

Heterometallic complexes and clusters with 2-boratanaphthalene ligands †

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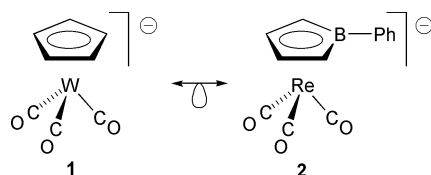
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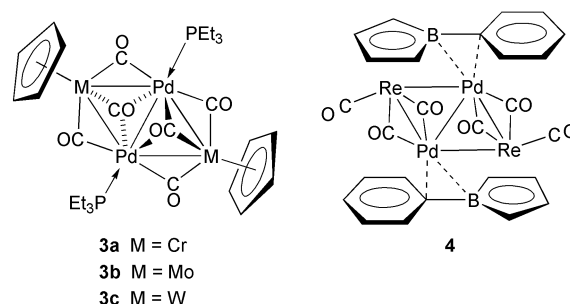
The new lithium (2-boratanaphthalene)tricarbonylmolybdates $\text{Li}[\text{Mo}(\text{RBn})(\text{CO})_3]$ ($\text{Bn} = \eta^5\text{-C}_{10}\text{H}_6\text{B}$; $\text{R} = \text{NiPr}_2$, Me) have been prepared and used as nucleophilic reagents for the synthesis of metal–metal bonded complexes and clusters. Dinuclear $[(i\text{Pr}_2\text{NBn})(\text{OC})_3\text{Mo}-\text{Au}(\text{PPh}_3)]$ (**7**) and trinuclear $[\text{Hg}\{\text{Mo}(\text{CO})_3(i\text{Pr}_2\text{NBn})\}_2]$ (**9**) and $[\text{Ag}\{\text{Mo}(\text{CO})_3(i\text{Pr}_2\text{NBn})\}\{\text{Mo}(\text{CO})_3[(i\text{Pr}_2\text{HN})\text{Bn}]\}]$ (**13**) chain complexes have been obtained in which the metalate behaves as an anionic two-electron donor metalloligand. In **13**, the other metal carbonyl fragment consists of an *N*-protonated (2-boratanaphthalene)tricarbonylmolybdenum fragment and acts therefore as a neutral, two-electron donor. In the 58-cluster valence electron planar, triangulated cluster $[\text{Mo}_2\text{Pd}_2(\text{MeBn})_2(\text{CO})_6(\text{PEt}_3)_2]$ (**15**), the $[\text{Mo}(\text{MeBn})(\text{CO})_3]^-$ fragment is however best viewed as a four-electron donor metalloligand bridging the Pd–Pd bond. These bonding situations are compared with those observed in complexes prepared from the metalate $[\text{MoCp}(\text{CO})_3]^-$ ($\text{Cp} = \eta^5\text{-C}_5\text{H}_5$) and this establishes experimentally the isoelectronic and isolobal character of the $[\text{Mo}(\text{MeBn})(\text{CO})_3]^-$ and $[\text{MoCp}(\text{CO})_3]^-$ fragments. The crystal structures of complexes **7**, **9**, **13** and **15** have been determined by X-ray diffraction.

In organometallic and coordination chemistry, the replacement of a uninegative, 6π electron cyclopentadienyl ligand with a dianionic 6π electron ligand may represent a way to obtain compounds with similar reactivity but with beneficial reduced Lewis acidity, a desirable feature in homogeneous Ziegler–Natta olefin polymerization metallocene-type catalysis.¹ This can be achieved *e.g.* by taking advantage of the isolobal analogy between CR and BR^- . In a different context, the isolobal analogy between the 6π electron borollide dianion $(\text{C}_4\text{H}_4\text{BR})^{2-}$ ($\text{R} = \text{Ph}$) and the cyclopentadienide ion $(\text{C}_5\text{H}_5)^-$ has been recently used to compare the reactivity and bonding properties of isolobal metal-centred organometallic nucleophiles of two adjacent columns of the Periodic classification, such as $[\text{WCp}(\text{CO})_3]^-$ (**1**) and $[\text{Re}(\eta^5\text{-C}_4\text{H}_4\text{BR})(\text{CO})_3]^-$ (**2**).^{2,3}



This led *inter alia* to the synthesis of heterodinuclear complexes with Re–Cu, Re–Ag, Re–Au or Re–Hg bonds in which the borole-containing carbonylmetalate behaves as a terminal, two-electron donor metalloligand. This bonding behaviour is also commonly encountered with the cyclopentadienyl-containing carbonylmetalates $[\text{MCp}(\text{CO})_3]^-$ ($\text{M} = \text{Cr}$, Mo or W) in metal–metal bonded complexes.⁴ In the 58-cluster valence electron (CVE), planar, triangulated clusters $[\text{M}_2\text{Pd}_2\text{Cp}_2(\text{CO})_6(\text{PEt}_3)_2]$ ($\text{M} = \text{Cr}$, **3a**; $\text{M} = \text{Mo}$, **3b**; $\text{M} = \text{W}$, **3c**) however, the latter metalates behave as four-electron donor metalloligands

and bridge the Pd–Pd bond.^{5,6} A strict analogue of these clusters does not exist in the borole series since despite its planar, triangulated metal core, the cluster $[\text{Re}_2\text{Pd}_2(\text{C}_4\text{H}_4\text{BPh})_2(\text{CO})_6]$ **4** contains only 54 CVE.³



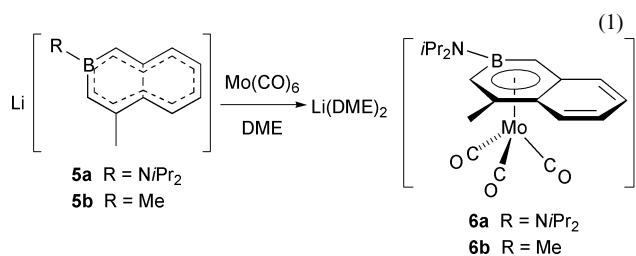
This unusual cluster displays a unique bonding situation for the borole ligand which not only binds to rhenium in the usual η^5 manner but also to the adjacent palladium *via* a $2e\text{--}3c$ B–C_{ipso}–Pd system. These intriguing similarities and differences led us to extend our studies with borole-containing carbonylmetalates to $[\text{HFe}(\eta^5\text{-C}_4\text{H}_4\text{BPh})(\text{CO})_2]^-$,^{2,7} $[\text{Fe}(\eta^5\text{-C}_4\text{H}_4\text{BPh})(\text{CO})_2\text{CN}]^-$,⁸ and now to the 2-boratanaphthalene system. Its boron-containing six-membered aromatic ring should behave as a neutral five-electron donor, like the five-membered Cp ligand and in contrast to the five-membered, four-electron donor borole ligand. This would therefore open the possibility to make further progress in the systematic comparison of the behaviour of organometallic building blocks in metal cluster chemistry⁹ since one could remain within the same column of the Periodic classification to form a carbonylmetalate with a boron-containing π -ligand that is isovalent to its Cp analogue.

