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ORGANICS

# Palladium-Catalysed Coupling Chemistry

# Palladium-Catalysed Coupling Chemistry

Palladium catalysis has gained widespread use in industrial and academic synthetic chemistry laboratories as a powerful methodology for the formation of C-C and C-Heteroatom bonds.



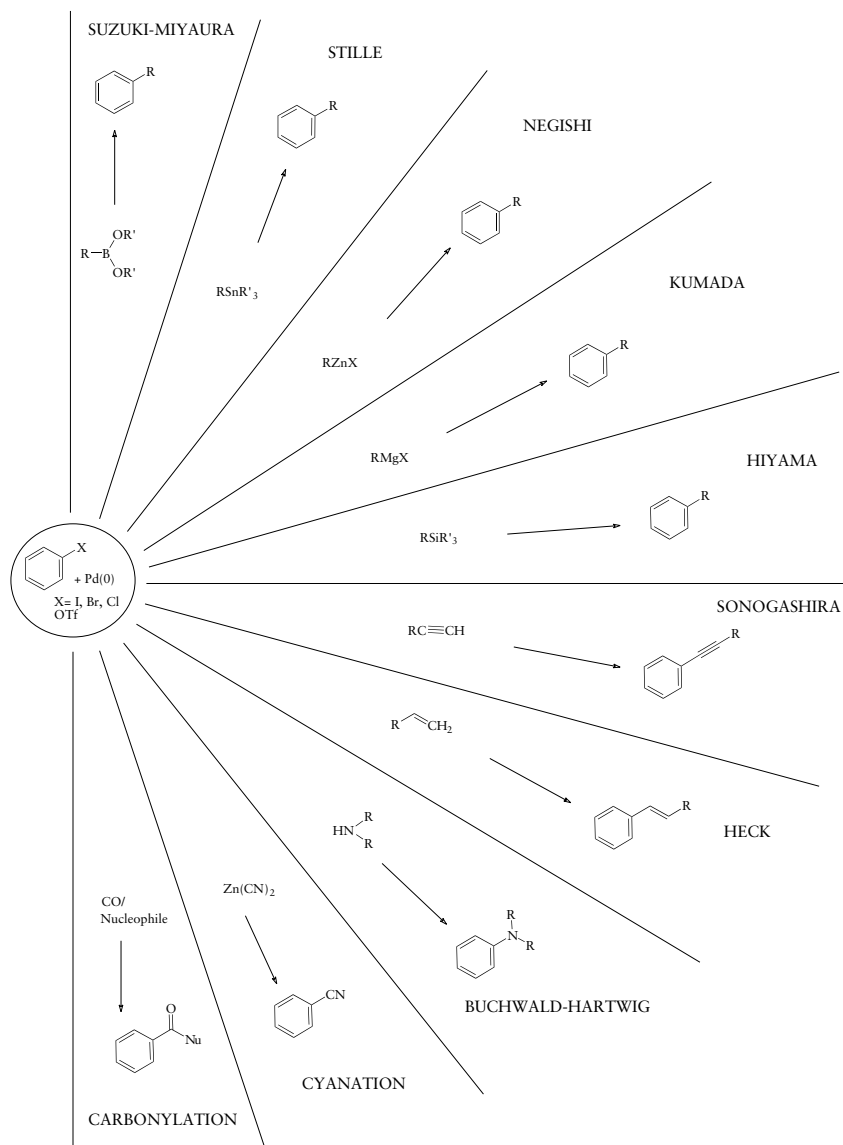
R = usually sp<sup>2</sup> hybridised carbon

X = usually I, Br, Cl or OTf

The nature of R' and M are dependant upon the specific coupling being performed

Several coupling reactions have been developed with different substrates:

1. SUZUKI-MIYAUURA
2. STILLE
3. NEGISHI
4. KUMADA
5. HIYAMA
6. SONOGASHIRA
7. HECK
8. BUCHWALD-HARTWIG
9. CYANATION
10. CARBONYLATION

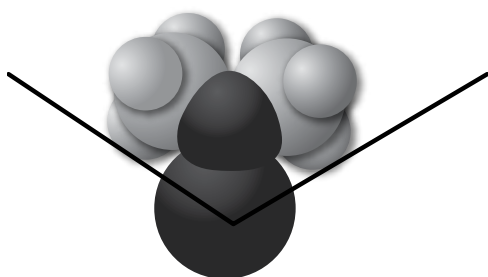
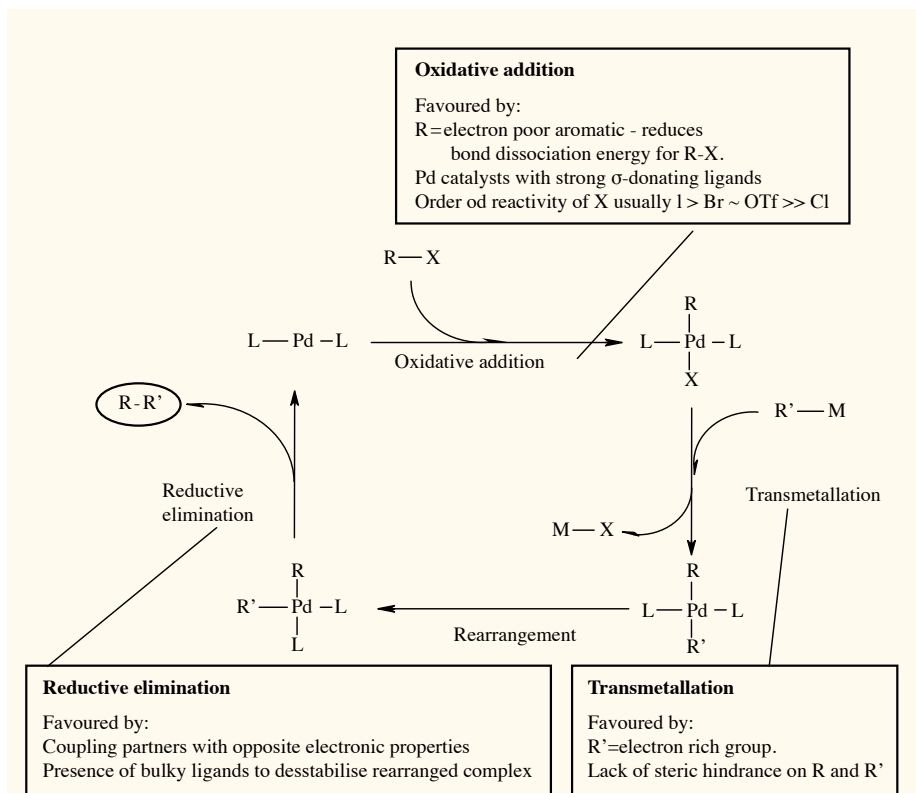


# Understanding the catalytic cycle

Most palladium catalysed reactions are believed to follow a similar catalytic cycle.

