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Gold-Catalyzed Hydroarylating Cyclization of 1,2-Bis(2-iodoethynyl)benzenes

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Abstract: 1,5-Diynes bearing halogen-substituted alkynes were synthesized and converted in the presence of a gold catalyst. In contrast to the corresponding hydroarylating aromatization reaction with terminal alkynes, a totally different reaction mode was observed. Instead of the expected dual catalysis pathway, only one gold center is needed and a 1,2-halogen migration is initiated in which either a gold vinylidene species or a gold carbenoid is involved. By the incorporation of one solvent molecule, diiodinated aromatic products are obtained in high selectivity.

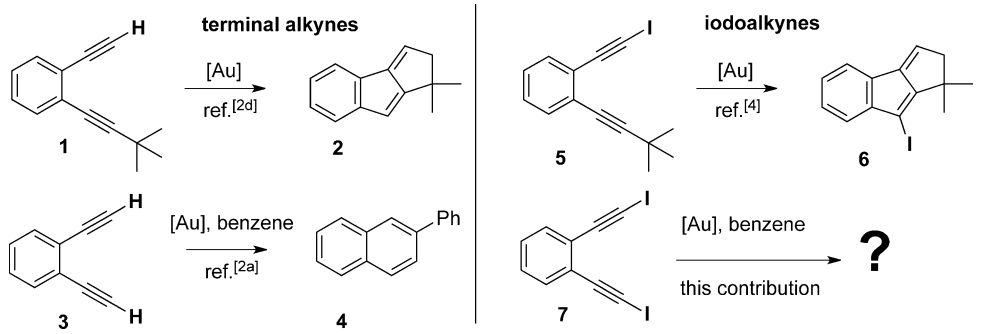
Keywords: gold catalysis; gold vinylidenes; 1,2-halogen migration; iodoalkynes; naphthalenes

Introduction

Amidst the continuous stream of new homogeneous gold-catalyzed reactions the field of diyne cyclizations is steadily gaining importance. The reactions can either be initiated by π-activation alone[1] or by a synergistic interplay between two gold fragments in which σ/π-activation (dual catalysis) takes place.[2] As an expansion of the dual catalysis approach, we could show that iodoalkynes were also suitable substrates for these kinds of transformations. This was demonstrated by the synthesis of iodofulvenes **6** which were formed from the corresponding iodoalkynes **5**. The crucial steps in this transformation were shown to be the formation of a gold acetylide. This only took place in the presence of an organogold compound as additive and a catalyst transfer from the aurated product to the iodoalkyne which closes the catalytic cycle. Based on these findings, we were curious if the same reaction principle was also feasible for related intermolecular processes. As model systems we investigated iododiyne **7** as suitable substrate. The corresponding terminal diyne system **3** has already been shown to add benzene in a β-selective fashion under dual catalysis conditions. The results of the cyclization with iodoalkynes as starting materials are summarized in this contribution (Scheme 1).

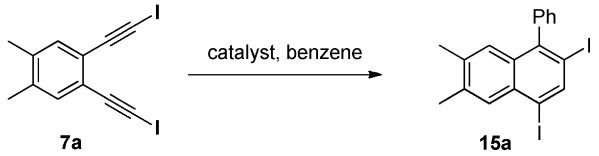
Results and Discussion

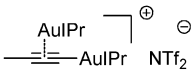
An initial screening was performed with the 1,2-bis-(iodoethynyl)-4,5-dimethylbenzene derivative **7a**. Indeed the formation of one major product was detected with most of the applied catalysts. A summary of the screening (GC yields) is depicted in Table 1. Our first choice of catalyst was DAC (dual activation catalyst) **8**[3] as these types of catalyst turned out to be the best choice for the synthesis of iodofulvenes **6**.[4] Indeed 48% of product was detected by GC with 2.5 mol% of **6**, which corresponds to 5 mol% of mononuclear gold(I) complexes. Shifting to the corresponding "normal" cationic gold complex $IPrAuNTf_2$ **9** [now 5 mol% instead of 2.5 mol% of **6** as **9** is a mononuclear gold(I) complex] delivered a rather unexpected picture. While in the case of the iodofulvene synthesis organogold additives or dual catalysts were crucial for the transformation of the iodoalkyne



Scheme 1. Overview of gold-catalyzed transformations of aromatic diynes.

