

Реакции образования связей C-N

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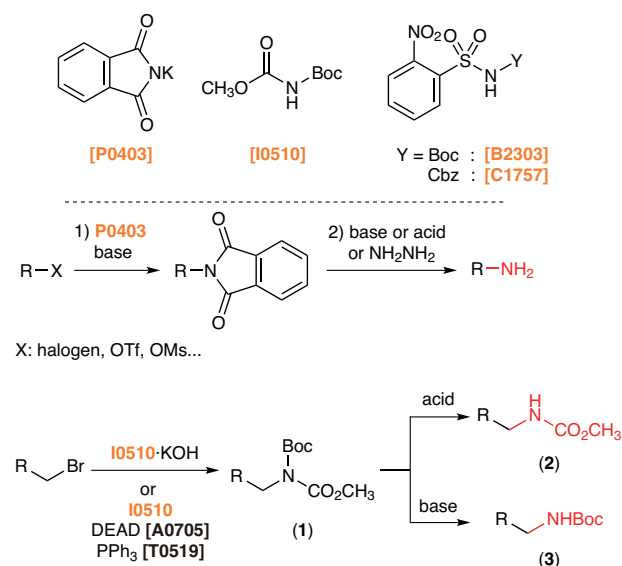
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C-N Bond Formation Reactions

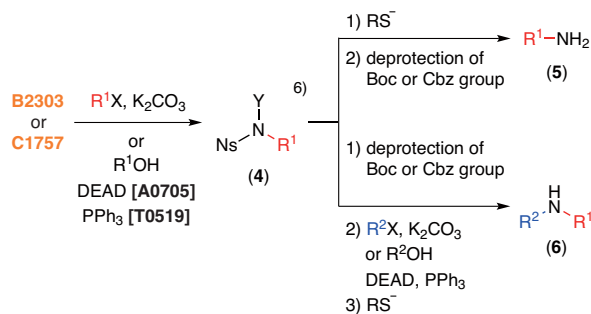
Nitrogen-containing compounds are found in wide fields from natural products such as amino acids, nucleic acids and alkaloids to synthetic compounds such as electronic materials and polyamides. Furthermore, various kinds of nitrogen-containing functional groups are known such as amino groups and nitro groups, which have different coupling schemes and oxidation states. Therefore, C-N bond formation reactions vary with each functional group. Since it is hard to introduce C-N bond formation reactions comprehensively because these reactions encompass many methods from simple introduction of functional groups to the construction of heterocyclic rings, this brochure mainly introduces TCI's reagents for installation of nitrogen-containing functional groups among the reagents for C-N bond formation reactions. Additionally, there are some cyanating agents in this brochure that may look strange to apply to C-N bond formation reactions, but these reagents are introduced because the cyano group can be converted into other nitrogen-containing functional groups.

● Reagents for Amination

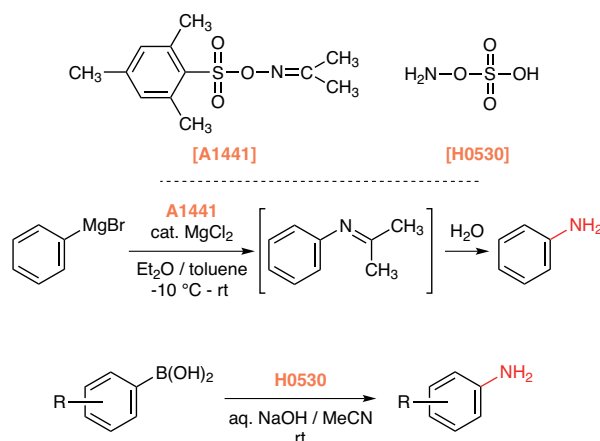
The Gabriel amine synthesis¹⁾ is widely used as a nucleophilic amination reaction and potassium phthalimide and similar aminating agents have been reported so far. The potassium salt of *tert*-butyl methyl iminodicarboxylate [I0510] reacts with an alkyl halide to give an imide (1).²⁾ 1 affords *N*-methoxycarbonylamine (2) and *N*-Boc amine (3) by treatment with acid or base, respectively. Furthermore, I0510 can be applied to the Mitsunobu reaction and convert a hydroxy group into an amino group.³⁾