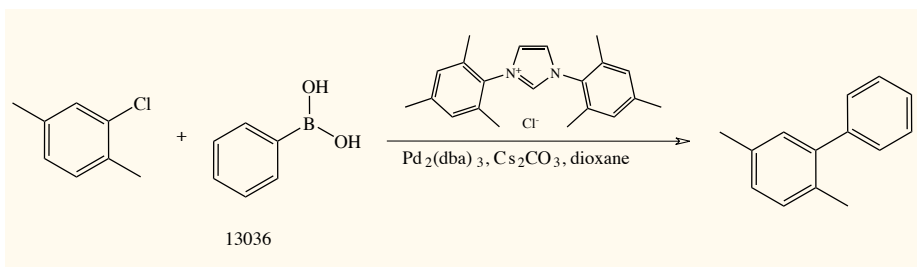
The catalytic species can be formed *in situ* using a palladium source, such as  $\text{Pd}_2(\text{dba})_3$  or  $\text{Pd}(\text{OAc})_2$  and the necessary ligand, or introduced as a preformed catalyst such as  $\text{Pd}(\text{PPh}_3)_4$  or  $\text{Pd}(\text{P}^t\text{Bu}_3)_2$ .

Careful choice of ligand can facilitate two steps of the catalytic cycle. The use of strong  $\sigma$ -donating ligands, such as trialkylphosphines, increases electron density around the metal, accelerating the oxidative addition of the catalyst to the substrate. This is most commonly believed to be the rate determining step. Choice of ligand also determines the mechanism by which oxidative addition occurs.<sup>1</sup> The elimination step is accelerated by the use of bulky ligands, in particular phosphine ligands exhibiting a large cone angle (also known as Tolman angle).<sup>2</sup>

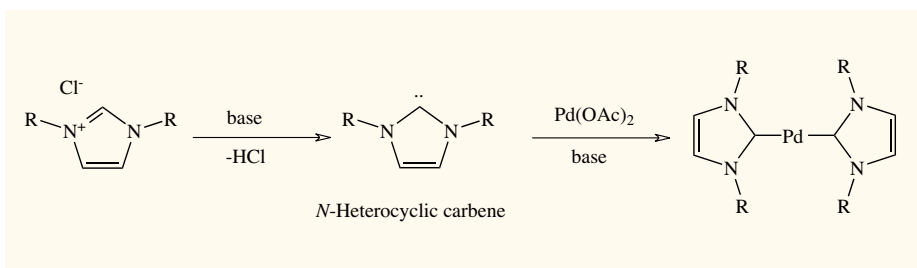


Ligand	Cone Angle (deg)	Cat. No.
dppm	121	29361
dppe	125	14791
dppp	127	31005
dcpe	142	36385
$\text{PPh}_3$	145	14042
$\text{P}(\text{c-hex})_3$	170	42161
$\text{P}^t\text{Bu}_3$	182	36089
$\text{P}(\text{C}_6\text{F}_5)_3$	184	31316
$\text{P}(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)_3$	212	32113

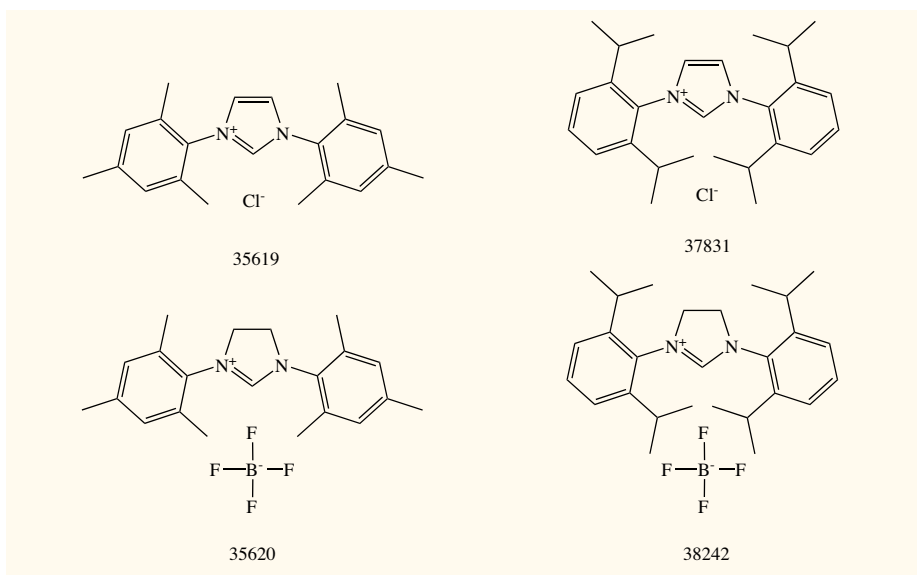
Phosphine ligands have recently been replaced in a number of palladium catalysed reactions with *N*-heterocyclic carbenes (NHCs).<sup>3</sup>



These ligands offer similar electronic properties to phosphines, being strongly  $\sigma$ -donating and weakly  $\pi$ -acidic. NHCs can offer very high catalytic activity combined with stability and longevity in comparison with phosphine ligands. The carbene is air sensitive but can be generated in situ to aid operational simplicity.



We offer a range of commonly used NHC precursors for use in cross coupling reactions.

