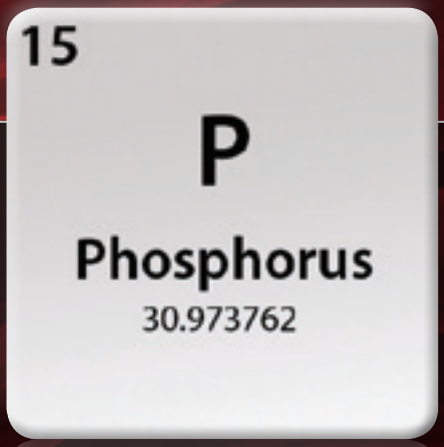
ALDRICH Chemistry



Phosphine Ligand Application Guide

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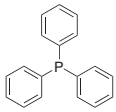
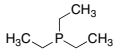
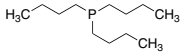
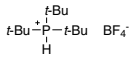
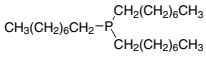
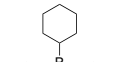
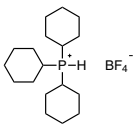
Phosphine Ligands

Transition-metal-catalyzed cross-coupling is an efficient tool for constructing C–C, C–N and C–O bonds in organic synthesis. Intensive interest has been focused on the development of the most effective ligands that improve catalyst performance. Ligands play a key role in stabilizing and activating the central metal atom and fine-tuning the selectivity of the transformation. Phosphines continue to be the most significant class of ligands for cross coupling. This brochure highlights the common applications and advantages of monodentate, bidentate, Buchwald, cataCXium®, and Dalphos ligands, all of which are commercially available from Aldrich Chemistry.

Monodentate Phosphine Ligands and Precursors

Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 93090 93092 T84409	PPh_3	603-35-0	262.29	X	X	X	X	X		• Morita-Baylis-Hillman reaction
 323322 324108 (1.0 M in THF) 324116 (1.0 M in toluene)	PMe_3	594-09-2	76.08							• Mitsunobu reaction • Transformation of azides into carbamates • Aziridines from azidoalcohols • Reagent for iminophosphoranes • Aza-Wittig reaction
 346179 (1.0 M in THF) 245275	PEt_3	554-70-1	118.16							• α-Arylation of ketones
 T49484 247049	$P(n\text{-Bu})_3$	998-40-3	202.32			X				• 1,4-addition • Acylation • Thioetherification of alcohols • Preparation of active esters
 570958 655325 (1.0 M in toluene)	$P(t\text{-Bu})_3$	13716-12-6	202.32	X	X	X	X	X	X	• Addition of organoboronic acids to aldehydes • Addition of alkynes to benzoylchlorides
 578940	$TTBP \cdot HBF_4$	131274-22-1	290.13	X	X	X	X	X	X	• Precusor for $P(t\text{-Bu})_3$ • Carbonylation • Addition of boronate esters to alkynes and alkenes
 117854 718165	$P(n\text{-Oct})_3$	4731-53-7	370.64							• Surfactant for nanomaterial and nanocrystal synthesis • A general phosphorus source for the low-temperature conversion of metals into metal phosphides
 261971	PCy_3	2622-14-2	280.43		X		X	X		• Hiyama coupling • Krische formal [4+2] of dienes and vicinal diols
 631493	$PCy_3 \cdot HBF_4$	58656-04-5	368.24		X	X		X		• Control regiochemistry of hydrostannations of 1-alkynes • Intramolecular arylation • Synthesis of indolines from *N*-alkyl-2-bromoanilines

