

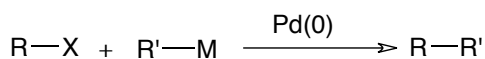


ACRŌS
ORGANICS

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.



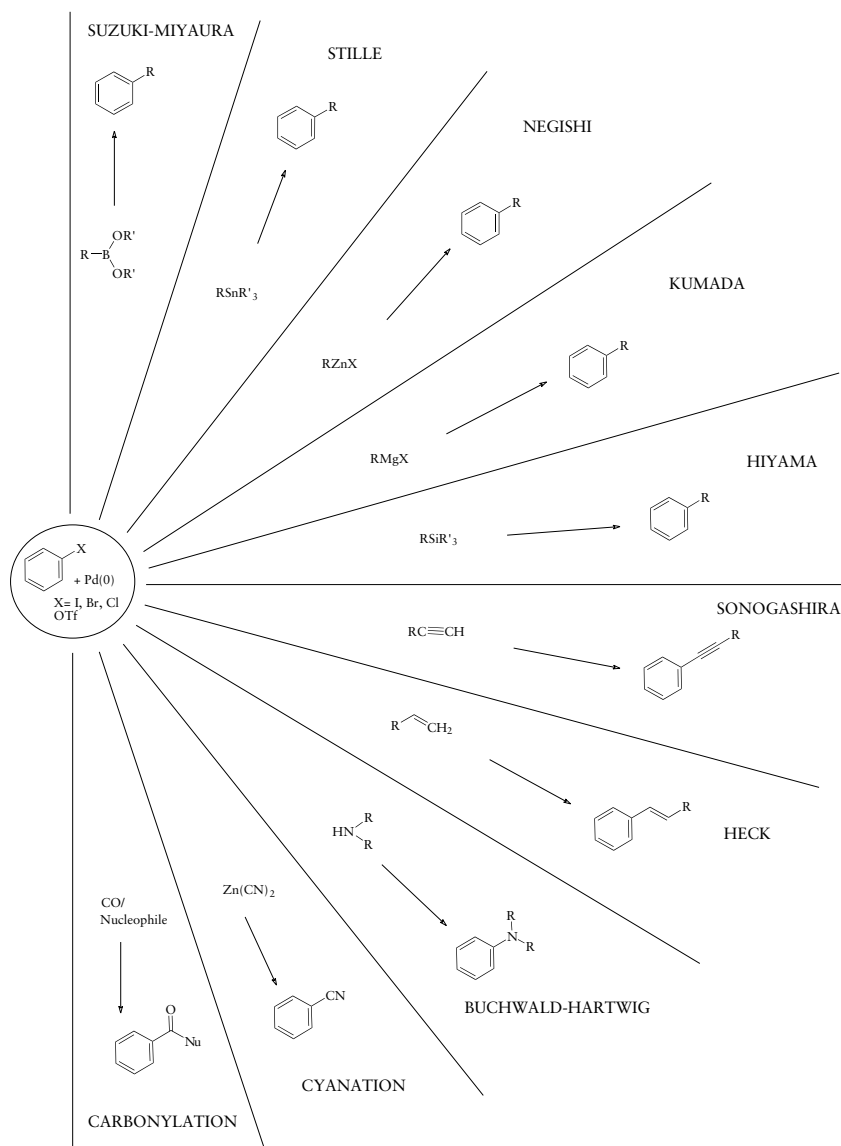
R = usually sp^2 hybridised carbon

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

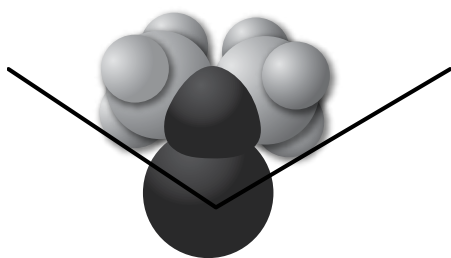
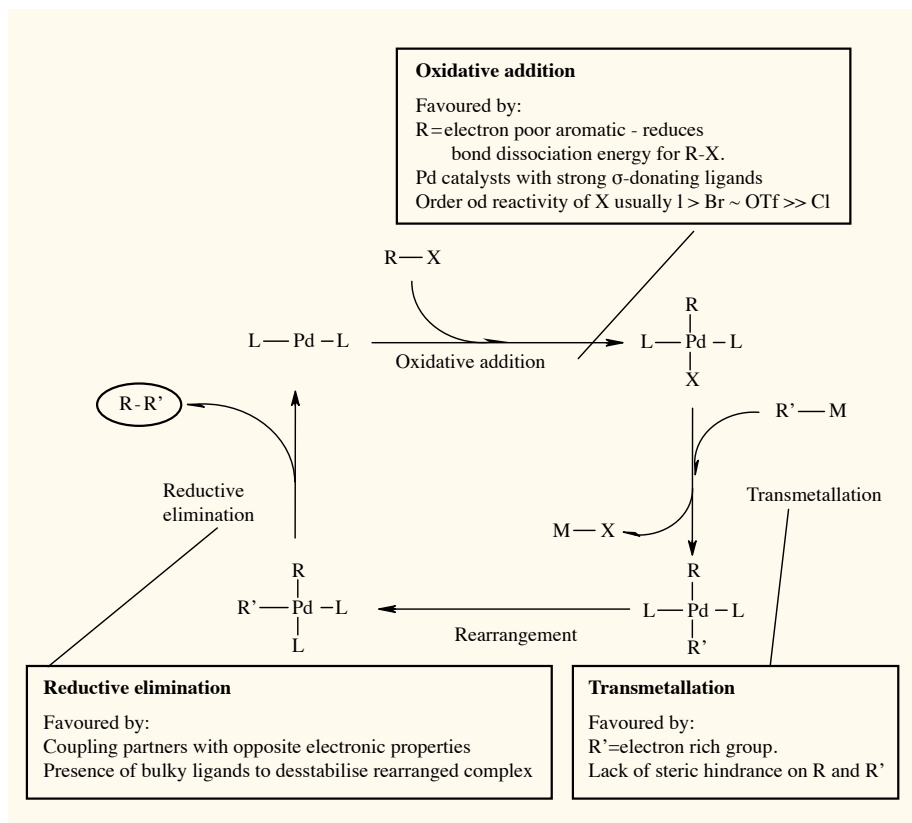