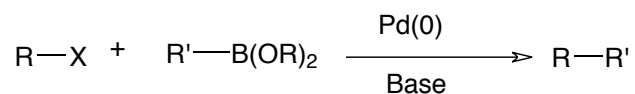


#### References

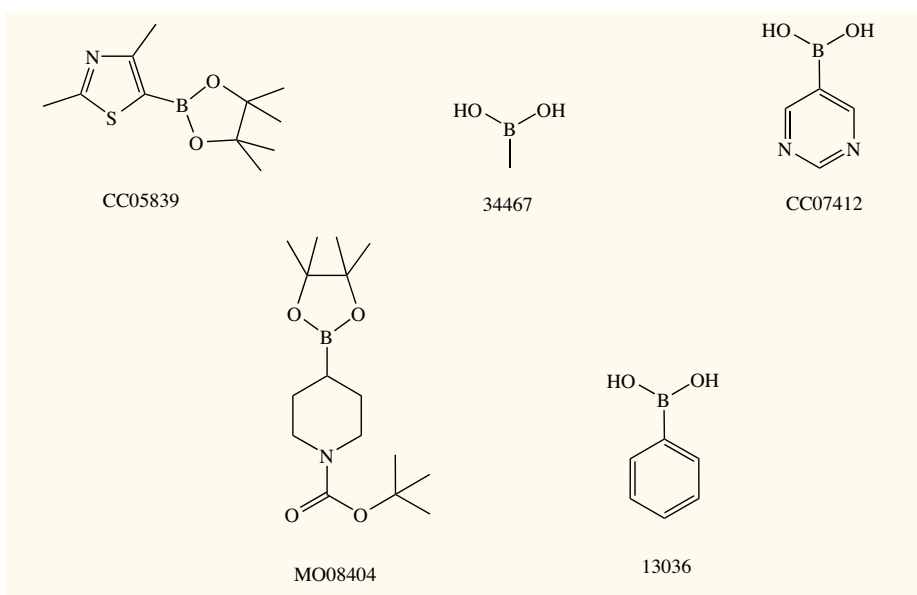
- Galardon, E.; Ramdeehul, S.; Brown, J. M.; Cowley, A.; Hii, K. K.; Jutand, A.; *Angew. Chem., Int. Ed.* **2002**, *41*, 1760-1763
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- For a review see: Hillier, A. C.; Grasa, G. A.; Viciu, M. S.; Lee, H. M.; Yang, C.; Nolan, S. P. *J. Organomet. Chem.* **2002**, 69-82

# Palladium Catalysed Reactions

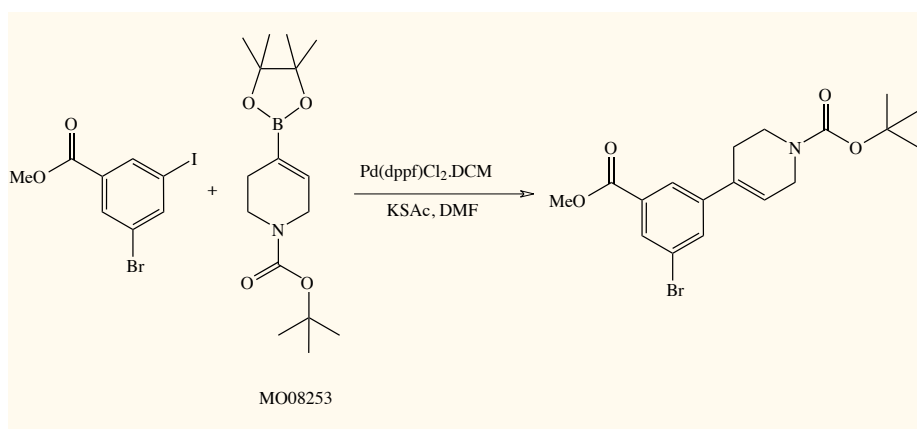
## 1) The Suzuki-Miyaura coupling



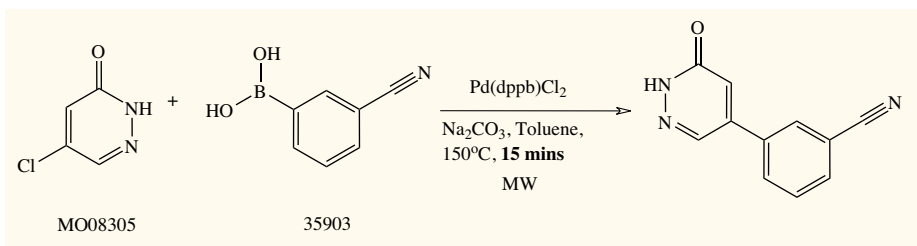
The Suzuki coupling reaction involves the cross coupling of organohalides (and their equivalents) with organoboron reagents. The organoboron reagent typically comes in the form of a boronic acid or ester, of which >300 structurally diverse examples are stocked under the Acros Organics and Maybridge brands, and requires activation by base or fluoride to enable it to undergo transmetalation.



The reaction is highly tolerant of many different functional groups, and boron containing by-products are easily removed by a simple alkali work-up. Although most commonly used to form aryl-aryl bonds the Suzuki reaction is just as effective for the synthesis of highly substituted styrene products.<sup>4</sup>

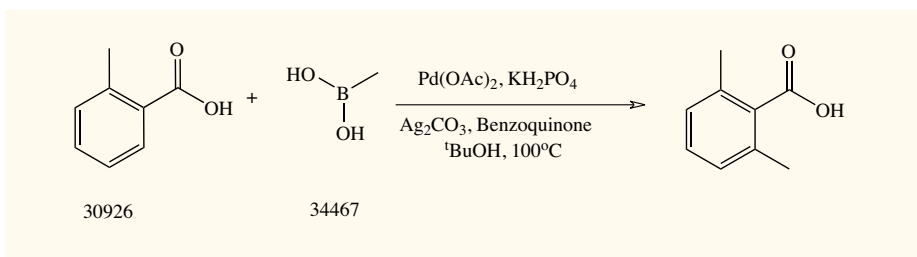


Suzuki chemistry is well known to be accelerated by the use of microwaves to heat the reaction.<sup>5</sup>

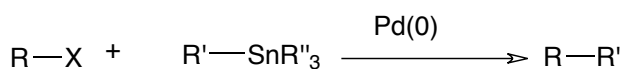


It can also be used to perform aromatic alkylations.<sup>6</sup> C-H insertion negates the necessity to begin with an aryl halide, improving the atom efficiency of the process.

