

Mono-indenyl and cyclopentadienyl derivatives of molybdenum(IV) with a 16 valence-electron configuration

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Abstract

Reactions of (ring)MoCl₃ [ring = cyclopentadienyl, η⁵-C₅H₅ (Cp) and indenyl η⁵-C₉H₇ (Ind)] with salts of the *N,N*-diethyldithiocarbamate (S₂CNEt₂)[−], *O,O'*-diethyldithiophosphate [S₂P(OEt)₂][−], *N,N'*-diphenylformamidinate [CH(NPh)₂][−], acetate (O₂CCH₃)[−] and acetylacetonate [CH₃COCH(O)CH₃][−] (acac)[−] anions yield the molybdenum(IV) complexes of the type (ring)MoCl₂X. Characterisation of new compounds by elemental analysis, IR and ¹H-NMR spectroscopy and magnetic susceptibility measurements is discussed.

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1. Introduction

To date, the chemistry of monocyclopentadienyl molybdenum(IV) complexes and its ring-substituted analogues remains relatively undeveloped and can be confined to a handful of compounds. Without being exhaustive, we believe that the earlier examples are the dicarbonyls CpMoX₃(CO)₂ (X = Cl, Br, I) obtained by X₂ oxidation of either Cp₂Mo₂(CO)₆ or CpMo(CO)₃X [1]. The cyanide derivative CpMo(CN)₃(CO)₂ was obtained by CN[−] substitution on the parent iodide [2]. Related phosphine derivatives, CpMoX₃L₂ (L₂ = dppe, dmpe; L = PMe₃, PMe₂Ph; X = Cl, Br, H) were also reported by the group of Green [3]. The trihydrides with L = dmpe, PMe₃, PMe₂Ph induce catalytic H/D exchange in sp³ and sp² C–H bonds under photolytic conditions [4]. However, the synthesis of the simple derivatives CpMoX₃ (X = Cl, Br or I) [5] and (η⁵-C₅Me₅)MoCl₃ by Poli and coworkers [6], is considerably more recent. It comprises a variety of synthetic methods

(oxidation of CpMCl₂; reduction of CpMCl₄ and conproportionation of CpMCl₂ and CpMCl₄ and covers a number of phosphine adducts of the type Cp^{*}MX₃L and Cp^{*}MX₃L₂ (L = dppe [7], dmpe [7] and PMe₂Ph [4]) either as the chlorides or hydrides. The more exotic Cp^{*}MoHCl(PPh₂)(PMe₃) also belongs to this family [8], as well as [CpMoCl₄][−] [9] and the related family of thiolates, [CpMo(SR)₄][−], e.g. [CpMo{S₂C₂(CN)₂]₂][−] [10] and [CpMo{SC₆F₅]₄][−] [11]. Recently, Royo and coworkers prepared the *O,O'*-diethyl-dithiophosphate derivative Cp^{*}MoCl₂{S₂P(OEt)₂} by reduction of Cp^{*}MoCl₃{S₂P(OEt)₂} [12].

The mono-indenyl analogues are still fewer. IndMoCl₃ was first cited in patent literature [13] and we recently reported its synthesis via decarbonylation of IndMoCl₃(CO)₂ according to the procedure discovered by Poli for CpMoX₃ [14].

In order to extend the scope of the chemistry of the (ring)Mo(IV) fragment, we decided to explore the synthesis of (ring)M(IV)L_{*n*} complexes directly from the available starting trihalides, (ring)MoCl₃ a method so far only used for some of the CpMoX₃(PR₃)₂ complexes mentioned above. In this work we report the synthesis of half-sandwich molybdenum(IV) complexes of the type (ring)MoCl₂X [ring = cyclopentadie-

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nyl, $\eta^5\text{-C}_5\text{H}_5$ (Cp) or indenyl $\eta^5\text{-C}_9\text{H}_7$ (Ind) and X = S_2CNET_2 , $\text{S}_2\text{P(OEt)}_2$, CH(NPh)_2 , O_2CCH_3 and acac.