Table 1. GC yields for the conversion of **7a** to **15a** employing various catalysts.



Entry	Catalyst		Yield [%]
1		**8** (2.5 mol%)	48
2	$IPrAuNTf_2$	**9** (5 mol%)	76
3	AuCl	**10** (5 mol%)	50
4	$Ph_3PAuNTf_2$	**10** (5 mol%)	39
5	$BrettPhosAuNTf_2$	**11** (5 mol%)	0
6	*t*-Bu-$XPhosNTf_2$	**12** (5 mol%)	0
7	$AuCl_3$	**13** (5 mol%)	51
8	$AgNTf_2$	**14** (5 mol%)	0

starting materials, for this transformation an even faster reaction was detected for the mono-metallic complex **9** and in addition yields were also better than for the corresponding DAC. As a consequence our further screening was conducted with monomeric gold complexes. Simple AuCl **10** was also efficient (entry 3), but yields turned out to be lower than for the NHC ligand. In a series of phosphane ligands only the common $Ph_3PAuNTf_2$ **10** showed significant product formation (entry 4), while Buchwald ligands turned out to be inefficient (entries 5 and 6). Gold in the oxidation state III was also effective, but yields were only moderate (entry 7). The control experiment with only silver showed no conversion (entry 8).

Next we performed the transformation on a preparative scale (190 μmol) under the optimized conditions in order to completely characterize the obtained species. Unfortunately, the isolated yield now was significantly lower than the GC yield (54%). This might result from a partly decomposition of the starting material, which turned out to be not stable under the reaction conditions. To suppress competing decomposition pathways, we performed a reaction under the same conditions except that the starting material was added *via* syringe pump to keep its concentration as low as possible (Table 2, entry 1). This procedure allowed the isolation of **15a** as pure compound in 70% yield. Crystals suitable for a single crystal X-ray structure analysis could be obtained for a secure structural assignment.[5] Figure 1 depicts the solid state molecular structure of compound **15a**. Indeed the expected benzene incorporation into the cyclization product took place, but unlike the related hydroarylating aromatization of the substrate not bearing iodine substituents,[6] for the diiodo starting material the aromatic solvent was selectively placed in the *peri*-position of the naphthalene system. Furthermore, a 1,2-iodine migration from the former position next to the alkyne into the second *peri*-position of the naphthalene system must have taken place. It is noteworthy that 5% of the product containing three iodine atoms on the naphthalene system was observed as disorder in the XRD structure.

In order to check the generality of this transformation different diiododiynes **7** were converted under the optimized conditions (Scheme 2, Table 2). First we examined substrate **7b** (entry 2) with no additional substituents at the benzene backbone. A clean conversion delivered the desired product **15b** in high yield. Electron-withdrawing halides at the benzene backbone were also tolerated. With the more electronegative fluoride substituent yields were only moderate (entry 3) but shifting to bromide substitution enabled good yields again (entry 4). Problems occurred with diester-substituted starting material **7e**. Due to low solubility no syringe pump addition was possible and furthermore decomposition of the catalyst $IPrAuNTf_2$ was observed. Shifting to 10 mol% AuCl delivered the target compound **15e** but yields were still poor (entry 5). Next we investigated substrate **7f** containing two electron-donating methoxy groups. These were tolerated well and the corresponding product was isolated in 69% yield (entry 6). 1,3-Benzodioxole derivative **7g** delivered only a complex mixture which might be due to competing reactions in-

Table 2. Conversion of various halodiynes under optimized conditions.

