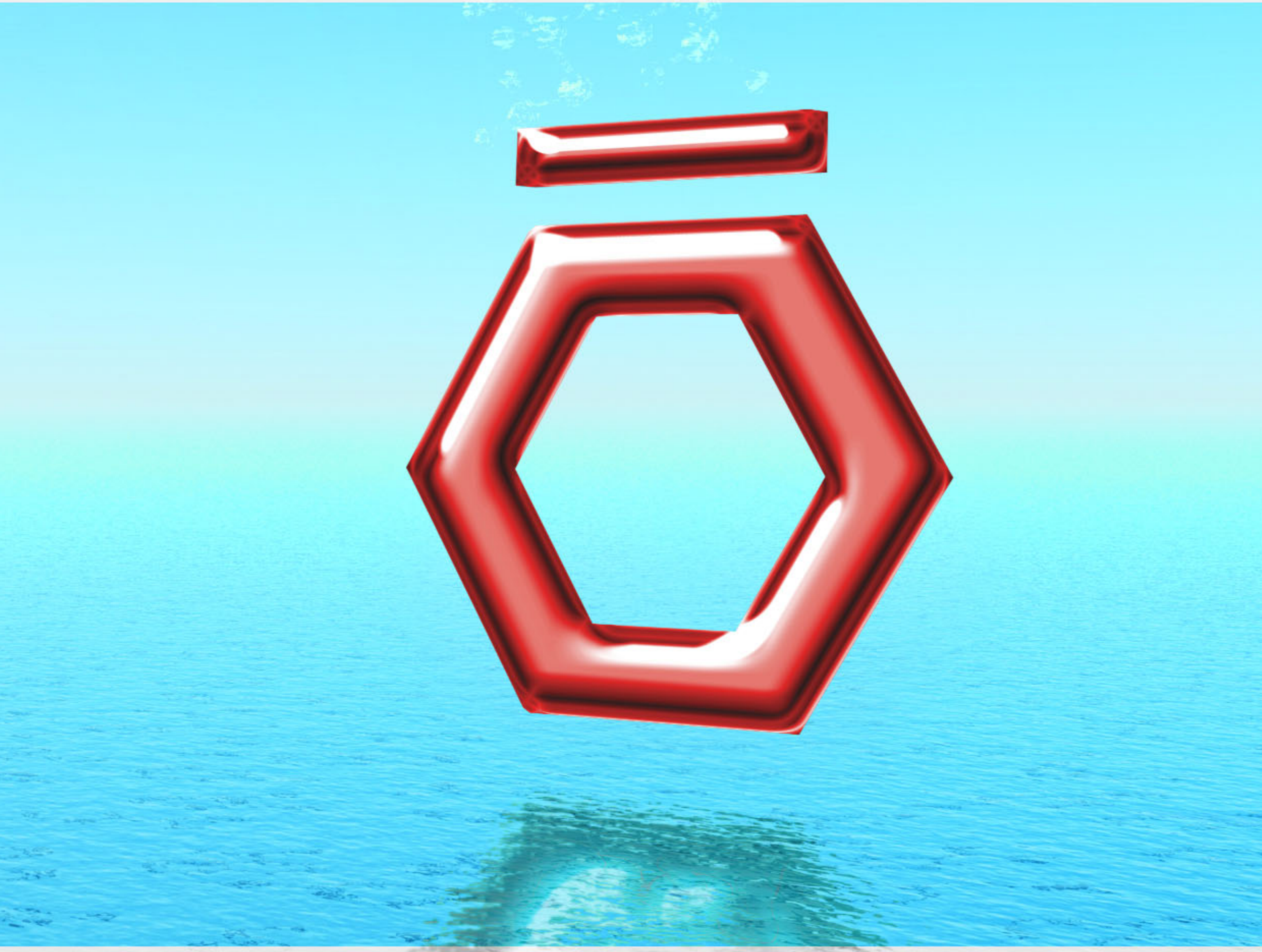
ORGANOLITHIUM COMPOUNDS



ACROS ORGANICS

A Fisher Scientific company

Organometallics

Organometallics

Available from Acros Organics

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Why Chemists choose Organolithium compounds from Acros Organics :

- **more convenient**
- **wider septum**
- **extended product range**
- **highest quality**

Organolithium reagents are sensitive to thermal decomposition.

By opening its new filling stations for metalorganics in Belgium, customers can now benefit from fresh material stored under ideal conditions and dispatched throughout Europe within 24-48 hours.

Acros Organics metalorganics come in sturdy, graduated reusable borosilicate bottles.

The wider size of the septum allows multiple punctures without any risk of damage.

The Teflon coated septum is compatible with the reagents in the bottle for maximum convenience and safety.

Instructions on how to open and use the product safely are clearly printed on every individual bottle stopper.

Over 20 new organometallics are available in a variety of formulations and pack sizes for the demanding chemist.

Consult **www.acros.com** for the latest product additions.

Contents

I. Introduction

Organometallic compounds are amongst the most often used reagents in organic synthesis.