Understanding the catalytic cycle

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

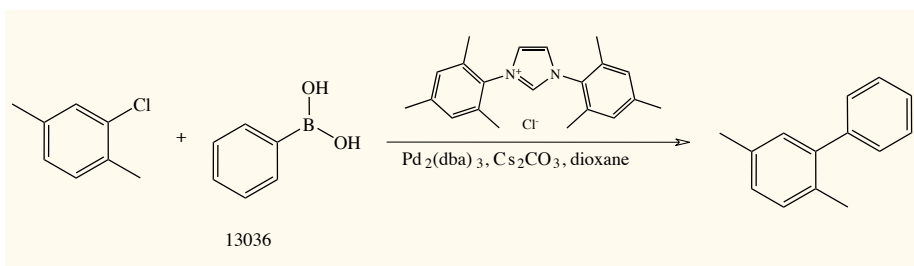
The catalytic species can be formed *in situ* using a palladium source, such as $\text{Pd}_2(\text{dba})_3$ or $\text{Pd}(\text{OAc})_2$ and the necessary ligand, or introduced as a preformed catalyst such as $\text{Pd}(\text{PPh}_3)_4$ or $\text{Pd}(\text{P}^t\text{Bu}_3)_2$.

Careful choice of ligand can facilitate two steps of the catalytic cycle. The use of strong σ -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.¹ 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).²

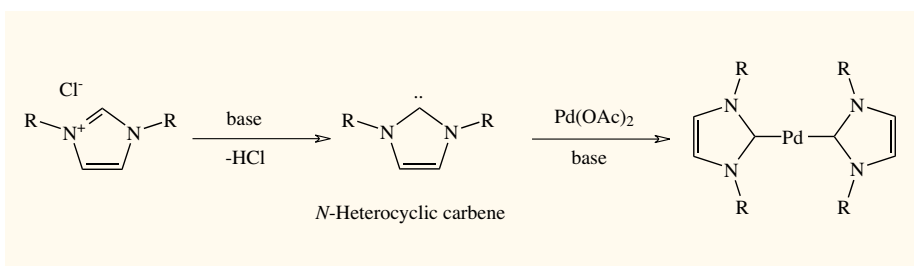


| Ligand | Cone Angle (deg) | Cat. No. |
|---|------------------|----------|
| dppm | 121 | 29361 |
| dppe | 125 | 14791 |
| dppp | 127 | 31005 |
| dcpe | 142 | 36385 |
| PPh_3 | 145 | 14042 |
| $\text{P}(\text{c-hex})_3$ | 170 | 42161 |
| $\text{P}(\text{t-Bu})_3$ | 182 | 36089 |
| $\text{P}(\text{C}_6\text{F}_5)_3$ | 184 | 31316 |
| $\text{P}(2,4,6\text{-Me}_3\text{C}_6\text{H}_2)_3$ | 212 | 32113 |