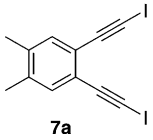
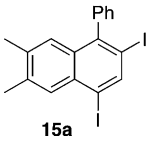
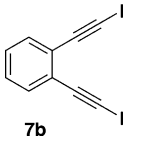
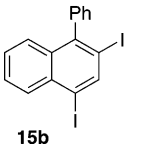
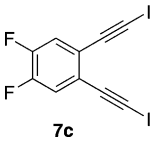
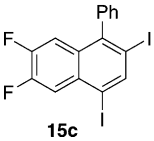
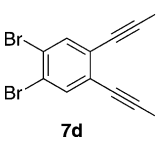
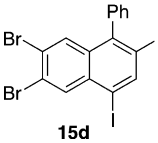
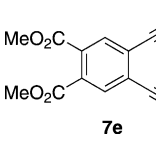
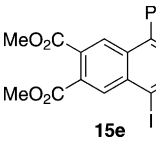
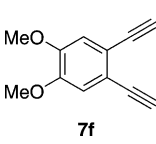
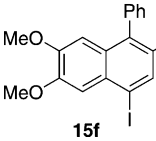
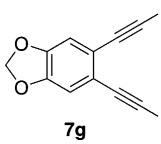
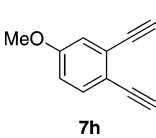
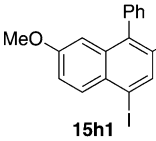
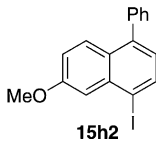
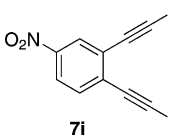
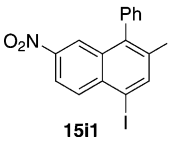
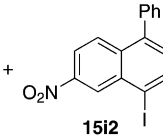
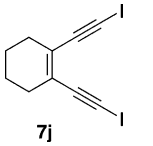
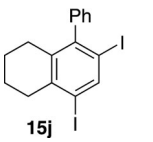
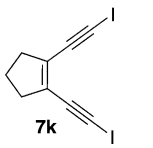
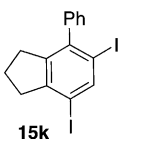
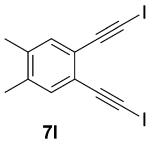
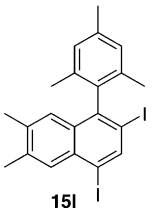
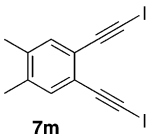
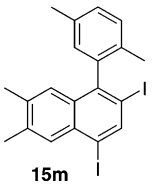
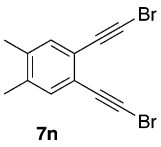
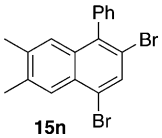
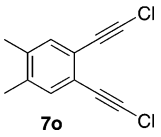
Entry	Substrate	Product	Yield [%]
1	 **7a**	 **15a**	70
2	 **7b**	 **15b**	85
3	 **7c**	 **15c**	48[b]
4	 **7d**	 **15d**	78
5	 **7e**	 **15e**	36[a]
6	 **7f**	 **15f**	69
7	 **7g**		unselective
8	 **7h**	 **15h1** +  **15h2**	53 (isomeric mixture 2:1)
9	 **7i**	 **15i1** +  **15i2**	45 (isomeric mixture 3:2)
10	 **7j**	 **15j**	74
11	 **7k**	 **15k**	43[b]

Table 2. (Continued)

Entry	Substrate	Product	Yield [%]
12	 **7l**	 **15l**	32
13	 **7m**	 **15m**	41
14	 **7n**	 **15n**	53[c]
15	 **7o**		no conversion[c]

[a] 10 mol% AuCl; no addition *via* syringe pump due to poor solubility in benzene.
[b] Contains traces of inseparable by-products.
[c] 20 mol% AuCl at 80 °C.