Other organoboron species such as trifluoroborate salts can also be used in this reaction.<sup>7</sup>



## 2) The Stille coupling

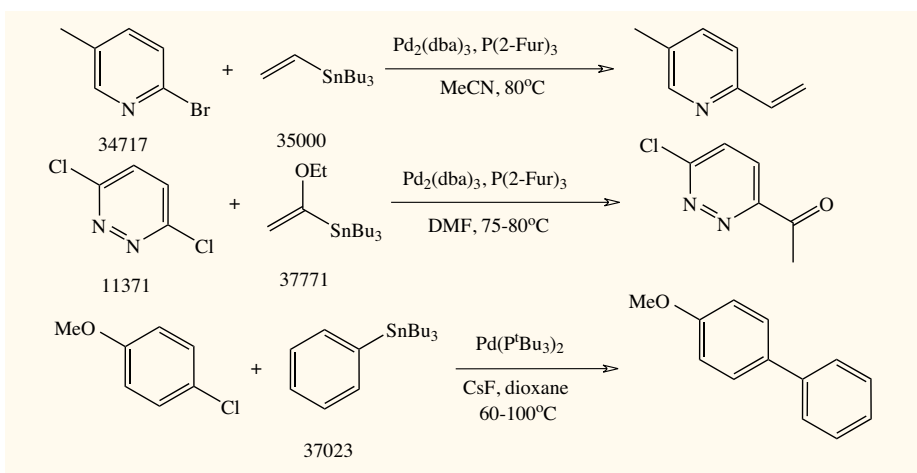


The Stille reaction is an extremely versatile alternative to the Suzuki reaction. It replaces the organoboron reagents with organostannanes. As the tin bears four organic functional groups, understanding the rates of transmetalation of each group is important.

Relative rate of transmetalation:

Alkynyl > vinyl > aryl > allyl ~ benzyl >> alkyl

The Stille coupling is particularly popular as organostannanes are readily prepared, purified and stored. The reaction also has the advantage that it is run under neutral conditions making it even more tolerant of different functional groups than the Suzuki reaction.



### References

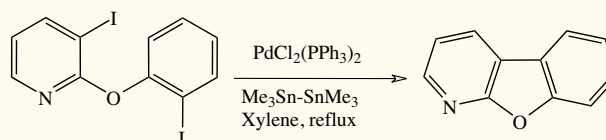
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It can be used to synthesise a wide range of compounds including styrenes,<sup>8</sup> aromatic ketones<sup>9</sup> and biaryl derivatives.<sup>10</sup>

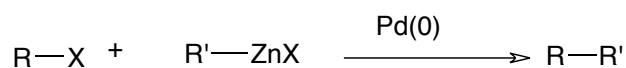
## The Stille-Kelly coupling

The Stille-Kelly coupling is a palladium catalysed intramolecular cross coupling using distannanes such as hexabutyldistannane or hexamethyldistannane.

The intermediate mono-halide mono-stannane cyclises under the reaction conditions to yield the desired product.<sup>11</sup>

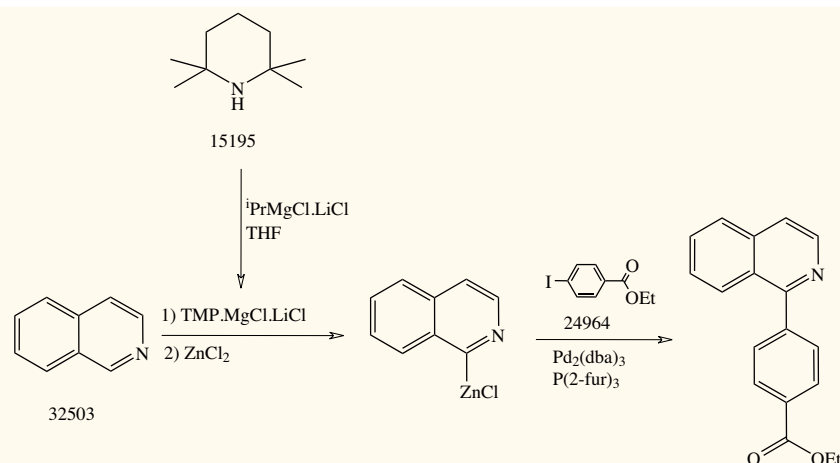


## 3) The Negishi coupling



The Negishi coupling utilises organo-zinc reagents as starting materials to cross couple with organohalides and equivalents.

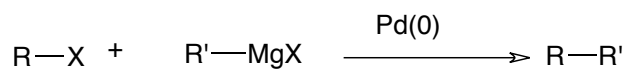
The method is compatible with a good range of functional groups on the organohalide including ketones, esters, amines and nitriles. The organo-zinc reagent can be prepared *in situ* by a variety of methodologies, such as transmetalation of the corresponding organo-lithium or Grignard reagent,<sup>12</sup> or *via* oxidative addition of activated Zn(0) to an organohalide.<sup>13</sup>



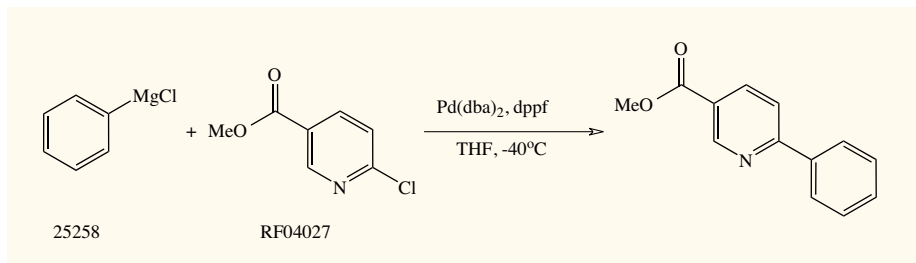
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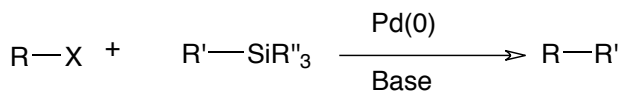
## 4) The Kumada coupling