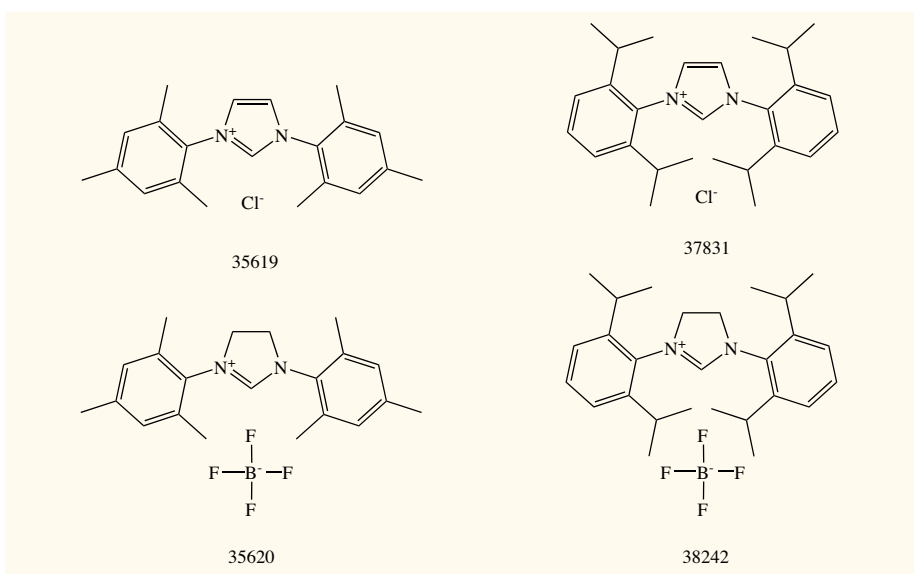
Phosphine ligands have recently been replaced in a number of palladium catalyzed reactions with *N*-heterocyclic carbenes (NHCs).³



These ligands offer similar electronic properties to phosphines, being strongly σ -donating and weakly π -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.

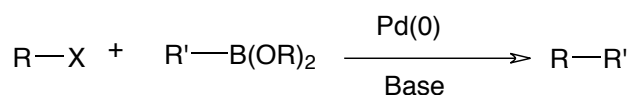


References

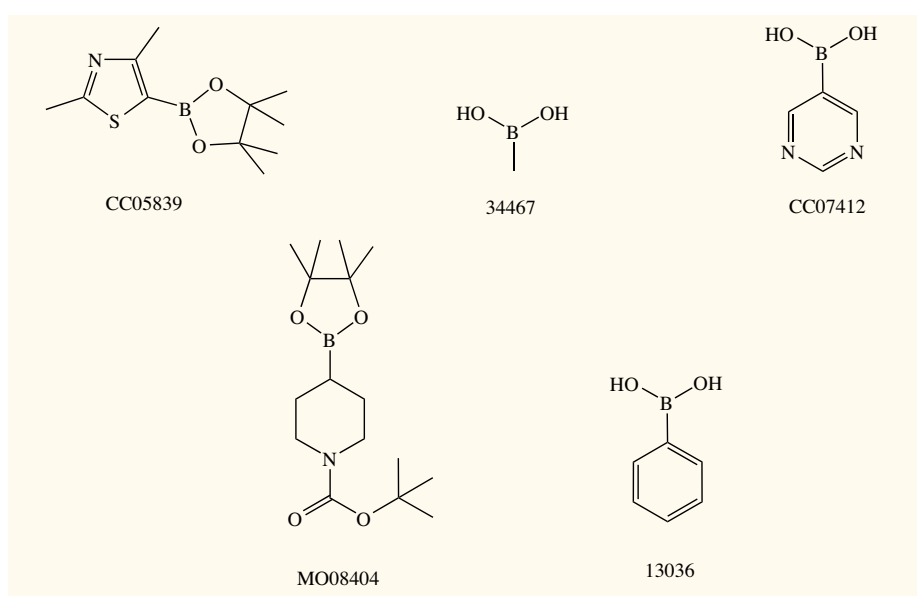
- Galardon, E.; Ramdeehul, S.; Brown, J.M.; Cowley, A.; Hii, K.K.; Jutand, A.; *Angew. Chem, Int. Ed.* **2002**, *41*, 1760-1763
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Palladium Catalysed Reactions