The earliest organometallic compound was already discovered in the early 19th century ("Zeise's salt"; a zinc-olefin complex was first reported in 1827!) and first examples of synthetic organometallic chemistry are the organozinc-compounds, discovered by Edward Frankland in 1849, the organo-magnesium compounds discovered by Victor Grignard and his teacher Philippe Barbier in 1901 and the organolithium compounds, discovered by Wilhelm Schlenk in 1917(1)

But only since the 1950th, based on the pioneering work of Georg Wittig and Henry Gilman, organometallic reagents became a routinely used tool in the synthetic organic laboratory.

A very early but still invaluable application of organometallic reagents is the olefin-polymerisation with the so-called Ziegler-Natta-catalyst (Invented in 1953, Nobel price for Karl Ziegler and Giulio Natta in 1963).(2)

The usage of organometallic compounds can be classified in several major applications:

- *Strong bases*
- *Nucleophiles*
- *Metal exchange*
- *Reducing agents*
- *Polymerisation initiator/catalyst*
- *Catalysts for organic reactions*
- *Material sciences*

The following brochure will give an overview of the properties and typical reactions of organolithium compounds in some fields of organic chemistry and the product range offered by Acros Organics.

Literature References:
1 A biography about Wilhelm Schlenk in Angew. Chem 2001, 113, 343
2 Karl Ziegler, Nobel lecture Dec. 12th, 1963

II. Organolithium compounds[3,4], properties & structures

Alkyllithium compounds are widely used as very strong bases, nucleophiles and reagents for metallations in organic synthesis. The following table[5] shows, that alkanes and arenes are very weak acids, hence their corresponding lithium-derivatives are extremely strong bases.

The most common members of the alkyllithium-family are n-butyllithium, methyllithium and tert-butyllithium (for which 2-methylpropane is the corresponding acid).

Simple alkyllithium-compounds are soluble in hydrocarbon- and in ether-solvents (although the more basic compounds can react with the latter) forming aggregates of mostly dimeric, tetrameric or hexameric species[6,7]. The aggregation[8] and, as a consequence, the reactivity of organolithiumcompounds can be strongly influenced by the solvent and/or complexing co-solvents[9,10] and additives[11,12,13].

Compound	pKa
2-Methylpropane	53.0
Ethane	50.0
Methane	48.0
Ethene	44.0
Benzene	43.0
Ammonia	38.0
Ethyne	25.0
Ethanol	16.0
Water	15.7

Strong complexing agents like i.e. N,N,N',N'-tetramethylethylendiamine are able to cleave the aggregates to form monomeric alkyllithium-complexes which are then much more reactive.

The mixture of n-butyllithium with potassium-tert-butanolate is called "Superbase"[11,12,13,14].

Aggregation of Organolithium-reagents in various solvents

Compound	Solvent	Aggregation number
MeLi	THF	4
MeLi	DEE	4
n-BuLi	Cyclohexane	6
n-BuLi	THF	4 / 2
sec-BuLi	Cyclopentane	4
sec-BuLi	THF	2 / 1
tert-BuLi	Hexane	4
tert-BuLi	DEE	2
tert-BuLi	THF	1

MeLi = Methyllithium • n-BuLi = n-Butyllithium • sec- BuLi = sec-Butyllithium • tert-BuLi tert.-Butyllithium • DEE = Dietylether • THF = Tetrahydrofuran

Literature References:

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III. Reactions of organolithium compounds

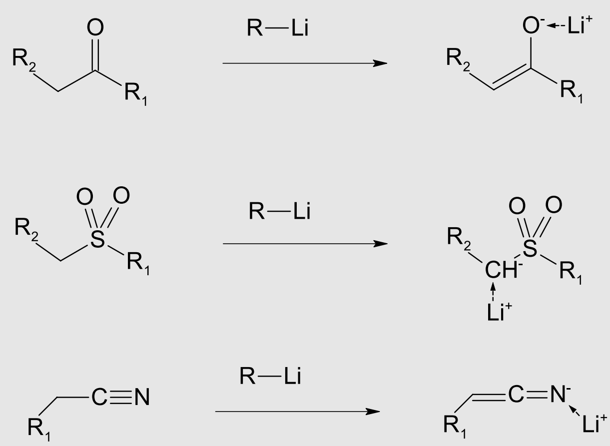
a. Metallation

Many hundreds of functionalised organolithium compounds have been prepared by the metallation reaction with n-butyllithium (or other alkyllithium compounds.

R-H + n-Butyl-Li → R-Li + Butane

The metallation uses the fact that the strong bases like n-butyllithium are capable to deprotonate organic molecules if they have "acidic" protons.