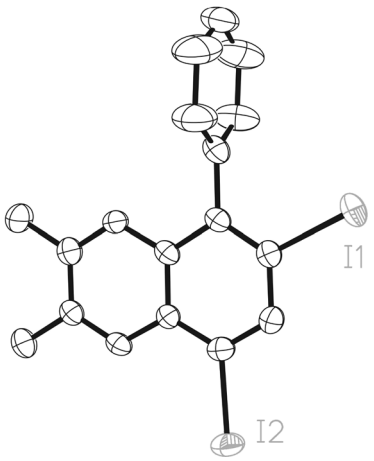
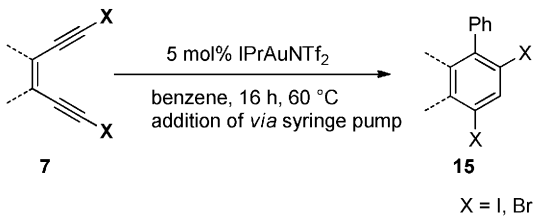


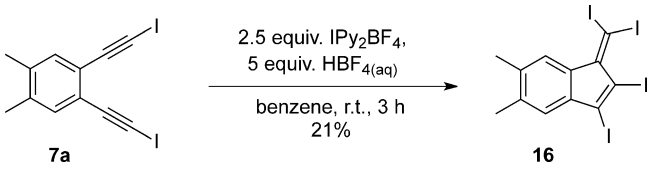
Figure 1. Solid state molecular structure of compound **15a**.[5]



Scheme 2. Hydroarylating cyclization of halodiynes **15**.

duced by the acetal functionality.[7] Unsymmetrically substituted starting materials **7h** and **7i** delivered the corresponding products in acceptable yields (entries 8 and 9) but no strong effect on the regioselectivity could be induced *via* the electronic nature of the substituents and therefore inseparable regioisomeric mixtures were obtained for both test substrates. Next we turned our focus to non-aromatic backbones as possible precursors for diiodobenzene derivatives. The gold-catalyzed transformation of cyclohexene substrate **7j** smoothly delivered the desired benzene derivative **15j** in 74% (entry 10). Unfortunately, the reaction with the corresponding cyclopentene substrate **7k** only delivered a moderate yield and furthermore the product was contaminated by traces of inseparable by-products (entry 11).

Next we evaluated the possibility to apply other aromatic solvents. When used as solvents, both mesitylene (entry 12) and *para*-xylene (entry 13) delivered the expected products but the yields turned out to be significantly lower than in the case of benzene as solvent. Finally, we tested the suitability of other halogens for this transformation. With dibromo derivative **7n** (entry 14) a corresponding reactivity was obtained, but in this case higher temperatures combined with higher catalyst loadings were necessary (in this case AuCl turned out to be the catalyst of choice). The di-



Scheme 3. Formation of the tetraiodinated product **16**.

chloro derivative **7o** was completely unreactive (entry 15).

As electrophilic iodine itself is known to be a good π-activator as well,[8] we also performed test reactions with diiodoalkyne **7a** in the presence of electrophilic iodine sources instead of the gold catalyst (Scheme 3). In the presence of 2.5 equivalents of the Barluenga reagent and an excess of aqueous HBF_4 for activation the tetraiodinated compound **16** was formed in low yield (Scheme 6). The surprising structure of **16** was confirmed by crystal structure analysis (Figure 2).[5]

Our next efforts focused on the elucidation of more mechanistic features. Scheme 4 displays the reaction in deuterated benzene under the normal reaction conditions. About 80% of the deuterium label was incorporated at the position between the two iodine atoms.