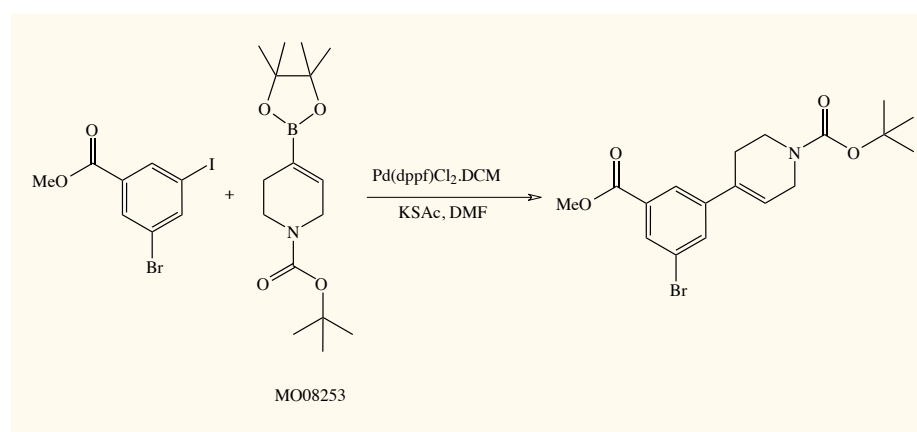
1) The Suzuki-Miyaura coupling



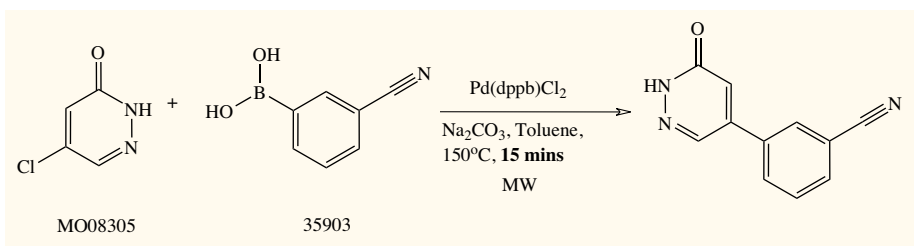
The Suzuki coupling reaction involves the cross coupling of organohalides (and their equivalents) with organoboron reagents. The organoboron reagent typically comes in the form of a boronic acid or ester, of which >300 structurally diverse examples are stocked under the Acros Organics and Maybridge brands,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



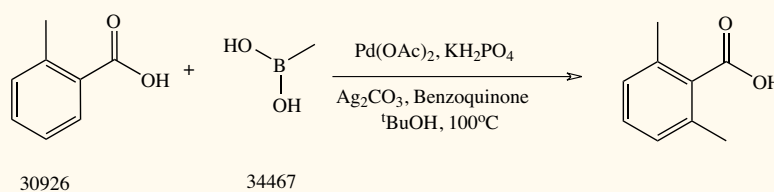
The reaction is highly tolerant of many different functional groups, and boron containing by-products are easily removed by a simple alkali work-up. Although most commonly used to form aryl-aryl bonds the Suzuki reaction is just as effective for the synthesis of highly substituted styrene products.⁴



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

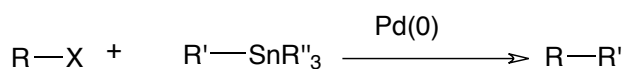


It can also be used to perform aromatic alkylations.⁶ C-H insertion negates the necessity to begin with an aryl halide, improving the atom efficiency of the process.



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

2) The Stille coupling

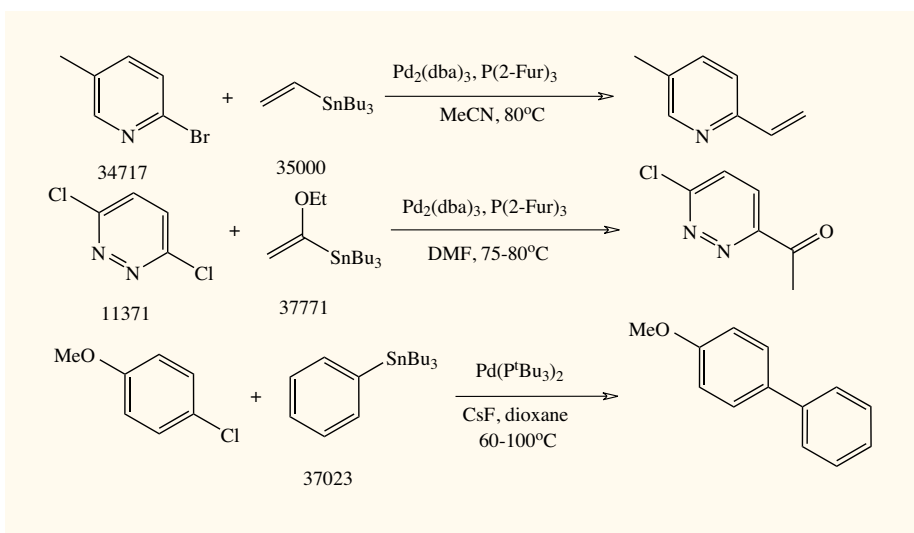


The Stille reaction is an extremely versatile alternative to the Suzuki reaction. It replaces the organoboron reagents with organostannanes. As the tin bears four organic functional groups, understanding the rates of transmetalation of each group is important.

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

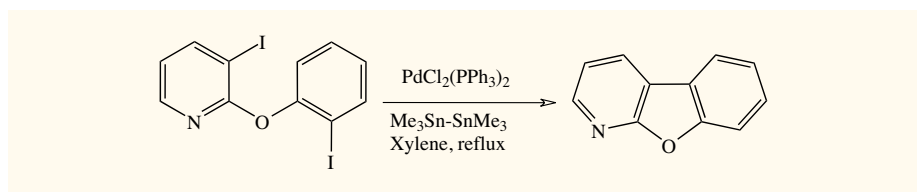
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It can be used to synthesise a wide range of compounds including styrenes,⁸ aromatic ketones⁹ and biaryl derivatives.¹⁰

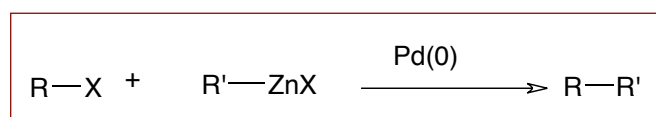
The Stille-Kelly coupling

The Stille-Kelly coupling is a palladium catalysed intramolecular cross coupling using di-stannanes such as hexabutyl-distannane or hexamethyldistannane.