Such "acidic" protons in organic molecules are mostly found at positions where a negatively charged anion is stabilised by a suitable functional group. Such molecules are i.e.:



- *carbonylcompounds like aldehydes, ketones, esters, imines etc*
- *sulfones, sulfoxides, sulfoximides*
- *nitriles*
- *terminal acetylenes*
- *nitro-compounds*
- *benzylic systems*

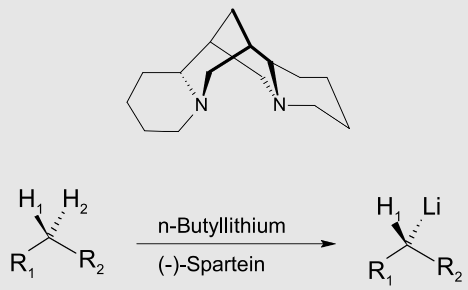
The stabilisation of carbanions by functional groups follows the order[15]:

$-NO_2$ > -C(O)-R > -COOR > $-SO_2$> -CN ~ CONHR > Halogenide > H > R

The stabilisation of such carbanions is influenced by mesomeric and inductive effects and the hybridisation of the anionic carbon.

The resulting enolates, sulfonyl[16,17]- and sulfinyl[18]-carbanions are valuable intermediates for aldol-condensations and many other reactions.

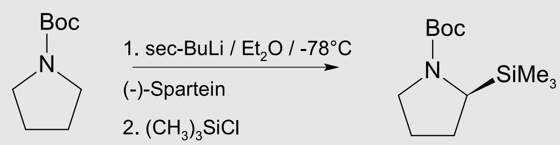
By using chiral-chelating ligands like (-)-spartein it is possible to deprotonate a prochiral substrate with high enantiomeric excess[19,20] to chiral organolithium compounds[21].



Products available from Acros Organics

(-)-Spartein sulfate pentahydrate 21562
n-Butyllithium 1.6 M solution in hexanes 18127

An example for the enantioselective deprotonation is the synthesis of (S)-2-trimethylsilyl-N-Boc-pyrrolidine[22].



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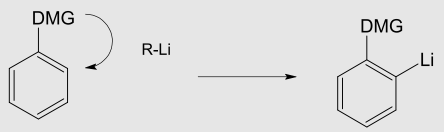
The metallation of diisopropylamine with n-butyllithium yields the very important organic base lithium-diisopropylamide (LDA). LDA is strong enough to deprotonate most organic molecules, but due to its bulky, sterically hindered substituents, it is not nucleophil. LDA is the preferred base[23] for the deprotonation of substrates, where a nucleophilic attack is also possible.

Products available from Acros Organics

Diisopropylamine, 99% 15907
Lithiumdiisopropylamide,
2 M in THF/Heptane 26883
sec-Butyllithium 1.3 M solution in
cyclohexane/hexane (92/8) 18754

b. Ortho-metallation[24]

Metallation of an aromatic ring near a substituent, which acts as a "Directed Metallation Group", is called "Ortho-Metallation". Several groups can function as DMG's, i.e. sulfones, sulfonamides, amines[25], amides[26], carbamates[27], thio-[28] and methoxy groups, they have in common the ability to coordinate the approaching cation (= lithium-ion) and/or to increase the acidity of the ortho-hydrogen.

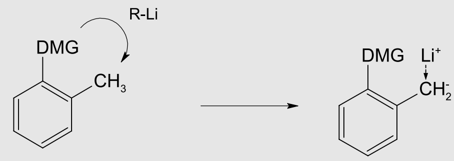


Ortho-Lithiation

The relative rate of directed metallation follows the order[29]

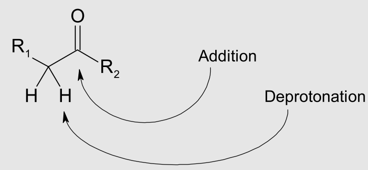
$$SO_2NR_2,\ C(O)N\text{-}R,\ CH_2NR_2 > OCH_3 > CH_2CH_2NR_2,\ CF_3,\ F,\ NR_2$$

A special form of ortho-lithiation is the "lateral-metallation". In this reaction a benzylic hydrogen is abstracted due to the higher acidity compared with the ring-hydrogens. Besides butyllithium also lithium-diisopropylamide is a useful base for this reaction.



Lateral metalation

c. Nucleophilic addition and substitution



Stabilized organolithium compounds like enolates and sulfonylcarbanions can react as nucleophiles[30] with alkyl-halogenides and carbonyl-compounds in a wide range of reactions:

- *Alkylation of Alkylhalogenides*
- *Addition to Carbonylcompounds*[31]
- *Alkylation of Allylhalogenides*
- *Epoxide-Ring Opening*[32]
- *Conjugate Addition*[33]
- *Addition to Carbondioxide*[34]

Literature References:

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32 R.C.Larock, Comprehensive Organic Transformations VCH, New York **1989**. p 512.