In this work, we describe the synthesis of (2-boratanaphthalene)tricarbonylmolybdates and their use for the synthesis of metal–metal bonded complexes to be compared to those obtained in the Cp series.

† Part of the PhD Thesis of E. C. Dedicated to Professor Antonio Tiripicchio on the occasion of his 65th birthday, with our most sincere congratulations and warmest wishes.

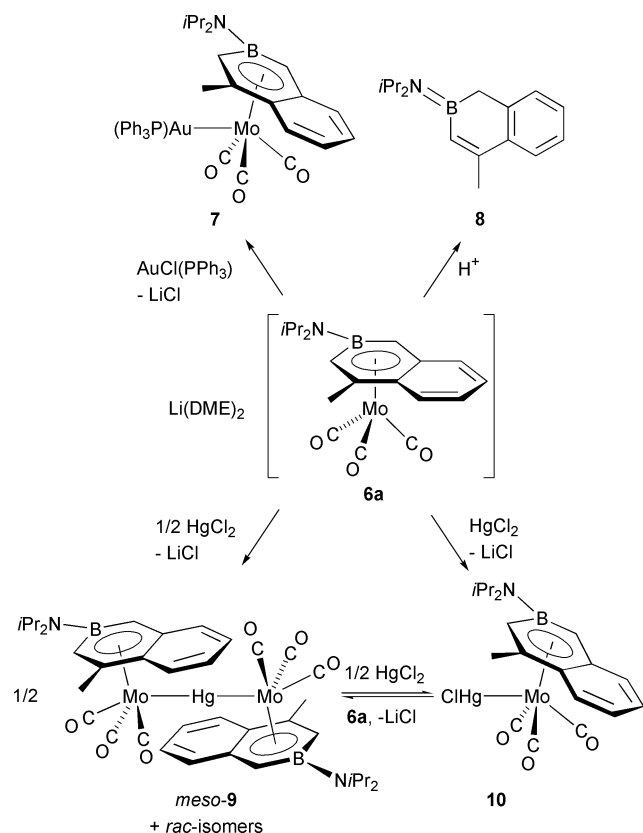
Results and discussion

The DME-solvated lithium salts of the (2-boratanaphthalene)tricarbonylmolybdates **6a,b** were prepared by reaction of $[\text{Mo}(\text{CO})_6]$ with the corresponding lithium 2-boratanaphthalenes **5a,b**.¹⁰ The reaction was performed under reflux, using conditions very similar to those described for the preparation of $\text{Na}[\text{MoCp}(\text{CO})_3] \cdot 2\text{DME}$ [eqn. (1)].¹¹



Synthesis of heterometallic Mo–Au, Mo–Ag and Mo–Hg complexes

The reaction of **6a** with one equivalent of $[\text{AuCl}(\text{PPh}_3)]$ yielded the air-stable, orange bimetallic complex $[(\text{OC})_3(i\text{Pr}_2\text{NBn})\text{Mo} - \text{Au}(\text{PPh}_3)]$ (**7**) (Scheme 1). Single crystals suitable for X-ray



Scheme 1 Reactions of the (2-boratanaphthalene)tricarbonylmolybdate **6a**.

diffraction were obtained by slow diffusion of pentane in a toluene solution. A view of the molecular structure is shown in Fig. 1 and selected bond distances and angles are given in Table 1.

The four-legged piano-stool geometry around the Mo centre is as expected and the Mo–Au distance of 2.7248(2) Å compares with that in $[(\text{OC})_3(\eta^5\text{-C}_5\text{H}_5)\text{Mo} - \text{Au}(\text{PPh}_3)]$ [2.710(1) Å]¹² or in $[(\text{OC})_3(\eta^5\text{-C}_5\text{H}_4\text{CHO})\text{Mo} - \text{Au}(\text{PPh}_3)]$ [2.7121(5) Å].¹³ The carbonyl ligands are all terminal, including C(19)–O(1) which, despite a slightly longer C–O distance [1.171(3) Å] is too far from the Au centre [$\text{Au} - \text{C}(19) = 2.485(3)$ Å] and makes an Mo–C(19)–O(1) angle too wide [171.5(2)°] to be considered as even semi-bridging. The double-bond character of the B–N

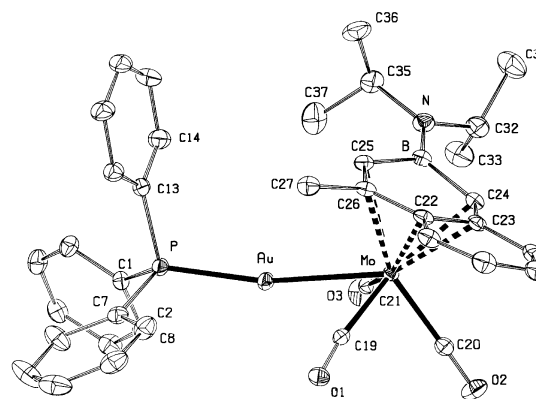


Fig. 1 ORTEP view of the molecular structure of **7**.

Table 1 Selected bond lengths (Å) and angles (°) for **7**

Au–Mo	2.7248(2)	Mo–C19	1.980(2)
Au–P	2.2750(7)	B–C24	1.538(4)
Au–C19	2.485(3)	B–C25	1.566(4)
C19–O1	1.171(3)	C24–C23	1.435(4)
C20–O2	1.153(3)	C23–C22	1.447(4)
C21–O3	1.163(3)	C22–C26	1.456(4)
B–N	1.422(4)	C26–C25	1.402(4)
Mo–B	2.670(3)	Mo–Au–P	166.16(2)
Mo–C24	2.379(2)	Mo–C19–O1	171.5(2)
Mo–C23	2.403(2)	B–N–C32	121.5(2)
Mo–C22	2.438(3)	B–N–C35	123.9(2)
Mo–C26	2.419(2)	C32–N–C35	114.5(2)
Mo–C25	2.462(2)		

