LITHIUM

ANNUAL SURVEY COVERING THE YEAR 1971

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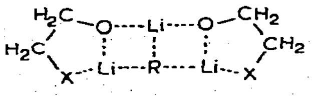
CONTENTS

I. Structure and Bonding Studies

Further evidence on the nature of benzylic lithiums has appeared. Bywater and Worsfold [1] have presented UV and PMR data on several compounds of this type in benzene and THF. The charge on the phenyl ring is estimated to be 0.29 e and 0.53 e for benzyllithium in C_6D_6 and THF-d_8 respectively, and is found to increase with increasing alkyl substitution on the side-chain. Styryl and α-methylstyryl compounds are said to be largely ionic ionic in character in both polar and non-polar solvents. On the other hand, McKeever and Waack have reiterated their position that the α-carbon atoms in 1,1-diphenylhexyllithium and benzyllithium have very little excess charge in non-polar solvents. ^1H and ^{13}C data have been interpreted by these authors to indicate a substantial degree of sp^3 character at the α-carbon atom [2, AS 69; 1]. No ^{13}C-^7Li coupling was observed in THF as low as -100°C, however. Okamoto and Yuki have reported PMR data on 1,1-diphenylbutyllithium (DPB^-) in THF/C_6H_6 mixtures [3]. Charge densities at the various ring positions were measured by the same method used in the two previous papers. Very striking is the observation that the total charge density on the ring (as measured by PMR shifts) is a minimum at $[THF]/[DPB^-] \simeq 2$, indicating some type of specific complex formation. The PMR spectrum of α-picolyllithium (I) is similar to that of benzyllithium [4]. Ring charge is somewhat greater in I, however, indicating that the carbon-lithium bond may be more ionic than in benzyllithium.

Electronic spectra of 1,1-diphenylhexyllithium and 9-fluorenyllithium are markedly effected by the addition of $CH_3OCH_2CH_2OLi$ [5]. With fluorenyllithium, the agents cause the disappearance of the contact ion pair and the formation of a "coordination-agent separated complex" RLi • $2XCH_2CH_2CH_2OLi$ [X = CH_3O, $(CH_3)_2N$], proposed to have the following structure:



The spectrum of fluorenyllithium in ethylamine has been interpreted in terms of separated ion pair formation [6].

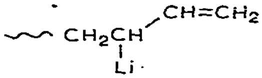
Further information on the structure of allylic lithium species has been published this past year. Burley and Young [7, AS 70; 256] have published more information pertaining to ion pairs of II:


II

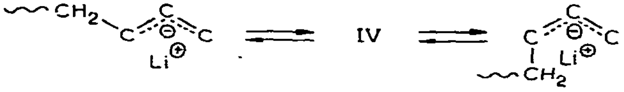
PMR data on "living" butadienyllithium has been reported by Dolinskaya et al. [8] and by Morton [9]. Both authors conclude that only the primary form III is present in hydrocarbon solvents. However, in basic solvents the secondary form IV may

$\sim\sim CH_2-CH=CH-CH_2Li$

III


$\sim\sim CH_2CH(Li)CH=CH_2$

IV

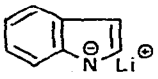

V VI

exist as an intermediate in the cis - trans isomerization of the (largely) ionic forms V and VI.

Sinn and coworkers [10] have proposed that the addition of dienes to form species such as these is exclusively cis-1,4 in all cases, and that trans-1,4 and 1,2 in-chain-units result from bimolecular isomerization processes. As proof, they cite the increase in cis-1,4 content in polybutadiene and polyisoprene as the concentration of initiator is decreased.
PMR has also been used to study the reaction products of iso-

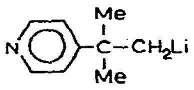
prene and 1,1-diphenylbutyllithium (DPBLi) [11]. Cis-1,4 and cis-4,1 addition products, but no trans forms, were found [AS 70; 256]. Addition of THF causes not only the isomerization of these products, but also the reversal of the reaction to isoprene and DPBLi. In another report conductivities of polydienyllithiums in THF have been used to calculate constants for dissociation into free ions [12].

Indolyllithium VII was reported to exist primarily as solvent-separated ion pairs in THF [13]. PMR spectra show that VII undergoes rapid exchange with indole above -30°C.

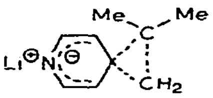


VII

Ring proton shifts for VIII are typical of pyridines and give no indication of the participation of homoconjugated species such as IX [14].



VIII



IX

Lithium-7 nmr spectra of fluorenyllithium, cyclopentadienyllithium, indenyllithium, and phenylallyllithium have been reported [15]. Variations in the ^{7}Li chemical shifts in different solvents are interpreted in terms of increasing cation-anion distance as the solvent changes in the series Et_2O, DME, THF, and HMPA. Lithium-7 shifts of thirteen dianions also were reported [16].

PMR, UV, and IR for cyclopentadienyllithium indicate that the compound has a delocalized, not a diene-type structure [17]. Fluorine-19 and proton spectra for (m- and p- fluorophenyl) cyclopentadienyl anions confirm this view, in general [18]. However, a considerable portion of the negative charge of the $C_5H_4^-$ substituent probably is present in its σ orbitals.

Lithium-7 spectra of mixtures of phenyllithium and p-tolyllithium in Et_2O at reduced temperatures have been interpreted in terms of an exchange process involving only monomeric species [19]. The ebulliometrically determined degree of

$$^{*}\mathrm{LiPh} + \mathrm{LiTol} \rightleftharpoons \mathrm{LiPh} + {}^{*}\mathrm{LiTol}$$

association of PhLi (i = 2), reported earlier, was concluded to be in error.

Infrared spectra of n-butyllithium in several ethers have been interpreted in terms of decreased C-Li bonding as compared to the case in non-donor solvents [20]. Stability constants for complexes of n-BuLi hexamers and tetramers with diethyl ether have been determined [21]. Complexes with n-$C_{12}H_{25}Li$ tetramers were also reported.

A vibrational analysis of t-BuLi based on IR and Raman data has confirmed the proposed tetrameric structure [22]. No Li-Li bonding was indicated, in agreement with previous findings based on ^7Li spectra [AS 68; 189]. The PMR spectrum of s-butyllithium has also been reported [23].

Vapor pressure lowering of benzene solutions of n-pentyllithium and n-octyllithium indicate that both compounds are predominantly hexameric over the range of concentrations studied [24]. Some further information regarding aggregates of this type has been obtained from ^7Li spin-lattice relaxation times

[25]. Temperature dependence of T_1 for several organolithiums indicate that tetrameric species have slower quadrupolar relaxation rates than do hexamers, presumably due to the greater symmetry of the tetramers. An unexpected change in the magnitude of T_1 for methyllithium in Et_2O near 0°C was attributed to some type of structural change of the aggregate.

Extended Hückel calculations for MeLi and $(MeLi)_4$ have also appeared [26]. The results are in general agreement with previous reports [AS 69; 2].

The crystal structure of $LiB(CH_3)_4$ has been reported and shows some unusual features of interest [27]. The compound contains two Li-C-B bridges, the first reported, one of which

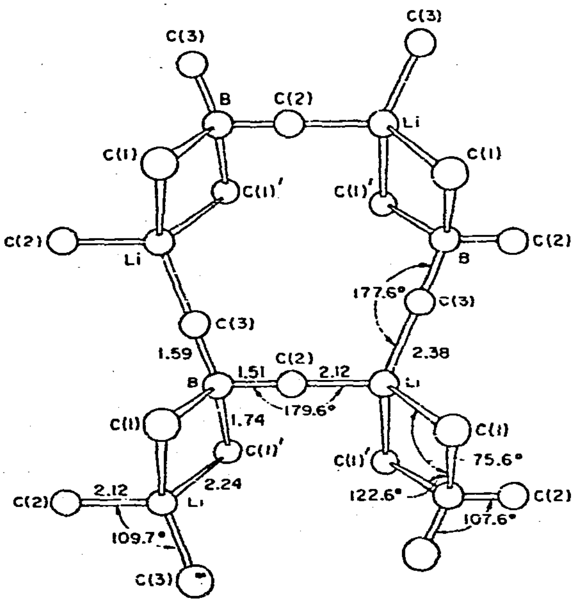


Fig. 1. Molecular structure of $LiB(CH_3)_4$. All of the atoms except C(1) and C(1)' lie in a crystallographic mirror plane which has its normal perpendicular to the plane of the paper. C(1) is related to C(1)' by the mirror plane.

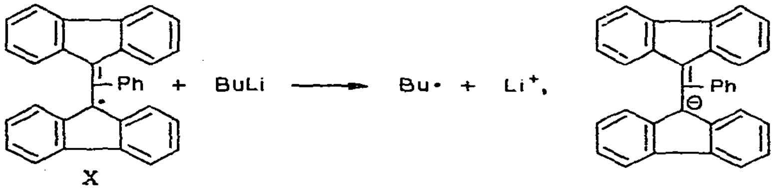
is bent (Li-C(1)-B angle 75.6°) the other nearly linear (Li-C(2)-B angle 179.6°). The B-C and Li-C distances are very short in the nearly linear bridge bond, suggesting a strong linkage.

II. Kinetics and Mechanisms

Reviews have appeared this year concerning the kinetics and mechanisms of anionic polymerizations [28,29], acidities of carbon acids [30], electron transfer reactions involving radical anions and alkyl halides [31], radical anions as intermediates in organic chemistry [32], and chemically induced dynamic nuclear polarization [33,34,35]. Some reference to organolithium chemistry is contained in each paper.

Further kinetic data on the addition of organolithium reagents to 1,1-diphenylethylene in Et_2O has appeared [36, AS 69; 4]. Relative reactivities of the lithium reagents vary with concentration due to differences in the kinetic order with respect to RLi. Uraneck has discussed the effect of temperature on the microstructure of alkyllithium-initiated polybutadiene [37]. The composite mechanism of Morton [9] was used by the author to explain the observed variations. Selman [38] has measured relative rates of polymerization of isoprene and styrene initiated by s-butyllithium and menthyllithium. The latter is an extremely rapid initiator [AS 69;8] and the usual induction period in cyclohexane is not observed. Kinetic studies of the rate of propagation of isoprene by polyisoprenyllithium has been shown to be first and 0.25 order respectively in cyclohexane [39]. The accelerating effect of 2,3-dimethoxybutane has also been reported [40].

Free radical chain processes have been demonstrated for the reactions of 2-lithio-2-nitropropane with 2-bromo-2-nitropropane and with 2,2-dinitropropane in the presence of light [41]. Several similar systems were also investigated. Screttas [42] has reported the reaction of n-BuLi with the stable free radicals 1,3-bis(biphenyl-2,2'-diyl)-2-phenylallyl X and triphenylmethyl. The radicals are converted to their corresponding carbanions, which processes are accelerated by the presence of Lewis bases.