2. Results and discussion

The reaction of (ring)MoCl₃ (ring = Cp and Ind) with the ammonium salt of the *O,O'*-diethyldithiophosphate [$\text{S}_2\text{P(OEt)}_2$][−] anion affords the molybdenum(IV) dithiophosphate complexes (ring)MoCl₂[$\text{S}_2\text{P(OEt)}_2$] [ring = Cp (**1**); Ind (**2**)] (see Scheme 1). Their IR spectra show a $\nu(\text{PS})$ absorption at 640 and 644 cm^{-1} attributable to a chelated dithiophosphate ligand [12]. Similar reaction carried out using the silver salt of *N,N*-diethyldithiocarbamate ligand $\text{AgS}_2\text{CNET}_2$ in dichloromethane affords the dithiocarbamate complexes (ring)MoCl₂[S_2CNET_2] [ring = Cp (**3**); Ind (**4**)] (see Scheme 1). The IR spectra of both dithiocarbamate complexes **3** and **4** show a strong $\nu(\text{CN})$ absorption at 1518 and 1512 cm^{-1} and a $\nu(\text{CS})$ absorption at 1076 and 1075 cm^{-1} , respectively, attributable to a bidentate [S_2CNET_2] ligand [12].

We have extended our studies to the synthesis of other molybdenum(IV) complexes with isoelectronic pseudo-allylic ligands containing nitrogen and oxygen atoms instead of sulphur. The reaction of (ring)MoCl₃ with one equivalent of thallium acetylacetonate or silver

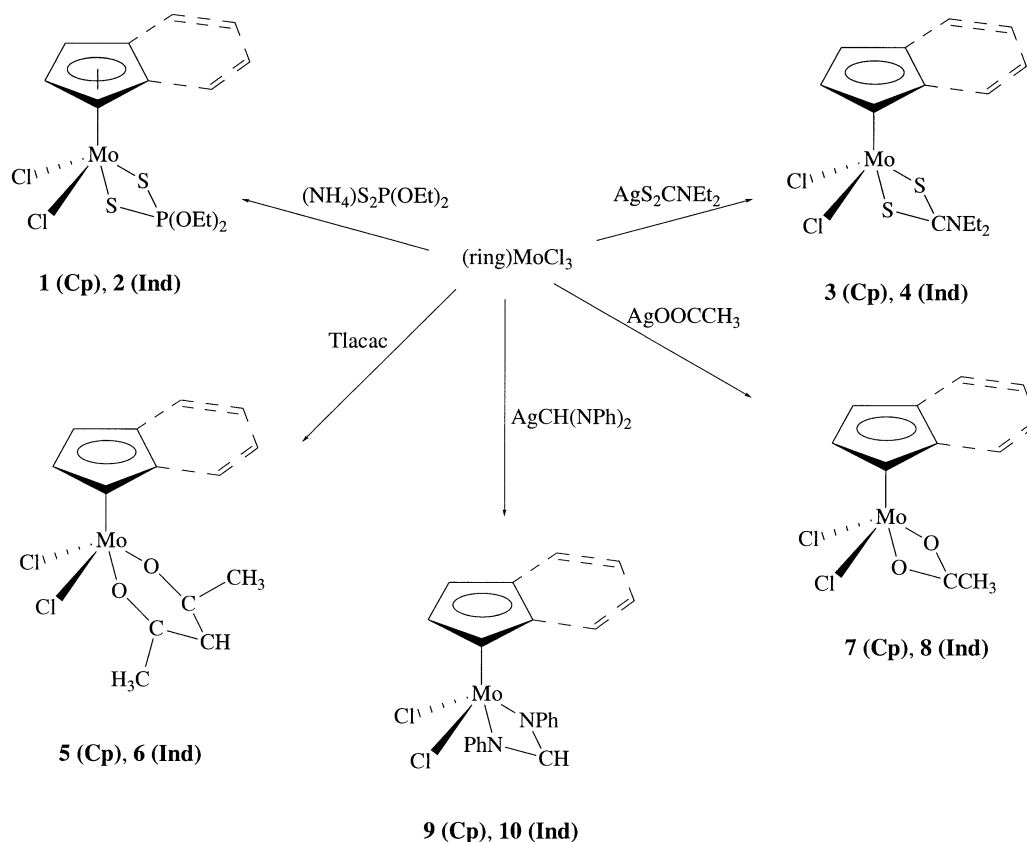
acetate in dichloromethane yields the corresponding (ring)MoCl₂(acac) [ring = Cp (**5**); Ind (**6**)] and (ring)MoCl₂(OOCCH₃) [ring = Cp (**7**); Ind (**8**)], respectively (see Scheme 1).

The formulation of complexes **1–8** as mononuclear 16 electron compounds is consistent with their spectral properties and magnetic behaviour. Thus, all these complexes are paramagnetic with magnetic values in accord with the spin-only value calculated for two unpaired electrons (2.83 μ_{B}). The paramagnetic behaviour for complexes **1–8** with values between 2.14 and 2.83 BM at 300 K is consistent with the spin triplet state expected for a pseudo-square pyramidal compound [15].