Meanwhile, Fukuyama *et al.* reported another method of amine synthesis using sulfonamides [B2303] [C1757], which are protected by the *o*-nitrobenzenesulfonyl (Ns) group.⁴⁾ These products afford the protected amines (4) under basic conditions with alkyl halides or under Mitsunobu conditions with alcohols, respectively. Each protective group is selectively removed and the primary amine (5) is given by the removal of both protecting groups. However, when the Ns group of 4 is left remaining, the secondary amine (6) can be given after alkylation and successive removal of it.

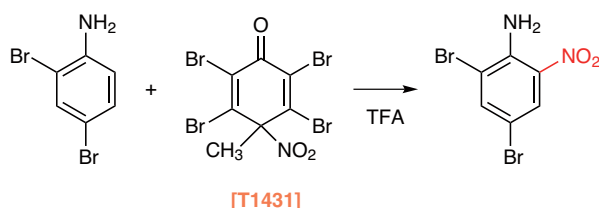


Acetoxime O-(2,4,6-trimethylphenylsulfonate) [A1441] is regarded as an electrophilic aminating reagent.⁶⁾ A1441 reacts with Grignard reagents under a catalytic amount of magnesium chloride to give primary amines in good yields.⁷⁾ Furthermore, hydroxylamine-O-sulfonic acid (HSA) [H0530] is also utilized as an aminating reagent. HSA behaves as an aminocation and gives primary anilines by the treatment with phenylboron derivatives. It is also an advantage that the reaction can proceed without any transition metal catalysts and with easy handling.⁸⁾



● Reagents for Nitration

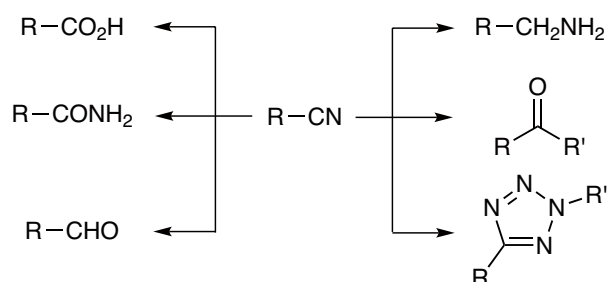
The nitro group is a strong electron-withdrawing functional group which can decrease the electron density of aromatic rings and increase the acidity of the proton at the α -position of the nitroalkane. Therefore, compounds with nitro groups can undergo unique reactions such as the Henry reaction and the Nef reaction. Generally, nitration of an aromatic ring is proceeded by electrophilic substitution reaction with concentrated nitric acid and concentrated sulfuric acid. However, 2,3,5,6-tetrabromo-4-methyl-4-nitro-2,5-cyclohexadien-1-one **[T1431]** is a mild nitrating reagent for aromatic compounds.^{9,10} For instance, electron-rich aromatic compounds like anilines give nitrated compounds substituted at the *ortho*- or *para*-position by treatment with **T1431**.



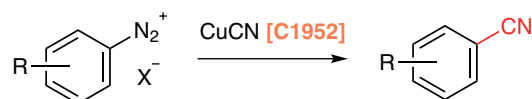
● Reagents for Cyanation

The cyano group is a strong electron-withdrawing group and a number of cyanating reagents are widely used in organic synthesis. The cyano group is converted to other functional groups such as carboxylic acids or amides by hydrolysis, and is also converted to amines or aldehydes by reduction with some reducing reagents. Furthermore, nitriles are transformed into the corresponding asymmetric ketones through nucleophilic addition reactions with Grignard reagents or organolithiums. On the other hand, the cyano group can be used for cycloaddition reactions with other multiple bonds. For instance, alkyl nitriles are cyclized with azides to give tetrazoles.

