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GROUP IB ORGANOMETALLIC CHEMISTRY

XXIII *. REACTIONS OF $Ar_4Cu_2Li_2$ AND $Ar_4Au_2Li_2$ WITH Rh^I COMPLEXES: SYNTHESIS OF 2-[(DIMETHYLAMINO)METHYL]PHENYLRHODIUM DICARBON MONOXIDE, AND ELECTRON-TRANSFER INDUCED SELECTIVE FORMATION OF DIARYL KETONES ArC(O)Ar

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Summary

The 1/1 reaction of $(2\text{-}Me_2NCH_2C_6H_4)_4M_2Li_2$ (M = Cu or Au) with $(CO)_2ClRh$-dimer affords $(2\text{-}Me_2NCH_2C_6H_4M)_n$ and the novel $2\text{-}Me_2\dot{N}CH_2C_6H_4\dot{R}h(CO)_2$. In contrast, the reaction of $(x\text{-tolyl})_4M_2Li_2$ (x = 2, M = Cu; x = 4, M = Cu or Au) under a CO atmosphere results in the formation of ditolyl ketone and bitolyl in 69, 92 and 31, 4% yield, respectively. In the presence of TMED (x = 4, M = Cu) ditolyl ketone is formed exclusively. The reaction of $(4\text{-tolyl})_4Cu_2Li_2$ with $(C_2H_4)_2ClRh$-dimer under C_2H_4 atmosphere gives 4,4′-bitolyl, quantitatively.

A reaction pathway is proposed which involves formation of $[(CO)_2Rh(aryl)_2M]_n$ (M = Cu or Au) as a key intermediate.

Introduction

Arylmetal IB-lithium clusters $Ar_4M_2Li_2$ (M = Cu, Ag or Au; Ar = 2-tolyl [2], 4-tolyl [2], $2\text{-}Me_2NC_6H_4$ [3,4], $2\text{-}Me_2NCH_2C_6H_4$ [2,5—7]) are well-defined species consisting of a tetranuclear M_2Li_2 core to which the aryl groups are bound via 2e—3c carbon-to-metal bonds. Recently, we found that such compounds are excellent starting materials for the synthesis of mixed-metal IB species, as is illustrated by reactions 1 and 2 [3,7]. These results led us to study the possibility of using M_2Li_2 cluster species for the synthesis of cluster compounds, containing

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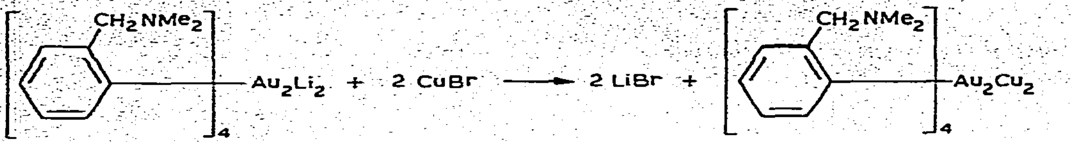
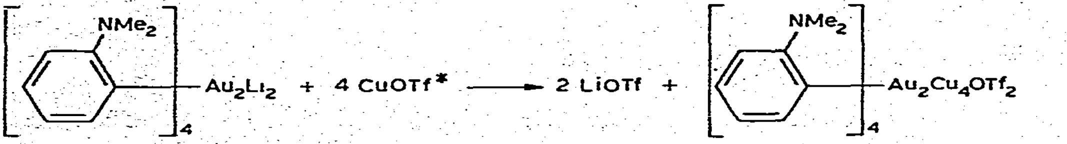
* For Par XXII see ref. 1.