Magnetic moments for (ring)Mo(IV) compounds with this geometry have only been reported for the following 16 electron species: Cp*MoCl₃(PMe₃) [6] ($\mu_{\text{eff}} = 2.63 \mu_{\text{B}}$) and Cp*MoCl₂[$\text{S}_2\text{P(OEt)}_2$] [12] ($\mu_{\text{eff}} = 2.75 \mu_{\text{B}}$).

In the case of the dithiophosphate derivative **1**, the monomeric formulation has also been confirmed by MALDI-TOF mass spectrometry.

¹H-NMR was recorded for compounds **1–8**. Their spectra showed broad resonances, in the range between δ 1 and 8 ppm, with unresolved H–H coupling due to line broadening, except for the dithiocarbamates CpMoCl₂[S_2CNET_2] (**3**) and IndMoCl₂[S_2CNET_2] (**4**). The ¹H-NMR spectrum of **3** showed one signal for the cyclopentadienyl ring protons and two sets of signals for



non-equivalent ethyl groups, due to the different *endo* and *exo* orientations of the two ethyl groups. The $^1\text{H-NMR}$ spectrum of **4** showed three resonances for the indenyl ligand at δ 5.41, 5.61 and 7.18–7.31 and two sets of signals for non-equivalent ethyl groups.

The reaction of $(\text{ring})\text{MoCl}_3$ with one equivalent of silver formamidinate $\text{AgCH}(\text{NPh})_2$ in dichloromethane affords $(\text{ring})\text{MoCl}_2[\text{CH}(\text{NPh})_2]$ [$\text{ring} = \text{Cp}$ (**9**); Ind (**10**)]. In contrast to the other mononuclear $(\text{ring})\text{Mo(IV)}$ complexes described herein, the amidinato complexes **9** and **10** are diamagnetic, as evidenced by sharp, well resolved, $^1\text{H-NMR}$ spectra. This quite likely reflects the presence of a strong field ligand. A pseudo-square pyramidal molybdenum with a 16 electron configuration (a d^2 metal) can in principle be found in two different spin states: a diamagnetic state, where the two metal electrons are coupled in the d_{xy} orbital and the d_{z^2} orbital remains empty, or a triplet state, where the two metal frontier orbitals hold one electron each [16]. $(\text{ring})\text{Mo(IV)}$ complexes of the types I, II and III are usually paramagnetic with a triplet state (see Fig. 1). Exceptions have been reported in the literature when strong π -donor ligands are present. This is the case of $\text{Cp}^*\text{MoHCl}(\text{PPh}_2)(\text{PMe}_3)$ [8] which has a singlet ground state, certainly the result of the strong π -interaction of the phosphido ligand. Similar diamagnetic behaviour is observed for the tetrathiolato derivatives $[\text{CpMo}(\text{SR})_4]^-$ [17]. The isoelectronic $\text{Cp}^*\text{ReCl}_3\text{Me}$ derivative has been reported to exhibit spin-crossover behaviour [18]. The $^1\text{H-NMR}$ spectrum of the amidinato complex **10** was recorded at 90°C showing unshifted resonances which indicates a singlet ground state. At higher temperature decomposition of the sample was observed.

The $^1\text{H-NMR}$ spectrum of **9** shows a singlet at δ 8.10 for the proton of the CH and a multiplet at δ 7.17–7.38 for the phenyl protons of the amidinato ligand. The cyclopentadienyl protons give a resonance at δ 5.23. Compound **10** gives a singlet at δ 8.35 for the proton of the CH and a multiplet at δ 7.07–7.48 for the phenyl protons of the amidinato ligand and a doublet at δ 5.59 ($\text{H}^{1/3}$), a triplet at δ 5.41 (H^2) and a multiplet at δ 6.88 (H^{5-8}) for the indenyl ligand (see Fig. 2).