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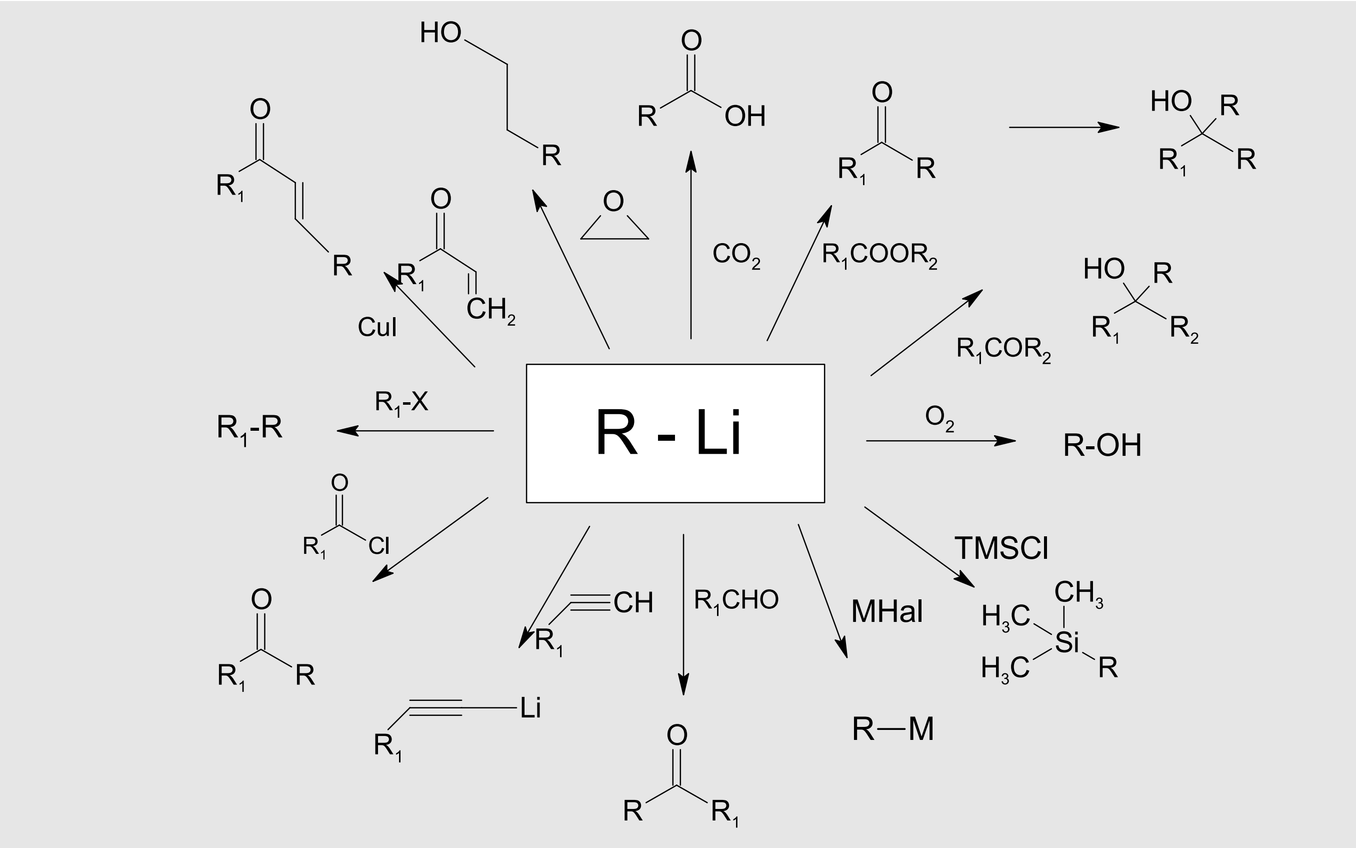
34 Leads with excess of RLi to ketones: T.M.Bare, H.O.House, Org.Synth. 49 (**1969**) 81.

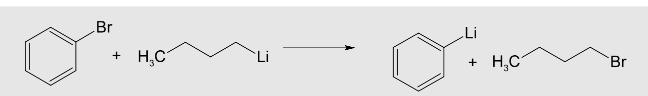


Simple, non-stabilized, alkyllithium reagents can react in the same way, but often they have to be transferred to more nucleophilic and less basic reagents like dialkyllithiumcuprates[43] to avoid deprotonation as a side reaction.

The addition to prochiral carbonyl-compounds can be enantioselective by using chiral ligands[35,36].

d. Halogen-Metal exchange

The Halogen metal-exchange reaction was discovered in the late 1930th by Gilman[37] and Wittig[38]. The reaction is often used to prepare vinyl- and aryl-lithium compounds from the more reactive alkyl-lithium species[39].

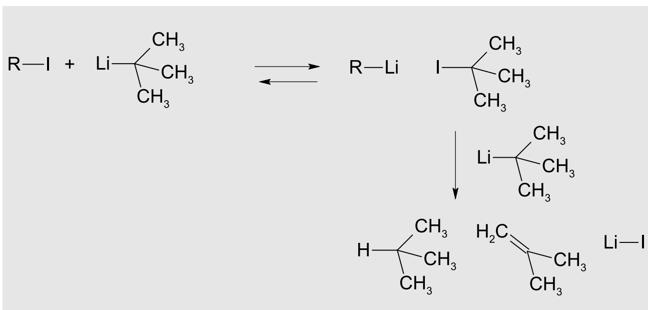


A sequence of halogen-metal exchanges - stannylation - Stille coupling reactions with pyridines has been used for the synthesis of oligo-pyridine ligands[40].