(1)



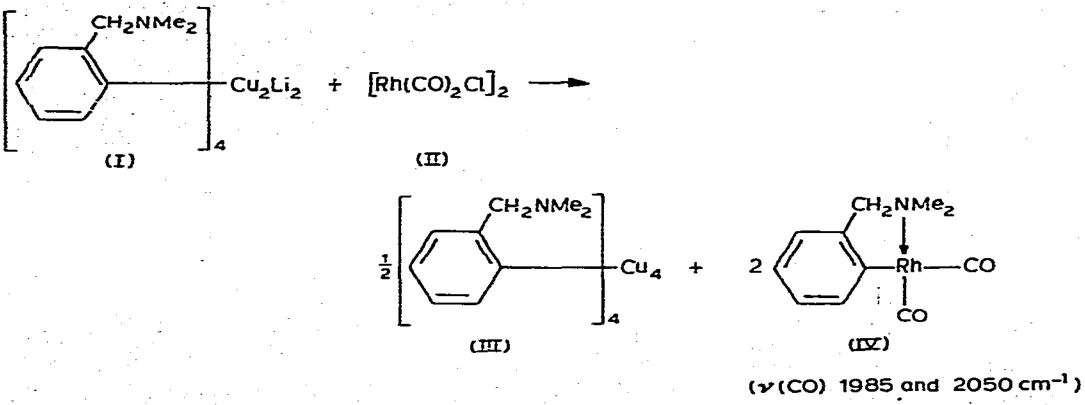
(2)

Group V—VIII transition metals in addition to a Group IB metal.

In this paper we report reactions of $Ar_4M_2Li_2$ (M = Cu or Au) species with rhodium(I) complexes, which were carried out with the aim of synthesizing CuRh and AuRh cluster species.

Results

The 1/1 reaction of $(2\text{-}Me_2NCH_2C_6H_4)_4Cu_2Li_2$ (I) with $(CO)_2ClRh$-dimer (II) in benzene affords a red solution. NMR spectroscopy revealed that in addition to $(2\text{-}Me_2NCH_2C_6H_4)_4Cu_4$ (III) [8], a second product containing a M-to-$C_6H_4CH_2NMe_2$ bond ($\delta(H_6)$ 7.95 ppm broadened multiplet in C_6D_6) had been formed. This product was isolated in 46% yield and appeared to be the novel $2\text{-}Me_2NCH_2C_6H_4Rh(CO)_2$ (IV). Its molecular formula was confirmed by elemental analysis, while molecular weight determinations established that $2\text{-}Me_2NCH_2C_6H_4Rh(CO)_2$ exists in benzene as a monomer. IR spectroscopy indicated that the CO ligands are in *cis* position (1985vs and 2050vs cm^{-1}). These results point to the structure IV, in which the $2\text{-}Me_2NCH_2C_6H_4$ ligand is bonded to rhodium(I) both via C and via N (eq. 3).



A similar reaction pattern was observed when the corresponding gold-lithium compound [7] $(2\text{-}Me_2NCH_2C_6H_4)_4Au_2Li_2$ (V) was used instead of the copper-lithium compound (I). In this reaction insoluble $(2\text{-}Me_2NCH_2C_6H_4Au)_n$ (VI) [2,9] precipitated from the reaction mixture. $2\text{-}Me_2NCH_2C_6H_4Rh(CO)_2$ was

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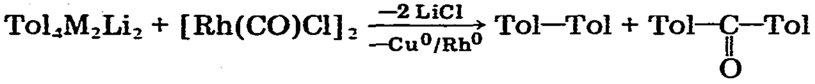
* CuOTf = triflate = copper trifluoromethanesulphonate.

isolated from the solution in nearly quantitative yield.

Similar reactions were carried out with 2- and 4-tolyl-copperlithium (VII) and -goldlithium (VIII) [2] cluster compounds. These reagents, which unlike I or V do not contain an intramolecular ligand, reacted with II to give entirely different products (see Table 1).