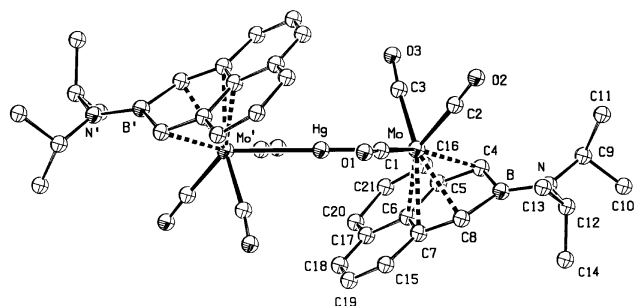
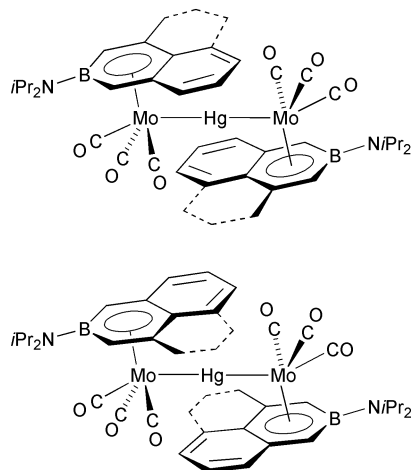
bond is reflected in the distance of 1.422(4) Å,¹⁴ and in the sum of the angles around N being equal to 359.9°. A similar situation was also encountered in the free ligand **5a**.¹⁰ The B–N moiety tends to deviate out of the ligand plane and be further away from molybdenum. Thus, the boron-containing heterocycle is bonded to Mo in a manner that is closer to η^5 .

It is interesting that despite the isolobal analogy between $[\text{Au}(\text{PPh}_3)]^+$ and H^+ , their reactions with **6a** and $[\text{MoCp}(\text{CO})_3]^-$ give very different products. Protonation of **6a** (HCl in Et_2O solution at -80°C or $[\text{HNEt}_3]\text{Cl}$) did not afford the metal hydride complex analogous to $[\text{HMoCp}(\text{CO})_3]$ ¹⁵ but led to ligand protonation, metal decoordination and liberation of **8** (Scheme 1). In contrast, the chromium hydrido borole complex $[\text{HCr}(\text{C}_5\text{H}_5\text{B-Me})(\text{CO})_3]$ has been obtained by reaction of $\text{Na}[\text{Cr}(\text{C}_5\text{H}_5\text{B-Me})(\text{CO})_3]$ with H^+ . This emphasizes the influence of the substituent at boron (and that of the metal) on the reactivity of the carbonylmolybdate. The hydride $[\text{HCr}(\text{C}_5\text{H}_5\text{B-Me})(\text{CO})_3]$ has been reported as sensitive and unstable, with a tendency to dimerize into $[\text{Cr}(\text{C}_5\text{H}_5\text{B-Me})(\text{CO})_3]_2$.¹⁶

The reaction of **6a** with HgCl_2 in a 2 : 1 ratio in toluene led to the formation of two orange trinuclear complexes of formula $[\text{Hg}\{\text{Mo}(i\text{Pr}_2\text{NBn})(\text{CO})_3\}_2]$ (**9**) in the form of a 50 : 50 diastereomeric mixture (NMR spectroscopy) (Scheme 1). This results from the racemic nature of the molybdenum salt **6a**. The reaction of **6a** with excess HgCl_2 or of **9** with HgCl_2 led to the heterodinuclear complex $[(\text{OC})_3(i\text{Pr}_2\text{NBn})\text{MoHgCl}]$ (**10**). Recrystallization of the diastereomeric mixture **9** gave rod-like single crystals which were analysed by X-ray diffraction. The molecular structure possesses crystallographic centrosymmetry and, in addition, displays disordered boratanaphthalene ligands (Fig. 2). The disorder relates to the bonding of the metal to the two enantiotopic faces of the planar chiral boratanaphthalene and is illustrated for the *meso* isomer in Scheme 2. Superposition of the two alternative dispositions of the molecule produces apparent lateral symmetry of the metal-to-ligand bonding. Note that this superposition may equally well comprise the two racemic isomers. Selected bond distances and angles are collected in Table 2.

Table 2 Selected bond lengths (Å) and angles (°) for **9**

Mo–Hg	2.7483(9)	Mo1–B	2.672(6)
B1–N1	1.425(7)	Mo1–C4	2.380(6)
N1–C9	1.470(8)	Mo1–C5	2.388(6)
N1–C12	1.486(7)	Mo1–C6	2.406(6)
C1–O1	1.140(8)	Mo1–C7	2.384(5)
C2–O2	1.150(8)	Mo1–C8	2.401(5)
C3–O3	1.130(8)	B–N–C12	122.8(5)
Mo1–C1	1.990(7)	C9–N1–C12	115.2(4)
Mo1–C2	1.967(6)	B–N–C9	122.0(5)
Mo1–C3	1.995(7)		

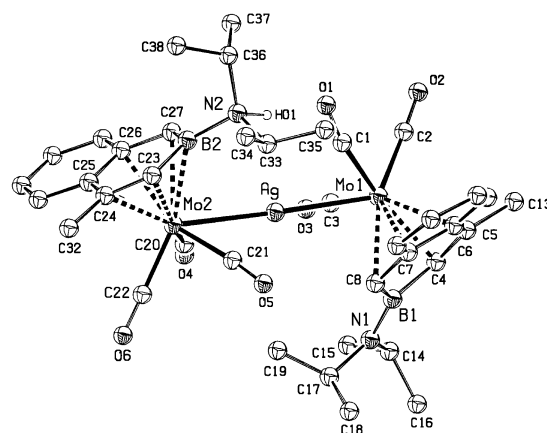
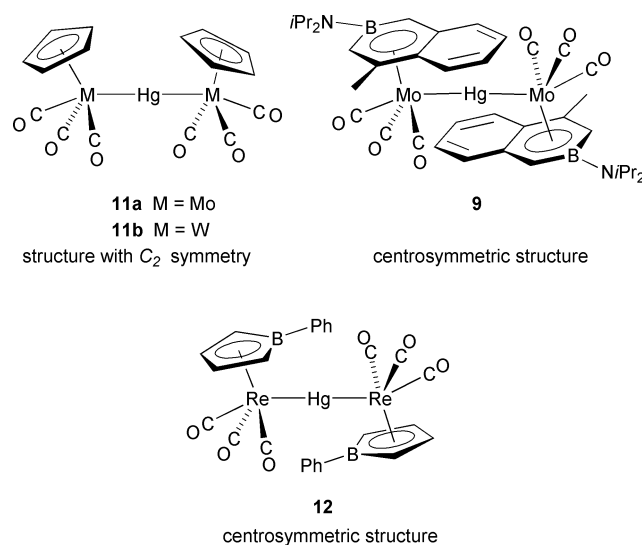
**Fig. 2** ORTEP view of the molecular structure of **9**. The carbon sites C(18), C(19) and C(20), C(21) have occupancies of 0.5 due to disorder (see also Scheme 2).**Scheme 2** Two dispositions of the *meso*-isomer of **9** resulting in disorder in the crystal.