The halogen-metal exchange between aliphatic substrates is less common and has experimental limitations due to the fact, that the reaction is most often an equilibrium:

$$R\text{-}Li + R'\text{-}X \leftrightarrow R\text{-}X + R'Li$$

and also side reactions like eliminations, couplings and α-metallations are possible. Useful halogen-metal-exchange reactions between aliphatic substrated can be achieved by shifting the equilibrium to the product side. Here tert-butyllithium plays a prominent role as reagent. By addition of a second equivalent tert-butyllithium the resulting tert-butyliodide is removed instantly from the equilibrium[41].



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The halogen-metal exchange is preferably done with iodides or bromides, while chlorides are less common. Addition of an ethereal solvent helps to remove excess of tert-butyllithium because it reacts with this solvent at higher temperatur.

Products available from Acros Organics

tert-butyllithium 1,5 M in Pentane . . . 18128

e. Transmetallation

The organolithium compounds are very often used to prepare other metallorganic compounds through the transmetallation reaction:

R-Li + M-X → R-M + Li-X
(M = Metalion, X = Halide)

Products available from Acros Organics

$CeCl_3$ anhydrous	36975
CuI, 99,995%	20150
CuI 98%	19490
CuCN 99%	20208
$TiCl_4$ 99,9%	19723
$Ti(OiPr)_4$ 98%	19470
$ZnCl_2$ 0,5M in THF	37006
$ZnCl_2$ 1M in Et_2O	37005

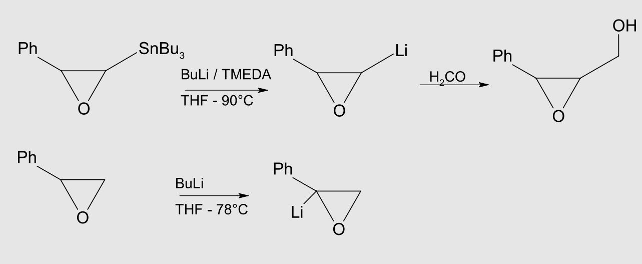
This reaction has been used to prepare the important organo-copper[42,43,44] (A) and organo-titanium[45] (B) and many other metallorganic compounds[46] which have often higher selectivity than the organolithium compounds.

(A) 2 R-Li + CuX → [R-Cu + R-Li + LiX] → R2CuLi + LiX

(B) R-Li + TiCl4 → R-TiCl3 + LiCl

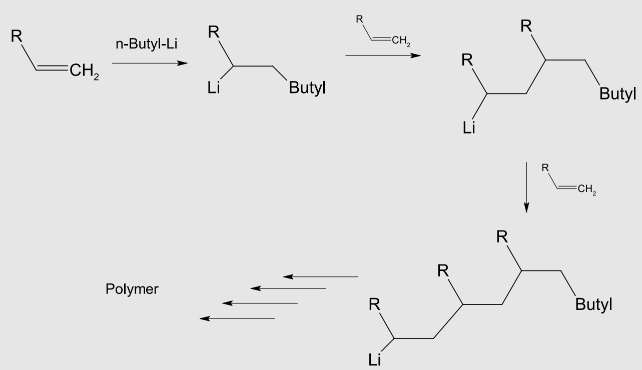
Highly functionalized mixed cuprates have been synthesized recently by halogen-metal exchange of sterically hindered alkylhalogenides with tert-butyllithium and subsequent transmetallation to the cuprate followed by a second halogen-metal-exchange[47].

An example of transmetallation to prepare complex and sensitive organolithium reagents is the following reaction, which gives the β-lithio-styreneoxide,whereas the direct metallation yields the α-isomer[49].



f. Anionic Polymerisation

A major industrial use of alkyllithium compounds, specifically n-Butyllithium, is the catalysis of the anionic polymerization of butadiene[50], isoprene and styrene.



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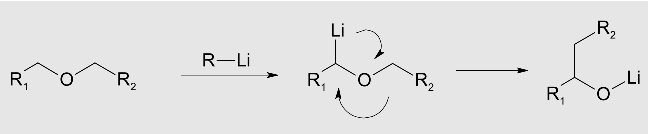
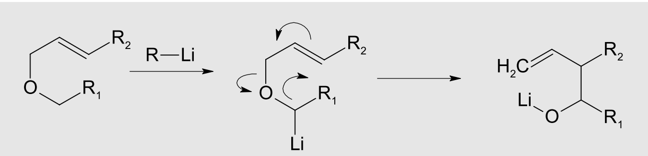
IV. Named organic reactions with organolithium compounds