In a preliminary reaction, in which II was added to a suspension of di-4-tolyl-copperlithium (VII) in benzene, nitrogen was used as an inert atmosphere. An immediate reaction occurred yielding a black reaction mixture containing, according to GC/MS analysis, the dimer 4,4'-bitolyl (X) and 4,4'-ditolylketone (XI) in 31 and 69% yield. Products resulting from hydrogen abstraction (toluene) were essentially absent:



$$\mathrm{Tol_4M_2Li_2} + [\mathrm{Rh(CO)Cl}]_2 \xrightarrow[-\mathrm{Cu^0/Rh^0}]{-2\,\mathrm{LiCl}} \mathrm{Tol{-}Tol} + \mathrm{Tol{-}}\underset{\underset{\mathrm{O}}{\|}}{\mathrm{C}}\mathrm{{-}Tol}$$

(X) (XI)

The formation of 4,4'-ditolylketone reveals a reaction sequence in which coordinated CO becomes a building block of one of the reaction products. Therefore, this reaction was repeated in a CO atmosphere, and the amount of ketone formed was increased to 83%, while the yield of the dimer decreased to about 11%. Other products, which were formed in low yield, were 4-methylbenzaldehyde (3%) and 4,4'-ditolyldiketone (1%). This amounts to a total recovery of tolyl groups of at least 98%.

Various other experiments were carried out in order to get more information about the nature of the reaction (see Table 1). The reaction is stoichiometric in nature, because an increase of the 4-$\mathrm{Tol_4Cu_2Li_2}$/[$\mathrm{RhCl(CO)_2}$]$_2$ molar ratio from

TABLE 1

REACTIONS OF DIARYLMETAL IB-LITHIUM COMPOUNDS $\mathrm{Ar_4M_2Li_2}$ WITH $\mathrm{Rh(CO)_2Cl}$-DIMER

Reagents	Ratio Rh/Li	Atmosphere	Products[a]		
			ArAr	$\mathrm{Ar}\underset{\underset{\mathrm{O}}{\|}}{\mathrm{C}}\mathrm{Ar}$	Other
Ar = 2-$Me_2NCH_2C_6H_4$					
1. $\mathrm{Ar_4Cu_2Li_2}$	1/1	$\mathrm{N_2}$	—	—	$\mathrm{Ar_4Cu_4 + ArRh(CO)_2}$
2. $\mathrm{Ar_4Au_2Li_2}$	1/1	$\mathrm{N_2}$	—	—	$\mathrm{Ar_2Cu_2 + ArRh(CO)_2}$
Ar = 4-tolyl					
3. $\mathrm{Ar_4Cu_2Li_2}$	1/1	$\mathrm{N_2}$	31	69	$\mathrm{Ar(C{=}O)_2Ar}$, $\mathrm{Ar(C{=}O)_2H}$, $\mathrm{Ar(C{=}O)_3H}$[b]
4. $\mathrm{Ar_4Cu_2Li_2}$[c]	1/1	CO	11	83	$\mathrm{Ar(C{=}O)_2H}$ (4%), $\mathrm{Ar(C{=}O)_2Ar}$ (1%)
5. $\mathrm{Ar_4Cu_2Li_2}$	1/2	CO	43	39	ArH (19%)
6. $\mathrm{Ar_4Cu_2Li_2 \cdot 2\ TMED}$	1/1	CO	—	95	
7. $\mathrm{Ar_4Cu_2Li_2}$	1/1[d]	Ethene	95	—	
8. $\mathrm{Ar_4Cu_2Li}$	1/2	CO	90	—	
9. $\mathrm{Ar_4Au_2Li_2 \cdot 2\ Et_2O}$	1/1	CO	69	31	
Ar = 2-tolyl					
10. $\mathrm{Ar_4Cu_2Li_2}$	1/1	CO	4	92	

[a] Calcd. (%) on the amount of Ar in the starting $\mathrm{Ar_4M_2Li_2}$ compound. [b] Trace amounts. [c] A similar result was obtained by using $\mathrm{Ar_4Cu_2Li_2 \cdot 2\ Et_2O}$. [d] *trans*-$\mathrm{PtCl_2(CH{=}CH_2)_2}$.

1/1 to 2/1 lowered the yield of the ketone considerably, whereas the yield of 4,4′-bitolyl increased to 43%. Furthermore, 20% of toluene was present in the reaction mixture; this is not the result of a radical-type reaction, but arises from hydrolysis of tolylmetal species during the work-up.