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

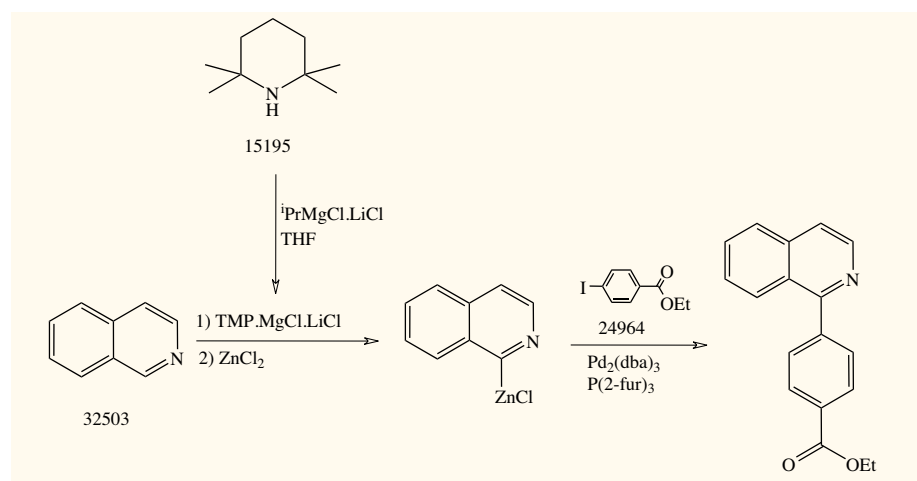


3) The Negishi coupling



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

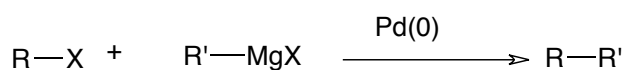
The method is compatible with a good range of functional groups on the organohalide including ketones, esters, amines and nitriles. The organo-zinc reagent can be prepared *in situ* by a variety of methodologies, such as transmetalation of the corresponding organo-lithium or Grignard reagent,¹² or *via*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



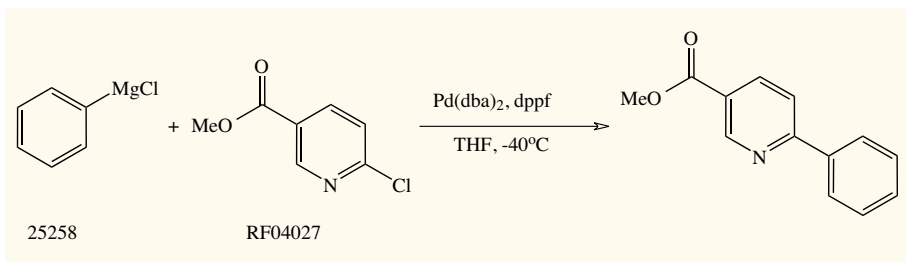
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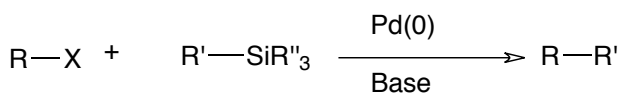
4) The Kumada coupling



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.¹⁴

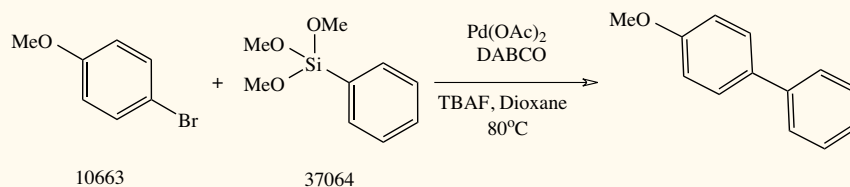


5) The Hiyama coupling



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.¹⁵

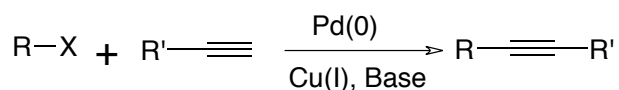
The use of a silanol as the organosilane is one recent method that has managed to negate the requirement for the reaction to contain fluoride as an activator.¹⁶ This has helped to enlarge the substrate scope available to organic chemists.