The cross coupling of organohalides with Grignard reagents is known as the Kumada coupling. Although it suffers from a limited tolerance of different functional groups, the higher reactivity and basicity of the Grignard reagent allows viable reactions to take place under mild conditions.<sup>14</sup>

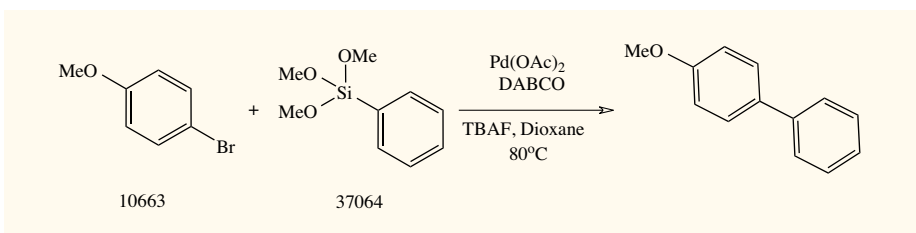


## 5) The Hiyama coupling



Organosilanes can also be coupled with organohalides (or their equivalents) using palladium catalysts. As with the Suzuki reaction the transmetalation will not occur without activation by base or fluoride.<sup>15</sup>

The use of a silanol as the organosilane is one recent method that has managed to negate the requirement for the reaction to contain fluoride as an activator.<sup>16</sup> This has helped to enlarge the substrate scope available to organic chemists.

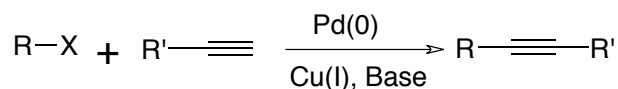


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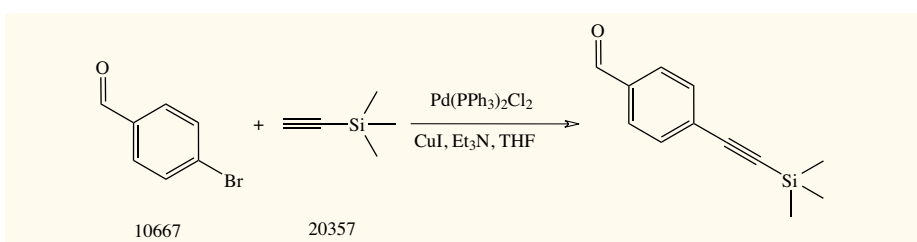


## 6) The Sonogashira coupling

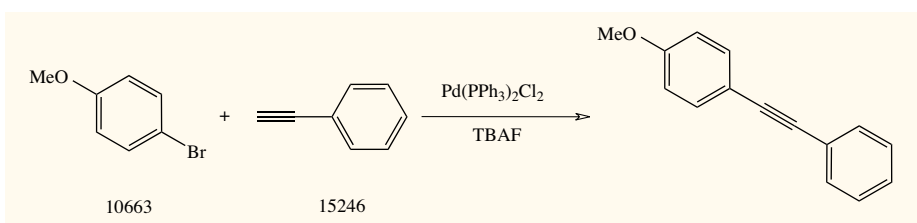


The Sonogashira reaction offers an extremely useful route into aryl- and alkenyl-alkynes. The alkyne moiety is usually introduced *via* its copper salt. This is generated *in situ* from a Cu(I) salt, such as CuI or CuCN, and a terminal alkyne in the presence of an amine base.<sup>17</sup>

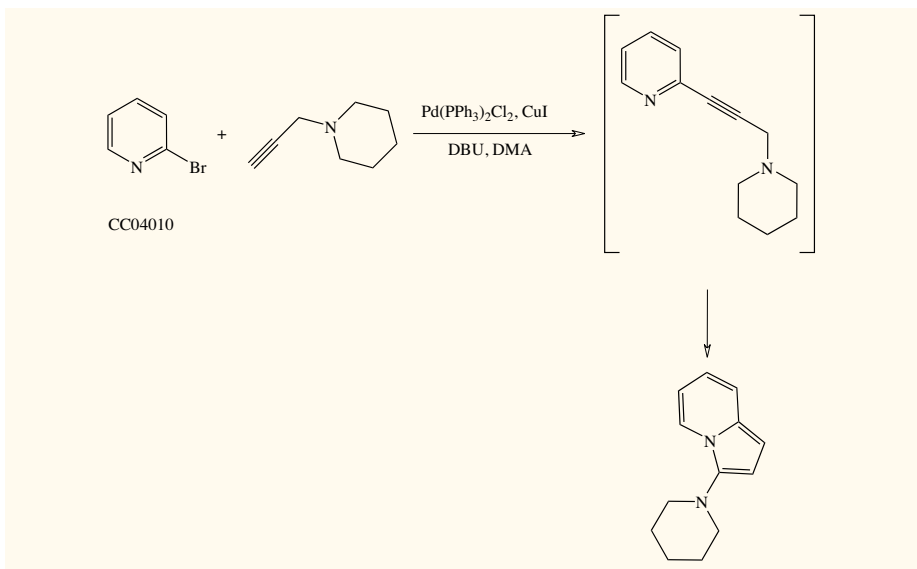
In this case, the TMS protecting group can be removed following the reaction to give the terminal alkyne product. This can be further functionalised, possibly *via* a second Sonogashira coupling.



Recent improvements in this reaction have led to the development of copper and amine free couplings.<sup>18</sup>



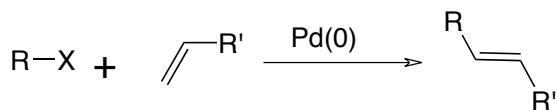
Other uses for this reaction involve the synthesis of intermediates that continue to react under the conditions to give more interesting products.<sup>19</sup>