The coupling reaction proceeds by intramolecular pathways rather than via free-radical routes, as can be inferred from the formation of 95% of 4,4′-bitolyl in the reaction of VII with $Rh(C_2H_4)_2Cl$-dimer (XII). In this reaction, which was carried out in an atmosphere of ethene, formation of the diaryl ketone cannot occur.

The reaction seems to be independent of steric factors (cf. results of 2- and $(4\text{-tolyl})_4Cu_2Li_2$ with II). However, the reaction course is determined by the presence of potential built-in ligands in the aryl groups. This is clearly illustrated by the result of the reaction of $(2\text{-tolyl})_4Cu_2Li_2$ (IX) with $Rh(CO)_2Cl$-dimer, which produces 2,2′-ditolyl ketone in 93% yield.

Furthermore, the nature of the Group IB metal in the metal IB-lithium species appears to play an important rcle during the product forming step. When di-4-tolylgoldlithium (VIII), which has exactly the same structural features [2] as di-4-tolylcopperlithium (VII), reacts with $Rh(CO)_2Cl$-dimer only 31% of the ketone is formed, while 4,4′-bitolyl is produced in 69% yield. The formation of 90% of 4,4′-bitolyl and the complete absence of 4,4′-bitolyl ketone in the reaction of $(4\text{-tolyl})_4Cu_2Li_2$ with *trans*-$PtCl_2(Pr_2S)_3$ in a CO atmosphere indicates that not only the Group IB metal but also the transition metal plays a specific rôle in the ketone formation.

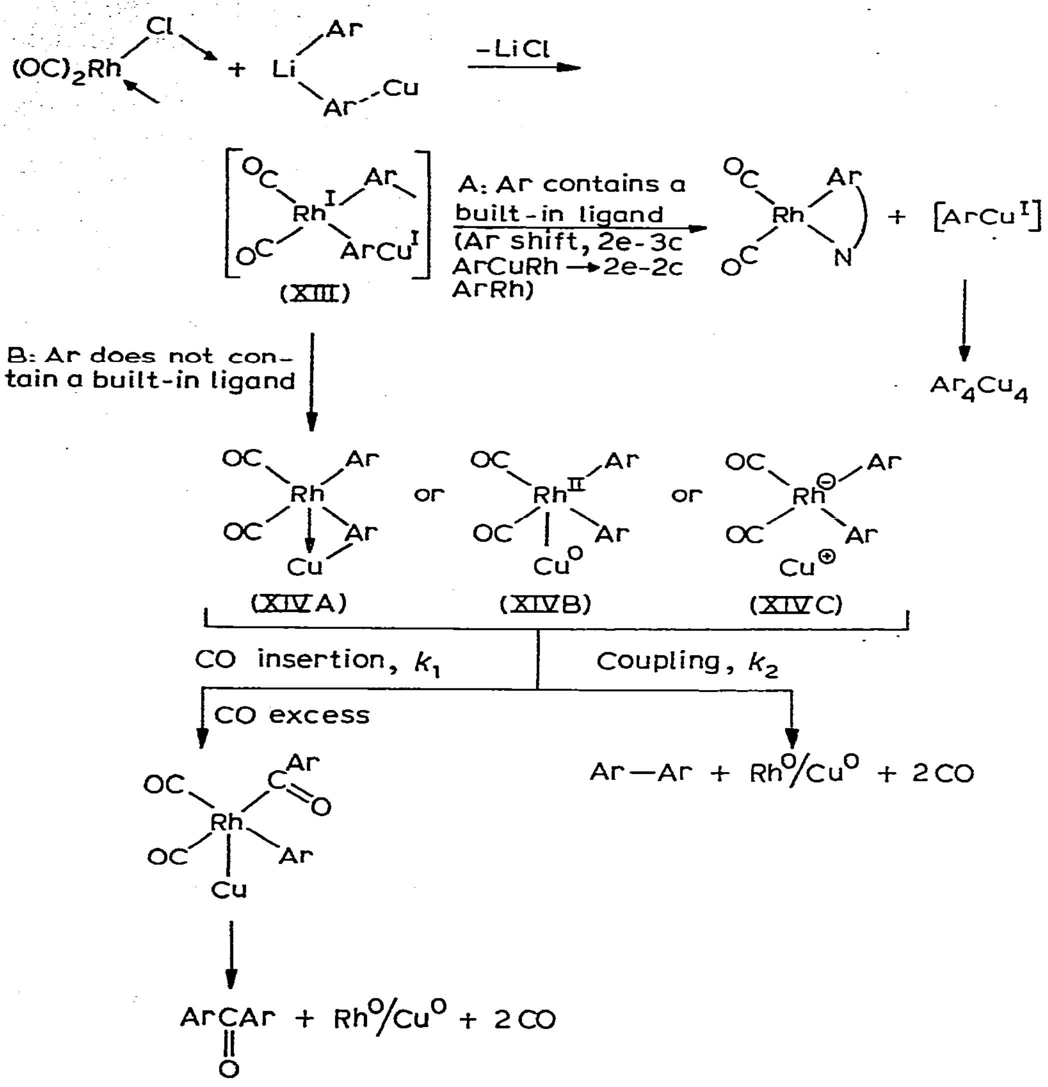
Finally, the best results were obtained when strong coordinating ligands were present in solution. Entries 4a and 4b show that both benzene-insoluble (4-$(4\text{-tolyl})_4Cu_2Li_2$ and benzene-soluble $(4\text{-tolyl})_4Cu_2Li_2 \cdot 2\,Et_2O$ give rise to the formation of 4,4′-bitolyl and 4,4′-ditolyl ketone in almost the same molar ratio. In contrast, the reaction of the complex of $(4\text{-tolyl})_4Cu_2Li_2$ with 1,2-bis(dimethylamino)ethane (TMED) [2] with rhodium dimer II produces the ketone XI in better than 95% yield.