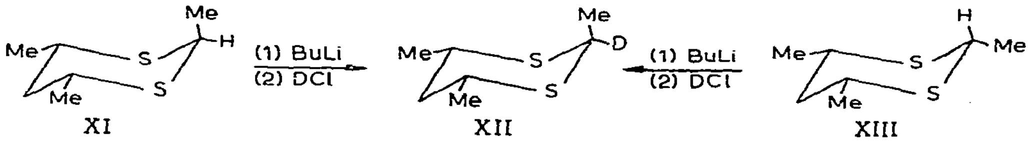
X

The kinetics of the addition of phenyllithium to benzonitrile in Et_2O is 0.67 order in PhLi [43]. A Hammett plot of rate constants for several m- and p-substituted phenyllithiums showed a very low effect of polar substituents (p = -0.035, irregular). A concerted electrophilic substitution mechanism of the type shown below was postulated:

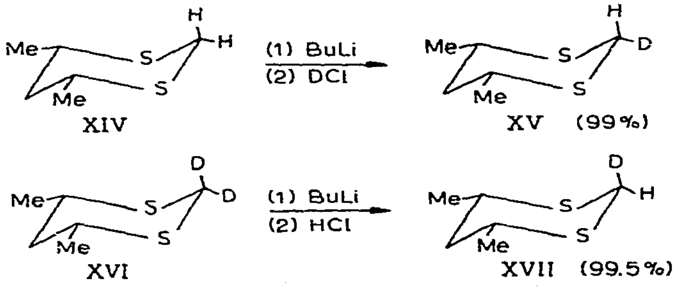
Batalov and coworkers continue to report their finding on the exchange reaction between phenyllithium and bromobenzene [AS 70; 264]. Tetraalkylammonium halides accelerate the exchange process [44] while lithium halides depress the rate [45,46]. An electron donating group (p-Me) in the aryllithium was shown to increase the exchange rate while p-Cl substitution decreased the rate [47]. Substituted bromobenzenes were also used to investigate polar effects, and a polar four center transition state for the exchange process has been postulated [48].

The reaction of n-BuLi with THF in hexane is first order in BuLi and 2.5 order in THF according to a recent report [49]. No particular mechanism was proposed, however. It should be noted, however, that other workers have pointed out secondary reactions which occur during the decomposition of THF and Et_2O by n-BuLi [50]. Multiple addition of ethylene yields higher alkyllithium compounds which undoubtedly complicates the interpretation of kinetic results.

Lithium derivatives of 1,3-dithianes have produced some interesting stereochemistry [51] . Lithiation of XI (fast), followed by reaction with DCl yields only XII, whereas lithiation of XIII (slow) yields the same product.

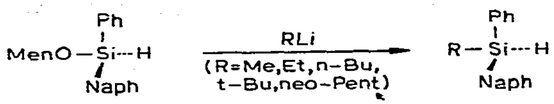


Lithiation of XIV followed by reaction with DCl yields only the equatorially deuterated product XV, while the lithium derivative

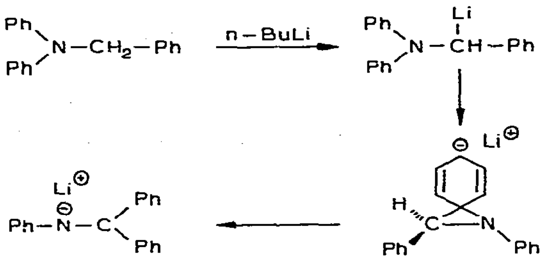


of XVI yields only the equitorially protonated product XVII. Thus, lithiation and protonation of these lithium derivatives are highly stereoselective processes [cf. reference 85 below].

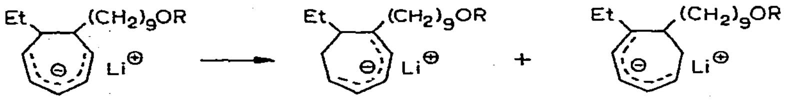
A series of chiral organosilanes has been prepared by the reaction of XVIII (Men = menthyl; Naph = α-naphthyl) with various RLi and RMgX compounds [52, AS 70; 387].



Anionic rearrangements [53] have been the subject of some recent work in which the bridging intermediate XIX was indicated [54]. Further information on the occurrence of 1,6-hydrogen

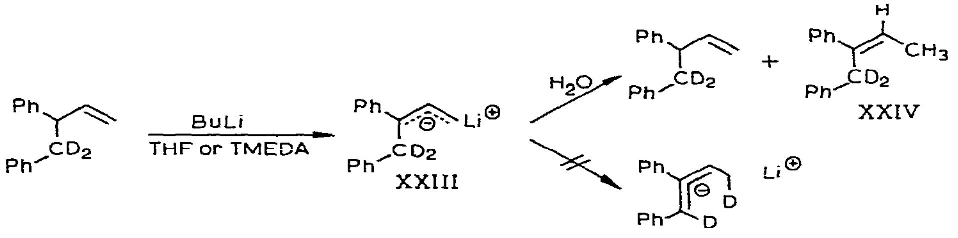


shifts in pentadienyl anions has also been reported [55, AS 70; 264]. Metalation of linolenyl alcohol gives heptatrienyllithium compounds which cyclize on standing. Rearrangements such as that of XX to XXI and XXII, which are forbidden by orbital

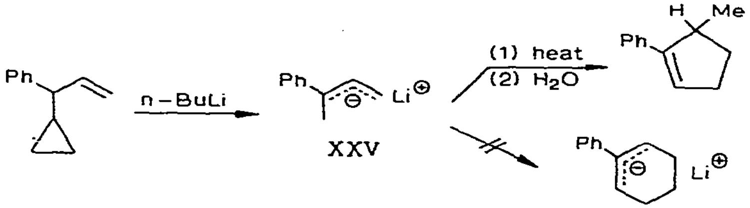


XX XXI XXII

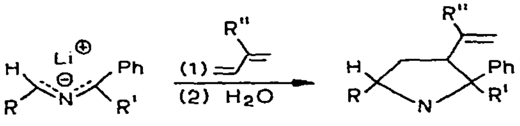
correlation rules, nevertheless do occur. The authors feel that the participation of the lithium atom may cause the migration process to be allowed. Lithiation of the monomethylamide of linoleic acid was observed at the doublic allylic position and/or the site α to the amide group [56]. Long exposure also resulted in 1,6-sigmatropic migrations. Sigmatropic [1,4] shifts have been searched for using the allylic lithium compound XXIII [57].



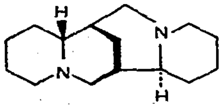
However, analysis of XXIV showed no evidence for deuterium scrambling which would be required by a [1,4]-hydrogen shift. Similarly, no [1-4]-carbon shift was observed from XXV.



1,3-Cycloaddition reactions of 2-azaallyllithium to dienes have been reported to occur in moderate to high yields [58].

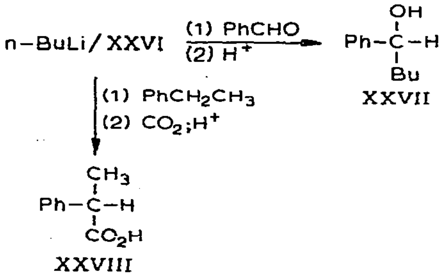


Asymmetric synthesis by means of alkyllithiums (or Grignards) complexed with (−)-sparteine XXVI has been reported [59].

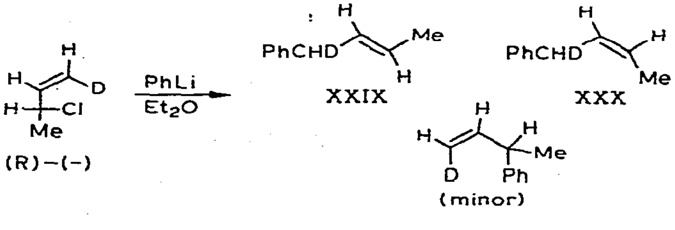


XXVI

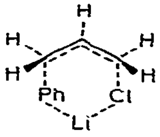
For example, reaction of n-BuLi - XXVI with benzaldehyde yields alcohol XXVII with 6% optical purity, while lithiation of ethyl benzene followed by carbonation gave XXVIII with 30% optical purity.



More details on the reaction of phenyllithium with allylic chlorides have appeared [60,61, AS 70; 287]. γ-Coupling with optically active 3-chloro-cis-1-butene-1-d yields two products



XXIX and XXX with optical purities indicative of a stereospecific syn attack as shown in XXXI.

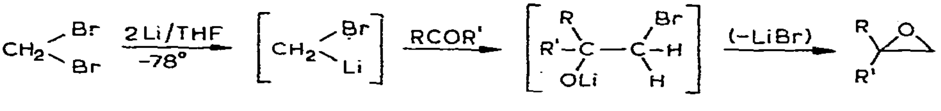


XXXI

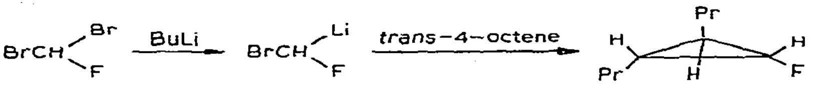
The mechanism of the reaction of organolithium reagents with triarylsulfonium salts has been discussed by Trost [62]. Benzyne, radicaloid, and direct nucleophilic aromatic substitution processes are rejected in favor of a mechanism involving a sulfurane intermediate.

III. Lithium Carbenoids and Other Halogen-Substituted Organolithiums

Metal carbenoids, including those of lithium, have been the subject of a recent review [63]. The solvent system THF/Me_2O has been used for the preparation of 1,1-dichloro-2,2,2-trifluoroethyllithium and 1,1-dichloro-2-X-2,2-(ethylenedioxy)-ethyllithium [X = Ph, H, and Me] [64,65, AS 69; 18]. Various derivatives of the latter are described. Reactions of $LiCH_2Br$, prepared <u>in situ</u>, with carbonyl compounds have been shown to yield epoxides, whereas α,β-unsaturated carbonyl compounds do not react [66]. The yields with $LiCH_2Cl$ are somewhat lower.

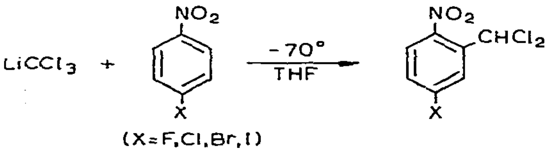


Schlosser and coworkers have described the preparation of some cyclopropanes derived from various lithium fluorocarbenoids [67]. Reaction of LiCHBrF (or :CHF) with olefins is stereospecific with respect to olefin configuration and stereoselective with preference to the *syn*-fluorocyclopropane [68]:

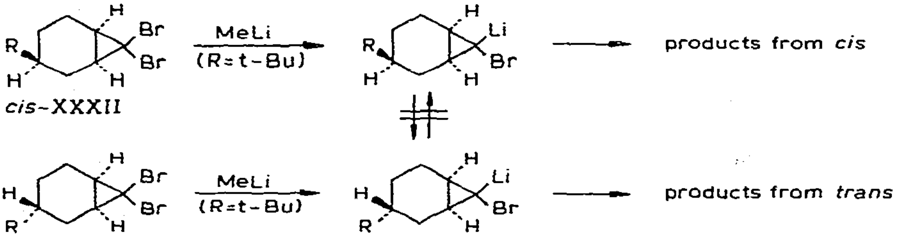


The system is quite complex, however, and low yields of the fluorocyclopropanes are the result.

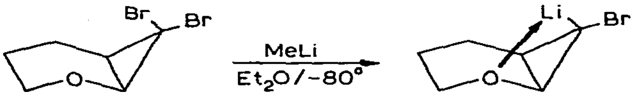
The reaction of $LiCCl_3$ with 4-halonitrobenzenes yields α,α-dichloro-5-halo-2-nitrotoluenes in quite good yields [69].



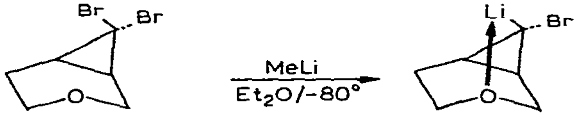
Carbenoids derived from *gem*-dibromocyclopropanes, *cis*- and *trans*-XXXII, yield different products indicating that no



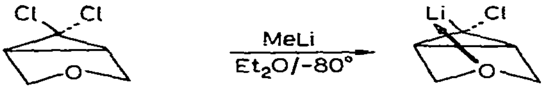
crossover occurs (e.g., through 1,2-cycloheptadiene). Carbenoids prepared from compounds such as XXXIII and XXXIV are stabilized by the neighboring oxygen and therefore are useful as nucleophilic reagents [71, AS 68, 206]:



XXXIII

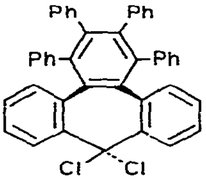


XXXIV

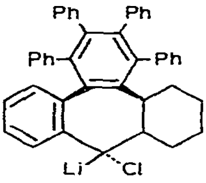


XXXV

The dichlorocyclopropane XXXV also yielded a relatively stable carbenoid, but in low yield. The dichloride XXXVI leads with high preference to the carbenoid with a quasiaxial C-Li bond [72].