All the complexes **1–10** are air-sensitive compounds. They can be stored as a solids under inert atmosphere

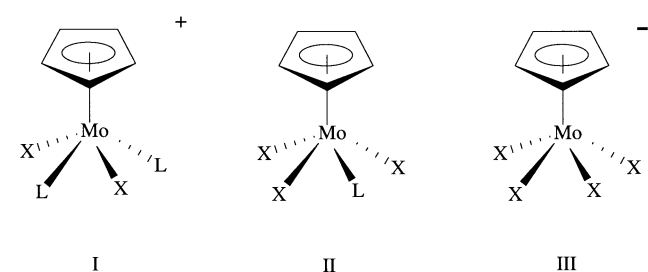


Fig. 1. Mo(IV) complexes of the type $(\text{ring})\text{MoX}_2\text{L}_2$ (I), $(\text{ring})\text{MoX}_3\text{L}$ (II) and $(\text{ring})\text{MoX}_4$ (III).

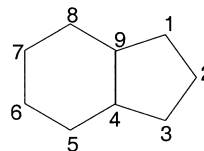


Fig. 2. Labelled carbon atoms of the indenyl ring.

for months but decomposition is observed if stored in solution. They are soluble in dichloromethane and insoluble in hexane, pentane, toluene and ether. Unfortunately, efforts to obtain single crystals for X-ray diffraction studies were unsuccessful.

3. Conclusion

The easily available $(\text{ring})\text{MoCl}_3$ compounds can be used directly to prepare a number of derivatives in which one bidentate ligand replaces one chloride forming pentacoordinate complexes of general formula $(\text{ring})\text{MoCl}_2\text{X}$ ($\text{X} =$ bidentate anionic N_2 , O_2 , S_2 donors). With the exception of the formamidinate derivatives all complexes are paramagnetic with two unpaired electrons.

4. Experimental

4.1. Materials and procedures

All operations were carried out under an atmosphere of dinitrogen with standar Schlenk-line and glove-box techniques. Solvents were purified by conventional methods and distilled under nitrogen prior to use. NMR spectra were measured on a Bruker CXP 300 spectrometer, and IR spectra were measured on a Unicam Mattson model 7000 FTIR spectrometer. Elemental analyses were performed in our laboratories (ITQB). Magnetic moments have been determined at room temperature (r.t.) with a Johnson–Matthey magnetic susceptibility balance and were corrected for diamagnetic contributions of the ligands by using Pascal's constants before conversion to magnetic moments. MALDI mass measurements were performed on a Reflex time-of-flight instrument (Bruker-Franzen Analytik, Bremen, Germany) equipped with a SCOUT ion source, operating in the positive reflection mode with pulsed extraction. Ions were formed by a pulsed UV laser beam (nitrogen laser, $\lambda = 337$ nm). No matrix was required for the ionization of the sample. External mass calibration was made by using the $[\text{M} + \text{H}]^+$ ion of α -cyano-4-hydroxycinnamic and angiotensin II.

All common chemicals and solvents were purchased from commercial suppliers. The synthesis of N,N' -diphenylformamidinato [19], CpMoCl_3 [5] and In-

dMoCl_3 [14] was prepared according to literature procedure. Thallium acetylacetonate was prepared by reaction of acetylacetone with thallium ethoxide in toluene at r.t.

4.2. Preparation of $\text{CpMoCl}_2[\text{S}_2\text{P}(\text{OEt})_2]$ (1)

Solid ammonium diethyldithiophosphate (0.21 g, 1.04 mmol) was added to a suspension of CpMoCl_3 (0.28 g, 1.04 mmol) in 40 ml of CH_2Cl_2 at r.t. The purple-brown color of the suspension changed to give a brown suspension after stirring for 10 h. The reaction mixture was filtered and the filtrate was concentrated to dryness. The solid residue was washed with Et_2O (3×5 ml). Compound **1** was isolated as a brown solid. Yield: 0.35 g (82%). Selected IR (KBr, cm^{-1}): 1006, vs, 960, vs $\nu(\text{POC})_{\text{asym}}$, 812, vs, 792, vs $\nu(\text{POC})_{\text{sym}}$, 640, vs, $\nu(\text{PS})$. Anal. Calc. for $\text{C}_9\text{H}_{15}\text{Cl}_2\text{MoO}_2\text{PS}_2$: C, 25.91; H, 3.62. Found: C, 26.02; H, 3.52%. $\mu_{\text{eff}} = 2.45 \mu_{\text{B}}$. MS: 418 [M^+].