Discussion

The present results show that substitution of the lithium atom in arylmetal IB-lithium cluster species of the type $Ar_4M_2Li_2$ by $Rh(CO)_2$ moieties gives rise to Cu^I—Rh^I products of low stability. The decomposition patterns of these compounds are dependent on whether or not intramolecular ligands are present in the aryl nuclei bound to the Cu^I—Rh^I core. Cluster disproportionation into the tetranuclear organocopper (III) [8] and the mononuclear rhodium compound (IV) is observed when the phenyl group contains a Me_2NCH_2 ligand in the *ortho* position with respect to the metal—carbon bond. However, when built-in ligands are absent release of aryl/acyl or aryl/aryl pairs from the Cu^I—Rh^I intermediate takes place. The absence of arenes amongst the reaction products, which would arise from a free-radical reaction pathway, supports the view that the reaction proceeds via an intramolecular pathway.

The two reaction patterns (A and B in Scheme 1) can be rationalized on the basis of electronic and geometric constraints of the metal IB—Rh^I intermediates formed.

SCHEME 1



In contrast with the metals present in the previously reported mixed metal clusters, e.g. $R_4Au_2Cu_4OTf_2$ [1,3], $R_4Au_2Ag_4OTf_2$ [1,3], $R_4Au_2Li_2$ [3,7], $R_4Au_2Cu_2$ [7] and $R_6Au_2Zn_2$ [10], Rh^I has considerable Lewis basicity. Rhodium(I) complexes, which have planar tetracoordinate structures, can form donor bonds to metals via the filled d_{z^2} orbital [11]. The proposed reaction pattern in Scheme 1 takes into account this important difference.

The first step involves substitution of the lithium atom in the $Ar_4M_2Li_2$ species by $Rh(CO)_2$ units, affording intermediates of type XIII, of which the aggregation state is unknown. The rhodium atom is assumed to take part in two 2e—3c aryl-to-metal bonds whereby it becomes coordinatively saturated.

