in this crystallographic position. Owing to the close proximity of O(33') and Cl(33), the coordinates of these two atoms are probably subject to considerable errors. This would, at least, explain the relatively high unsymmetry of the bridging system of this molecule.

In the early stages of structure solution, molecule 1 seemed to be free from disorder: The Fourier synthesis showed three large peaks in the bridging region; their positions and intensities led to a straightforward assignment as a Cl and an NO bridge. However, when anisotropic refinement of all atoms had converged at an *R*-value of 8.2%, the following observations indicated that there was a certain degree of disorder for molecule 1 also: an unusual distribution of temperature factors within the NO-bridge ($U(\text{eq})_N = 1.6 \cdot U(\text{eq})_O$); a distinctly undervalued N–O distance (96 pm), accompanied by relatively large Co–N distances (191 pm); and a relatively high residual electron density ($1.5 \text{ e}/10^6 \text{ pm}^3$) within the Co(1)–Co(2)–Cl(12) triangle.

A refinement including a partial disordering of Cl(12), N(12) and O(12) and using constrained lengths for chemically equivalent bonds within molecule 1 gave the following results: a normal distribution of temperature factors for the NO

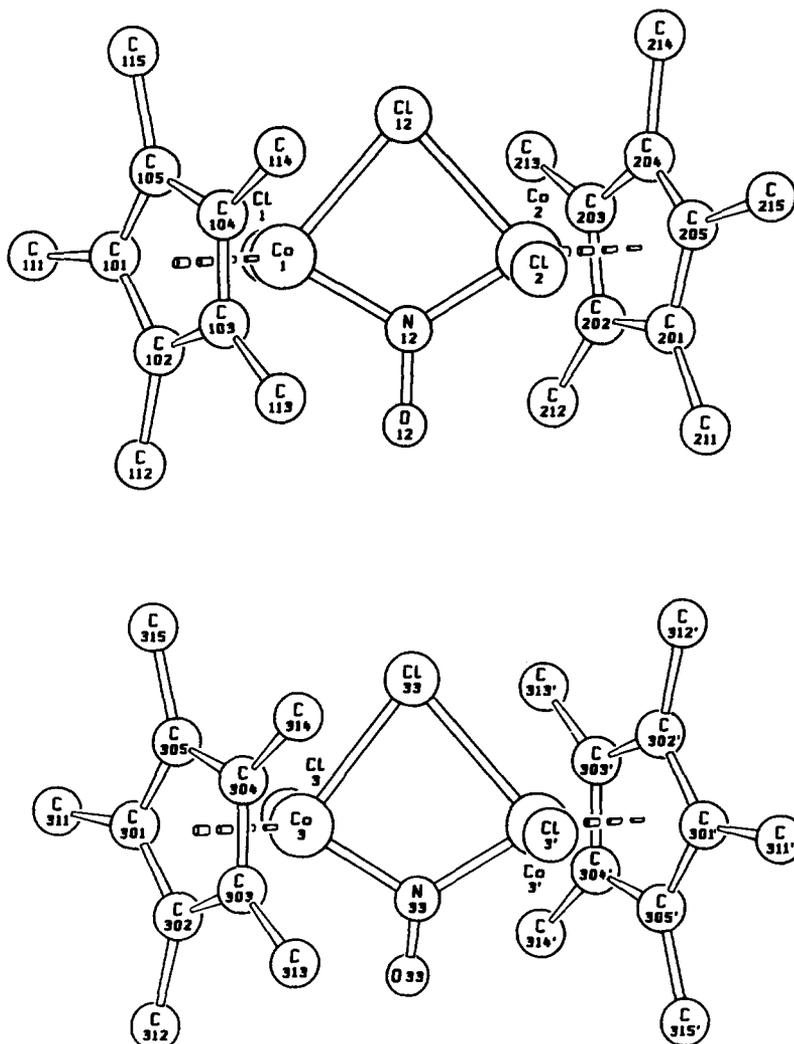


Fig. 1. Molecular structures of the two crystallographically independent molecules of $[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)$.

bridge; more reasonable bond lengths; and a reduced maximum residual electron density ($1.2 e/10^6 \text{ pm}^3$). The population ratio refined to 0.72/0.28; the final R value was 7.8%.