The mercury atom resides on an inversion centre and the Mo–Hg distance of 2.7481(5) Å is similar to that found in [Hg{MoCp(CO)₃}₂] (**11a**) [2.746(2) Å].^{17,18} In the latter complex, the Hg atom is situated on a crystallographic C₂ axis. Similarly, the Re–Hg–Re complex **12**² is centrosymmetric whereas its isolobal analogue **11b** processes a C₂ axis.

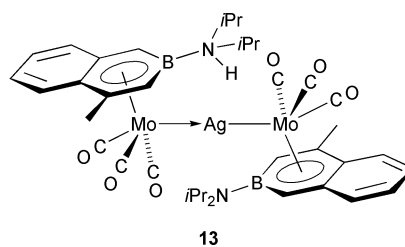
These differences can be explained on steric grounds since the bulkier ligands in **9** and **12** prevent a molecular conformation of the type encountered with the Cp analogues. As in **7**, the *i*Pr₂NBn ligand is coordinated in an η⁵ manner to Mo and the B–N bond possesses double bond character. By analogy with the reaction of HgCl₂ with two equivalents of **6a**, we expected that a similar reaction with [AgOTf] would afford an anionic Mo–Ag–Mo chain complex, as observed in the Cp series with [Ag{MoCp(CO)₃}₂][–].¹⁹ The reaction turned out to be more complicated than expected and a mixture was obtained whose analysis by mass spectrometry suggested the presence of oligomeric species (see Experimental section). In the course of purification procedures, one of the complexes present, **13**, was obtained as single crystals from toluene–hexane. The quantity of pure material was unfortunately too small to allow full characterization by analytical and spectroscopic methods.

Table 3 Selected bond lengths (Å) and angles (°) for **13**

Ag–Mo1	2.8097(4)	Mo1–Ag–Mo2	174.95(1)
Ag–Mo2	2.8395(4)	B1–N1–C14	121.8(3)
Mo1–C8	2.425(3)	B1–N1–C17	124.5(3)
Mo1–B1	2.703(3)	C14–N1–C17	113.5(2)
B1–N1	1.437(4)	B2–N2–C33	115.8(2)
Mo2–C27	2.406(3)	B2–N2–C36	112.8(2)
Mo2–B2	2.502(3)	C33–N2–C36	114.2(2)
B2–N2	1.570(4)	C33–N2–H01	97.25
N2–H01	1.0536	C36–N2–H01	107.72

**Fig. 3** ORTEP view of the molecular structure of **13**.

However, its crystal structure was determined by X-ray diffraction and revealed features of sufficient interest to be reported. If a metal chain Mo–Ag–Mo was indeed present, the complex turned out to be neutral and not anionic, as originally anticipated. A view of the molecular structure is shown in Fig. 3 and selected bond distances and angles are given in Table 3.



The two *i*Pr₂NBnMo fragments are chemically non-equivalent and a proton is bound to N(2), which has clearly become sp³ hybridized in contrast to N(1) which remains unchanged. The B(2)–N(2) distance of 1.570(4) Å corresponds to a single

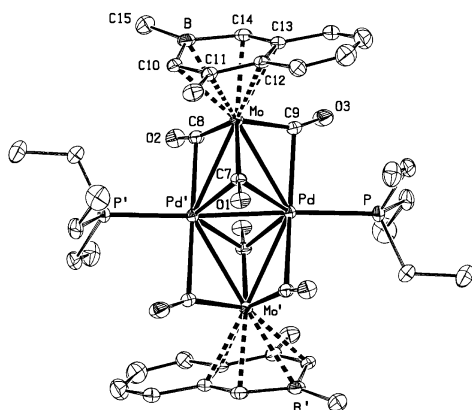
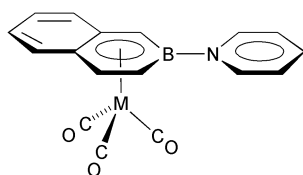


Fig. 4 ORTEP view of the molecular structure of **15**.

Table 4 Selected bond lengths (Å) and angles (°) for **15**

Mo–Pd	2.8815(4)	C7–O1	1.169(4)
Mo–Pd	2.8334(3)	C8–O2	1.183(5)
Pd–Pd'	2.5955(4)	C9–O3	1.179(5)
Pd–P	2.358(1)	B–C10	1.517(6)
Mo–B	2.547(5)	B–C14	1.531(6)
Mo–C10	2.409(4)	C10–C11	1.400(5)
Mo–C11	2.440(4)	C11–C12	1.453(5)
Mo–C12	2.446(4)	C12–C13	1.442(6)
Mo–C13	2.392(4)	C13–C14	1.414(6)
Mo–C14	2.353(4)	B–C15	1.577(7)
Mo–C7	2.066(4)		
Mo–C8	1.972(4)	Pd–Mo–Pd'	54.01(1)
Mo–C9	1.994(4)	Mo–Pd–Mo'	125.99(1)
Pd–C7	2.442(4)	P–Pd–Pd'	176.75(3)
Pd'–C7	2.331(3)	Mo–C7–O1	158.7(2)
Pd'–C8	2.409(4)	Mo–C8–O2	166.3(3)
Pd–C9	2.367(4)	Mo–C9–O3	158.7(3)

bond, as observed in amineboranes^{14,20} and is similar to that of 1.541(4) Å in the 2-pyridine-2-boratanaphthalene complexes **14** where the BC₅ ring is hexahapto coordinated to the metal.^{21,22}



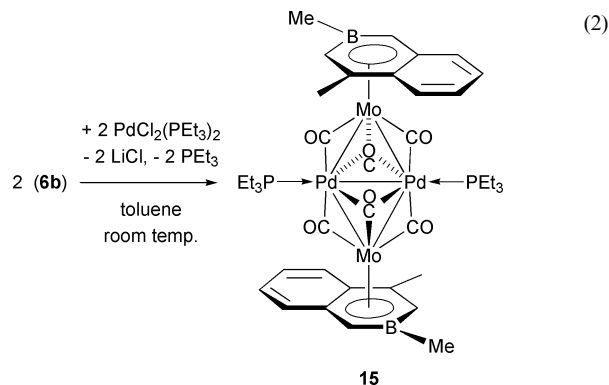
14 M = Cr, Mo

Since the vacant orbital on boron is no longer stabilized through π -bonding with nitrogen, the boron atom comes much closer to the electron-rich molybdenum atom [Mo(2)–B(2) = 2.502(3) Å]. The heterocycle is now bonded to Mo in an η^6 manner. The Mo(1)(*i*Pr₂NBn)(CO)₃ fragment is, in contrast, very similar to those in **7** and **9**. Its N(1) atom has a trigonal planar environment and the B(1)–N(1) distance of 1.437(4) Å indicates a double bond character, with an η^5 coordination of the Bn ligand.