In case the aryl group contains a built-in ligand such as CH_2NMe_2 the aryl group shifts from a 2e—3c aryl-to-metal bonded situation in XIII to a 2e—2c aryl—Rh interaction. This aryl shift is assisted in the case of Ar = $C_6H_4CH_2NMe_2$ by attack of the amino ligand at the fourth coordination site of the rhodium thereby displacing the second 2e—3c aryl-to-metal interaction in which rhodium takes part with formation of [ArCu].

Similar subtle shifts of 2e—3c to 2e—2c aryl-to-metal bonding have been observed in $(2\text{-}Me_2NCH_2C_6H_4)_4Cu_4$ ($2\text{-}Me_2NCH_2C_6H_4$ bridges two Cu atoms via 2e—3c $C{-}Cu_2$ bonds [8]) as compared with $[2\text{-}Me_2NCH(CH_3)C_6H_4Au]_2$ (2e—2c C-Au bonds [2,9]) and in $(2\text{-}Me_2NC_6H_4)_4Cu_6X_2$ ($2\text{-}Me_2NC_6H_4$ bridges two Cu atoms via 2e—3c $C{-}Cu_2$ bonds [12]) as compared with $(2\text{-}Me_2NC_6H_4)_4Au_2Cu_4OTf_2$ (contribution of canonical structures such as $R_2Au^{-}Cu_4X_2{}^{2+}{}^{-}AuR_2$ with 2e—2c C—Au bonds to the total bonding [1,3]).

When built-in ligands are absent a similar shift in the bonding pattern of XIII takes place. Possible formulations for the resulting intermediates are XIVA, XIVB or XIVC. Structures of type XIVA containing Rh-to-Cu donor bonds with one of the aryl groups bridging Rh and Cu have recently been reported for $(Ph_3P)_2(CO)RhCu(Me{-}\bar{N}\overset{-}{-}\bar{N}\overset{-}{-}\bar{N}{-}Me)Cl$ [11a] (X-ray: Rh—Cu 2.73 Å [11b]).

Structures of type XIVB or XIVC in which the second aryl group is shifted from Cu to Rh, leading either to five-coordinated Rh^{II} and a Rh—Cu bond (XIVB) or to an "ate"-like species XIVC, have precedents in $[(PPh_3)(CO)_3Re(C_2C_6F_5)_2]^{-}(CuPPh_3)^{+}$ [13], $\{(PPh_3Cu)^{+}[RuC_5H_5(C_2C_6F_5)_2]^{-}\}_2$ [14] and $[Ru(C_2C_6F_5)_5(PPh_3)]^{2-}(AgPPh_3)_2{}^{+}$ [15]. These compounds result from reactions in which acetylide groups, initially bound to copper or to silver, migrate to rhenium or to ruthenium.

In the intermediate (XIVA—XIVC) either CO insertion into the Ar—Rh bond takes place followed by *e*-transfer from Rh to Cu with concomitant release of the diaryl ketone, or coupling of two aryl groups occurs leading to biaryl.

On the basis of this scheme the observed influence of the nature of the IB metal and of the presence of internal and external ligands on the products formed can be rationalized.

The use of a CO atmosphere promotes the CO insertion step by stabilizing the arylacylrhodiumcopper intermediate. In the presence of excess CO the rhodium atoms again become coordinatively saturated after the insertion step. In the absence of excess CO (reactions in an N_2 atmosphere) competitive biaryl formation takes place to a larger extent, while in the reaction of the rhodium ethylene complex XII biaryl is released exclusively.

From the viewpoint of copper chemistry it is noteworthy that none of the arylmetal IB-lithium species undergoes CO insertion:

$$Ar_4M_2Li_2 \xrightarrow{CO} \text{no reaction } (M = Cu \text{ or } Au)$$

The use of $(4\text{-tolyl})_4Au_2Li_2$ (VIII) [2] species instead of the copper-lithium compound (VII) leads to a gold-rhodium intermediate which is less stable than the copper-rhodium intermediate (XIVA—XIVC) for two reasons: (i) the inherently greater Lewis acidity of Cu^{I} as compared with Au^{I} [16], which results in a weaker Rh-to-Au interaction and (ii) as compared with Cu^{I}, Au^{I} has a lower tendency to transfer both aryl groups to Rh. Accordingly, in this reaction coupling of aryl groups is faster than CO insertion (cf. formation of 69% of X and 31% of XI).