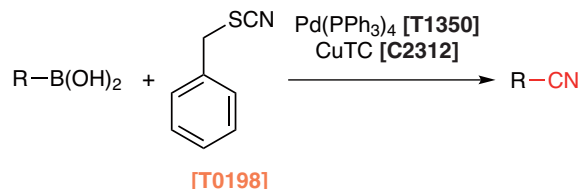
To introduce the cyano group, the reaction of potassium cyanide **[P1613]** with alkyl halides is a typical synthetic method and the copper cyanide-mediated Sandmeyer reaction and the Rosenmund-von Braun reaction have been known for a long time. Recently, cyanation reactions using palladium catalysts with some cyanating reagents have been developed. Benzyl thiocyanate **[T0198]**,¹¹ ethyl cyanoacetate **[C0441]**,¹² *tert*-butyl isocyanide **[B1274]**¹³ and acetone cyanohydrin **[M0361]**¹⁴ work as cyanide ion equivalents. Recently, some groups have reported that acetonitrile¹⁵ and dimethylmalononitrile **[D5514]**¹⁶ can be utilized as cyano sources. In this way, a variety of cyanating reagents are used for the direct cyanation of various substances.



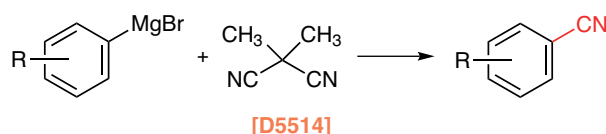
Sandmeyer reaction



Pd-Catalyzed cross-coupling cyanation

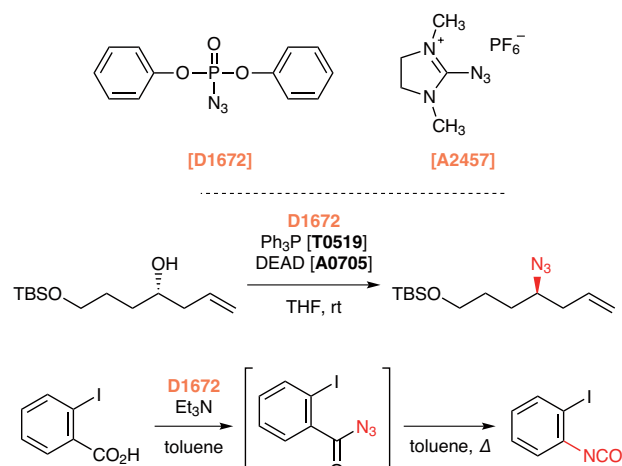


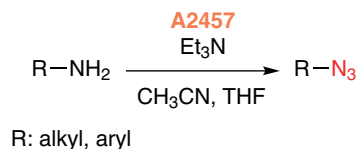
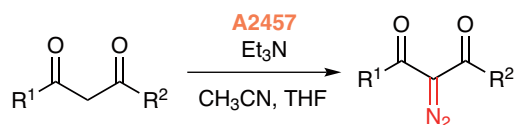
Cyanation of Grignard reagent



● Reagents for Azidation and Diazotization

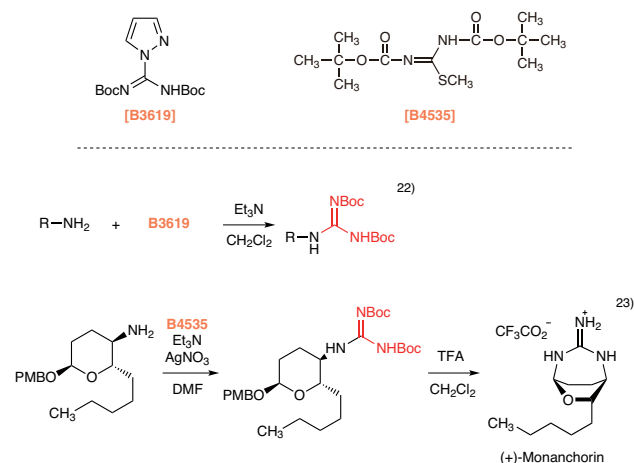
Organic azide compounds can be synthesized in a simple manner by the reaction of sodium azide **[S0489]** with halogenated alkyl compounds, or the reaction with trifluoromethanesulfonyl azide and primary amines. However, these azide sources have highly explosive characters, which makes them difficult to handle. In contrast, Shioiri *et al.* developed a stable and easy-to-handle reagent for azidation, DPPA **[D1672]**.¹⁷ When an alcohol is treated with DPPA under Mitsunobu conditions, the inverted azide compound is given in high yield. DPPA is utilized not only in the Curtius rearrangement but also as a condensation reagent.¹⁸ Furthermore, 2-azido-1,3-dimethylimidazolinium hexafluorophosphate (ADMP) **[A2457]**, which was developed by Kitamura *et al.*, is a crystalline diazotransfer reagent with high thermal stability and low reactivity to impact and friction. ADMP reacts with a variety of primary amines to afford azides¹⁹ as well as with 1,3-dicarbonyl compounds to afford α -diazo compounds in high yields.²⁰