Complex **13** is best viewed as resulting from the combination of the metalate [Mo(*i*Pr₂NBn)(CO)₃][–] with the Ag⁺ cation and the coordination of the neutral two-electron donor fragment [Mo(*i*Pr₂HNBn)(CO)₃] whose HOMO is centred on the metal. This fragment has never been isolated before and only observed in **13** as a metalloligand. Note however that protonation of the nitrogen atom of diisopropylaminoborole complexes was first observed with Cr, Fe, Ru and Co and afforded stable salts with no B–N bond dissociation.²³ This was then extended to the case of Zr, Hf^{24,25} and Ta^{26,27} complexes.

Synthesis of the first boratanaphthalene metal cluster

The reaction of two equivalents of **6b** with [PdCl₂(PEt₃)₂]

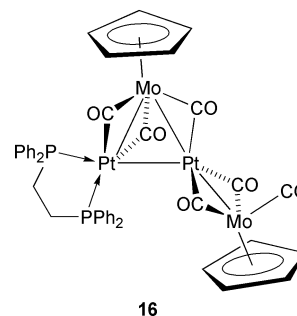


yielded a dark green complex whose crystal structure could be established by X-ray diffraction [eqn. (2)]. This first metal cluster with a boratanaphthalene ligand, [Mo₂Pd₂(MeBn)₂(CO)₆(PEt₃)₂] (**15**), contains a planar triangulated metal core with a centre of symmetry in the middle of the Pd–Pd bond. Selected bond distances and angles are given in Table 4 and a view of the molecule is shown in Fig. 4.

The Pd–Pd' distance of 2.5955(4) Å is short relative to the sum of the covalent radii (2.98 Å) or that in Pd metal (2.751 Å) or in other complexes.^{5,28} It is however similar to that found in the related cluster [Mo₂Pd₂Cp₂(CO)₆(PEt₃)₂] (**3b**) (2.582(1) Å) which also contains a linear R₃P←Pd–Pd→PR₃ moiety bridged by two MoCp(CO)₃ fragments. The disposition of the carbonyl ligands C(8)–O(2) and C(9)–O(3), which form asymmetric bridges and C(7)–O(1), which is semi-triply bridging, is similar to that in [Mo₂Pd₂Cp₂(CO)₆(PEt₃)₂]. The Mo–Pd distances of 2.8815(4) and 2.8334(3) Å are in the range of those found for this bond in other dinuclear and cluster complexes.^{5,29}

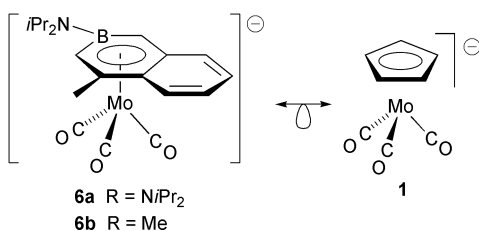
With the hope of characterizing a 54 CVE cluster, with an Mo₂Pd₂ core but no phosphine ligand, that would be related to cluster **4**, we reacted two equivalents of **6a** with [PdCl₂(NCPh)₂] in toluene. Although a major, deep-blue product displayed ν_{CO} absorptions in KBr at 1956s, 1881m, 1841s, 1808w cm^{–1}, it could not be further characterized owing to decomposition and formation of paramagnetic species.

Both **3b** and **15** are 58 CVE butterfly-type clusters and the deviation from the 62 CVE count expected on the basis of the Wade–Mingos rules originates from the 16 electron rather than 18 electron count for the palladium centres. Note however that a butterfly structure with five metal–metal bonds involving these metals is not always observed with the 58 CVE count, as exemplified by the spiked-triangular structure of [Mo₂Pd₂Cp(CO)₆(dppe)] (**16**) whose composition differs from that of **3b** only by the nature of the phosphine ligands.³⁰



Cluster **3b** has been adequately described as containing a [R₃P→Pd–Pd←PR₃]²⁺ d⁹–d⁹ unit bridged by two four-electron donor [MoCp(CO)₃][–] metalates. This emphasized the relationship between clusters **3** and phosphido-, allyl- or halide-bridged dinuclear Pd(I) complexes.³¹ The crystal structures of **7**, **9**, **13** and **15** now experimentally establish that the terminal (two-electron donor) or bridging (four-electron donor) bonding modes observed previously with the metalloligand [MoCp–

(CO)₃][−] can be similarly extended to the isolobal and isovalent anions of the type [Mo(RBn)(CO)₃][−]. Such relationships help rationalize the synthesis and structures of transition metal clusters.⁹



Experimental

General procedures

All reactions were performed in standard Schlenk-type flasks under nitrogen. Solvents were dried by conventional methods and freshly distilled under nitrogen. Deuterated solvents for NMR spectroscopy were degassed, dried and stored over molecular sieves (4 Å Merck). The Kieselguhr was heated overnight at 300 °C, cooled under vacuum and kept under N₂. Electron impact mass spectra were recorded on a Finnigan-MAT 95 spectrometer with a nominal electron energy of 70 eV (Aachen), or on a Fisons ZAB-HF (Strasbourg). Melting points were determined in sealed capillaries on a Büchi 510 melting point apparatus and are uncorrected. Elemental analyses were performed by the analytical laboratories of Prof. H. Malissa and G. Reuter, D-51789 Lindlar, Germany, of Prof. M. Veith, Universität des Saarlandes, D-66041 Saarbrücken, Germany and the Service Central de Microanalyses du CNRS, Institut de Chimie, F-67008 Strasbourg, France. The NMR spectra were recorded on a Varian Unity 500 spectrometer (¹H, 500 MHz; ¹³C{¹H}, 126 MHz; ¹¹B, 160 MHz), on a FT Bruker AC 300 spectrometer (¹H, 300 MHz; ³¹P{¹H}, 121 MHz), or on an FT Bruker 400 spectrometer (¹³C{¹H}, 100 MHz; ¹¹B, 128 MHz). Chemical shifts are given in ppm; they were measured at ambient temperature and are relative to internal TMS for ¹H and ¹³C{¹H} and relative to BF₃·OEt₂ as external reference for ¹¹B. ³¹P{¹H} NMR spectra were externally referenced to 85% H₃PO₄ in H₂O, with downfield chemical shift reported as positive. The ¹H and ¹³C spectra show a fairly constant pattern for the phenyl ring of boratanaphthalene derivatives. Assignments are based on numerous H,H-COSY, H,C-COSY (HETCOR, HMQC), NOE, APT and DEPT spectra which are not detailed here. The IR spectra were recorded in the region 4000–400 cm^{−1} on a Perkin-Elmer 1720 X FT-IR or a Bruker IFS66/113 (FT-IR) instrument. [Li(MeBn)] was prepared as reported in the literature.¹⁰