### References

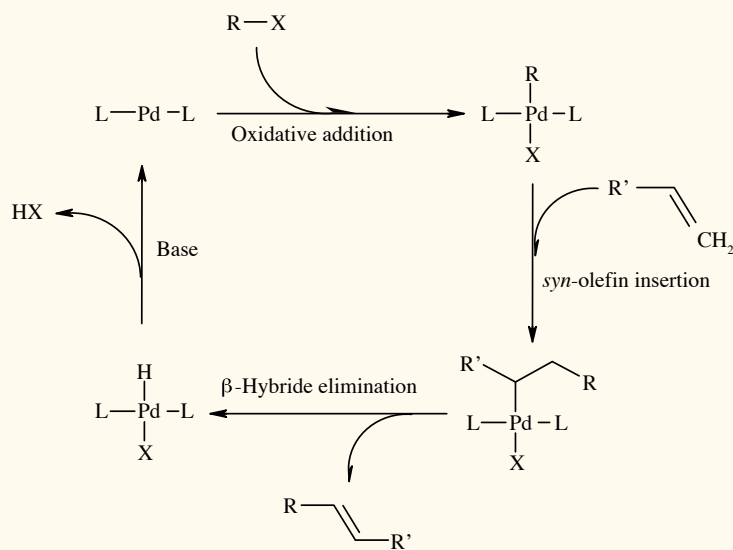
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## 7) The Heck reaction

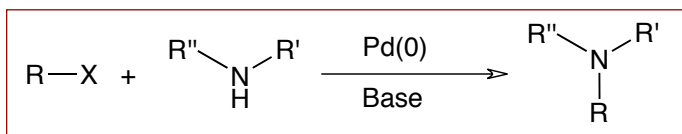


The Heck reaction follows a slightly different pathway to other palladium catalysed couplings.

For intermolecular reactions with mono-substituted olefins, the olefin insertion step is usually directed by steric hindrance. This intermediate then undergoes  $\beta$ -hydride elimination under thermodynamically controlled conditions, leading to preferential formation of the *E* product.

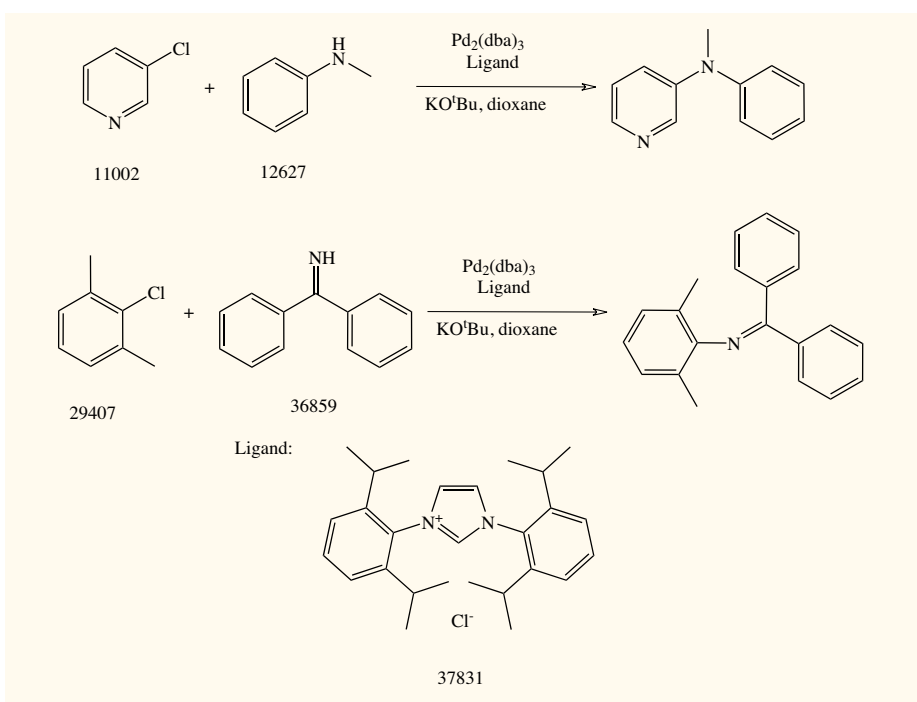


## 8) The Buchwald-Hartwig coupling

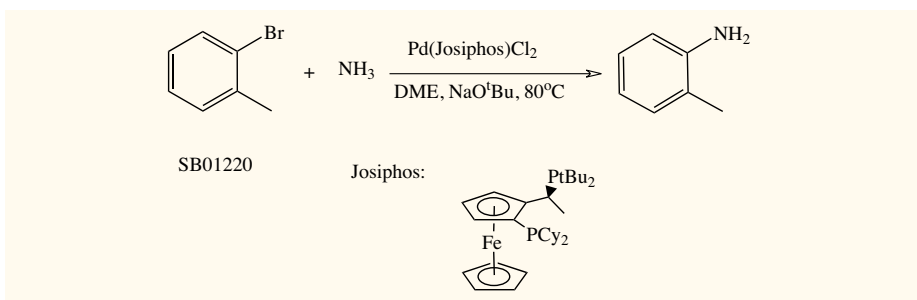


Palladium catalysis has also been expanded to the formation of C-N bonds. In 1995 Buchwald and Hartwig independently reported the palladium catalysed coupling of aryl halides with amine nucleophiles in the presence of stoichiometric amounts of base.<sup>20</sup>

The coupling of aryl chlorides with amine nucleophiles, including anilines and ammonia surrogates, has been reported in high yields using an NHC ligand.<sup>21</sup>



Hartwig has reported that the use of a Josiphos based catalyst can facilitate the direct coupling of ammonia with aryl bromides, giving predominantly the monoarylamine.<sup>22</sup>

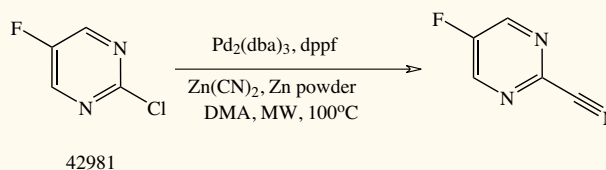


### References

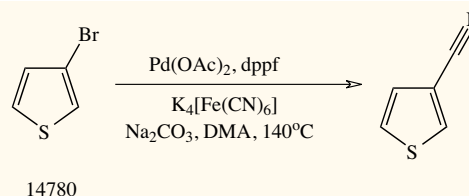
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21. Hillier, A.C.; Grasa, G. A.; Viciu, M.S.; Lee, H. M.; Yang, C; Nolan, S. P. *J. Organomet. Chem.* 2002, 69-82
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## 9) Palladium catalysed cyanation