Reagents for Guanidinylation

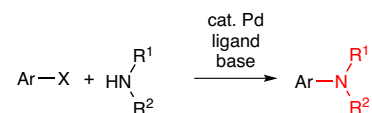
The guanidine moiety has a stronger basicity among organic bases and it is seen in many kinds of bioactive compounds as well as in arginine. For instance, saxitoxin and tetrodotoxin derived from puffer toxin, and batzelladine A as an HIV inhibitor in sea sponge are known as compounds bearing a guanidino group. Furthermore, pharmaceutical compounds like antimalarial and antimicrobial agents often contain guanidine structures. In this way, guanidinylation reagents are utilized in the synthesis of guanidine derivatives in the drug discovery field.⁽²¹⁾ The guanidinylation group is mainly introduced by an addition reaction to an amino group.



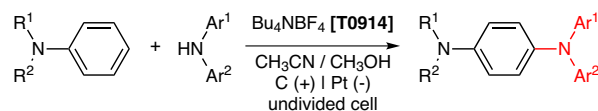
C-N bond formation via Cross-Coupling Reaction

Buchwald⁽²⁴⁾ and Hartwig⁽²⁵⁾ have independently reported a new type of coupling reaction between amines and aryl halides in the presence of palladium catalyst and strong base. This reaction is called the Buchwald-Hartwig cross-coupling reaction. This reaction introduces amino groups to aromatic compounds but it can also be applied to the construction of nitrogen-containing heteroaromatic rings.⁽²⁶⁾ Therefore, the reaction is widely used in the total synthesis of natural products, medicinal chemistry and process chemistry. Very recently, the new electro-oxidative C-N cross-coupling reaction has been reported.⁽²⁷⁾ This reaction has some advantages: *para*-selective cross-coupling; metal-catalyst free reaction; and being regarded as a green reaction since the sole byproduct is hydrogen gas generated through C-H activation.

Buchwald-Hartwig cross-coupling



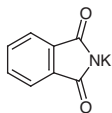
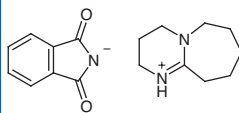
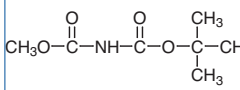
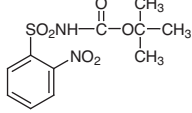
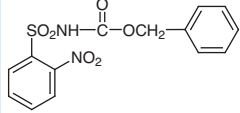
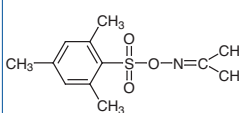
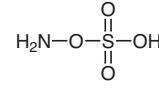
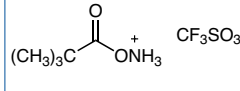
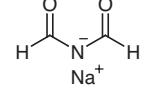
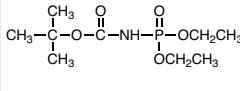
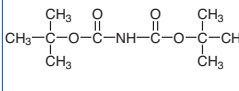
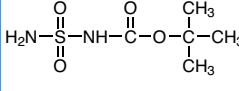
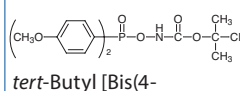
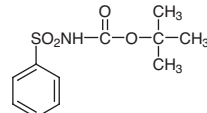
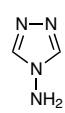
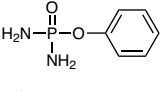
Electrooxidative C-N cross-coupling