Syntheses

Lithium tricarboxyl(2,4-dimethyl-2-boratanaphthalene)molybdate-bis(1,2-dimethoxyethane) Li[Mo(CO)₃(MeBn)]·2DME (6b). A solution of [Li(MeBn)] (5b) (3.498 g, 21.7 mmol) in 60 mL of DME was added to a suspension of [Mo(CO)₆] (5.73 g, 21.7 mmol) in 100 mL of DME. The mixture which turned orange was refluxed for 24 h with constant stirring. The disappearance of [Mo(CO)₆] was followed by IR spectroscopy. The solution was filtered through Kieselguhr. After concentration of the solution and precipitation with pentane (200 mL), compound Li[Mo(CO)₃(MeBn)]·2DME (6b) was isolated by filtration, washed with pentane (3 × 50 mL) and dried *in vacuo*. Yield: 10.2 g, 90%. 6b is a bright yellow powder, insoluble in pentane but soluble in toluene, DME or THF. Anal. Calc. for C₂₂H₃₂BLiMoO₇: C, 50.60; H, 6.18. Found: C, 50.33; H, 5.98. ¹H NMR (THF-*d*₈): δ 7.68 (d, *J* = 8.8 Hz, 1H, 5-H), 7.11 (d, *J* = 8.5 Hz, 1H, 8-H), 6.89 (ddd, *J* = 8.5, 6.4, 1.2

Hz, 1H, 7-H), 6.73 (ddd, *J* = 8.5, 6.4, 1.2 Hz, 1H, 6-H), 4.80 (d, ⁴*J*₁₃ = 1.6 Hz, 1H, 3-H), 4.74 (d, ⁴*J*₁₃ = 1.6 Hz, 1H, 1-H), 2.54 (s, 3H, 4-Me), 0.49 (s, 3H, BMe). DME: 3.43 (s, 4H, CH₂), 3.27 (s, 6H, Me). ¹³C NMR (THF-*d*₈): δ 230.7 (CO), 132.2 (C-8), 128.7 (C-5), 125.4 (C-7), 122.5, 117.7 and 104.1 (C-4, C-4a, C-8a), 119.8 (C-6), 108.0 (br, C-3), 90.1 (br, C-1), 23.8 (4-Me), 2.2 (br, BMe). DME: 72.7 (CH₂), 58.9 (Me). ¹¹B NMR (THF-*d*₈): δ 24. IR (12-crown-4, DME): ν_{CO} = 1933s, 1911s, 1808m.

Lithium tricarboxyl(2-diisopropylamino-4-methyl-2-boratanaphthalene)molybdate-bis(1,2-dimethoxyethane) Li[Mo(CO)₃(iPr₂NBn)]·2DME (6a). In a similar manner to 6b, [Li(iPr₂NBn)] (5a) (2.744 g, 11.1 mmol) was reacted with [Mo(CO)₆] (2.930 g, 11.1 mmol) to give Li[Mo(CO)₃(iPr₂NBn)]·2DME (6a). Yield: 5.90 g, 87%. 6a is a yellowish powder, insoluble in pentane, slightly soluble in toluene and very soluble in DME or THF. Anal. Calc. for C₂₇H₄₃BLiMoO₇: C, 53.40; H, 7.14; N, 2.31. Found: C, 53.32; H, 6.96; N, 2.25. ¹H NMR (THF-*d*₈): δ 7.55 (dd, *J* = 8.8, 0.6 Hz, 1H, 5-H), 7.02 (dd, *J* = 8.5, 0.6 Hz, 1H, 8-H), 6.76 (ddd, *J* = 8.5, 6.4, 1.2 Hz, 1H, 7-H), 6.53 (ddd, *J* = 8.8, 6.4, 1.2 Hz, 1H, 6-H), 4.60 (d, ⁴*J*₁₃ = 2.5 Hz, 1H, 3-H), 4.17 (d, ⁴*J*₁₃ = 2.5 Hz, 1H, 1-H), 2.54 (s, 3H, 4-Me). NiPr₂: 3.60 (m, ³*J* = 6.7 Hz, 2H, NCH), 1.22 and 1.20 (d, ³*J* = 6.8 Hz, 12H, Me). DME: 3.42 (s, 4H, CH₂), 3.27 (s, 6H, Me). ¹³C NMR (THF-*d*₈): δ 232.4 (CO), 132.7 (C-8), 129.0 (C-5), 125.2 (C-7), 124.2, 120.1 and 99.8 (C-4, C-4a, C-8a), 117.8 (C-6), 96.4 (br, C-3), 74.3 (br, C-1), 24.6 (4-Me). NiPr₂: 46.3 (NC), 23.2 and 23.1 (Me). DME: 72.7 (CH₂), 58.9 (Me). ¹¹B NMR (THF-*d*₈): δ 23. IR (12-crown-4, DME): ν_{CO} = 1904s, 1806s, 1790s.

[Tricarboxyl(2-diisopropylamino-4-methyl-2-boratanaphthalene)molybdate](triphenylphosphine)gold [(iPr₂NBn)(OC)₃MoAu(PPh₃)] (7). Li[Mo(CO)₃(iPr₂NBn)]·2DME (6a) (0.181 g, 0.298 mmol) and [AuCl(PPh₃)] (0.148 g, 0.299 mmol) were stirred in 20 mL toluene for 1 h. After complete evaporation of the volatiles under reduced pressure, the residue was dissolved in toluene and filtered through Kieselguhr to give a clear orange solution. The solvent was then removed *in vacuo* to give 7 in quantitative yield as an air stable, yellow powder or large orange-red crystals, slightly soluble in pentane and very soluble in toluene. Mp: decomposition 125 °C. Anal. Calc. for C₃₇H₃₈AuBMoPNO₃: C, 50.54; H, 4.36; N, 1.59. Found: C, 50.89; H, 4.24; N, 1.46. ¹H NMR (C₆D₆): δ 7.57 (d, *J* = 8.8 Hz, 1H, 5-H), 7.41 and 6.98 (m, 15H, PPh₃), 7.25 (d, *J* = 8.8 Hz, 1H, 8-H), 6.85 (ddd, *J* = 8.6, 6.5, 0.8 Hz, 1H, 7-H), 6.58 (ddd, *J* = 8.8, 6.4, 0.8 Hz, 1H, 6-H), 5.12 (d, ⁴*J*₁₃ = 2.4 Hz, 1H, 3-H), 4.93 (d, ⁴*J*₁₃ = 2.4 Hz, 1H, 1-H), 2.51 (s, 3H, 4-Me). NiPr₂: 3.69 (s br, 2H, NCH), 1.34 (d, ³*J* = 5.6 Hz, 12H, Me). ¹³C NMR (C₆D₆): δ 228.3 (CO), 124.1, 122.2 and 100.9 (C-4, C-4a, C-8a), 134.6 (C-8), 130.0 (C-5), 128.2 (C-7), 122.8 (C-6), 96.7 (br, C-3), 76.8 (br, C-1), 25.5 (4-Me). NiPr₂: 46.9 (NC), 23.5 and 23.4 (Me). PPh₃: 135.1 and 130.0 (d, *J*_{PC} = 15 and 11 Hz, *ortho* and *meta*), 132.1 (d, *J*_{PC} = 2 Hz, *para*), 131.7 (d, *J*_{PC} = 51 Hz, *ipso*). ¹¹B NMR (C₆D₆): δ 24. ³¹P NMR (C₆D₆): δ 47.8. IR (KBr): ν_{CO} = 1948s, 1869m, 1835s.