a. [1,2] and [2,3]-Wittig rearrangement



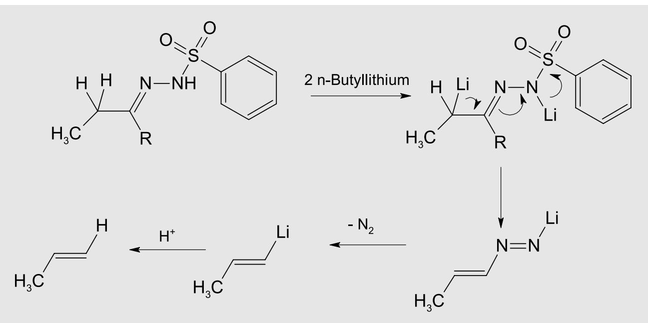
The [1,2] Wittig-rearrangement of ethers with alkyl lithiums to yield alcohols via a [1,2]-shift[51]. Usually a strong base like phenyllithium is used. The groups R_1 and R_2 may be alkyl, aryl or vinyl. The facility of migration follows the order allyl > benzyl > ethyl > methyl > phenyl[52].



The [2,3]-Wittig rearrangement[53] is a sigma-tropic rearrangement of a α-deprotonated allylether.

b. Shapiro Olefination[54]

The Shapiro Olefination is a decomposition of a p-tosylhydrazone with two equivalents of a strong base (usually methyllithium). The reaction is used to produce the olefins or vinyllithium-compounds.

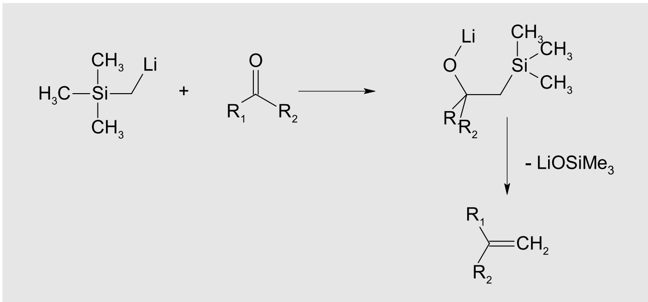


Products available from Acros Organics

p-Toluenesulfonylhydrazine 15786

c. Peterson Olefination[55]

The Peterson Olefination is the addition of a α-Silylcarbanion to a carbonyl-compound which yields after elimination of Lithiumtrimethylsilanoate the olefine.

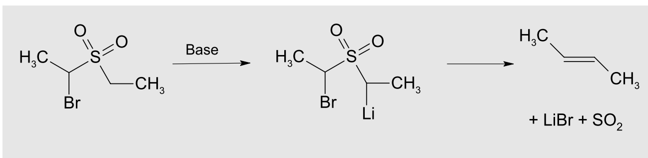


Products available from Acros Organics

(Trimethylsilyl)methyllithium,
1 M in Pentane 37745
(Trimethylsilyl)methylmagnesium
bromide, 1 M in Diethylether 37746

d. Ramberg-Bäcklund-Reaction[56]

In this reaction is a α-Halogensulfone is treated with a strong base to give an olefine.



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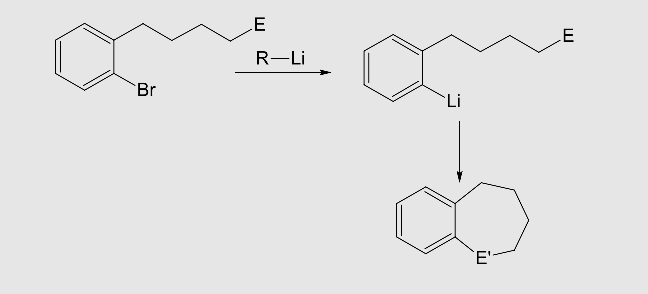
e. Parham Cyclization[57]

The Parham cyclisation is an example for a halogen-metal exchange on an aromatic ring followed by a ring-closure reaction with the electrophile as part of a side chain.

E can be CH_2-Br, CH_2-Cl, Epoxide, Carbonyl etc.



Literature References:
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VI. Indicators for the titration of organolithium compounds

Organolithium reagents are used almost always in solution. Knowing the exact content of reagent in the solvent is crucial for an exact dosage of the reagent.

Several methods have been developed to examine the titer of organolithium compounds.

The oldest method is the **Gilman double titration**.[58]

Products available from Acros Organics

Benzylchloride, 99,5+%	18085
Methyl-orange-xylene-cyanol, solution in Water	41467
Hydrochloric acid, 1 N standard solution	12421
Hydrochloric acid, 0.1 N standard solution	12420

This method determines both the organolithium compounds and also the lithium-alcoholate content in solution. The latter can be produced if oxygen comes in contact with the organolithium product.

In the Gilman method the butyllithium is first derivatized with benzylchloride to neutral pentylbenzene leaving all other basic impurities unaffected. The reaction mixture is hydrolysed and titrated with a hydrochloric acid standard solution to a methyl-orange-xylene-cyanol end point. The result represents the amount of the basic impurities, which are not organolithiums.