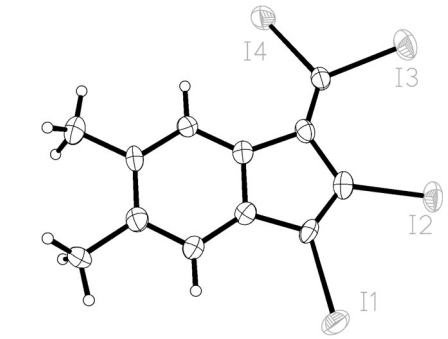
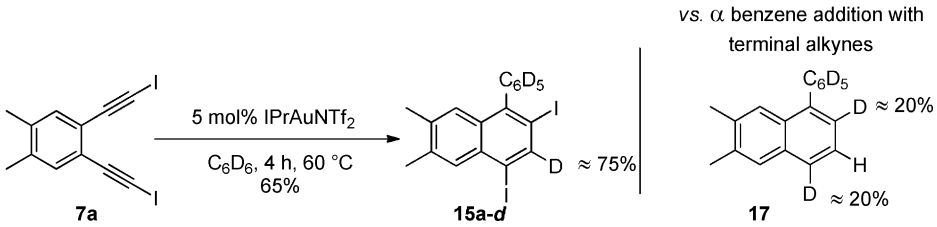
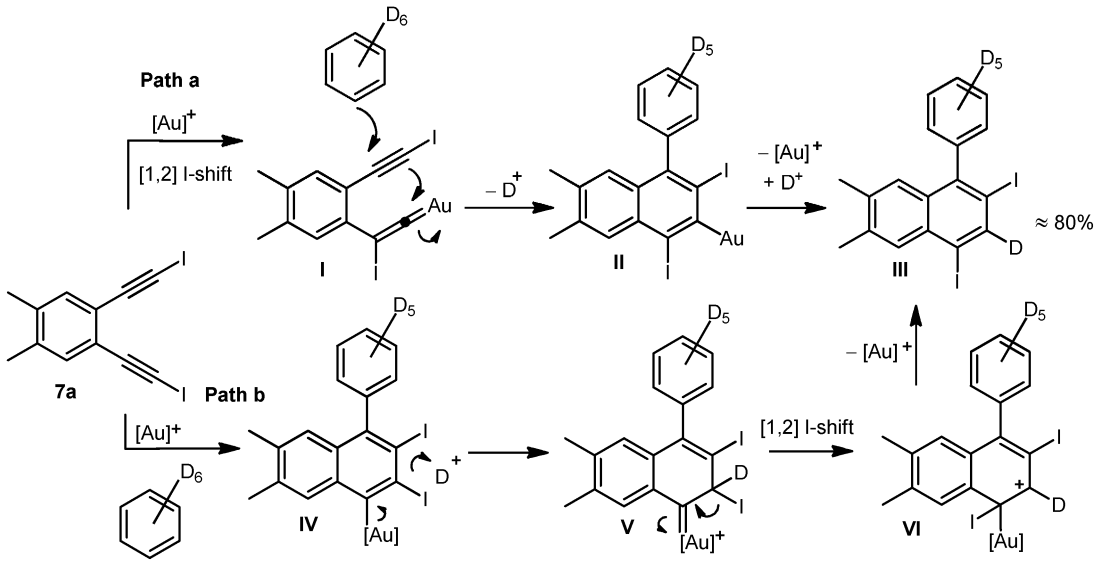


Figure 2. Solid state molecular structure of compound **16**.[5]

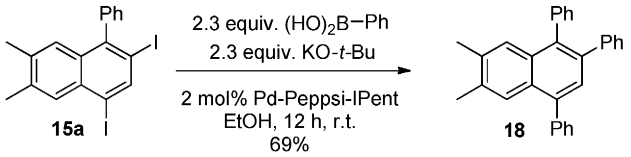
It is highly likely that the incorporation takes place *via* protodeauration, thus a gold fragment should be located at this position at a late stage of the reaction. If one compares the deuterium labelling of the iodo reaction with the related hydroarylating reaction towards α-substituted naphthalenes (that are formed if high catalyst loadings are applied) (Scheme 4, right), it becomes obvious that a completely different reaction mechanism must take place as in this case no deuterium incorporation at this position takes place, instead the two adjacent naphthalene positions are



Scheme 4. Deuterium labelling experiments.



Scheme 5. Possible mechanisms.



Scheme 6. Suzuki cross-coupling of substrate **15a** with phenylboronic acid.