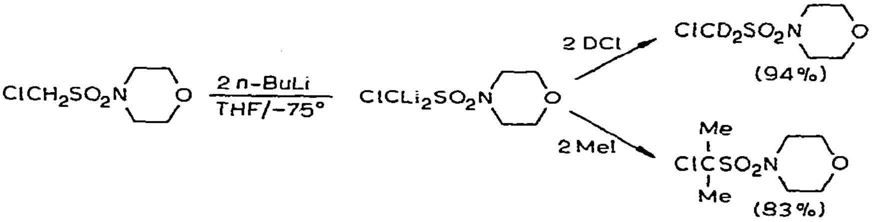
XXXVI



XXXVII

One is tempted to suggest interaction of the lithium atom in XXXVII with the π electrons of the neighboring benzene rings.

The first 1-chloro-1,1-dilithium compound has been reported [73].

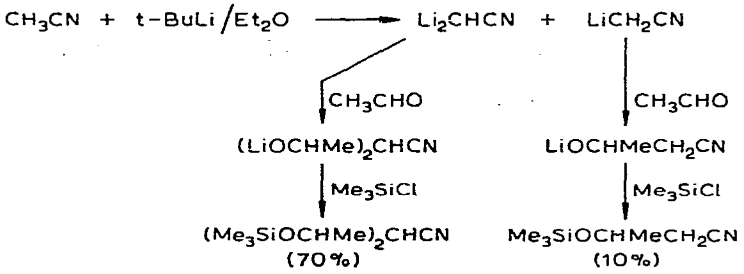


Other gem-dilithium compounds have been reported by Kaiser *et al.* [74].

$$PhCH_2CN \xrightarrow[\text{THF/hexane}\ 25°]{2.3\ n\text{-}BuLi} PhCLi_2CN \xrightarrow{D_2O} \underset{(90\%)}{PhCD_2CN}$$

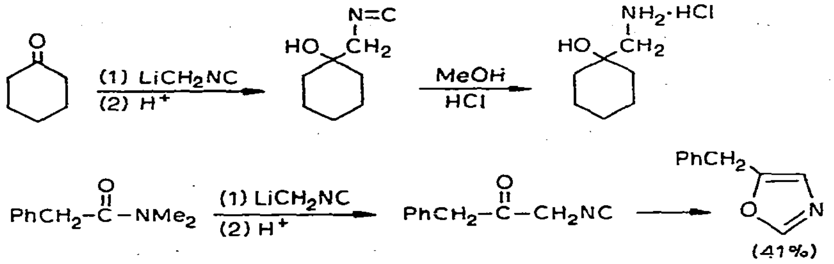
$$PhSO_2CH_2Ph \xrightarrow[\text{THF/hexane}\ 25°]{2.5\ n\text{-}BuLi} PhSO_2CLi_2Ph \xrightarrow{D_2O} \underset{(94\%)}{PhSO_2CD_2Ph}$$

Polylithiation of acetonitrile [75] produces the corresponding dilithio derivative (no evidence for Li_3CCN).

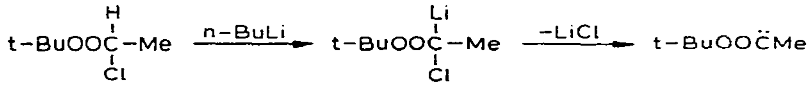


The formation of 5% $(Me_3SiOCHMe)_3CN$ from the system was attributed to metalation of either $(LiOCHMe)_2CHCN$ or $(LiOCHMe)LiC_2HN$ during derivatization. The infrared spectra of metalated nitriles were also investigated and the principal species were identified.

Further use of isocyanomethyllithium for synthetic purposes has been reported [AS 70; 272]. Cyclic ketones with $LiCH_2NC$ yield aminomethylcyclohexanoles [76] and acyl halides or amides yield 2-unsubstituted oxazoles [77]. Two examples are given below:



α-Lithiated isocyano-acetic and -propionic esters may also be utilized in these reactions. Lithiated carboxylic acids are also useful intermediates, as evidenced by their use in the preparation of α-hydroxycarboxylic acids by aeration [78], and in the preparation of β-hydroxycarboxylic acids by condensation with carbonyl compounds [79]. The stereochemistry of the reaction of dilithiopropionate to a cyclic ketone (dehydroisoandrosterone) has also been reported [80]. The intermediacy of the α-lithio derivative XXXVIII from the reaction of BuLi with α-chloroethyl tertiary butyl peroxide has been suggested [81]:

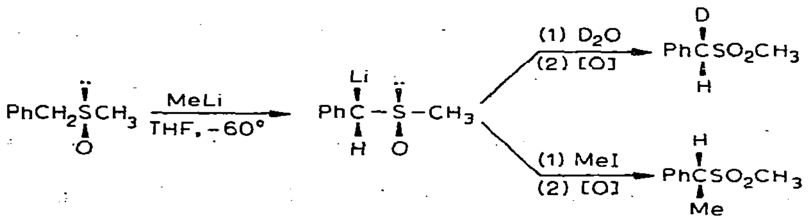


XXXVIII

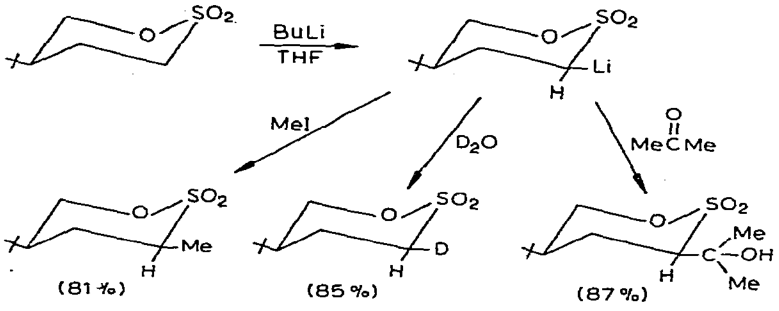
$$t\text{-BuOOC}(H)(Cl)\text{-Me} \xrightarrow{n\text{-BuLi}} t\text{-BuOOC}(Li)(Cl)\text{-Me} \xrightarrow{-LiCl} t\text{-BuOO}\ddot{C}\text{Me}$$

Lithium N,N-dimethylacetamide has also been utilized for the synthesis of tryptamines [82].

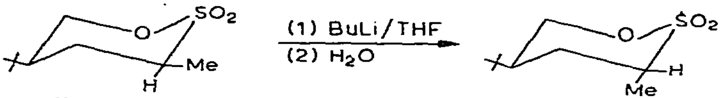
The stereochemical course of reactions of α-lithio sulfoxides with D_2O, MeI, and ketones has been established.

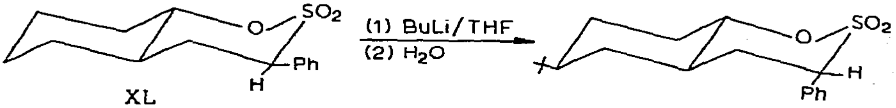


Assuming that deuteriation proceeds with retention, then reaction with MeI must occur with inversion of configuration [83]. Reaction with ketones occurs with retention [84]. α-Lithio-δ-sultones have also been studied in the same laboratory [85; cf reference 51 above]. There apparently is a strong preference for lithium in the equatorial position as evidenced by the formation of only equatorially-substituted derivatives.



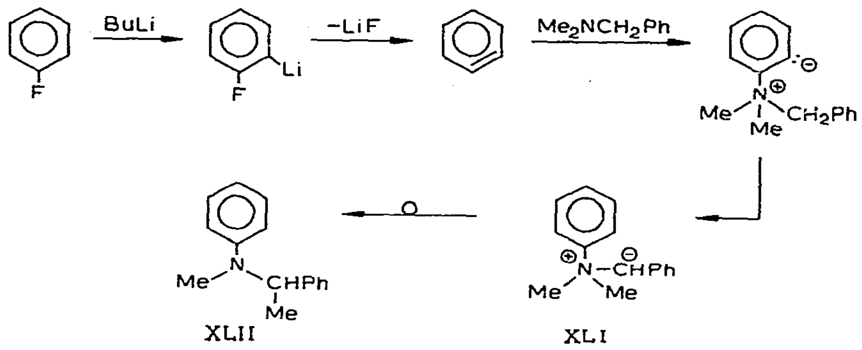
Moreover, XXXIX and XL rapidly rearrange to the corresponding axial isomers upon brief treatment by BuLi/THF.


XXXIX


XL

The author estimates that lithium in the equatorial position of XL is more stable by approximately 2.7 Kcal, which is appreciably more than that found for cyclohexyllithium compounds.

Halophenyllithium compounds continue to be used as a source for benzyne, as, for example, in the addition to N,N-dimethylbenzylamine [86].

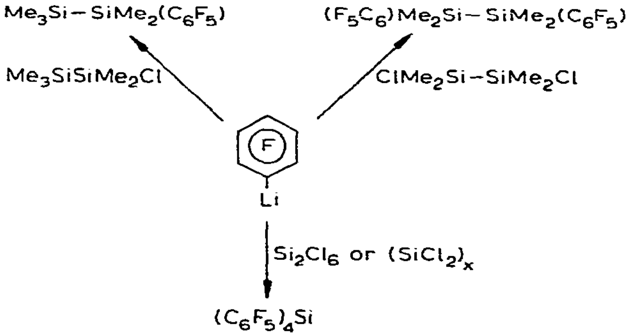


The rearrangement of XLI to the major product XLII occurs via a radical pair process.

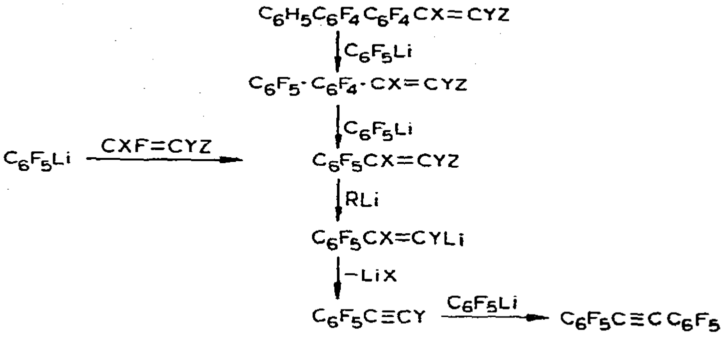
The decomposition of o-halophenyllithium has been investigated by thermal analysis [87] and NMR methods [88]. These workers consider lithium halide elimination to be a synchronous process rather than a two step ionic process.

The explosive tendency of polyhalophenyllithium compounds should be noted by all workers in this area [89,90]!