References

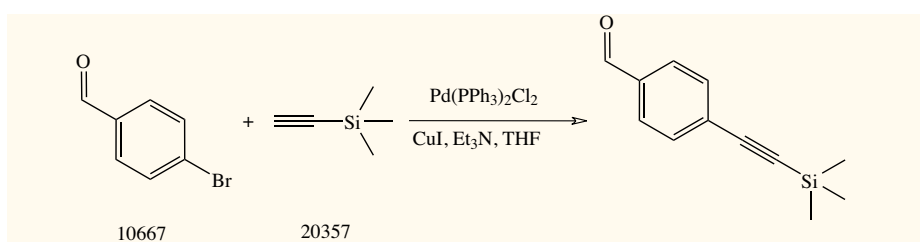
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6) The Sonogashira coupling

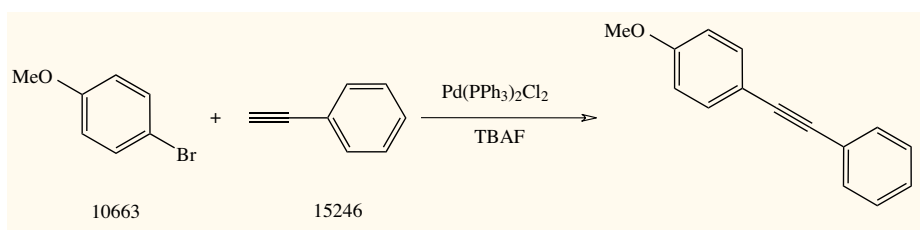


The Sonogashira reaction offers an extremely useful route into aryl- and alkenyl-alkynes. The alkyne moiety is usually introduced *via* its copper salt. This is generated *in situ* from a Cu(I) salt, such as CuI or CuCN, and a terminal alkyne in the presence of an amine base.¹⁷

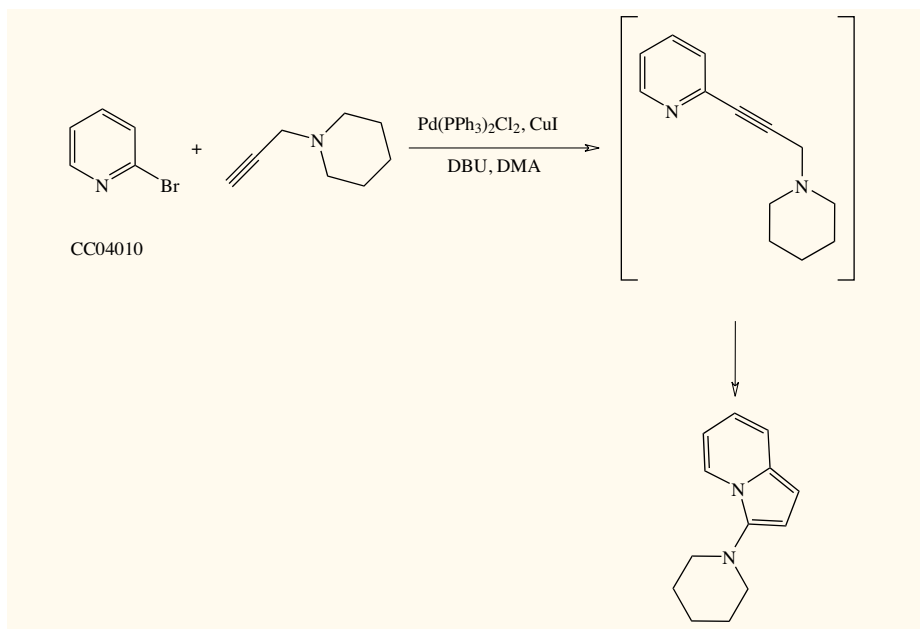
In this case, the TMS protecting group can be removed following the reaction to give the terminal alkyne product. This can be further functionalised, possibly *via* a second Sonogashira coupling.



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



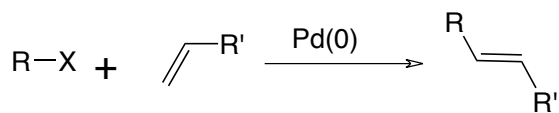
Other uses for this reaction involve the synthesis of intermediates that continue to react under the conditions to give more interesting products.¹⁹



References

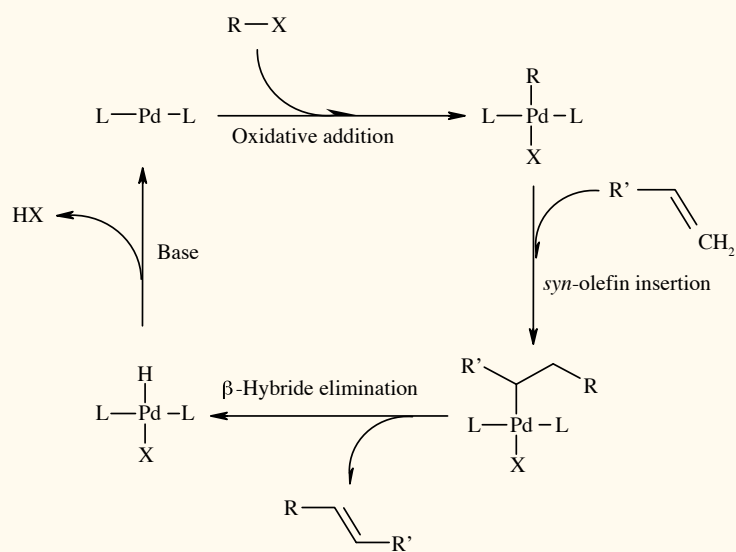
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7) The Heck reaction

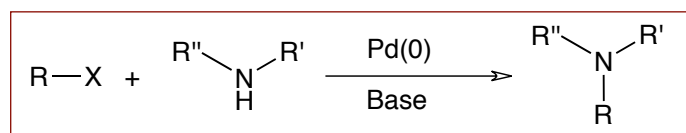


The Heck reaction follows a slightly different pathway to other palladium catalysed couplings.