4.3. Preparation of $\text{IndMoCl}_2[\text{S}_2\text{P}(\text{OEt})_2]$ (2)

Solid ammonium diethyldithiophosphate (0.19 g, 0.94 mmol) was added to a stirred suspension of IndMoCl_3 (0.3 g, 0.94 mmol) in 30 ml of CH_2Cl_2 at r.t. The reaction was completed after 10 h stirring leading a brown solution. The solution was filtered and the filtrate was concentrated to dryness to give **2** as a brown solid, which was washed with Et_2O (3×5 ml). Yield: 0.35 g (80%). Selected IR (KBr, cm^{-1}): 1008, vs, 956, vs, $\nu(\text{POC})_{\text{asym}}$, 758, vs, $\nu(\text{POC})_{\text{sym}}$, 644, vs $\nu(\text{PS})$. Anal. Calc. for $\text{C}_{13}\text{H}_{17}\text{Cl}_2\text{MoO}_2\text{PS}_2$: C, 33.42; H, 3.67. Found: C, 33.26; H, 3.58%. $\mu_{\text{eff}} = 2.77 \mu_{\text{B}}$.

4.4. Preparation of $\text{CpMoCl}_2(\text{S}_2\text{CNEt}_2)$ (3)

Solid silver diethyldithiocarbamate (0.28 g, 1.12 mmol) was added to a stirred suspension of CpMoCl_3 (0.3 g, 1.12 mmol) in 40 ml of CH_2Cl_2 at r.t. The reaction mixture was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness to yield the title compound **3** as a brown solid, which was washed with Et_2O (3×5 ml). Yield: 0.36 g (86%). Selected IR (KBr, cm^{-1}): 1518, vs, $\nu(\text{CN})_{\text{asym}}$, 1076, vs, $\nu(\text{CS})$. Anal. Calc. for $\text{C}_{10}\text{H}_{15}\text{Cl}_2\text{MoNS}_2$: C, 31.59; H, 3.98; N 3.68. Found: C, 31.59; H, 3.83; N, 3.73%. $^1\text{H-NMR}$ (C_6D_6 , 25 °C, δ ppm): 4.89 (s, Cp); 3.10 (br m, ethyl); 0.66 (br m, ethyl), 0.78 (br m, ethyl). $\mu_{\text{eff}} = 2.14 \mu_{\text{B}}$.

4.5. Preparation of $\text{IndMoCl}_2(\text{S}_2\text{CNEt}_2)$ (4)

Solid silver dithiocarbamate (0.24 g, 0.94 mmol) was added to a stirred suspension of IndMoCl_3 (0.3 g, 0.94 mmol) in 40 ml of CH_2Cl_2 at r.t. The reaction mixture

was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness to yield the title compound **4** as a brown solid which was washed with Et_2O (3×5 ml). Yield: 0.34 g (85%). Selected IR (KBr, cm^{-1}) 1512, vs, $\nu(\text{CN})_{\text{asym}}$, 1075, vs, $\nu(\text{CS})$. Anal. Calc. for $\text{C}_{14}\text{H}_{17}\text{Cl}_2\text{MoNS}_2$: C, 39.08; H, 3.98; N, 3.26. Found: C, 39.02; H, 3.83; N, 2.97%. $^1\text{H-NMR}$ (C_6D_6 , 25 °C, δ ppm): 7.18–7.31 (m, H^{5-8}); 5.61 (d, $\text{H}^{1/3}$); 5.41 (t, 1H, H^2); 3.93 (br m, ethyl); 1.55 (br m, ethyl), 1.26 (br m, ethyl). $\mu_{\text{eff}} = 2.63 \mu_{\text{B}}$.

4.6. Preparation of $\text{CpMoCl}_2(\text{acac})$ (5)

To a suspension of CpMoCl_3 (0.3 g, 1.12 mmol) in 50 ml of CH_2Cl_2 was added $\text{Ti}(\text{acac})$ as a solid (0.34 g, 1.12 mmol) at r.t. The reaction mixture was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness and the residue was washed with Et_2O (3×5 ml) to yield the title compound **5** as a brown solid. Yield: 0.28 g (76%). Selected IR (KBr, cm^{-1}): 1521, vs, $\nu(\text{CO})$. Anal. Calc. for $\text{C}_{10}\text{H}_{12}\text{Cl}_2\text{MoO}_2$: C, 36.28; H, 3.65. Found: C, 36.10; H, 3.72%. $\mu_{\text{eff}} = 2.21 \mu_{\text{B}}$.