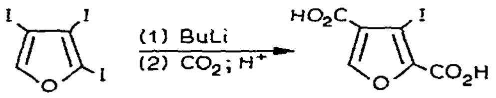
Coupling of pentafluorophenyllithium with two methylchlorodisilanes and with perhalo-di- and poly-silanes has been reported [91]:



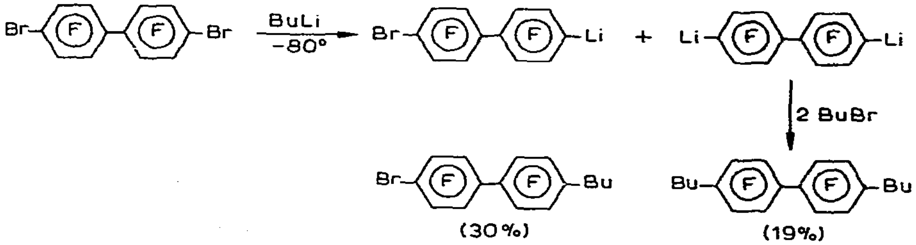
With halogenated olefins, C_6F_5Li gives products corresponding to pentafluorophenyl-fluorine exchange, sometimes followed by metalation and elimination to the corresponding acetylene [92]:



Various adducts of tetrachlorobenzyne, prepared from pentachlorophenyllithium, have been reported [93]. More details on the reaction of C_6Cl_5Li with nitriles have also appeared [94, AS 70; 21]. Also reported were procedures for the preparation of trichloro-2-thienyllithium [95] and various 2-substituted silane derivatives. The reaction of 2,3,4-triiodofuran with excess BuLi yields the dilithiated species [96]:

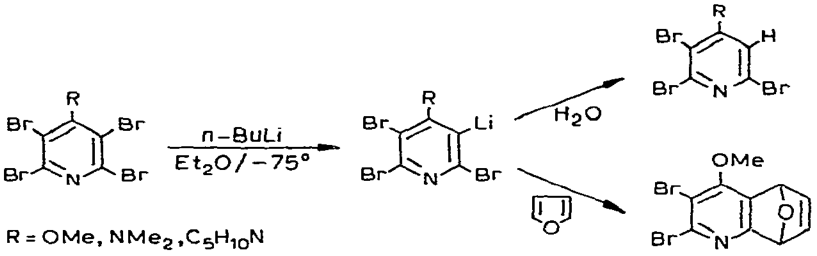


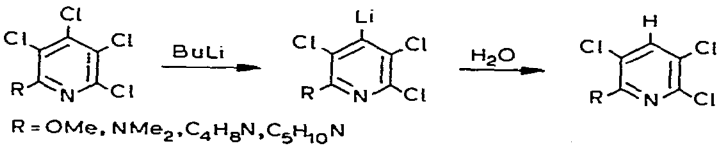
Lithiation of 4,4'-dibromooctafluorobiphenyl leads to a variety of products depending on the reaction temperature [97, AS 70, 274].



Furan adducts were formed which revealed the presence of several benzyme intermediates.

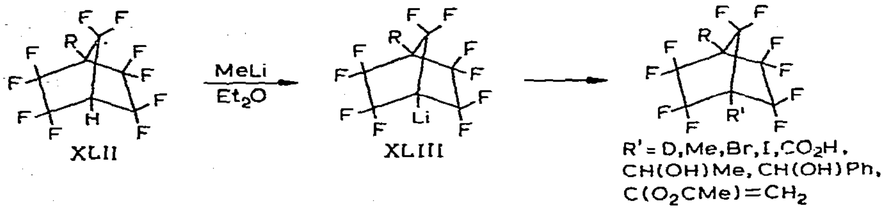
Tetrabromo-4-pyridyl derivatives react with BuLi in similar fashion [98]. However, only the 4-methoxy compound yielded a furan adduct. Tetrachloro-2-pyridyl derivatives also gave products characteristic of halogen-lithium exchange. The authors have summarized the results of several studies of this type, pointing out the factors which determine the site of halogen-lithium exchange [AS 69, 22].



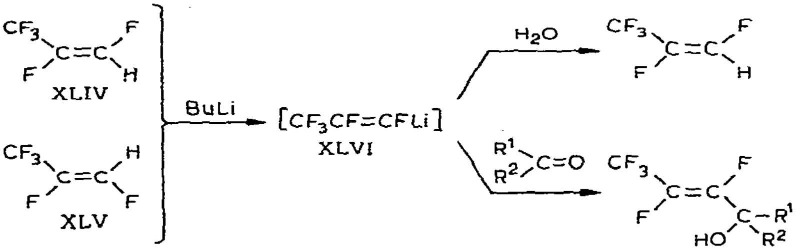


4-Lithiotetrachloropyridine has been derivatized to the 4-trimethylsilyl derivative [99]. With excess t-BuLi, pentachloropyridine yields, after reaction with Me_3SiCl, a tris(trimethylsilyl)-derivative.

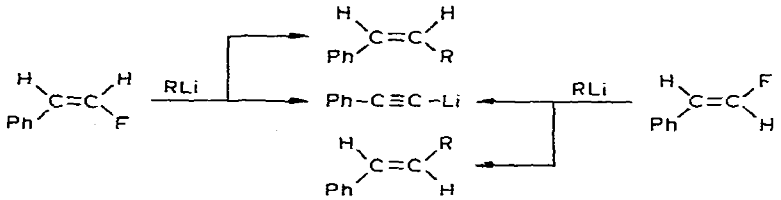
Lithiation of the fluorinated hydrocarbon XLIII (R = F) has been used to prepare a number of derivatives [100]. Similar derivatives of XLII (R = H) were reported in another paper [101]. Decomposition of the lithium derivatives XLIII has been proposed to occur via a transient bridgehead radical.



Pentafluorylpropenyllithium has been prepared by the reaction of a 1:1 mixture of XLIV and XLV with BuLi [102]:



The yields of the carbinols were greater than 50% indicating loss of stereochemical identity of XLVI during the reaction. The reaction of <u>cis</u>- and <u>trans</u>-styrylfluoride yields a mixture of substitution and elimination products [103]. The olefins are

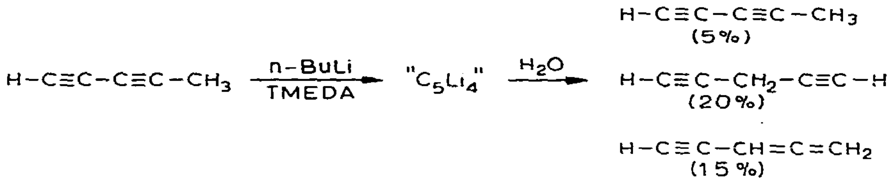


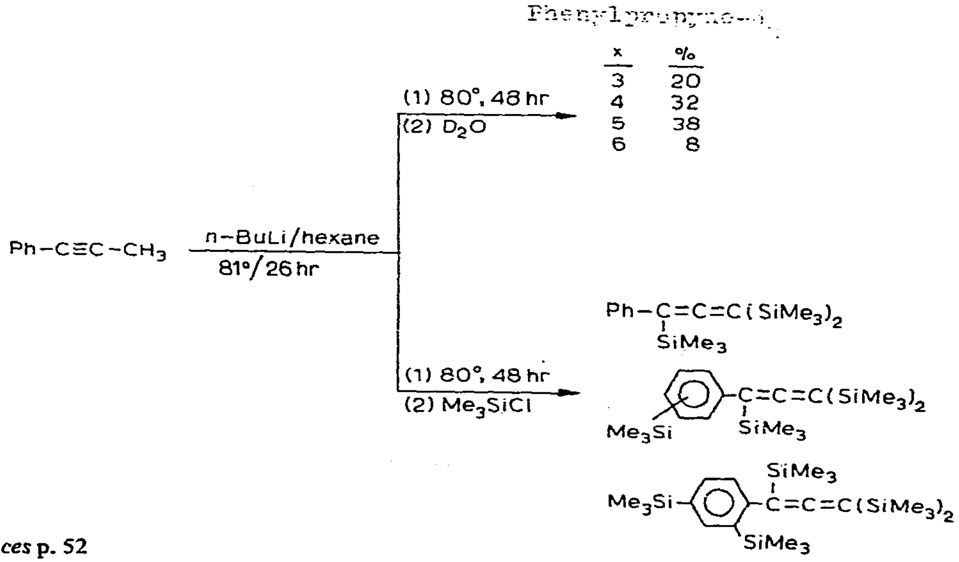
formed with retention of configuration. A large α-hydrogen isotope effect for elimination indicates this to be an E_2cb process, although a direct E_2 process cannot be ruled out for the <u>trans</u>-isomer

IV. <u>Lithiations (Hydrogen-Lithium Exchange Reactions)</u>

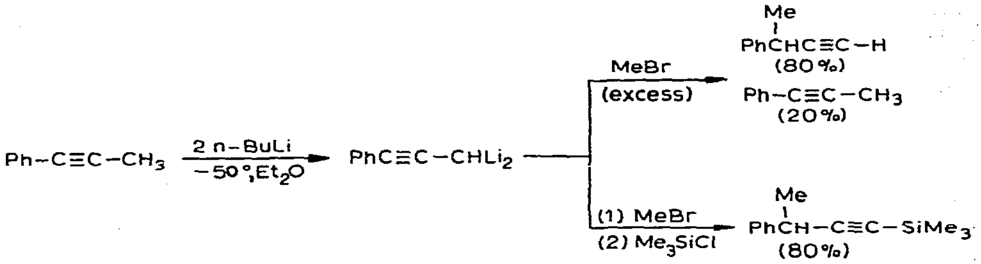
Several groups have continued to exploit the metalation of organic materials by organolithium compounds, particularly when activated by diamines and other complexing agents. Halasa [104] has examined the polylithiated products from anthracene, biphenyl, fluorene, and indene prepared from n-BuLi • TMEDA. The polylithiated products were examined by reaction with D_2O and Me_3SiCl. As many as ten lithium atoms per (fluorene or anthracene) molecule were indicated.

R. West and coworkers also have contributed to this area, most recently by the study of polylithiated phenylpropynes [105] and penta-1,3-diyne [106]. C_5Li_4, obtained from the diyne, is the second reported perlithium compound. Quenching with water yields the three C_5H_4 isomer shown below; with D_2O the same types of derivatives are formed with at least 66% of the species having four deuterium atoms. With 1-phenylpropyne, one obtains $C_9Li_6H_2$ and $C_9Li_5H_3$ species as the major products. No TMEDA

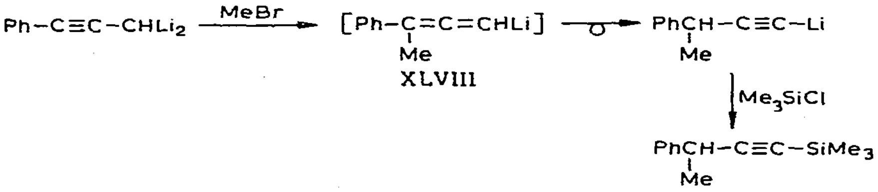




or other complexing agent is required. Although the rate of metalation of the propyne is accelerated with such agents, the yield of polylithiated species is decreased due to metalation of the agent. Klein and Brenner [107] and Balyan et al. [180] have investigated this system under somewhat milder conditions [AS 70, 279]. The former have shown that the dilithio derivative of 1-phenylpropyne reacts with one mole of methyl bromide rapidly, but that reaction with a second mole is slow enough so that Me_3SiCl may be used to trap the half-methylated product [107]:

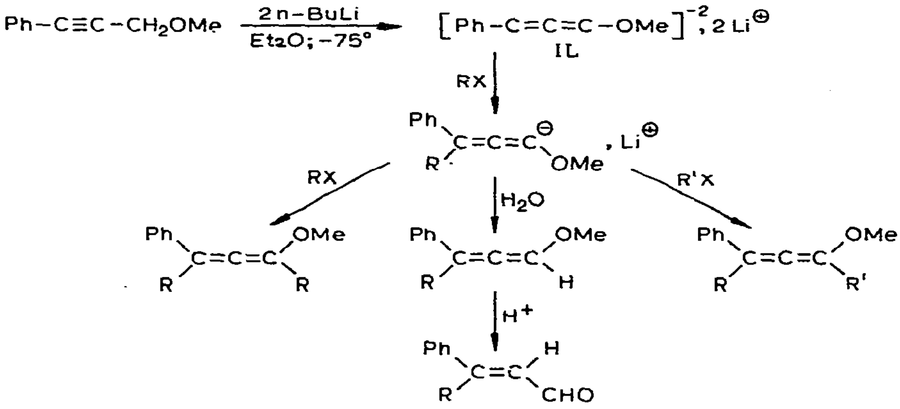


The authors conclude that methylation occurs at the benzylic position followed by rearrangement of the mono-methylated lithiopropyne XLVIII by hydrogen migration:



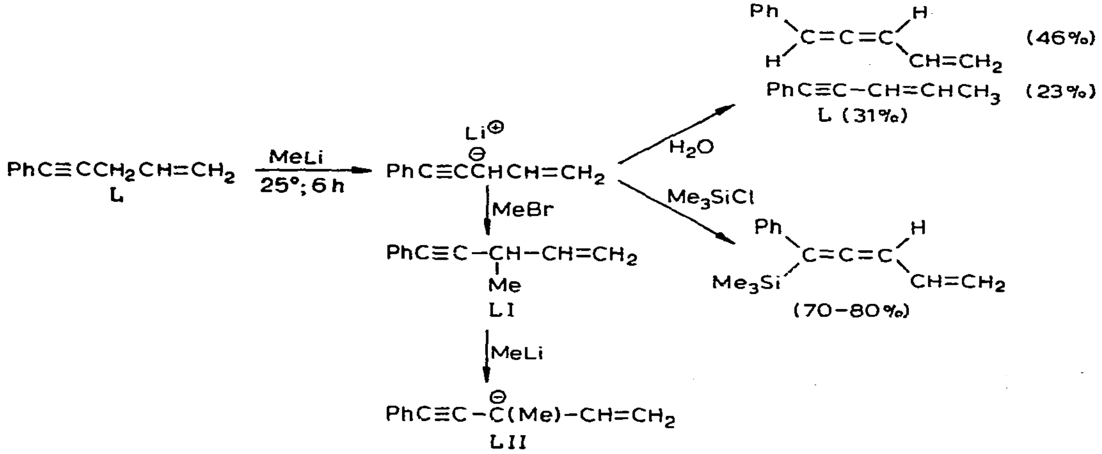
The present data regarding the nature of the lithiated species in this system, whether allenic or acetylenic, is incomplete and somewhat confusing to this reviewer.

The lithiation of 3-methoxy-1-phenyl-1-propyne under conditions similar to those used by Klein and Brenner yields allene derivatives as the major products [109]. The dilithiated compound IL, which the authors write as an allenic derivative, may be sequentially alkylated as shown below:



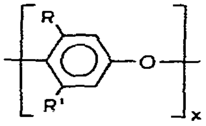
Yields of the allenes (R = Me, SiMe$_3$; R' = Me, SiMe$_3$) were reported between 70 and 82%. α-Ethylenic aldehydes and ketones were prepared in high yield by acid hydrolysis of this monoalkylated product.

Metalation of enynes has also been reported from two laboratories. Klein and coworkers [110, AS 69, 26] have used MeLi to monometalate a series of enynes (BuLi quickly dimetalates the same substrates). The major products from protolysis



and silylation were allenes, whereas methyl bromide gave only the unconjugated enyne LI. The monometalated derivative of LI was also examined by PMR; its spectrum was consistent with structure LII, although substantial delocalization of charge was indicated by the chemical shifts of the olefin and aromatic protons. Also reported was the metalation of MeC≡C-CH=CHMe and EtC≡C-CH=CHMe [111].

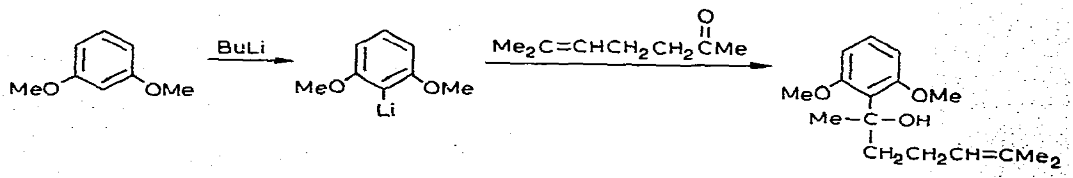
Lithiation of polybutadiene and polyisoprene were reported to be smooth, efficient processes if n-BuLi/TMEDA was used [112]. Poly(2-methyl-6-phenyl-1,4-phenylene ether) [113] and poly(2,6-diphenyl-1,4-phenylene ether) [114] lithiate more easily in THF and no chain scission takes place in the process in contrast to lithiation of dienes. Very interesting is the report [113] that LIII (R=R'=Me) apparently metalates on a ring



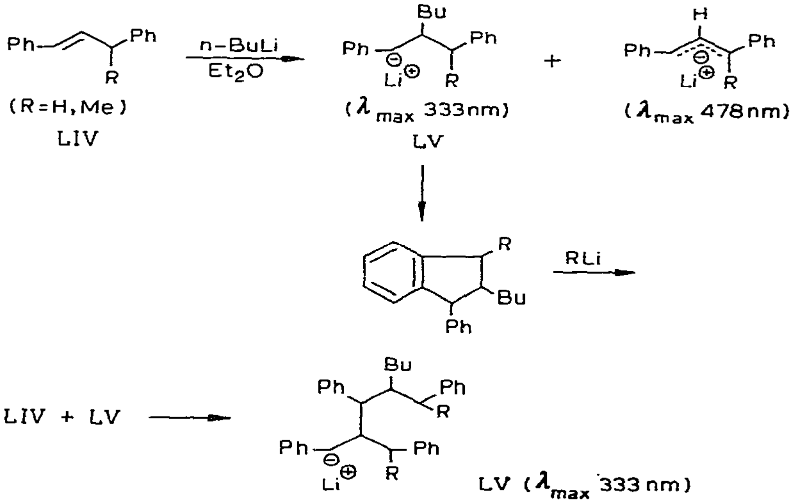
R = Me,Ph; R'=Me,Ph
LIII

position initially, but isomerizes to side-chain metalation with time.

Lithiation of a series of methoxy-substituted benzenes, followed by condensation with $Me_2C=CHCH_2CH_2COMe$, has been used for the synthesis of some aromatic derivatives [115].



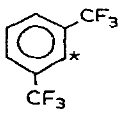
Reaction of the olefins LIV with n-BuLi in THF [116] yields exclusively the mono-lithiated products whose spectra have been reported earlier. In Et_2O, however, addition is a significant side reaction as shown below:

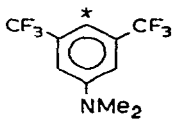


Also reported was the lithiation of some benzyl amines [117]. The intermediate dilithioamines LVI were derivatized with several ketones.

$$\underset{(R=Me,Ph)}{PhCH_2NHR} \xrightarrow{n\text{-BuLi/TMEDA}} \underset{LVI}{PhCHLiNHLi}$$

Lithiation of LVII was reported to occur only at the position between the substituents(*) if n-BuLi/TMEDA was used [118].


LVI


LVII

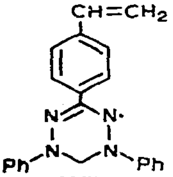
With n-BuLi/Et_2O a mixture of products (unresolved) results. LVII with n-BuLi/Et_2O gave mainly the 4-lithiated product. Also reported [119] were lithiations of some ring methoxylated N,N-dimethyl(naphthylmethyl) amines.

V. Polymerization Reactions

Two reviews have appeared on polymerizations initiated by organolithium compound [120, 121].

Copolymerization of 1,3-butadiene and o- and p-chlorostyrenes with n-BuLi initiator has been studied by Halasa and coworkers [122]. Unlike the unsubstituted case, these styrenes have reactivities approximately equal to that of butadiene and are incorporated into the polymer chain with uniform distribution.

Polymerization of the stable radical LVIII with n-BuLi or NaNaph yields a paramagnetic polymer of low molecular weight [123].



LVIII

Also reported is the copolymerization of p-anisaldehyde and dimethyl ketene with benzophenone-Li adduct [124] in which it was shown that ketene addition takes place across the C=C bond. In the polymerization of optically active 2-[(S)-1-methylpropyl)-1,3-butadiene with n-BuLi in n-heptane [125], the specific rotation of the polymer was of the same order of magnitude as that of the monomer.

The reaction of alkyllithium compounds with carbon black, followed by addition of styrene or isoprene, results in the grafting of polymers to the carbon black surface [126]. The method is superior to reaction of "living" polymers with ester functionalities which may be introduced on the carbon surface.

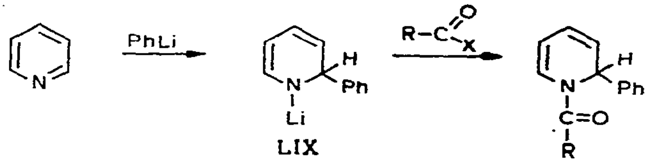
BuLi-initiated polymerization of 2,3-dimethylbutadiene in THF yields increasing amounts of 1,2-addition as the temperature is lowered. A steric argument was used to rationalize the observations [127]. Similarly, the ratio of cis-1,4 to 3,4-addition of isoprene in benzene was found to decrease from ∿ 200 to near zero as the temperature was increased [128] and also as the ionic character of the aryllithium initiator was increased [129].

Isoprene was homo- and copolymerized with oligomeric dilithium initiators produced from lithium metal and a diene [130] or styrene [131].

Other reports have appeared concerning the polymerization of the following monomers with lithium initiators: (2-methoxy)-ethyl methacrylate and (2-dimethylamino)ethyl methacrylate [132]; methyl methacrylate [133]; methyl α-phenylacrylate [134, 135]; 3-methyl-but-3-ene-2-one [136]; cyclic sulfides [137, 138]; phenyl and methyl vinyl sulfones [139]; and styrene [140, 141, 142]. The last report describes the effect of impurities (presumably oxygen, carbon dioxide, and water) on polymerization rates of polystyryllithium.

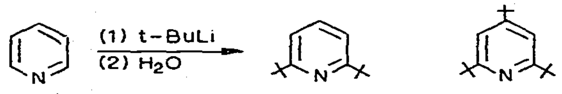
VI. Reactions with Heterocyclic Compounds

A new procedure for the preparation of N-substituted-2-phenyl-1,2-dihydropyridines has been developed which involves the reaction of 1-lithio-2-phenyl-1,2-dihydropyridine LIX with acid halides [143, AS 70, 289]:

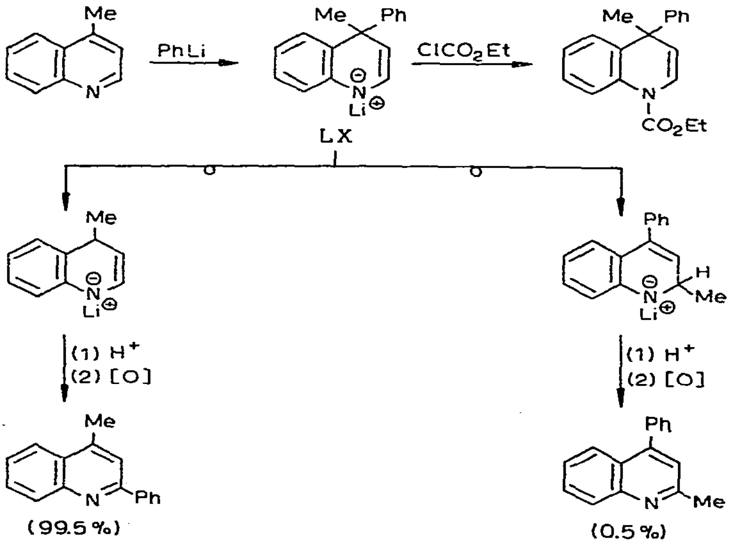


The 5-C-substituted derivative is also formed, and with R = CF_3 becomes the major product.