Finally, the highly selective formation of diaryl ketone in the presence of TMED can be explained on the basis of complex formation of the TMED molecule with copper, i.e., by stabilization of structure XIVC. This observation may indicate that it is in an intermediate of type XIVC that the CO insertion takes place.

Recently Eaborn et al. [17] have reported the formation of ketones from the reaction of acyl chlorides with triethylsilane in the presence of rhodium complexes. Product formation was explained in terms of a catalytic cycle involving a sequence of oxidative addition and reductive elimination steps one of which was the elimination of R_2CO from a $Rh^{III}(R)$ (COR) species. Hegedus et al. [18] have reported the alkylation of acyl chlorides by alkylrhodium(I) complexes, which is thought to occur via oxidative addition of acyl chloride to alkylrhodium(I) followed by elimination of R_2CO from a $Rh^{III}(R)(COR))$ species. These results seem to support the importance of intermediates in which both aryl groups are bound to rhodium in the present coupling reactions.

Conclusions

The synthesis of novel $2\text{-}Me_2\overline{NCH_2C_6H_4Rh}(CO)_2$ in almost quantitative yield via the 1/1 reaction of $ClRh(CO)_2$-dimer with $(2\text{-}Me_2NCH_2C_6H_4)_4M_2Li_2$ (M = Cu or Au) indicates that these IB metallithium compounds are excellent arylating agents for rhodium(I). Precedents of the use of organocopper compounds for the synthesis of organo-transition metal compounds are limited in number and so far were restricted to the above mentioned reactions of copper acetylide compounds with transition metal carbonyl halide complexes [13—15].

The proposed scheme provides a rationale for the occurrence of transarylation in the case where built-in ligands are present as well as for the selective formation of diaryl ketones in the case where built-in ligands are absent. The latter reaction represents a novel route for ketone formation by a IB metal assisted aryl—acyl coupling reaction on a transition metal center.

Experimental

General

All reactions were carried out in a dry oxygen-free atmosphere (either nitrogen, ethylene or carbon monoxide). Solvents were carefully purified, dried and distilled before use under dry, oxygen-free nitrogen. ^1H NMR spectra were recorded on a Varian Associates HA 100 or T60 NMR spectrometer. IR spectra were recorded on a Perkin—Elmer 577 Grating IR spectrometer. Combined GC/MS analysis were carried out with a Finnigan 3100 D GC/MS-data system.

^1H NMR, IR and GC/MS measurements were carried out by Mrs. G.M. Bijlsma-Kreuger, Mrs. T. van Montfort-Volp, Mrs. G.G. Versluis-de Haan and Mr. J.W. Marsman.

Bis{2-[(dimethylamino)methyl]phenyl}-copperlithium (I) and -goldlithium (V) were prepared by published methods [5,7,19]. Synthesis and characterization of ditolyl-copperlithium (VII or IX) and -goldlithium (VIII) compounds are described in a separate paper [2].

Interaction of bis{2-[(dimethylamino)methyl]phenyl}copperlithium (I) with di-μ-chlorotetracarbonyldirhodium (II)

Solid II (1.48 mmol) was added to a stirred solution of I (1.48 mmol) in benzene (40 ml). The mixture immediately turned red and a white precipitate (LiCl) was formed. NMR spectroscopy of the solution revealed that 2-[(dimethylamino)methyl]phenylcopper (III; 43% *), {*C*,*N*-2-[(dimethylamino)methyl]phenyl}dicarbonylrhodium (IV; 50% *) and *N*,*N*-dimethylbenzylamine (7% *) were present.