Tricarboxyl(chloromercury)(2-diisopropylamino-4-methyl-2-boratanaphthalene)molybdenum [(iPr₂NBn)(OC)₃MoHgCl] (10). Solid HgCl₂ (large excess, 0.150 g, 0.552 mmol) was added to a suspension of Li[Mo(CO)₃(iPr₂NBn)]·2DME (6a) (0.254 g, 0.418 mmol) in 30 mL of toluene. The mixture was stirred for 1 h and the resulting greenish suspension was filtered through Kieselguhr. The yellow filtrate was then concentrated under reduced pressure. The product 10, [(iPr₂NBn)(OC)₃MoHgCl] was obtained in quantitative yield after precipitation with pentane. 10 is a yellow, air stable powder, light-sensitive in solution, slightly soluble in pentane and very soluble in toluene. Mp: 132 °C. Anal. Calc. for C₁₉H₂₃BClHgMoNO₃: C, 34.78; H, 3.53; N, 2.13. Found: C, 35.07; H, 3.51; N, 1.94. ¹H NMR (C₆D₆): δ 7.19 (d, *J* = 9.0 Hz, 1H, 5-H), 6.73 and 6.59 (m, 3H,

Table 5 Summary of the crystallographic details

	7	9	13	15
Empirical formula	C ₃₇ H ₃₈ AuBMoNO ₃ P	C ₃₈ H ₄₆ B ₂ HgMo ₂ N ₂ O ₆	C ₃₈ H ₄₇ AgB ₂ Mo ₂ N ₂ O ₆ ·C ₇ H ₈	C ₄₀ H ₅₄ B ₂ Mo ₂ O ₆ P ₂ Pd ₂
Formula weight	879.41	1040.89	1041.32	1119.12
Crystal system	Monoclinic	Triclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 1̄	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> /Å	17.8519(8)	9.2428(6)	12.0187(2)	8.8882(4)
<i>b</i> /Å	10.3946(6)	9.7821(6)	18.5098(6)	20.8012(8)
<i>c</i> /Å	19.1659(8)	12.3798(5)	20.4035(6)	11.9776(6)
<i>α</i> /°		78.225(5)		
<i>β</i> /°	100.148(8)	87.103(5)	98.989(5)	103.372(5)
<i>γ</i> /°		67.449(5)		
<i>V</i> /Å ³	3500.9(6)	1011.5(1)	4483.3(4)	2154.4(3)
<i>Z</i>	4	1	4	2
Crystal size/mm	0.18 × 0.15 × 0.12	0.20 × 0.16 × 0.02	0.20 × 0.08 × 0.06	0.10 × 0.04 × 0.02
<i>D</i> /g cm ^{−3}	1.67	1.71	1.54	1.73
<i>F</i> (000)	1728	510	2112	1116
<i>μ</i> /mm ^{−1}	4.625	4.440	1.031	1.508
Temperature/K	294	173	173	173
<i>θ</i> limits/°	2.5/27.48	2.5/27.50	2.5/27.47	2.5/27.50
Reflns. collected	8427	6602	19098	8976
No. of variables	406	250	523	244
<i>R</i>	0.022	0.037	0.029	0.029
<i>R</i> _w	0.031	0.050	0.031	0.043
GOF	1.031	1.021	1.053	1.004
Largest peak in final diff. map/e Å ^{−3}	0.611	1.182	0.429	0.621
No. of data with <i>I</i> > 3σ(<i>I</i>)	6860	3395	6353	3678
Weighting scheme	4 <i>F</i> _o ² /(σ ² (<i>F</i> _o ²) + 0.0016 <i>F</i> _o ⁴)	4 <i>F</i> _o ² /(σ ² (<i>F</i> _o ²) + 0.0049 <i>F</i> _o ⁴)	4 <i>F</i> _o ² /(σ ² (<i>F</i> _o ²) + 0.0001 <i>F</i> _o ⁴)	4 <i>F</i> _o ² /(σ ² (<i>F</i> _o ²) + 0.0025 <i>F</i> _o ⁴)

6-H, 7-H, 8-H), 4.80 (d, *J* = 1.8 Hz, 1H, 3-H), 4.56 (d, *J* = 2.4 Hz, 1H, 1-H), 1.94 (s, 3H, 4-Me). NiPr₂: 3.46 and 3.26 (sept, ³*J* = 7.2 and 6.6 Hz, 2H, NCH), 1.18 (m br, 12H, Me). ¹³C NMR (C₆D₆): δ 227.8, 223.2 and 219.4 (CO), 134.1, 128.2, 127.3 and 126.6 (C-5, C-6, C-7, C-8), 119.3, 116.3 and 101.2 (C-4, C-4a, C-8a), 91.1 (br, C-3), 75.8 (br, C-1), 22.7 (4-Me). NiPr₂: 46.7 and 45.5 (NC), 22.4, 22.2, 21.7 and 21.3 (Me). ¹¹B NMR (C₆D₆): δ 23. IR (KBr): ν_{CO} = 2007s, 1946m, 1919s. MS: *m/z* (*I*_{rel}): 656 (M⁺, 32%); 642 (M⁺ − CH₃, 8%); 422 ([Mo(CO)₃(iPr₂NBn)]⁺, 94%).

Bis[tricarbonyl(2-diisopropylamino-4-methyl-2-boratanaphthalene)molybdenum]mercury [Hg{Mo(CO)₃(iPr₂NBn)}₂] (**9**). Solid HgCl₂ (0.135 g, 0.497 mmol) was added to a suspension of Li[Mo(CO)₃(iPr₂NBn)]·2DME (**6a**) (0.630 g, 1.037 mmol) in 30 mL of toluene. The mixture was stirred for 1 h, the volatiles were removed under reduced pressure to give a brown residue which was dissolved in toluene and filtered twice through Kieselguhr. The resulting red solution was concentrated *in vacuo*. After precipitation with pentane, **9** was isolated as a mixture of two diastereomers. Yield: varying from 70 to 98%. Slow diffusion of pentane into a solution of the isomeric mixture in toluene afforded orange crystals suitable for X-ray diffraction. The product is an orange air stable powder, light-sensitive in solution, slightly soluble in pentane and very soluble in toluene. Mp: decomposition 190 °C. Anal. Calc. for C₃₈H₄₆B₂HgMo₂N₂O₆: C, 43.85; H, 4.45; N, 2.69. Found: C, 44.24; H, 4.34; N, 2.70.