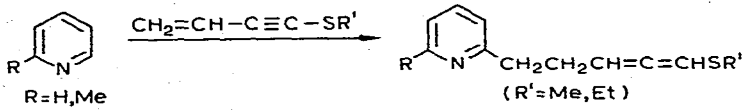
2,6-Di-t-butyl- and 2,4,6-tri-t-butylpyridine were prepared by direct alkylation by pyridine [144]:



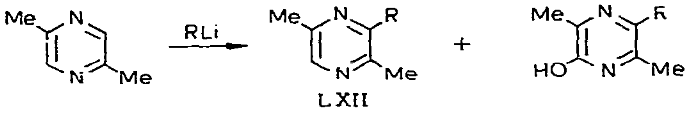
The mechanism of the reaction of quinolines with organolithium has been reexamined [145]. The initial product from PhLi with lepidine was proposed to be 1-lithio-4-methyl-4-phenyl-1,4-dihydroquinoline (LX) which may rearrange by phenyl or methyl migration:



Also prepared were adducts of α-picolyl- and 2,6-lutidyllithium wigh alkylthiovinylacetylenes [146] to yield allenic thio esters:

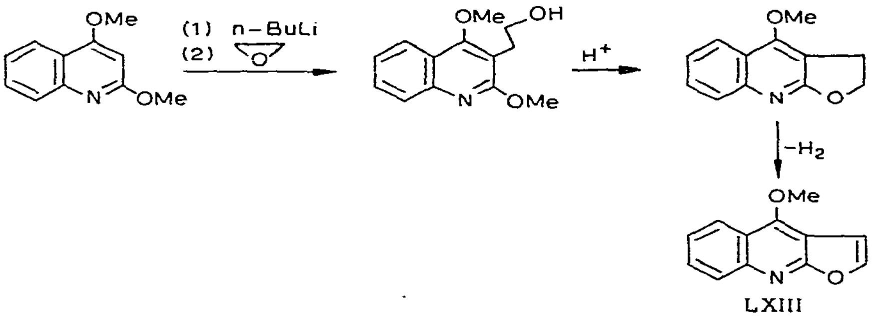


Pyrazine, methylpyrazine, and 2,5-dimethylpyrazine react with MeLi, EtLi, and 2-methylbutyllithium [147]. Some dialkylated and hydroxylated products were also found, the latter probably due to lithiation and subsequent oxidation of the major product LXII.

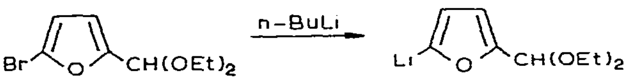


1- and 2-alkylindazoles have been shown to metalate primarily in the 1- and 3-positions respectively [148, 149].

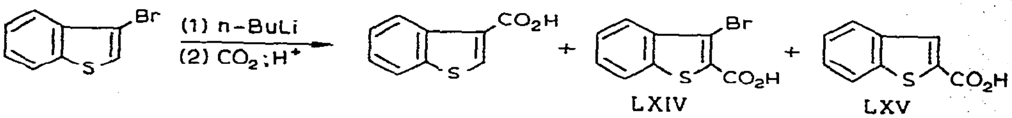
Furoquinoline alkaloids have been prepared by a new route involving lithiated intermediates [150]. The route to dictamnine LXIII is shown as an example.



Halogen-lithium exchange has been utilized to prepare the 5-lithio derivatives of some furfural acetals [151]. These were subsequently carbonated and also used to prepare the corresponding cadmium reagents with $CdCl_2$.

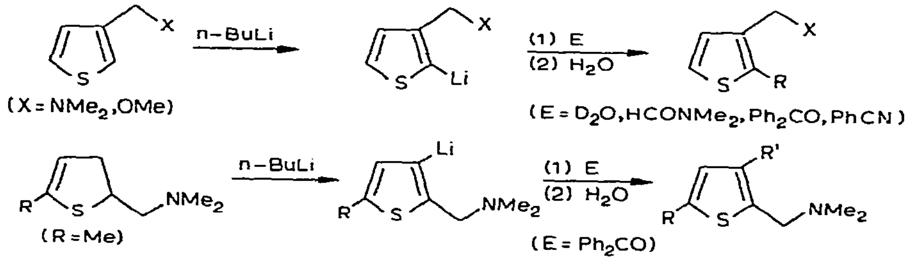


More information on the stability of 3-benzo[b]thienyllithium has appeared [152, AS 70; 290]. In THF, 3-bromobenzo[b]thiophene when treated with n-BuLi undergoes lithiation by hydrogen as well as halogen exchange reactions.

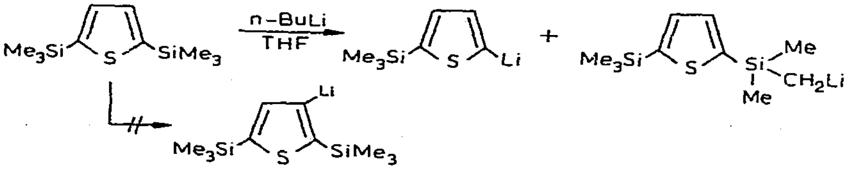


The lithium precursor of compound LXV presumably comes from a halogen-lithium exchange reaction between the lithium precursor of LXIV with 3-benzo[b]thiophene. Yields of methylation reactions also were compared in ether and THF in this paper.

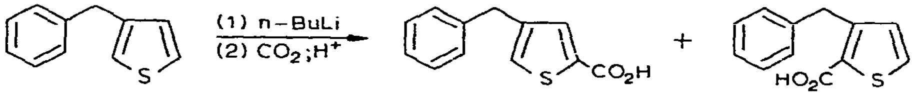
New routes to 2,3-disubstituted and 2,3,5-trisubstituted thiophenes have been revealed by Slocum and Gierer [153] by directed metalation reactions.



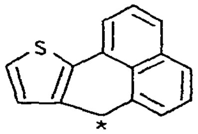
Reaction of 2,5-bis(trimethylsilyl)thiophene with n-BuLi/THF does not yield the expected 3-lithio derivative; rather, cleavage of $-SiMe_3$ groups and metalation are the result [154]:



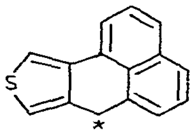
Metalation of 3-benzylthiophene occurs on the thiophene ring rather than at the benzylic position or on the benzene ring [155]:



Also reported by the same group was the synthesis and metalation of two phenalenothiophenes LXVI-LXVII [156]. Each metalated exclusively at the benzylic position (*).

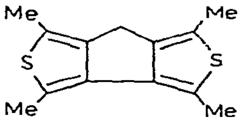


LXVI

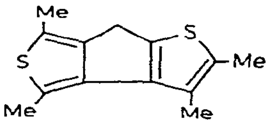


LXVII

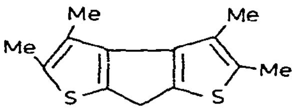
Competitive metalations of LXVIII-LXX with EtLi have been reported by Gronowidz [157]. The relative rates of metalation



LXVIII



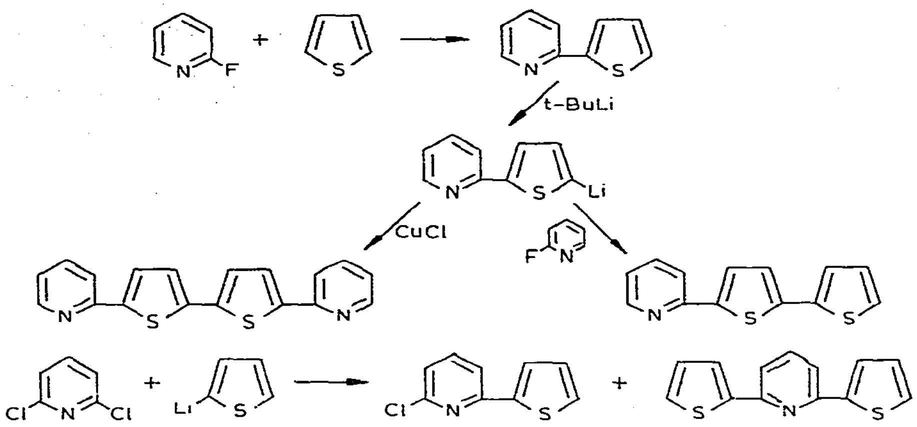
LXIX



LXX

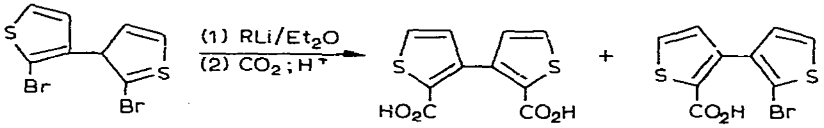
at the bridging methylene were found to be: LXVIII (1); LXIX (16.8); fluorene (371); 3-methylfluorene (754); LXX (1360).

Several thiophene-pyridine compounds were prepared by Kauffmann and associates using various organolithiums en route [158]. Some examples are shown below:



Similar couplings between thiophene and quinoline ring systems were also reported [159].

A rather striking difference between EtLi and n-BuLi has been demonstrated in their reactions with some dibromo-3,3'-bithienyls [160]. EtLi gave 55% LXXI and 45% LXXII while n-BuLi gave only 10% LXXI and 90% LXXII.

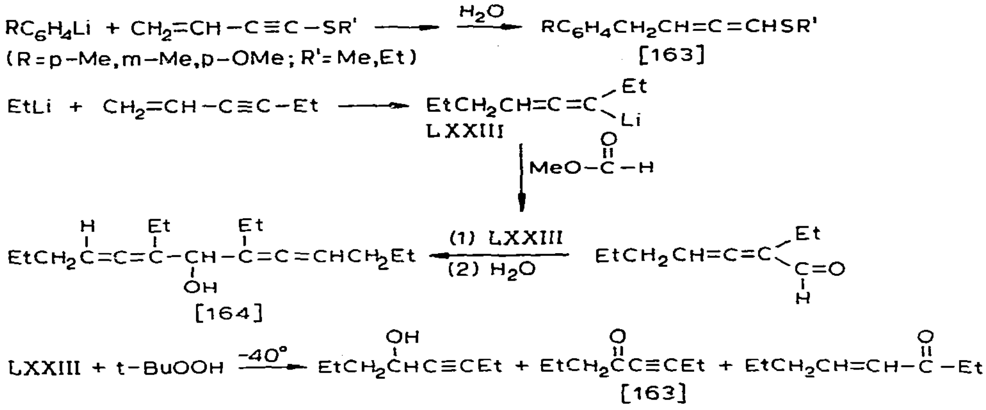


LXXI LXXII

Lithio-1,3-dithianes continue to serve as useful intermediates [see Mechanisms section above]. New reports this year relate to the reactions of these compounds with aromatic halides [161] and to the relative tendencies of metalation of five and six membered rings containing two sulfur atoms [162].