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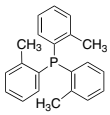
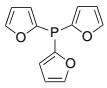
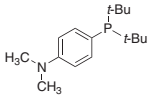
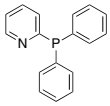
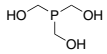
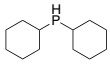
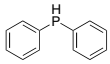
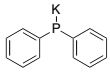
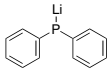
 287822	$P(o\text{-tol})_3$	6163-58-2	304.37	X	X	X	X		X	• Silylation
 265020	Me_2PPh	672-66-2	138.15							• Ligand for transition metal complexes
 383767	TFP	5518-52-5	232.17	X	X	X	X	X	X	• Cycloisomerization/oxidation of alcohols
 677264	APhos	932710-63-9	265.37		X					• A-Phos ligand for cross-coupling
 392960	Diphenyl-2-pyridylphosphine	37943-90-1	263.27							• Carbonylations • Dehydrogenative coupling • Distannylation • Silylation • Carbostannylation • Hydration • Mitsunobu reaction
 177881	Tris(hydroxymethyl)phosphine	2767-80-8	124.08							• Precursor for metal complexes
 666572	$HPCy_2$	829-84-5	198.28		X					• Precursor for ligands and metal complexes
 252964	$HPPh_2$	829-85-6	186.19							• Precusor for the phosphorus-based alkenation reagents • Source of phosphorus-centered radical
 380768	$KPPh_2$	15475-27-1	224.28							• Precusor for novel sulfur and phosphorus containing oxazolines used in asymmetric catalysis
 464872	Ph_2PLi	65567-06-8	192.12							• Dealkylation of alkyl aryl ethers • Dehydroxylation of α-hydroxy ketones
 337773	$P(i\text{-Pr})_2Cl$	40244-90-4	152.60							• *O*-Arylation of phenols • Precusor for ligands and metal complexes
 362719	$HP(t\text{-Bu})_2$	819-19-2	146.21		X		X		X	• Precusor for ligands and metal complexes
 301558	$P(t\text{-Bu})_2Cl$	13716-10-4	180.66		X					

Monodentate Phosphine Ligands and Precursors – continued

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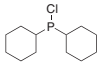
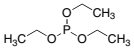
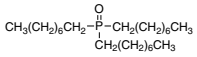
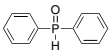
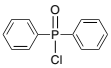
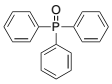
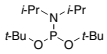
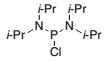
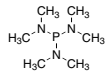
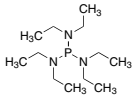
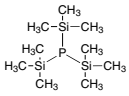
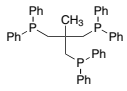
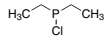
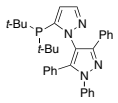
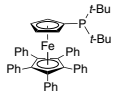
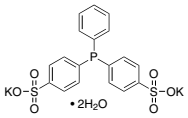
 481408	Cy_2PCl	16523-54-9	232.73							• Precusor for phosphine ligands
 92730 240907	$P(OMe)_3$	121-45-9	124.08		X		X			• Mild desulfurization reagent
 T61204	$P(OEt)_3$	122-52-1	166.16							• Reducing agent • Precusor for phosphonates or phosphates • Used with copper(I) iodide to form a stable metal complex
 T84654	$P(OPh)_3$	101-02-0	310.28			X	X			• Conversion of alcohols to halides • Wittig-type reaction • Deoxygenation • Cycloaddition reactions • Allylic alkylations • Cyclohydrocarbonylation/CO insertion • Wittig-type olefinations
 447536	2-Chloro-4,4,5,5-tetramethyl-1,3,2-dioxaphospholane	14812-59-0	182.59							• Reagent for the phosphitylation of alcohols and heteroatomic nucleophiles
 223301 346187	TOPO®	78-50-2	386.63							• Agent for extraction of metals • Capping ligand for the production of quantum dots • Solvent for the synthesis and solubilizes the growing nanoparticles
 287881	$HPOPh_2$	4559-70-0	202.19		X				X	• Hydrophosphinylation of terminal alkynes • Selective hydrophosphinylation of olefins • Precursor for diphenylphosphino-containing chiral ligands
 230235	Ph_2POCl	1499-21-4	236.63							• Acid activation • Alkylphosphine oxide formation • Amine protection
 T84603	TPPO	791-28-6	278.28							• Catalyst for allylation of aldehydes or *N*-aryl hydrazones • Promoter for coupling reaction of 1-alkenyl with sulfonyl chlorides • Cocatalyst for enantioselective cyanosilylations • Tuning ligand for asymmetric epoxidation • Scavenger for enantioselective Michael reactions of enones
 389560	$PhPOCl_2$	824-72-6	194.98							• Precursor for phosphonate ligands and metal complexes
 419362	Di-*tert*-butyl *N,N*-diisopropylphosphoramidite	137348-86-8	277.38							• Precursor for ligands • Reagent for the synthesis of nucleosides and nucleotides
 341347	Bis(diisopropylamino)chlorophosphine	56183-63-2	266.79							• Reagent for the synthesis of oligonucleotides

Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 393290	HMPT	1608-26-0	163.20		X					• Synthesis of epoxides, arene oxides, and carbonates • Conversion of alcohols to alkyl chlorides • Conversion of disulfides to sulfides • Deoxygenation of sulfoxides and azoxyarenes • Reduction of ozonides • Replaces Ph_3P in the Wittig reaction
 253189	$P(NEt_2)_3$	2283-11-6	247.36							• Precursor for phosphine ligands and metal complexes
 333670	$P(SiMe_3)_3$	15573-38-3	250.54							• Phosphorus source • Alternative to phosphine gas • Precursor of $(Me_3Si)_2PLi$
 380741	Triphos	22031-12-5	624.67							• Reagent for metal complexes
 D71984	$PhPCl_2$	644-97-3	178.98							• Precursor for phosphine ligands
 573949	Et_2PCl	686-69-1	124.55							• Precursor for phosphine ligands
 676632	BippyPhos	894086-00-1	506.62						X	• Amidation • Coupling of hydroxylamines with aryl halides
 675784	QPhos	312959-24-3	710.71		X			X	X	• α-Arylation of esters, amides and aldehydes
 695467	PTA	53597-69-6	157.15		X			X		• Morita–Baylis–Hillman reactions • Hydrogenations • Olefin hydroformylations • Cyclization
 698539	Bis(*p*-sulfonatophenyl) phenylphosphine dihydrate dipotassium salt	151888-20-9	534.62		X					
 RNI00061	6-DPPon	64741-28-2	279.1							• *New* • Breit's Regioselective Hydroformylation Ligand

Bidentate Phosphine Ligands and Precursors

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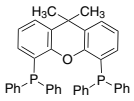
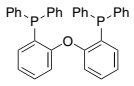
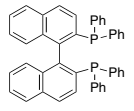
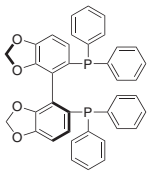
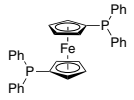
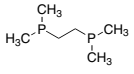
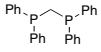
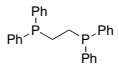
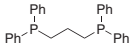
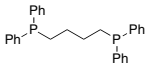
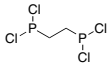
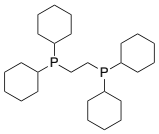
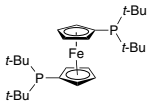
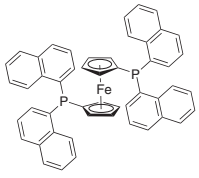
 526460	XantPhos	161265-03-8	578.62		X	X			X	• C-P bond formation • Regioselective hydroformylation of allylamines • Borylation of nitriles
 510017	DPEPhos	166330-10-5	538.55	X	X		X		X	• Hydroamination
 481084	(±)-BINAP	98327-87-8	622.67			X			X	• Intramolecular acylation of aryl bromides • Minisci reaction with alcohols • Decarboxylative benzylation of diphenylglycinate Imines • Regioselective hydroarylation • Decarboxylative cycloaddition • S-arylation
 693006	(S)-SEGPHOS®	210169-54-3	610.57							• Stereoselective preparation of homoallylic alcohols via Ir-catalyzed stereoselective transfer hydrogenative crotylation of an allylic acetate with alcohols or aldehydes
 460273	dppbenz	13991-08-7	446.46							• Cycloadditions • Addition of arylboronic acids • Baeyer-Villiger oxidation • Reduction of propargyl oxiranes and hydroboration of styrene
 177261	dppf	12150-46-8	554.38	X	X	X	X	X	X	• Tsuji-Trost allylation • Carbonylation of vinyl triflates • Enantioselective ene reaction • Addition of organoboronic acids to aldehydes
 261939	dmpe	23936-60-9	150.14							• Ligand for metal complexes
 127566	dppm	2071-20-7	384.39		X		X			• Ligand for metal complexes

Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 106496	dppe	1663-45-2	398.42	X	X		X			• Allylation of ketones • Decarboxylation of allyl esters • [3 + 2] cycloaddition • Carbonylation • Homo-Diels–Alder cycloaddition of norbornadiene with alkynes
 262048	dppp	6737-42-4	412.44	X	X			X		• Deoxygenation of phenols via the corresponding triflate • Substitution of aryl bromides with vinyl ethers • Carbonylation of aryl triflates • Addition of β-dicarbonyl enolates to dienes • Coupling of aryl and alkyl halides with Grignard reagents
 261947	dppb	7688-25-7	426.47	X	X	X	X	X		• Lactonization of unsaturated alcohol • Salkylation of allyl acetates • Hydroacylation • Alkyne Isomerizations • Intramolecular allylation of alkenes • Alkyne–CO_2 coupling
 261920	1,2-Bis(dichlorophosphino) ethane	28240-69-9	231.81							• Precusor for ligands and transition metal complexes
 479500	dcpe	23743-26-2	422.61	X	X					• Decarboxylative coupling • Ligand for metal complexes
 695149	D^{t}BPF	84680-95-5	474.42		X				X	• Arylation • Indolization
 L511501	dnpf	N/A	754.61				X			• Zhou regioselective arylation of α-olefins

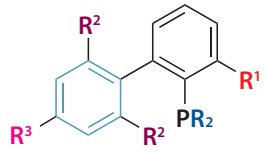
Buchwald Ligands

Buchwald ligands are bulky electron-rich dialkylbiaryl phospines and are known to improve reactivity in palladium catalysis. These highly active reagents have been extensively applied in the synthesis of pharmaceuticals, natural products, polymers, and new materials. The structure of the dialkylbiaryl ligand is directly correlated to the efficiency of catalysts containing these ligands.

Structural features of Dialkylbiaryl Phosphine Ligands

R^2: Increases catalyst stability by preventing cyclometalation, Encourages formation of $L_1Pd(0)$

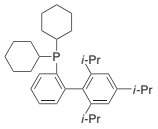
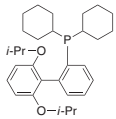
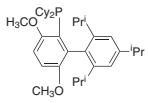
R^3: When not equal to H, usually only for ease of synthesis

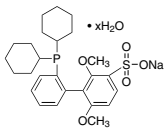
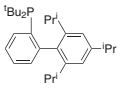
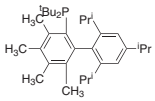
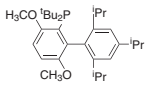
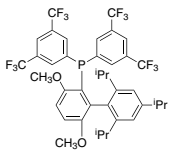


R^1: Substitution promotes reductive elimination

R: Electron-rich groups accelerate rate of oxidative addition

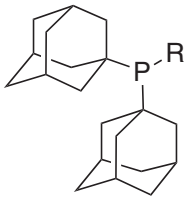
: Prevents oxidation at P by O_2, Accelerates reductive elimination

Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 638099	Cyclohexyl JohnPhos	247940-06-3	350.48	X	X				X	• Hiyama coupling • Methylation of aryl halides • C-B bond formation • Oxidation of alcohols to ketones • α-Arylation of ketones
 638021	DavePhos	213697-53-1	393.54	X	X				X	• Kumada-Corriu coupling and α-Arylation of ketones • Hydroamination
 638064	XPhos	564483-18-7	476.72	X	X	X	X	X	X	• Hiyama coupling • Carbonyl enolate coupling
 638072	SPhos	657408-07-6	410.53	X	X		X		X	• Kumada-Corriu coupling
 695262	MePhos	251320-86-2	364.5		X				X	• α-Arylation of ketones
 663131	RuPhos	787618-22-8	466.64	X	X			X	X	• α-Arylation of oxindoles
 718742	BrettPhos	1070663-78-3	536.77						X	• Trifluoromethylation of aryl chlorides