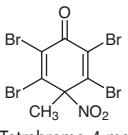
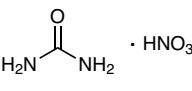
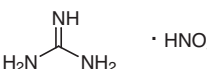
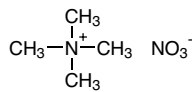
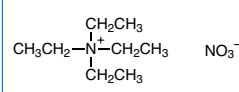
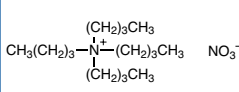
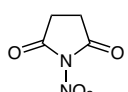
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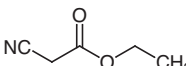
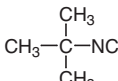
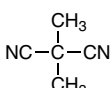
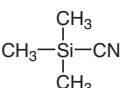
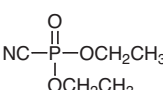
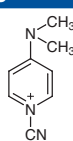
Reagents for Amination

Reagents for Amination		P0403 25g 500g  Phthalimide Potassium Salt CAS RN: 1074-82-4	P1235 25g  Phthalimide DBU Salt CAS RN: 119812-51-0	I0510 5g  tert-Butyl Methyl Iminodicarboxylate CAS RN: 66389-76-2
B2303 1g 5g 25g  N-Boc-2-nitrobenzenesulfonamide CAS RN: 198572-71-3	C1757 5g 25g  N-Cbz-2-nitrobenzenesulfonamide CAS RN: 245365-64-4	A1441 5g 25g  Acetoxime O-(2,4,6-Trimethylphenyl)sulfonate) CAS RN: 81549-07-7	H0530 25g 100g 500g  Hydroxylamine-O-sulfonic Acid CAS RN: 2950-43-8	P2856 1g  O-Pivaloylhydroxylamine Triflic Acid CAS RN: 1293990-73-4
D2479 25g  Sodium Diformylamide CAS RN: 18197-26-7	B1734 1g 5g  N-Boc-phosphoramidic Acid Diethyl Ester CAS RN: 85232-02-6	I0497 5g 25g  Di-tert-butyl Iminodicarboxylate CAS RN: 51779-32-9	B5402 1g 5g  tert-Butyl Sulfamoylcarbamate CAS RN: 148017-28-1	B2857 1g 5g  tert-Butyl [Bis(4-methoxyphenyl)-phosphinyloxy]carbamate CAS RN: 619333-95-8
B1648 10g 25g  N-Boc-p-toluenesulfonamide CAS RN: 18303-04-3	A1137 25g 250g  4-Amino-1,2,4-triazole CAS RN: 584-13-4	P2909 1g 5g  Phenyl Phosphorodiamidate CAS RN: 7450-69-3		

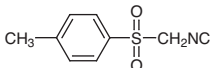
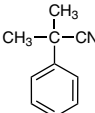
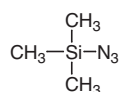
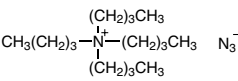
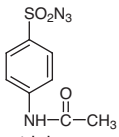
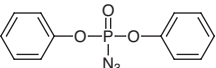
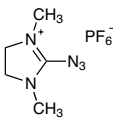
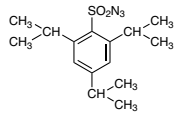
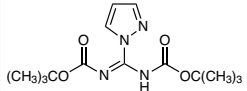
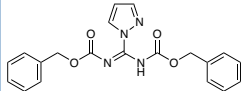
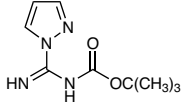
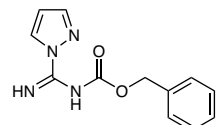
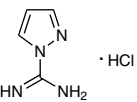
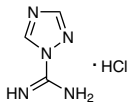
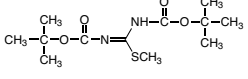
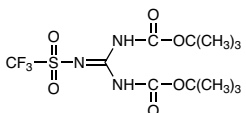
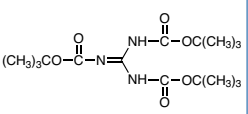
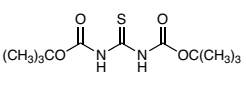
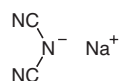
Reagents for Nitration