A second aliquot of the organolithium solution is hydrolysed and titrated to give the total value for alkaline compounds.

Products available from Acros Organics

1,10-phenanthroline	15753
2,2'-biquinoline	10630
Toluene, extra dry	32698
2-Butanol, p.a.	22029

The **Watson-Eastham method** uses the fact, that organolithium compounds form coloured, stable charge-transfer (CT) complexes with indicators like 1,10-phenanthroline (green-yellow-CT-complex) or 2,2'-biquinoline (red-brown CT complex) in dry toluene solution. The titration uses 2-butanol in dry toluene as titrant. The CT complex is only destroyed when all unreacted organolithium compound is already reacted with the alcohol, then at the end-point the solution decolourises.

Substances, which form coloured mono-anions with organolithium compounds, can be used also as indicators. The metallorganic base is added to an excess of the indicator, which forms the coloured anion. Titration with an alcohol in a dry solvent until the colour disappears shows the end point. An example is α-Methylstyrene, which forms a red adduct with n-Butyllithium, which can be titrated with n-Butanol.

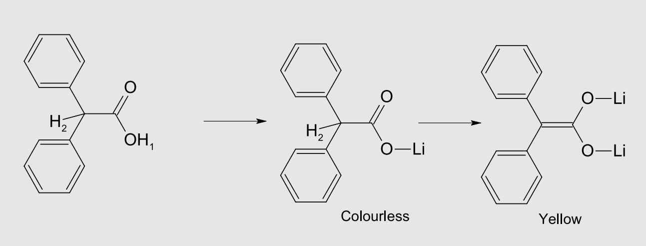
Products available from Acros Organics

α-Methylstyrene	12771

One very common, simple and fast method uses the fact the some compounds form very deeply coloured di-anions after double deprotonation with organolithium compounds. Usually the (dry!) indicator is weighed exactly and dissolved in dry THF. Then the organolithium-solution is added directly by a syringe until the color of the end point appears.

Literature References:
58 H.Gilman, A.H.Haubein, J.AmChem.Soc. 66 (**1944**) 1515.

An example is diphenylacetic acid. The first equivalent of base is used to deprotonate H1, the resulting carboxylate-ion is colourless. Only when H2 is also deprotonated the intensive yellow colour of the di-anion appears.



Similar reagents are N-Pivaloyl-O-toluidine and N-Pivaloyl-O-benzylaniline[59], two crystalline, non-hygroscopic and stable compounds which form intensive yellow and yellow-orange dianions.

Products available from Acros Organics

Diphenylacetic acid 11715
N-Pivaloyl-O-toluidine 34673
N-Pivaloyl-O-benzylaniline 34674

Literature References:

59 J.Suffert, J.Org.Chem. 54 (1989) 509.

V. Organolithium compounds available at Acros Organics

ORGANOMETALLICS	FORMULATION		PRODUCT NO	AVAILABLE PACKSIZE		
n-Butyllithium	1.6 M	solution in hexanes	18127	100ml	800ml	
n-Butyllithium	2.5 M	solution in hexanes	21335	100ml	800ml	
n-Butyllithium	2.2 M	solution in cyclohexane	37749	100ml	800ml	
n-Butyllithium	2.6 M	solution in toluene	37893	100ml	800ml	
iso-Butyllithium	1.6 M	solution in heptane	37759	100ml	800ml	
sec-Butyllithium	1.3 M	solution in cyclohexane/hexane (92/8)	18754	100ml	800ml	
tert-Butyllithium	1.5 M	solution in pentane	18128	100ml	800ml	
Ethyllithium	1.7 M	solution in dibutyl ether	37757	100ml	800ml	
n-Hexyllithium	2.3 M	solution in n-hexane	30165	100ml	800ml	
Lithium acetylide, ethylenediamine complex		powder 85%	18125	50g	100g	
Lithium amide		powder 95%	19986	5g	100g	500g
Lithium bis(trimethylsilyl)amide		powder 95%	33814	10g		
Lithium bis(trimethylsilyl)amide	1.0 M	solution in THF	34770	100ml	800ml	
Lithium bis(trimethylsilyl)amide	1.0 M	solution in methyl tert-butyl ether	34567	100ml	800ml	
Lithium bis(trimethylsilyl)amide	1.3 M	solution in toluene/ethylbenzene	37748	100ml	800ml	
Lithium bis(trimethylsilyl)amide	1.3 M	solution in hexanes/ethylbenzene	37747	100ml	800ml	
Lithium tert-butoxide		powder 95%	30122	available soon		
Lithium tert-butoxide	1.0 M	solution in hexanes	38015	available soon		
Lithium tert-butoxide	2.2 M	solution in THF	38016	available soon		
Lithium cyclopentadienide		powder 97%	31658	5g		
Lithium diisopropylamide	2.0 M	solution in heptane/THF/ethylbenzene	26883	100ml	800ml	
Lithium ethoxide	1.0 M	solution in ethanol	38017	available soon		
Lithium ethoxide	1.0 M	solution in THF	38018	available soon		
Lithium hydride		powder 98%	19119	10g	100g	500g
Lithium isopropoxide		powder 95%	30124	available soon		
Lithium isopropoxide	2.0 M	solution in THF	38019	available soon		
Lithium isopropoxide	1.0 M	solution in hexanes	38020	available soon		
Lithium methoxide		powder 98%	30123	available soon		
Lithium methoxide	2.0 M	solution in methanol	33675	100ml	800ml	
Lithium pentamethylcyclopentadienide		powder 98%	35324	1g	5g	
Methyllithium	1.6 M	solution in diethyl ether	18875	100ml	800ml	
Methyllithium, as complex with lithium bromide	2.2 M	solution in diethyl ether	18129	100ml	800ml	
Phenyllithium	2.0 M	solution in dibutyl ether	36515	100ml	500ml	
(Trimethylsilylmethyl)lithium	1.0 M	solution in hexanes	37745	available soon		