NMR data for the first diastereoisomer: ¹H NMR (C₆D₆): δ 7.38 (d, *J* = 8.4 Hz, 1H, 5-H), 6.85 (m, 3H, 6-H, 7-H, 8-H), 4.65 (d, ⁴*J*₁₃ = 2.5 Hz, 1H, 3-H), 4.55 (d, ⁴*J*₁₃ = 2.5 Hz, 1H, 1-H), 2.19 (s, 3H, 4-Me). NiPr₂: 3.5 (br, 2H, NCH), 1.21 (m br, 12H, Me). ¹³C NMR (C₆D₆): δ 225 (vbr, CO), 133.6, 127.2, 126.2 and 123.4 (C-5, C-6, C-7, C-8), 121.6, 116.9 and 100.5 (C-4, C-4a, C-8a), 93.6 (br, C-3), 76.0 (br, C-1), 23.0 (4-Me). NiPr₂: 44.8 (NC), 21.2 (br, Me). ¹¹B NMR (C₆D₆): δ 24.

NMR data for the second diastereoisomer: ¹H NMR (C₆D₆): δ 7.51 (d, *J* = 8.6 Hz, 1H, 5-H), 6.85 (m, 3H, 6-H, 7-H, 8-H), 4.70 (d, ⁴*J*₁₃ = 2.6 Hz, 1H, 3-H), 4.50 (d, ⁴*J*₁₃ = 2.6 Hz, 1H, 1-H), 2.27 (s, 3H, 4-Me). NiPr₂: 3.5 (br, 2H, NCH), 1.21 (m br, 12H, Me). ¹³C NMR (C₆D₆): δ 225 (vbr, CO), 133.6, 126.9, 126.3 and

123.0 (C-5, C-6, C-7, C-8), 121.3, 116.7 and 100.8 (C-4, C-4a, C-8a), 93.6 (br, C-3), 76.0 (br, C-1), 23.0 (4-Me). NiPr₂: 44.8 (NC), 21.2 (br, Me). ¹¹B NMR (C₆D₆): δ 24. IR (KBr): ν_{CO} = 1970s, 1915m, 1889s, 1820vw, 1800vw. MS: *m/z* (*I*_{rel}): 1041 (M⁺, 12%); 422 ([Mo(CO)₃(iPr₂NBn)]⁺, 100%).

[Tricarbonyl(2-diisopropylamino-4-methyl-2-boratanaphthalene)molybdenum][tricarbonyl(2-diisopropylamine-4-methyl-2-boratanaphthalene)molybdenum]silver [Ag{Mo(CO)₃(iPr₂NBn)}{Mo(CO)₃(iPr₂HN)Bn}] (**13**). The reaction of one equivalent of **6a** with AgOTf in toluene afforded a mixture of products whose analysis by mass spectrometry suggested the formation of oligomeric compounds with:

m/z (*I*_{rel}): 1584 (22%): [{AgMo(CO)₃(iPr₂NBn)}₃]⁺
m/z (*I*_{rel}): 1165 (99%): [{AgMo(CO)₃(iPr₂NBn)}₂ + Ag + H]⁺
m/z (*I*_{rel}): 1056 (22%): [{AgMo(CO)₃(iPr₂NBn)}₂]⁺
m/z (*I*_{rel}): 950 (16%): [{Ag[Mo(CO)₃(iPr₂NBn)]₂} + H]⁺
m/z (*I*_{rel}): 530 (51%): [AgMo(CO)₃(iPr₂NBn)]⁺

During attempts of purification, one of them, **13**, could be isolated in the crystalline form from a mixture toluene–hexane (M⁺ = 950) and studied by X-ray diffraction.

Hexacarbonyl-bis(η⁶-2,4-dimethyl-2-boratanaphthalene)-bis-(triethylphosphine)-dimolybdenumdipalladium [Pd₂Mo₂(Me-Bn)₂(CO)₆(PEt₃)₂] (**15**). A mixture of **6b** (0.205 g, 0.392 mmol) and [PdCl₂(PEt₃)₂] (0.081 g, 0.196 mmol) in 30 mL of toluene was stirred for 2 days. A dark green solution with a black precipitate was formed. The solid was removed by filtration through Kieselguhr whereas the filtrate was cooled at −30 °C for crystallization. Cluster **15** was obtained as green crystals (suitable crystals for X-ray diffraction were obtained by diffusion from hexane in a toluene solution). Yield: 0.022 g, 20%. **15** forms air stable, dark green–red dichroic crystals, insoluble in pentane and diethyl ether, moderately soluble in toluene and very soluble in THF. Anal. Calc. for C₄₀H₅₄B₂Mo₂O₆Pd₂P₂: C, 42.93; H, 4.86. Found: C, 42.72; H, 4.89. ¹H NMR (THF-*d*₈): δ 7.67 (d, *J* = 8.5 Hz, 2H, 5-H), 7.36 (d, *J* = 8.3 Hz, 2H, 8-H), 7.27 (dd, *J* = 8.4, 6.5 Hz, 2H, 7-H), 7.15 (ddd, *J* = 8.1, 7.7, 0.8 Hz, 2H, 6-H), 4.95 (d, *J* = 1.6 Hz, 2H, 3-H), 4.74 (d, *J* = 2.1 Hz, 2H, 1-H), 2.48 (s, 6H, 4-Me), 0.66 (s, 6H, BMe). PEt₃: 1.50 (dm, ²*J*_{PH} = 48.5 Hz, 12H, CH₂), 0.84 (m, 18H, Me). ¹¹B NMR

(THF- d_8): δ 26. ^{31}P NMR (C_6D_6): δ 18.3. IR (KBr): ν_{CO} = 1856s, 1821(sh), 1806s, 1793(sh).

Crystal structure determinations

The X-ray data for all structures were collected on a Kappa CCD diffractometer, using graphite-monochromated Mo-K α radiation (0.71073 Å) and ψ -scan mode. The structures were solved using direct methods and refined against $|F|$. For all computations the Nonius OpenMoleN package was used.³² The hydrogen atoms were introduced as fixed contributors ($d_{\text{C-H}}$ = 0.95 Å, $B(\text{H})$ = $1.3B_{\text{equiv}}(\text{C})$ Å²). Crystallographic details are given in Table 5.

CCDC reference numbers 153364–153367.

See <http://www.rsc.org/suppdata/dt/b1/b101556g/> for crystallographic data in CIF or other electronic format.

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