deuterated to a degree of about 20% each.[9] Based on these findings, two mechanistic scenarios are reasonable (Scheme 5). Path **a** involves a gold(I) vinylidene species **I** that is generated *via* a 1,2-iodine shift. The formation of a gold(I) vinylidene species by a 1,2-iodine shift was already discussed by Fürstner's group in the gold-catalyzed synthesis of halophenanthrenes[10] and in a recent publication on the synthesis of 3-iodo-2*H*-chromene derivatives by the González group.[11] Induced by the high electrophilicity of the generated vinylidene species **I**, cyclization takes place in the next step. Upon addition of benzene to the α-position a proton/deuterium is released which protodeaurates the catalyst and closes the catalytic cycle. Path **b** is initiated by an arylating cyclization that would be related to the synthesis of α-substituted napthhalenes from the corresponding terminal starting materials under "α-conditions".[12] Upon addition of benzene a proton/deuterium would be released that could protonate in β-position to the gold fragment which would generate a gold carbene intermediate **V**. After selective 1,2-iodine migration and subsequent elimination of the catalyst, product **III** would also be formed. Like for path **a** this pathway would be in accordance with the observed deuterium labelling. Therefore we cannot rule out this mechanistic scenario even if it is highly speculative that instead of protodeauration, protonation and carbene formation should be favored.[8,13] The absence of protodeauration products originating from intermediate **IV** could be evidence in favor of path **a**. In the context of these reactions, it should be kept in mind that previous investigations on the iodine-induced cyclizations of dialkynylarenes gave different products, and also iodoalkynes have been dimerized or been used in cycloisomerization reactions.[14]

To demonstrate briefly that the iodinated catalysis products can be suitable substrates for further metal-mediated cross-couplings, substrate **15a** was coupled with phenylboronic acid in an acceptable yield.

Conclusions

In conclusion, we have demonstrated that diododiynes are suitable substrates for gold catalysis. For this kind of substrate no dual catalysis pathway takes place. Instead products are obtained in which one of the iodine centers undergoes a 1,2 iodine migration. This methodology provides a fast access towards synthetically useful diiodonaphthalene derivatives which can be further functionalized by metal-mediated cross-coupling strategies.

Experimental Section

General Procedure for the Gold-catalyzed Conversion

Five mol% of $IPrAuNTf_2$ were dissolved in 0.5 mL of benzene and heated to 60 °C. The diiodo compound **7** was dissolved in additional 1.5 to 2 mL of benzene (depending on the solubility) and added dropwise (about 4 drops per minute) using a syringe pump. The reaction mixture was stirred at 60 °C until completion was indicated by TLC. Then the mixture was taken up with DCM and adsorbed onto Celite®. The solvents were removed under reduced pressure and the crude product purified by flash column chromatography.

2,4-Diiodo-6,7-dimethyl-1-phenylnaphthalene (15a): According to the general procedure, 1.00 equiv. of 1,2-bis(iodoethynyl)-4,5-dimethylbenzene (**7a**, 76.0 mg, 187 μmol) dissolved in 1.5 mL of benzene were added dropwise to the catalyst (8.10 mg, 9.36 μmol). The mixture was stirred for 2 h at 60 °C. After flash column chromatography (SiO_2, PE) **15a** was obtained as a yellow solid was obtained; yield: 60.0 mg (124 μmol, 70%). R_f (PE/EA 10:1) = 0.64; decomposition at 200 °C; IR (KBr): ν = 3055, 2971, 2937, 1623, 1546, 1496, 1443, 1325, 1266, 1130, 1027, 869, 699 cm^{-1}; ^1H NMR (300 MHz, CD_2Cl_2): δ = 2.28 (s, 3H), 2.45 (s, 3H), 7.09 (s, 1H), 7.20–7.23 (m, 2H), 7.47–7.55 (m, 3H), 7.84 (s, 1H), 8.51 (s, 1H); ^{13}C NMR (75 MHz, CD_2Cl_2): δ = 20.1 (q), 20.3 (q), 97.1 (s), 98.8 (s), 127.7 (d), 128.4 (d), 128.8 (d, 2C), 130.3 (d, 2C), 132.1 (d), 132.7 (s), 132.9 (s), 138.3 (s), 138.7 (s), 143.2 (s), 144.7 (d), 145.0 (s); MS (EI^+, 70 eV): *m/z* (%) = 484 (100) $[M]^+$, 230 (26), 215 (23); HR-MS (EI^+, 70 eV): *m/z* = 483.9194 $[M]^+$, calculated for $C_{18}H_{14}I_2^+$: 483.9179 $[M]^+$.

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8 Gold-Catalyzed Hydroarylating Cyclization of 1,2-Bis(2-iodoethynyl)benzenes

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