DRY SOLVENTS

QUALITY DRY SOLVENTS IN TWO FORMATS

› Highest Quality extra dry solvents (down to 10 ppm water content), filtered over 0.2 micron PFTE filter for the demanding chemist.
› Quality dry solvents (down to 50 ppm water content), stored over 3 Å molecular sieve for daily use.

HIGHEST QUALITY EXTRA DRY SOLVENTS (DOWN TO 10 PPM WATER), FILTERED OVER 0.2 MICRON PFTE FILTER

Product name	Code 100ml	Code 1l	Code 2.5l
Acetone, extra dry, water < 50 ppm	326801000	326800010	
Acetonitrile, extra dry, water < 10 ppm	326811000	326810010	
Chloroform, extra dry, water < 50 ppm, stabilized	326821000	326820010	
Cyclohexane, extra dry, water < 50 ppm	326831000		
1,2-Dichloroethane, extra dry, water < 50 ppm	326841000	326840010	
Dichloromethane, extra dry, water < 30 ppm, stabilized	326851000	326850010	326850025
Dimethylformamide, extra dry, water < 50 ppm	326871000	326870010	326870025
1,4-Dioxane, extra dry, water < 50 ppm, stabilized	326891000	326890010	
Ether, extra dry, water < 50 ppm, stabilized	326861000	326860010	
Ethyl acetate, extra dry, water < 50 ppm	326901000		
n-Heptane, extra dry, water < 30 ppm	326911000		
n-Hexane, extra dry, water < 20 ppm	326921000	326920010	
Isopropanol, extra dry, water < 50 ppm	326961000	326960010	
Methyl alcohol, extra dry, water < 50 ppm	326951000	326950010	
1-Methyl-2-pyrrolidinone, extra dry, water < 50 ppm	326931000	326930010	
Methyl sulfoxide, extra dry, water < 50 ppm	326881000	326880010	
Pyridine, extra dry, water < 50ppm	339421000	339420010	
Tetrahydrofuran, extra dry, water < 50 ppm, stabilized	326971000	326970010	326970025
Toluene, extra dry, water < 30 ppm	326981000	326980010	
2,2,4-Trimethylpentane, extra dry, water < 30 ppm	326941000		

QUALITY DRY SOLVENTS (DOWN TO 50 PPM WATER), STORED OVER 3 Ä MOLECULAR SIEVE

Product name	Code 100ml	Code 1l	Code 2.5l
Acetonitrile, extra dry < 50 ppm	364311000	364310010	
Chloroform, extra dry < 50 ppm, stabilized	364321000	364320010	
Dichloromethane, extra dry, water < 50 ppm, stabilized	348461000	348460010	348460025
N,N,-Dimethylformamide, extra dry, water < 50 ppm	348431000	348430010	348430025
1,4-Dioxane, extra dry < 50 ppm, stabilized	364341000	364340010	
Ether, extra dry < 50 ppm, stabilized	364331000	364330010	
Ethyl acetate, extra dry < 50 ppm	364351000	364350010	
n-Heptane, extra dry < 50 ppm	364361000	364360010	
n-Hexane, extra dry < 50 ppm	364371000	364370010	
Isopropanol, extra dry < 50 ppm	364401000	364400010	
Methyl alcohol, extra dry < 50 ppm	364391000	364390010	
1-Methyl-2-pyrrolidinone, extra dry < 50 ppm	364381000	364380010	
Methylsulfoxide, extra dry, water < 50 ppm	348441000	348440010	348440025
Pyridine, extra dry < 50 ppm	364421000	364420010	
Tetrahydrofuran, extra dry, water < 50 ppm, stabilized	348451000	348450010	348450025
Toluene, extra dry < 50 ppm	364411000	364410010	

AcroSeal bottles are individually packaged in sealed aluminium bags for longer shelf life.

White graduations on amber bottle provide a useful way to track solvent use.

Clearly marked label identifies product, assay, water content, and physical properties.

The AcroSeal™ Septum Closure

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