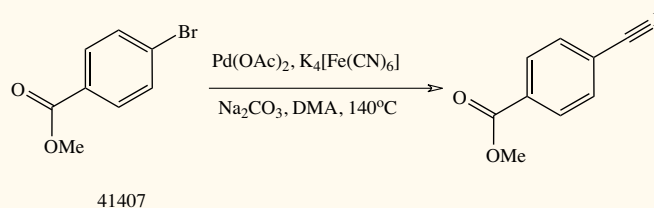
The palladium catalysed cyanation of aromatic halides offers a convenient alternative to the Rosemund-Von Braun reaction, which often employs harsh reaction conditions and can have a labour intensive work-up. As the cyanide nucleophile is a strong  $\sigma$ -donor and can poison the catalyst, it is necessary to keep its concentration low during the reaction. To achieve this  $\text{Zn}(\text{CN})_2$  is often employed as the cyanide source as its solubility in DMF (a common solvent for this reaction) is limited.<sup>23</sup>



An alternative, non-toxic, source of cyanide has also been reported.  $\text{K}_4[\text{Fe}(\text{CN})_6]$  can be used in combination with palladium catalysts to synthesise aryl nitriles from their corresponding halides.<sup>24</sup>



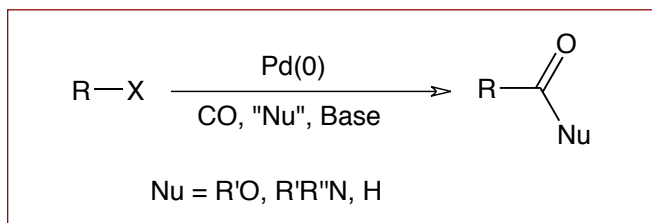
This work was later extended to enable the reaction to take place without the need for the phosphine ligand.<sup>25</sup>



### References

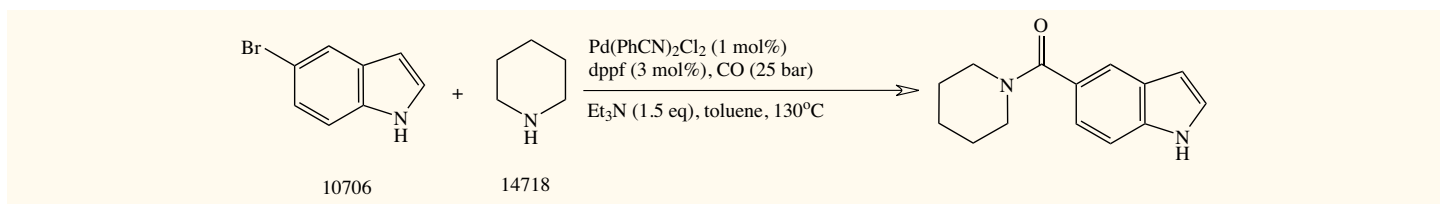
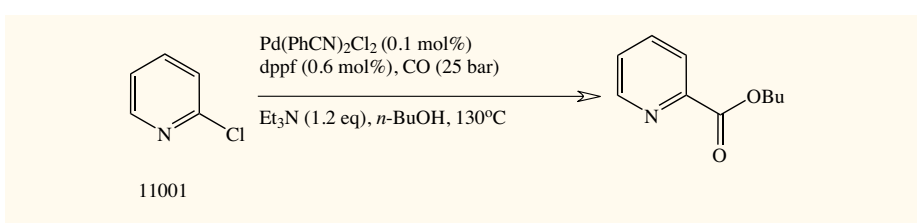
23.  $\text{Zn}(\text{CN})_2$  has a solubility of  $1.8 \times 10^{-4}$  g/mL in DMF at 80°C.
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## 10) Palladium catalysed carbonylation

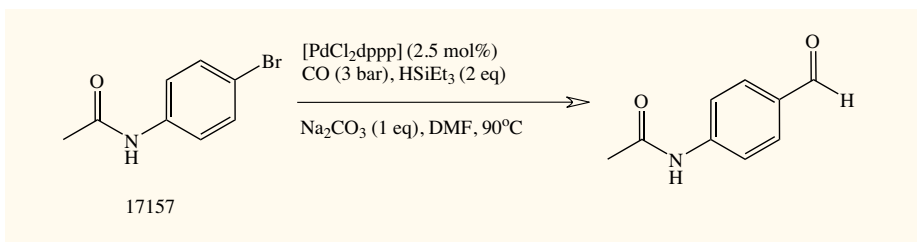


As with most palladium mediated C-C bond forming reactions palladium catalysed carbonylation is compatible with a range of functional groups. This gives it significant advantages over standard organolithium and Grignard chemistry for the synthesis of aryl aldehydes, acids, esters and amides.

Esters and amides are synthesised by carbonylation in the presence of the required alcohol<sup>26</sup> or amine nucleophile.<sup>27</sup>



The use of triethylsilane as the nucleophile gives the corresponding aldehyde as the product.<sup>28</sup>



### References

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## Monodentate Ligands

### General ligands

Cat. No.	Ligand Name	CAS No
14042	Triphenylphosphine	603-35-0
29480	Tri-(2-furyl)phosphine	5518-52-5
42232	Tri-o-tolylphosphine	6163-58-2
32113	Trimesitylphosphine	23897-15-6
42161, 38683, 42842, 42783	Tricyclohexylphosphine	2622-14-2
31733	Triisopropylphosphine	6476-36-4
13934	Tri-n-butylphosphine	998-40-3
38338	Di-tert-butylmethylphosphine	6002-40-0
36089, 36694	Tri-tert-butylphosphine	13716-12-6

### Buchwald type ligands

Cat. No.	Ligand Name	CAS No
38972	2-(Dicyclohexylphosphino)-2'-isopropylbiphenyl	251320-85-1
38714	2-(Dicyclohexylphosphino)-2',4',6'-triisopropylbiphenyl	564483-18-7
35621	2-(Di-tert-butylphosphino)biphenyl	224311-51-7
35622	2-(Dicyclohexylphosphino)biphenyl	247940-06-3
35623	2-Dicyclohexylphosphino-2'-(N,N-dimethylamino)biphenyl	213697-53-1
38009	2-Diphenylphosphino-2'-(N,N-dimethylamino)biphenyl	240417-00-9
38008	2-(Dicyclohexylphosphino)-2'-methylbiphenyl	251320-86-2
38007	2-(Di-tert-butylphosphino)-2'-methylbiphenyl	255837-19-5
38006	2-Di-tert-butylphosphino-2'-(N,N-dimethylamino)biphenyl	224311-49-3
42983	2-Dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl	787618-22-8
42984	2-Di-tert-butylphosphino-2',4',6'-triisopropylbiphenyl	564483-19-8