The solution was decanted and concentrated at reduced pressure. The yellow precipitate which was filtered off, washed with pentane and dried in vacuo, appeared to be pure III (NMR, benzene-d_6, δ(ppm): NCH_3, 1.90(s); NCH_2, 3.02(s); H_6, 8.45(m); IR identical to a pure sample of III [8]).

The pentane solution was concentrated and the resulting oily residue chromatographed over silica gel (column 30 cm/1.5 cm) with benzene as eluent. IV was isolated as an orange crystalline solid. M.p. 48—50°C. Found: C, 45.1; H, 4.1; N, 4.7; O, 10.9. $C_{11}H_{12}NO_2Rh$ calcd.: C, 44.8; H, 4.1; N, 4.6; O, 11.0%. Mol. wt. osmometric in benzene: found (calcd.), 285 (293). IR (Nujol, cm^{-1}), 1985vs and 2050vs. ^1H NMR vide supra.

Interaction of bis{2-[(dimethylamino)methyl]phenyl}goldlithium (V) with di-μ-chlorotetracarbonyldirhodium

Solid II (0.53 mmol) was added to a stirred solution of bis{2-[(dimethylamino)methyl]phenyl}goldlithium (V; 0.53 mmol). A white precipitate formed immediately. The solution was decanted and concentrated, affording crude IV in 97% yield. The white precipitate (0.180 g: 0.53 mmol VI and 0.53 mmol LiCl = 0.190 g) was washed with benzene, dried in vacuo and subsequently dissolved in pyridine-d_5. NMR spectroscopy revealed the presence in solution of 2-[(dimethylamino)methyl]phenylgold (VI), identical to that of an analytically pure sample [3] while the insoluble white solid appeared to LiCl (according to Cl analysis).

Reaction of di-tolyl-copperlithium or -goldlithium compounds with rhodium(I) salts

The copperlithium or goldlithium complex was dissolved (or suspended) in benzene under CO atmosphere. Solid rhodium salt (II or XII) was added to the stirred solution (suspension) at once. The resulting dark-coloured reaction mixture was stirred for 15 min. Subsequently, 10 ml of a 4 *N* HCl solution were added and the resulting mixture stirred for 10 min. The layers were separated and then filtered over silica gel (1.5 cm length). The clear yellow filtrate was concentrated and the residue analysed by NMR, IR and GC/MS techniques. 4,4'-ditolylketone: ^1H NMR in C_6D_6 (δ, ppm), 7.72 ($H_{2,6}$, d of d), 6.89 ($H_{3,5}$, d of d), 2.00 (4-CH_3, s); MS, parent ion *m/e* 210, RCO^+ *m/e* 119. 4,4'-bitolyl: ^1H NMR in C_6D_6 (δ ppm), 7.43 ($H_{2,6}$, d of d), 6.95 ($H_{3,5}$, d of d), 2.15 (4-CH_3, s); MS, parent ion *m/e* 182. Full data are presented in Table 1.

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* Calculated on the amount of 2-[(dimethylamino)methyl]phenyl in I.

Reaction of di-4-tolylcopperlithium/TMED complex with di-μ-chlorotetracarbonyldirhodium

VII (0.306 mmol) was dissolved in benzene (1 ml) under CO atmosphere. An equimolar amount of 1,2-bis(dimethylamino)ethane (TMED) was added to the pale-yellow solution which resulted in an immediate formation of a white precipitate of $(4\text{-tolyl})_2CuLi \cdot TMED$. NMR spectroscopy established the absence of organocopper species in solution. Subsequently, solid II (0.306 mmol) was added at once to the stirred suspension affording a dark-coloured reaction mixture which was stirred for an additional 15 min and then worked up as is described above. NMR spectroscopy ($H_{2,6}$, 7.72 d of d; $H_{3,5}$, 6.89 d of d; $4\text{-}CH_3$, 2.00 s ppm) pointed to the exclusive formation of 4,4′-ditolylketone (95% yield).

Acknowledgement

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