Reagents for Nitration		T1431 5g 25g  2,3,5,6-Tetrabromo-4-methyl-4-nitro-2,5-cyclohexadien-1-one CAS RN: 95111-49-2	N0806 300mL HNO_3 Nitric Acid (67%) CAS RN: 7697-37-2	U0015 25g  Urea Nitrate (wetted with ca. 25% Water) CAS RN: 124-47-0
G0164 25g  Guanidine Nitrate CAS RN: 506-93-4	T4131 5g 25g  Tetramethylammonium Nitrate CAS RN: 1941-24-8	T3082 5g 25g  Tetraethylammonium Nitrate CAS RN: 1941-26-0	T3651 5g 25g  Tetrabutylammonium Nitrate CAS RN: 1941-27-1	N1193 1g  N-Nitrosuccinimide CAS RN: 5336-95-8

Reagents for Cyanation

Reagents for Cyanation	T0198 25g 500g	C0441 25g 500g	B1274 5mL 25mL
	 Benzyl Thiocyanate CAS RN: 3012-37-1	 Ethyl Cyanoacetate CAS RN: 105-56-6	 tert-Butyl Isocyanide CAS RN: 7188-38-7
D5514 5g 25g	C1952 25g 300g	T0990 25mL 100mL 500mL	C1242 5g 25g
 Dimethylmalononitrile CAS RN: 7321-55-3	 Copper(I) Cyanide CAS RN: 544-92-3	 Trimethylsilyl Cyanide CAS RN: 7677-24-9	 Diethyl Cyanophosphonate CAS RN: 2942-58-7
			C2348 100mg
			 1-Cyano-4-(dimethylamino)-pyridinium Tetrafluoroborate CAS RN: 59016-56-7

C-N Bond Formation Reactions

T1046 5g 25g  TosMIC CAS RN: 36635-61-7	C3003 1g 5g  2-Chlorobenzyl Thiocyanate CAS RN: 2082-66-8	M3308 1g 5g  2-Methyl-2-phenylpropanenitrile CAS RN: 1195-98-8
Reagents for Azidation and Diazotization		
T0801 5g 25g 100g  Trimethylsilyl Azide CAS RN: 4648-54-8	T0920 5g 25g  Tetrabutylammonium Azide CAS RN: 993-22-6	S0489 100g NaN_3 Sodium Azide CAS RN: 26628-22-8
A1786 5g 25g 100g  4-Acetamidobenzenesulfonyl Azide CAS RN: 2158-14-7	D1672 5g 25g 250g  DPPA CAS RN: 26386-88-9	A2457 5g  2-Azido-1,3-dimethylimidazolium Hexafluorophosphate CAS RN: 1266134-54-6
T3434 1g 5g  2,4,6-Triisopropylbenzenesulfonyl Azide (wetted with ca. 10% Water) CAS RN: 36982-84-0	D2580 25g  Dodecylbenzenesulfonyl Azide (soft type) (mixture) CAS RN: 79791-38-1	
Reagents for Guanidinylation		
B3619 1g 5g  1-[N,N'-(Di-Boc)amidino]pyrazole CAS RN: 152120-54-2	B3605 1g 5g  1-[N,N'-(Di-Cbz)amidino]pyrazole CAS RN: 152120-55-3	B4028 5g  1-(N-Boc-amidino)pyrazole CAS RN: 152120-61-1
C2709 1g 5g  1-(N-Cbz-amidino)pyrazole CAS RN: 152120-62-2	A2055 5g 25g  1-Aminopyrazole Hydrochloride CAS RN: 4023-02-3	T3124 1g 5g  1-Carbamimidoyl-1,2,4-triazole Hydrochloride CAS RN: 19503-26-5
B4535 5g 25g  N,N'-Di-Boc-S-methylisothiourea CAS RN: 107819-90-9	B3625 1g 5g  Goodman's Reagent CAS RN: 207857-15-6	
T2964 1g 5g  1,2,3-Tri-Boc-guanidine CAS RN: 216584-22-4	B4559 1g 5g  1,3-Di-Boc-thiourea CAS RN: 145013-05-4	S0838 25g 100g  Sodium Dicyanamide CAS RN: 1934-75-4

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