As a final check for the correctness of the model, a ΔF synthesis was calculated based on the refined atomic parameters but with all Cl, N and O atoms removed from the model. The 6 strongest peaks are listed in Table 4, together with the corresponding atom(s) for the complete model. It is evident that the peak height relations are in fair agreement with the corresponding relations between the amounts of formally contributing electrons (an even better agreement would be obtained for a population ratio of 0.80/0.20 for molecule 1).

In each of the two crystallographically independent molecules of **1**, two $(C_5(CH_3)_5)CoCl$ units are bridged by one Cl and one NO bridge. The terminal Co–Cl bonds are shorter by about 15 pm than the Co–(μ -Cl) bonds, as expected

Table 4

Largest peaks in a difference Fourier synthesis with all bridging atoms omitted

| Peak No. | Electron density ($e/10^6 \text{ pm}^3$) | Contributing atoms | Distance to peak (pm) | Contributing electrons (e) |
|----------|--|---------------------------|-----------------------|----------------------------|
| 1 | 16.3 | 0.72 Cl(12) 0.28 O(12) | 3 31 | 14.5 |
| 2 | 10.6 | 0.50 Cl(33) 0.50 O(33) | 4 42 | 12.5 |
| 3 | 9.2 | 0.28 Cl(12) 0.72 O(12) | 21 20 | 10.5 |
| 4 | 5.3 | 0.72 N(12) | 5 | 5.0 |
| 5 | 3.5 | 0.50 N(33) | 10 | 3.5 |
| 6 | 1.7 | 0.28 N(12) | 23 | 2.0 |

from related data on other metal halide compounds *. The Co–N bond distances of 178–184 pm are close to those reported for other cyclopentadienyl Co complexes with bridging NO ligands [10–12].

The Co–Co distances of 306 and 311 pm in molecules 1 and 2, however, are far beyond the range of ca. 233 to 256 pm reported for binuclear cobalt compounds containing cobalt–cobalt bonds of various formal bond orders [13]. We have to conclude, in accord with the 18-electron rule, that the interaction between the two cobalt(III) centres of each molecule of 1 does not involve a cobalt–cobalt bond **. The mixed dimer 1 appears to be the first structurally characterised binuclear nitrosyl complex which contains an NO bridge between two non-bonded metal centres. Since halide ligands, on the other hand, occur quite frequently in bridging positions between otherwise non-bonded metal centers, the presence of the μ -Cl ligand in 1 may well be an important factor in stabilizing such an NO bridge. The ready conversion of the dimeric dichloride $[(C_5(CH_3)_5)CoCl]_2(\mu-Cl)_2$ into the mixed dimer $[(C_5(CH_3)_5)CoCl]_2(\mu-NO)(\mu-Cl)$ (1) upon exposure to $(C_5(CH_3)_5)Co(NO)Cl$ indicates that the μ -NO bridge in 1 serves as an even better point of attachment for the Lewis-acidic cobalt(III) centre of $(C_5(CH_3)_5)CoCl_2$ than does a Cl ligand in the dimer of this dichloride.

Experimental

All manipulations were carried out under argon in Schlenk-type glassware; all solvents were thoroughly dried and freed from air. $[(C_5(CH_3)_5)Co]_2(\mu-NO)_2$ was prepared as described in ref. 6, and $(C_5(CH_3)_5)Co(CO)_2$ by the CO-induced disproportionation of $[(C_5(CH_3)_5)Co]_2(\mu-Cl)_2$ (cf. ref. 7).

1. $[(C_5(CH_3)_5)Co]_2(\mu-Cl)_2$. The preparations reported in ref. 7 and 8 were modified as follows. To a suspension of 36.4 g (0.28 mol) anhydrous $CoCl_2$ in THF at room temperature are added 119.3 g (0.28 mol) of tri-*n*-butylpentamethylcyclopentadienyltin (prepared by reaction of 40 g pentamethylcyclopentadienyllithium with 120 g tri-*n*-butyltin chloride in 500 ml THF and subsequent distillation at

* See e.g. ref. 9 and literature cited therein.