VII. Addition Reactions To Carbonyls, Olefins, and Similar Substrates

Further work has appeared on the addition of organolithium compounds to vinylacetylenes [AS 69; 35, also see 110-111, 146 above]. The following reaction sequences illustrate the use of these reactions for the preparation of various allene derivatives:



$$RC_6H_4Li + CH_2{=}CH{-}C{\equiv}C{-}SR' \longrightarrow \xrightarrow{H_2O} RC_6H_4CH_2CH{=}C{=}CHSR'$$

(R = p-Me, m-Me, p-OMe; R' = Me, Et) [163]

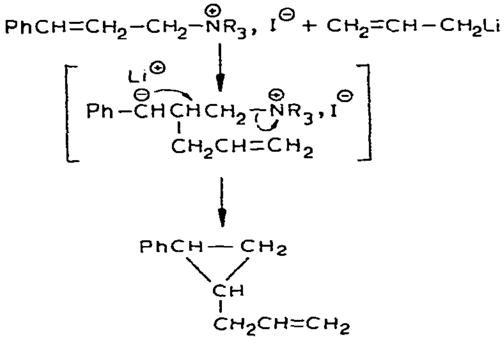
$$EtLi + CH_2{=}CH{-}C{\equiv}C{-}Et \longrightarrow \underset{\text{LXXIII}}{EtCH_2CH{=}C{=}C(Et)Li} \xrightarrow{MeO-\overset{O}{\overset{\|}{C}}-H} EtCH_2CH{=}C{=}C(Et){-}\underset{H}{\underset{|}{C}}{=}O \xrightarrow[(2)\ H_2O]{(1)\ \text{LXXIII}} EtCH_2\overset{H}{\overset{|}{C}}{=}C{=}\overset{Et}{\overset{|}{C}}{-}\underset{OH}{\underset{|}{C}}H{-}\overset{Et}{\overset{|}{C}}{=}C{=}CHCH_2Et$$

[164]

$$\text{LXXIII} + t\text{-}BuOOH \xrightarrow{-40°} EtCH_2\overset{OH}{\overset{|}{C}}HC{\equiv}CEt + EtCH_2\overset{O}{\overset{\|}{C}}C{\equiv}CEt + EtCH_2CH{=}CH{-}\overset{O}{\overset{\|}{C}}{-}Et$$

[163]

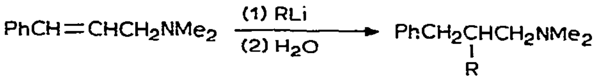
Cinnamyl ammonium salts have been reported to react with allyllithium to yield cyclopropanes by an addition-rearrangement process [165]:



(35 %)

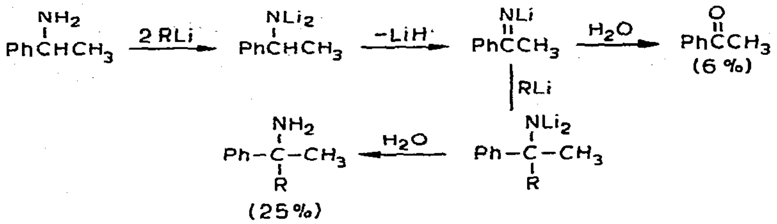
Metalation with subsequent Stephens rearrangement is a competing process.

The neutral cinnamyl amine reacts with n-BuLi and t-BuLi by a straightforward addition process [166]:



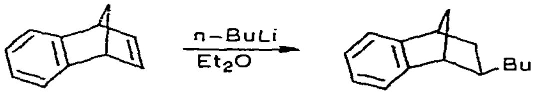
$$PhCH{=}CHCH_2NMe_2 \xrightarrow[(2)\ H_2O]{(1)\ RLi} PhCH_2\underset{R}{\underset{|}{C}}HCH_2NMe_2$$

With primary amines excess RLi yields ketones presumably by the following route (shown for α-methylbenzylamine):

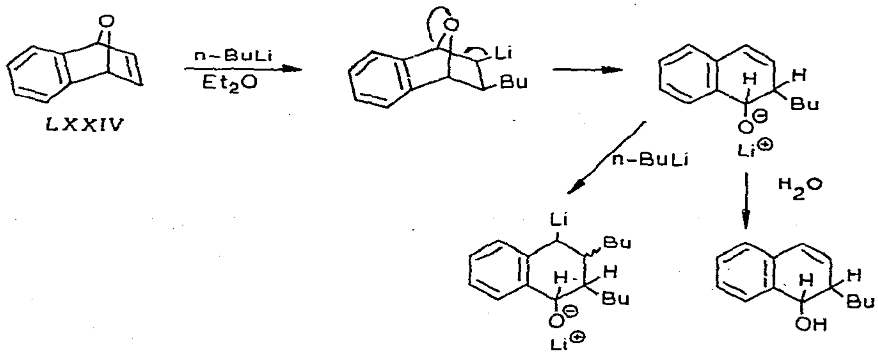


The reaction of isoprene with lithium naphthalene followed by oxidation with molecular oxygen has been reported to yield a mixture of at least eleven terpene alcohols [167]. Derivatization of the intermediate anions has also been carried out with ketones and epoxides [168].

Also reported was the addition of RLi to benzonorbornadiene [169]. In the same paper was described the reaction of t-BuLi



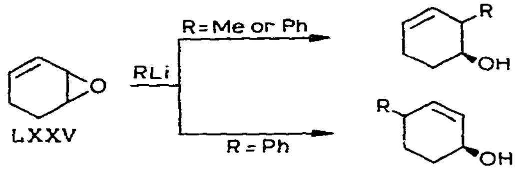
and n-BuLi with oxide LXXIV in which ring-opening of the initial adduct occurs:



Excess n-BuLi caused the formation of a diadduct due to addition to the C=C bond.

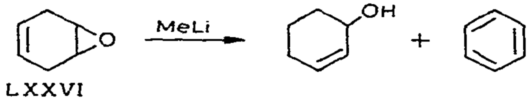
Other reports have also appeared concerning additions of

RLi to epoxides. MeLi and PhLi have been compared in their reactivity with cyclohexene oxides to the corresponding organomagnesium and copper compounds. With LXXV, MeLi is unusual in that it gives only the trans-1,2-addition product, whereas PhLi and the other methylmetal compounds give products arising from 1,4-conjugate addition as well [170, 171]:



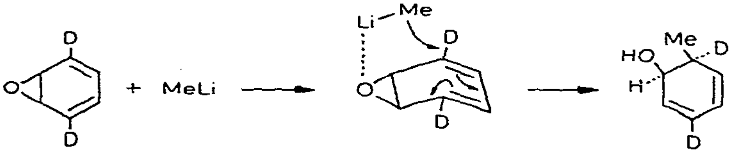
LXXV

With LXXVI, MeLi gives 75% benzene hydrate and 25% benzene [171], in sharp contrast to MeMgCl, Me_2Mg, and Me_2CuLi:

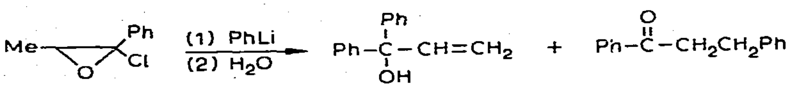


LXXVI

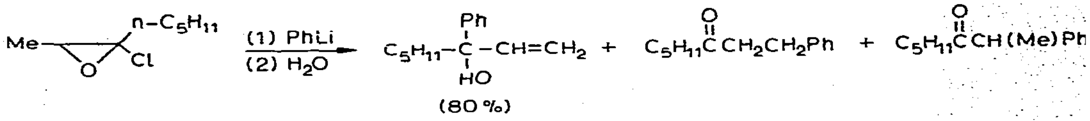
1,6-Addition to LXXVII has been shown to occur in a cis-fashion:



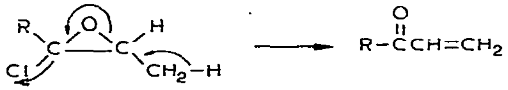
Formation of the trans product as indicated in an earlier report apparently was due to the presence of halide in the MeLi. Chloroepoxides reportedly react with PhLi by different routes depending on the substituents present [173]:



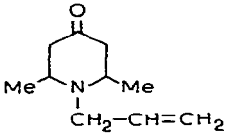
LXXVIII (60%) LXXIX (40%)



Products LXXVIII and LXXIX arise from $RCOCH=CH_2$, which is formed by a deprotonation-elimination process:

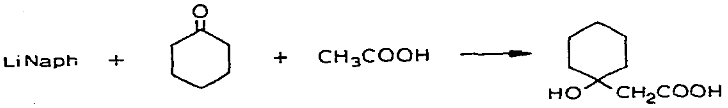


Reaction of organolithium compounds with carbonyl compounds continues to be a useful synthetic procedure. The reactions of EtLi with 2-methylcyclohexanone and LXXX reportedly yield

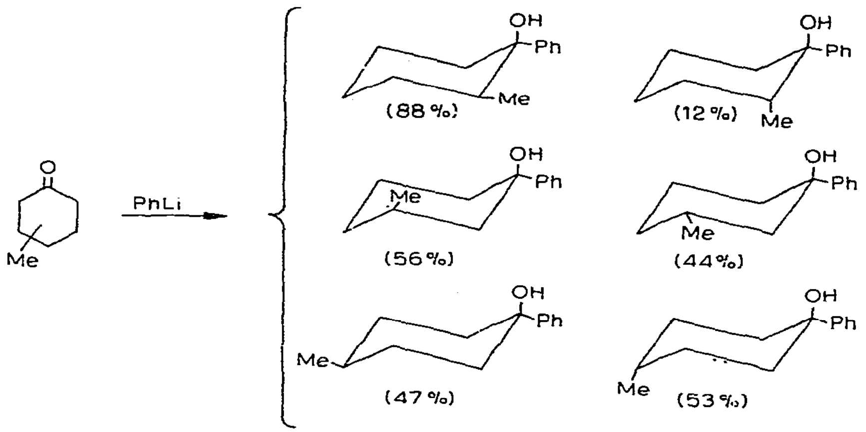


LXXX

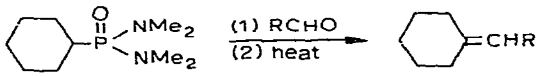
volatile products consisting of H, C_2H_4, C_2H_6, and $n\text{-}C_4H_{10}$; presumably <u>via</u> a radical process. Also reported was the addition of EtLi to the C=C bond of the sodium enolate of 2-methylcyclohexanone [174, 175]. Reaction of lithium naphthalene with a ketone (e.g., cyclohexanone) and acetic acid was reported to yield β-hydroxy acids [176]:



The stereochemistry of additions of PhLi to the three isomeric methylcyclohexanones has been reported [177].