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

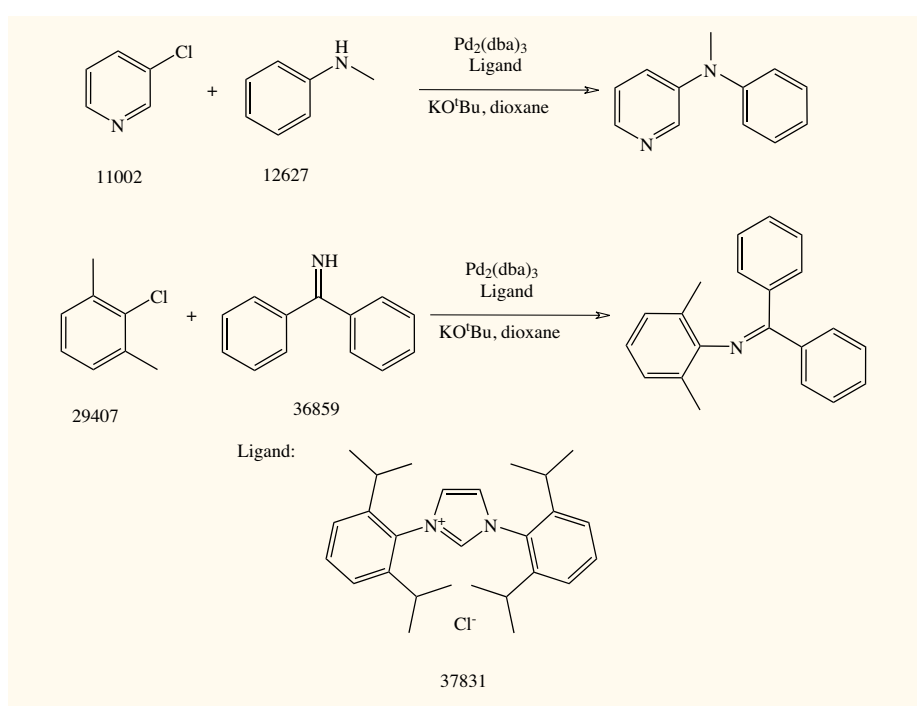


8) The Buchwald-Hartwig coupling

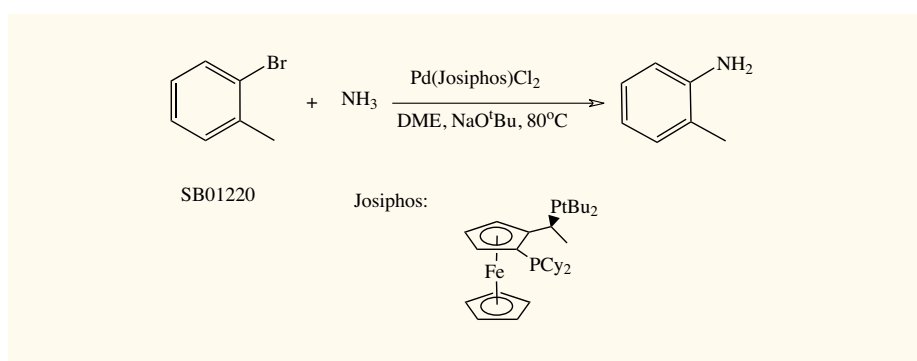


Palladium catalysis has also been expanded to the formation of C-N bonds. In 1995 Buchwald and Hartwig independently reported the palladium catalysed coupling of aryl halides with amine nucleophiles in the presence of stoichiometric amounts of base.²⁰

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



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.²²

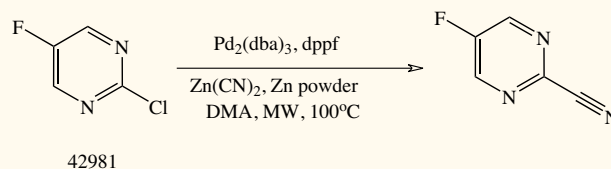


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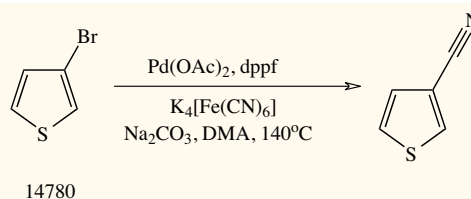
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9) Palladium catalysed cyanation