** An even larger Co–Co distance of 347.0 pm has been reported for a di- $(\mu-Cl)$ -bridged ethanediol complex of cobalt(II) (ref. 14).

110–115°C/0.01 Torr). The reddish-brown mixture is stirred overnight and the dark precipitate then filtered off, washed several times with 15 ml portions of n-pentane, and dried in vacuo to yield 60 g (93% theoretical yield) of $[(C_5(CH_3)_5Co)_2(\mu-Cl)_2]$ as a black powder.

2. $(C_5(CH_3)_5Co(NO)Cl)$. To a well-stirred solution of 300 mg (0.7 mmol) $[(C_5(CH_3)_5Co)_2(\mu-NO)_2]$ [6] in about 15 ml n-hexane at room temperature, 16 ml of Cl_2 gas are added with a syringe through a rubber septum during 1 h. The dark green precipitate is filtered off, washed twice with 5 ml n-hexane, and dried in vacuo, to yield 210 mg (ca. 60% theoretical yield) of $(C_5(CH_3)_5Co(NO)Cl)$. Elemental analysis: Found: C, 44.96; H, 5.15; N, 4.98; Cl 13.99. $C_{10}H_{15}ClCoNO$ calcd.: C, 46.26; H, 5.82; N, 5.40; Cl 13.66%. Mass spectrum with parent ion at m/e 259–262 and appropriate isotope distribution, and further peaks at m/e 229–232 ($M - NO$), 329–330 ($(C_5(CH_3)_5)_2Co$) and 448–449 ($[(C_5(CH_3)_5Co)NO]_2$).

3. $(C_5(CH_3)_5Co(NO)Br)$. Treatment of 280 mg (0.62 mmol) $[(C_5(CH_3)_5Co)(\mu-NO)_2]$ with a solution of 100 mg (0.62 mmol) Br_2 in 0.7 ml n-hexane, followed by work-up as in experiment 2 above yields 220 mg (58% theoretical yield) of $(C_5(CH_3)_5Co(NO)Br)$ as a black powder. The product can be further purified by sublimation at 80°C in vacuo. Elemental analysis: Found: C, 40.13; H, 4.96; N, 4.44. $C_{10}H_{15}BrCoNO$ calcd.: C, 39.50; H, 4.97; N, 4.61%. Mass spectrum with parent ion at m/e 303–306 with appropriate isotope distribution and further peaks at m/e 273–376 ($M - NO$) and 497–500 ($[(C_5(CH_3)_5Co)_2(NO)Br]$).

4. $(C_5(CH_3)_5Co(NO)I)$. To a stirred solution of 3.3 g (7.3 mmol) $[(C_5(CH_3)_5Co)_2(\mu-NO)_2]$ in 25 ml diethyl ether at room temperature a solution of 1.87 g (7.3 mmol) I_2 in 15 ml diethyl ether is added, during 2.5 h. Work-up as in section 1 yields 1.56 g (32% theoretical yield) of black $(C_5(CH_3)_5Co(NO)I)$, which can be further purified by sublimation at 140°C in vacuo. Elemental analysis: Found: C, 34.19; H, 4.60; N, 3.85. $C_{10}H_{15}CoINO$ calcd.: C, 34.21; H, 4.31; N, 3.99%. Mass spectrum with parent ion at m/e 351–353 with appropriate isotope distribution and a further peak at m/e 321–322 ($M - NO$).

5. $[(C_5(CH_3)_5CoCl)_2(\mu-NO)(\mu-Cl)]$. To a stirred solution of 1.9 g (4.1 mmol) $[(C_5(CH_3)_5Co)(\mu-Cl)_2]$ [7] in ca. 30 ml n-pentane at $-90^\circ C$, 185 ml (8.3 mmol) of NO gas is introduced from a gas syringe. Partial evaporation of solvent at room temperature yields a greenish-brown precipitate, which is filtered off and dissolved in ca. 5 ml CH_2Cl_2 . This solution is exposed under N_2 to the vapour of about 100 ml of n-pentane. After about 2 months of diffusion-induced crystallisation, 285 mg (18% theoretical yield) of 1 are formed as black crystalline platelets.

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