### NHC ligands

Cat. No.	Ligand Name	CAS No
35619	1,3-Bis(2,4,6-trimethylphenyl)imidazolium chloride	141556-45-8
37831	1,3-Bis(2,6-diisopropylphenyl)imidazolium chloride	250285-32-6
37832	1,3-Bis(adamant-1-yl)imidazolium chloride	131042-78-9
35620	1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate	245679-18-9
38242	1,3-Bis(2,6-diisopropylphenyl)imidazolidinium tetrafluoroborate	282109-83-5
37833	1,3-Bis(2,4,6-trimethylphenyl)imidazolidinium chloride	173035-10-4
37834	1,3-Bis(2,6-diisopropylphenyl)imidazolidinium chloride	258278-25-0

## Bidentate Ligands

### General ligands

Cat. No.	Ligand Name	CAS No
29361	Bis(diphenylphosphino)methane	2071-20-7
14791	1,2-Bis(diphenylphosphino)ethane	1663-45-2
36385	1,2-Bis(dicyclohexylphosphino)ethane	23743-26-2
31005	1,3-Bis(diphenylphosphino)propane	6737-42-4
38112	1,3-Bis(dicyclohexylphosphino)propane	103099-52-1
29646	1,4-Bis(diphenylphosphino)butane	7688-25-7
32085	1,5-Bis(diphenylphosphino)pentane	27721-02-4
38337	Bis(2-diphenylphosphinophenyl)ether	166330-10-5
34801	1,1'-Bis(diphenylphosphino)ferrocene	12150-46-8
36387	1,1'-Bis(diisopropylphosphino)ferrocene	97239-80-0
42971	1,1'-Bis(di-tert-butylphosphino)ferrocene	84680-95-5
36375	1,2-Bis(diphenylphosphino)benzene	13991-08-7
37806	9,9-Dimethyl-4,5-bis(diphenylphosphino)xanthene	161265-03-8

### BINAP ligands

Cat. No.	Ligand Name	CAS No
38235	(S)-(-)-2,2'-Bis(di-p-tolylphosphino)-1,1'-binaphthyl	100165-88-6
38234	(R)-(+)-2,2'-Bis(di-p-tolylphosphino)-1,1'-binaphthyl	99646-28-3
26554	(S)-(-)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl	76189-56-5
26553	(R)-(+)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl	76189-55-4
39223	rac-2,2'-Bis(di-p-tolylphosphino)-1,1'-binaphthyl	153305-67-0
39222	rac-2,2'-Bis(di(3,5-dimethylphenyl)phosphino)-1,1'-binaphthyl	145416-77-9
36864	(±)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl	98327-87-8

### Josiphos ligands

Cat. No.	Ligand Name	CAS No
37075	(R)-(-)-1-[(S)-2-Di-tert-butylphosphino]ferrocenyl]ethyl-di-(4-trifluoromethylphenyl)phosphine	246231-79-8
37070	(R)-(-)-1-[(S)-2-Diphenylphosphino]ferrocenyl]ethylbis(3,5-dimethylphenyl)phosphine	184095-69-0
37069	(R)-(-)-1-[(S)-2-Dicyclohexylphosphino]ferrocenyl]ethyl-dicyclohexylphosphine	167416-28-6
37068	(R)-(-)-1-[(S)-2-Diphenylphosphino]ferrocenyl]ethyl-di-tert-butylphosphine	155830-69-6
37067	(R)-(-)-1-[(S)-2-Diphenylphosphino]ferrocenyl]ethyl-dicyclohexylphosphine	155806-35-2

# Palladium catalysts and precursors

## Catalyst precursors

Cat. No.	Catalyst Precursor Name	CAS No
20683	Allylpalladium chloride dimer	12012-95-2
20945	Bis(acetonitrile)palladium(II) chloride	14592-56-4
20790	Bis(benzonitrile)palladium(II) chloride	14220-64-5
29197	Bis(dibenzylideneacetone)palladium	32005-36-0
19518	Palladium(II) acetate	3375-31-3
19519	Palladium(II) bromide	13444-94-5
19520, 36967	Palladium(II) chloride	7647-10-1
31702	Palladium(II) trifluoroacetate	42196-31-6
36352	Tetrakis(acetonitrile)palladium(II) tetrafluoroborate	21797-13-7
31877	Tris(dibenzylideneacetone)dipalladium(0)	51364-51-3
36934	Tris(dibenzylideneacetone)dipalladium-chloroform adduct	52522-40-4

## Catalysts

Cat. No.	Catalyst Name	CAS No
38403	[1,2-Bis(diphenylphosphino)ethane] dichloropalladium(II)	19978-61-1
34868	1,1'-Bis(diphenylphosphino)ferrocene-palladium(II)dichloride dichloromethane adduct	95464-05-4
36351	Bis(tricyclohexylphosphine)palladium(0)	33309-88-5
37797	Bis(triethylphosphine)palladium(II) chloride	28425-04-9
20927	Bis(triphenylphosphine)palladium(II) acetate	14588-08-0
19732, 29925	Bis(triphenylphosphine)palladium(II) chloride	13965-03-2
36350	Bis(tri-t-butylphosphine)palladium(0)	53199-31-8
21299	Bis[1,2-bis(diphenylphosphino)ethane] palladium(0)	31277-98-2
37796	Bis[tri(o-tolyl)phosphine]palladium(II) chloride	40691-33-6
39589	Dichlorobis(tricyclohexylphosphine) palladium(II)	29934-17-6
20238	Tetrakis(triphenylphosphine)palladium(0)	14221-01-3
36971	trans-Benzyl(chloro)bis(triphenylphosphine) palladium(II)	22784-59-4



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