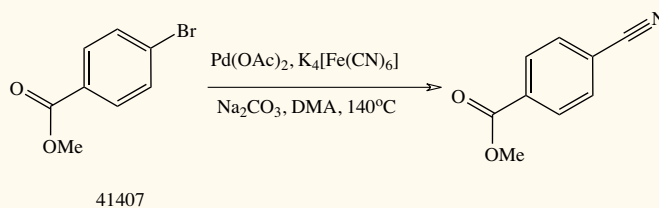
The palladium catalysed cyanation of aromatic halides offers a convenient alternative to the Rosemund-Von Braun reaction, which often employs harsh reaction conditions and can have a labour intensive work-up. As the cyanide nucleophile is a strong σ -donor and can poison the catalyst, it is necessary 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.²³



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.²⁴



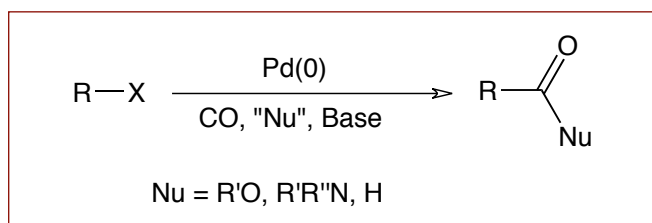
This work was later extended to enable the reaction to take place without the need for the phosphine ligand.²⁵



References

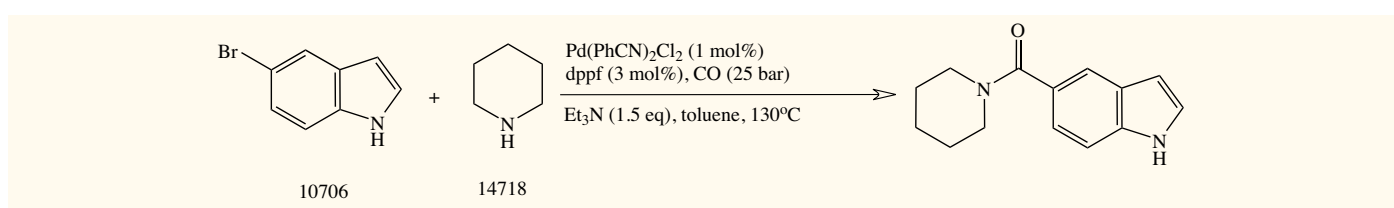
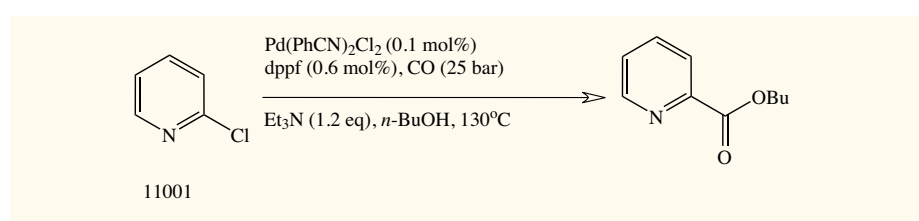
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10) Palladium catalysed carbonylation

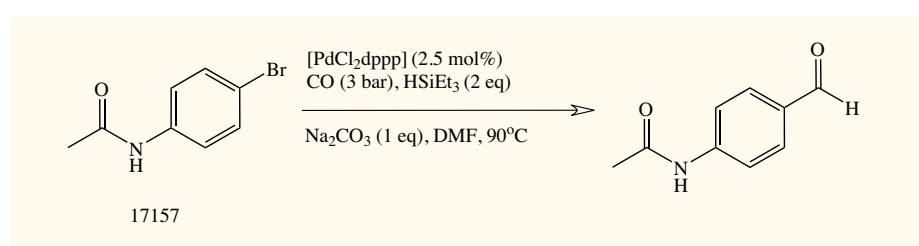


As with most palladium mediated C-C bond forming reactions palladium catalysed carbonylation is compatible with a range of functional groups. This gives it significant advantages over standard organolithium and Grignard chemistry for the synthesis of aryl aldehydes, acids, esters and amides.

Esters and amides are synthesised by carbonylation in the presence of the required alcohol²⁶ or amine nucleophile.²⁷



The use of triethylsilane as the nucleophile gives the corresponding aldehyde as the product.²⁸



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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)imidazolidium tetrafluoroborate | 282109-83-5 |
| 37833 | 1,3-Bis(2,4,6-trimethylphenyl)imidazolidium chloride | 173035-10-4 |
| 37834 | 1,3-Bis(2,6-diisopropylphenyl)imidazolidium 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]ethyldicyclohexylphosphine | 167416-28-6 |
| 37068 | (R)-(-)-1-[(S)-2-Diphenylphosphino]ferrocenyl]ethyl-di-tert-butylphosphine | 155830-69-6 |
| 37067 | (R)-(-)-1-[(S)-2-Diphenylphosphino]ferrocenyl]ethyldicyclohexylphosphine | 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- <i>t</i> -butylphosphine)palladium(0) | 53199-31-8 |
| 21299 | Bis[1,2-bis(diphenylphosphino)ethane] palladium(0) | 31277-98-2 |
| 37796 | Bis[tri(<i>o</i> -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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