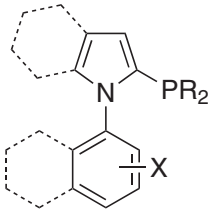
Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 677280	^{S}SPhos	1049726-96-6	512.57		X					• 1, 2-addition of arylboronic acids to aldehydes and ketones
 695882	PhDavePhos	240417-00-9	381.45						X	• Heteroarene benzylation
 638080	*t*BuXPhos	564483-19-8	424.64						X	• α-Arylation of tetramic acids • Decarboxylative coupling of aromatic acids and aryl iodides • Carboxylation of aryl bromides with CO_2
 638439	JohnPhos	224311-51-7	298.40		X		X		X	• C-Si bond formation
 675938	Tetramethyl di-*t*BuXPhos	857356-94-6	480.75						X	• Conversion of aryl halides to phenols
 695211	*t*BuMePhos	255837-19-5	312.43				X		X	• α-Arylation of ketones • Formate reduction of *N*-heterocyclic allylic acetates
 730998	*t*BuBrettPhos	1160861-53-9	484.69						X	• *O*-Arylation of ethyl acetohydroximate • C-F bond formation • Conversion of aryl and vinyl triflates to bromides and chlorides • Conversion of aryl chlorides, triflates, and nonaflates to nitroaromatics
 695874	*t*BuDavePhos	224311-49-3	341.47						X	• α-Arylation of esters • C-Si bond formation
 731013	JackiePhos	1160861-60-8	796.66						X	• Coupling of aryl nonaflates and triflates with secondary amides, ureas, carbamates, and sulfonamides • Coupling of aryl chlorides with secondary amides, carbamates, and sulfonamides

cataCXium® Ligands

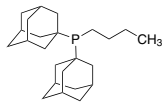
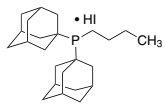
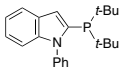
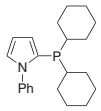
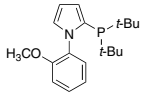
cataCXium Ligands are highly effective ligands for palladium catalyzed cross-coupling reactions. Di-adamantylalkylphosphine, known as cataCXium A, is a bulky and electron-rich phosphine ligand used for Heck and Suzuki couplings, Buchwald-Hartwig amination of arylchlorides, and α-arylation reactions of ketones. In reactions, these ligands allow for low catalyst loadings used under mild conditions. Recently, another class of basic, sterically hindered phosphines featuring phosphino-substituted *N*-aryl pyrroles (cataCXium P) has shown high catalyst turnover numbers for the Suzuki coupling of both electron-rich and electron-deficient aryl chlorides.



cataCXium® A



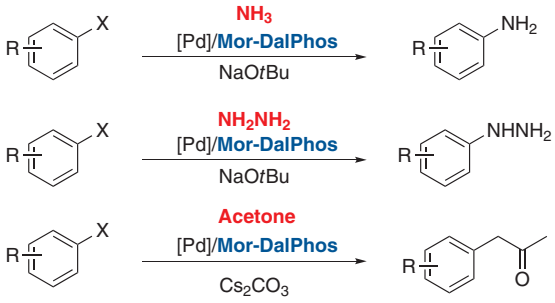
cataCXium® P

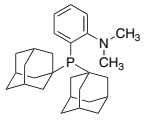
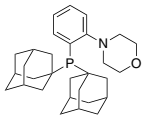
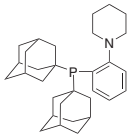
Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 671479	cataCXium® A	321921-71-5	358.54		X		X	X	X	• Formylation of aryl bromides • Arylation
 671584	cataCXium® AHI	714951-87-8	486.45		X				X	• Arylation
 672661	cataCXium® PtB	672937-61-0	287.38		X				X	• Dehydrogenation of iso-propanol
 672343	cataCXium® PIntB	740815-37-6	337.44		X			X	X	
 672017	cataCXium® PCy	672937-60-9	339.45		X		X			• Amination of alcohols with ammonia
 672564	cataCXium® POMetB	1053658-91-5	317.41						X	

DalPhos Ligands

The bulky di(1-adamantyl)phosphino [P(1-Ad)$_2$] fragment is common to the DalPhos ligand scaffold. These chelating N,P ligands are useful for Pd-catalyzed C-N and C-C bond formation. The more reactive Mor-DalPhos improves the scope and utility of ammonia coupling at room temperature, and is also effective in coupling of hydrazine and acetone.

Pd Catalyzed Monoarylation of Ammonia, Hydrazine and Acetone



Structure and Cat. No.	Common Name	CAS No.	Mol. Wt.	Negishi	Suzuki	Stille	Heck	Sonogashira	Buchwald-Hartwig (C–N or C–O)	Additional Applications
 751413	Me-DalPhos	1219080-77-9	421.6						X	
 751618	Mor-DalPhos	1237588-12-3	463.63						X	• Monoarylation of hydrazine and acetone
 759198	Di(1-adamantyl)-1-piperidinyl-phenylphosphine	1237588-13-4	461.66						X	• *New*

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