4.7. Preparation of $\text{IndMoCl}_2(\text{acac})$ (6)

To a suspension of IndMoCl_3 (0.20 g, 0.66 mmol) in 40 ml of CH_2Cl_2 was added $\text{Ti}(\text{acac})$ as a solid (0.20 g, 0.66 mmol) at r.t. The reaction mixture was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness and the residue was washed with Et_2O (3×5 ml) to yield the title compound **6** as a brown solid. Yield: 0.18 g (72%). Selected IR (KBr, cm^{-1}): 1523, vs, $\nu(\text{CO})$. Anal. Calc. for $\text{C}_{14}\text{H}_{14}\text{Cl}_2\text{MoO}_2$: C, 44.12; H, 3.70. Found: C, 43.91; H, 4.01%. $\mu_{\text{eff}} = 2.58 \mu_{\text{B}}$.

4.8. Preparation of $\text{CpMoCl}_2(\text{OOCMe})$ (7)

To a stirred suspension of CpMoCl_3 (0.3 g, 1.12 mmol) in 40 ml of CH_2Cl_2 was added AgOOCMe as a solid (0.18 g, 1.12 mmol) at r.t. The reaction was complete after 6 h stirring leading to a red–brown solution. The AgCl precipitate was filtered and the solvent was removed from the filtrate under vacuum to give a brown residue which after being washed with Et_2O (3×5 ml) yield the title compound **7** as a brown solid. Yield: 0.28 g (87%). Selected IR (KBr, cm^{-1}) 1548, vs, $\nu(\text{CO})$. Anal. Calc. for $\text{C}_7\text{H}_8\text{Cl}_2\text{MoO}_2$: C, 28.89; H, 2.77. Found: C, 28.86; H, 3.02%. $\mu_{\text{eff}} = 2.62 \mu_{\text{B}}$.

4.9. Preparation of $\text{IndMoCl}_2(\text{OOCMe})$ (8)

To a stirred suspension of IndMoCl_3 (0.17 g, 0.53 mmol) in 20 ml of CH_2Cl_2 was added AgOOCMe as a solid (0.09 g, 0.53 mmol) at r.t. The reaction mixture was stirred for 6 h and the solution was filtered. The filtrate

was taken to dryness and the residue was washed with Et₂O (3 × 5 ml) to yield the title compound **8** as a brown solid. Yield: 0.16 g (80%). Selected IR (KBr, cm⁻¹): 1537, vs, ν(CO). Anal. Calc. for C₉H₁₂Cl₂MoO₂: C, 38.74; H, 2.96. Found: C, 38.46; H, 2.68%. μ_{eff} = 2.84 μ_B.

4.10. Preparation of CpMoCl₂[CH(NPh)₂] (**9**)

Solid AgCH(NPh)₂ (0.49 g, 1.64 mmol) was added to a stirred suspension of CpMoCl₃ (0.44 g, 1.64 mmol) in 50 ml of CH₂Cl₂. The reaction mixture was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness and the residue was washed with Et₂O (3 × 5 ml) to yield the title compound **9** as a brown solid. Yield: 0.55 g (78%). Selected IR (KBr, cm⁻¹): 1689, vs, ν(NCN)_{asym}, 1589, vs, 1531, vs, ν(CN). Anal. Calc. for C₁₈H₁₆Cl₂MoN₂: C, 50.61; H, 3.78; N, 6.56. Found: C, 50.57; H, 3.56; N, 6.43%. ¹H-NMR (CD₂Cl₂, 25 °C, δ ppm): 8.10 (s, 1H); 7.17–7.38 (m, 10H); 5.23 (s, 5H).

4.11. Preparation of IndMoCl₂[CH(NPh)₂] (**10**)

Solid AgCH(NPh)₂ (0.28 g, 0.94 mmol) was added to a stirred suspension of IndMoCl₃ (0.3 g, 0.94 mmol) in 50 ml of CH₂Cl₂. The reaction mixture was stirred for 10 h and the solution was filtered. The filtrate was taken to dryness and the residue was washed with Et₂O (3 × 5 ml) to yield the title compound **10** as a brown solid. Yield: 0.36 g (82%). Selected IR (KBr, cm⁻¹): 1680, vs, ν(NCN)_{asym}, 1564, vs, ν(CN). Anal. Calc. for C₂₂H₁₈Cl₂MoN₂: C, 55.37; H, 3.80; N, 5.87. Found: C, 55.57; H, 3.98; N, 5.96%. ¹H-NMR (CD₂Cl₂, 25 °C, δ ppm): 8.35 (s, 1H); 7.07–7.48 (m, 10H); 6.88 (m, 4H, H⁵⁻⁸); 5.59 (d, 2H, H^{1/3}); 5.41 (t, 1H, H²).

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