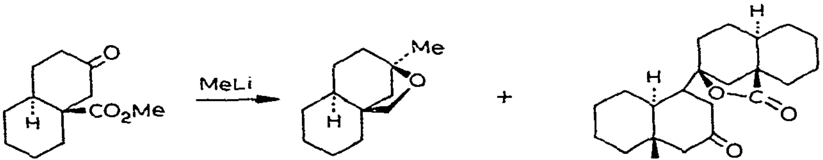


Reaction of LXXXI with cyclohexanone could not be effected. However, reaction with aldehydes can occur to give alkenes [178]:



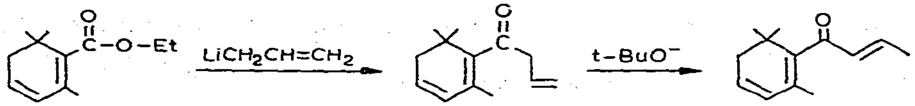
LXXXI

Also reported was the reaction of LXXXII with MeLi as shown [179]:



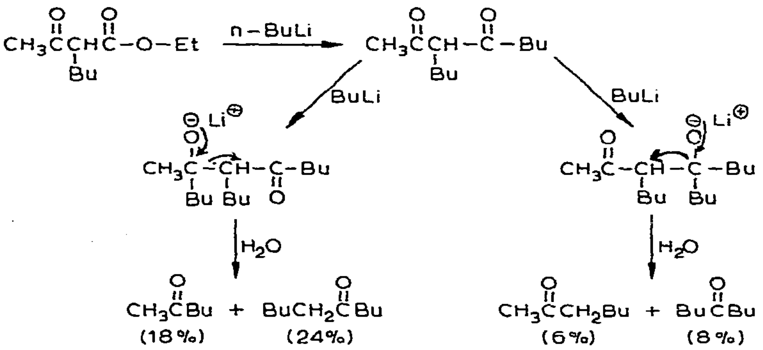
LXXXII

The preparation of ketones from the reaction of organolithium compounds with carboxylic acids has been reviewed by Jorgenson [180]. The reaction of the ester LXXXIII with allyllithium has been utilized to prepare β-damascenone, an odor principle [181]:

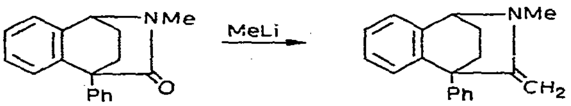


LXXXIII

Alkyllithiums with β-keto esters were found unexpectedly to produce ketones, rather than to undergo metalation [182]. The mechanism proposed involves the initial formation of a β-diketone as shown below for ethyl α-n-butyl acetoacetate:



Addition of alkyllithiums to bridged lactams such as LXXXIV yields the corresponding en namine in very high yield [183]:



LXXXIV

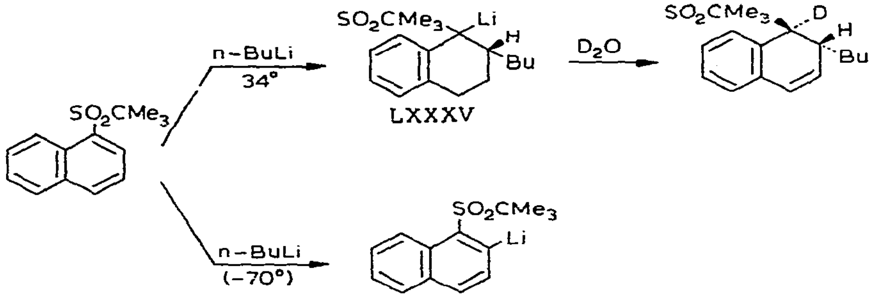
9-Methylanthracene is not stereospecifically alkylated by EtLi in THF [184], i.e. both cis- and trans-9-ethyl-10-methyl-9,10-dihydroanthracenes are formed. A radical pathway to these products was proposed:

$$\mathrm{RLi} + \mathrm{ArH} \rightleftharpoons \mathrm{ArH}^{\cdot -},\mathrm{Li}^{+} + \mathrm{R}\cdot$$

$$\mathrm{R}\cdot + \mathrm{ArH} \rightleftharpoons \mathrm{R{-}Ar{-}H}\cdot$$

$$\mathrm{RArH} + \mathrm{RLi} \longrightarrow \mathrm{RArH}^{\cdot -},\mathrm{Li}^{+} + \mathrm{R}\cdot$$

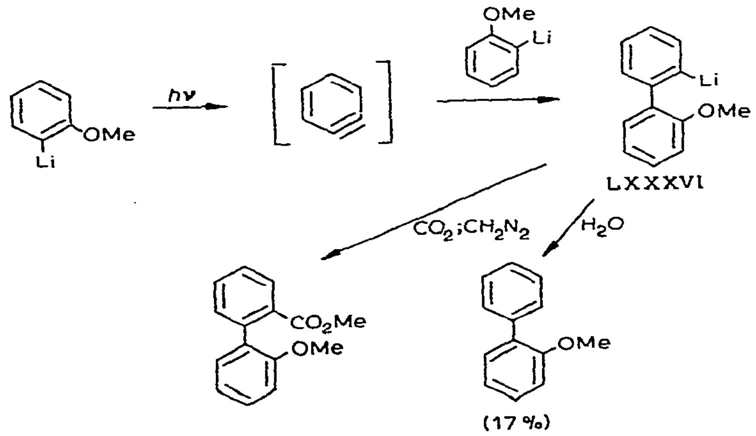
Reaction of arylsulfonium compounds with BuLi has been found to occur by addition at elevated temperatures whereas metalation predominates at -70°C [185]:



The intermediate LXXXV was trapped with D_2O (as shown) and with CO_2.

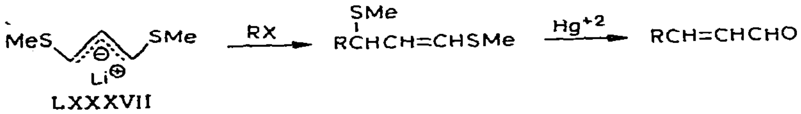
VIII. Miscellaneous Organic Reactions

Photolysis of aryllithiums to produce biphenyls has been reported in more detail [186, AS 65; 15]. Photolysis of o-anisyllithium [187] does not yield much coupling product (2,2'-dimethoxybiphenyl), however; 2-methoxybiphenyl is the primary product. The intermediate LXXXVI was trapped with carbon



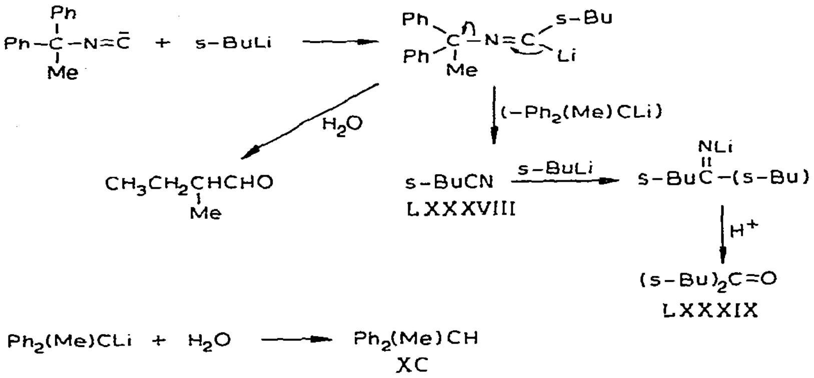
dioxide and D_2O. A benzyne mechanism was postulated although not proven conclusively.

A new synthesis of α,β-unsaturated aldehydes has been elucidated [188]. The key to the synthesis is the 1,3-bis(methythio)-allyl anion LXXXVII. Reactions of LXXXVII with ketones,



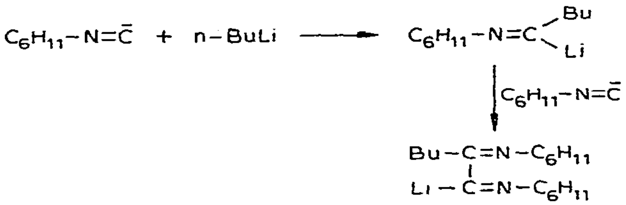
aldehydes, and epoxides were also reported.

The reaction of s-BuLi and 1,1-diphenylethylisonitrile proceeds in somewhat unexpected fashion as compared to other isonitriles [189, AS 70; 296]. In addition to 2-methylbutyraldehyde, three other products were isolated LXXXVIII-XC:

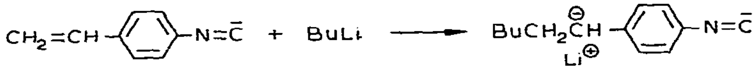


With triphenylmethylisonitrile, the only products are those resulting from cleavage of the intermediate lithium aldimine.

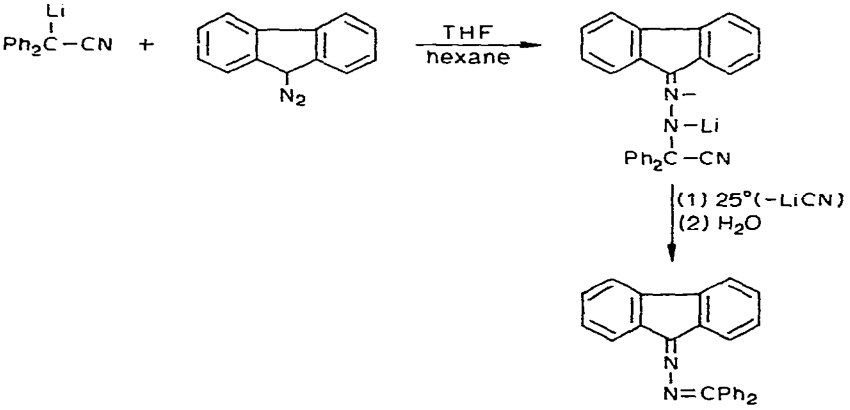
UV spectra of these adducts have been reported [190]. At high isonitrile/RLi ratios, dimeric adducts (and higher) may be formed:



With o- and p-isocyanostyrenes, BuLi or polystyryllithium adds only to the vinyl group without further polymerization:



Substituted alkyllithium compounds react with aliphatic diazo compounds by a similar route, thereby providing convenient access to a number of hydrazones [191].

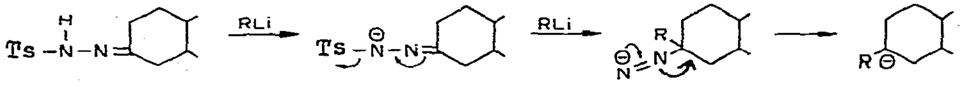


Reaction of RLi with 3,4-dimethoxybenzyl chloride reportedly gives 3,4-dimethoxyalkylbenzenes (simple coupling) and 1,2-bis(3,4-dimethoxyphenyl)ethane (halogen-lithium exchange followed by coupling) [192].

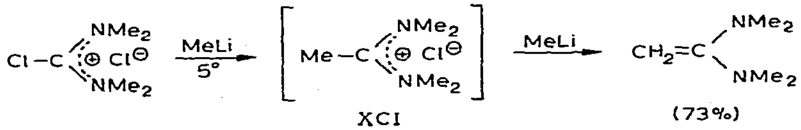
The autooxidation of organolithiums to peroxides has been reviewed [193].

Olefin formation, especially from steroid hydroxy ketones, via reaction of tosylhydrazones with RLi has been exploited

[194; AS 68; 200]. In the presence of an excess of alkyllithium, however, further reaction occurs by the following (proposed) mechanism to yield alkylated products [195]:



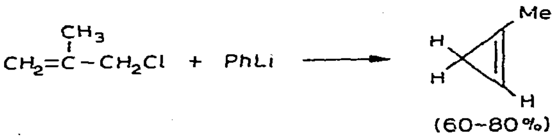
Enediamines have been synthesized by reaction of MeLi with formamidinium salts [196]:



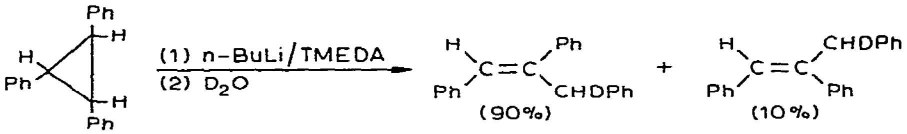
The acetamidinium ion XCl is the presumed intermediate. The reaction is not general, however, as evidenced by the isolation of 81% $(t\text{-Bu})_2CHNMe_2$ when t-BuLi was used. A radical process was proposed for the latter case.

Reaction of N,N-dimethyl-N-benzylanilinium ion with RLi has been reported to yield cleavage and rearrangement products [197]. A mechanism involving radical pairs [AS 69; 12] was employed to rationalize the results.

1-Methylcyclopropene has been synthesized by reaction of β-methylallyl chloride with PhLi (halide free) [198, see 60-61 above]:

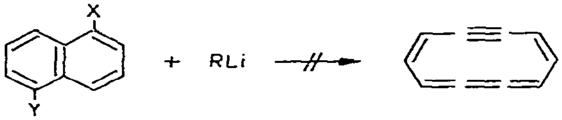


The attempted polylithiation of triphenylcyclopropyl compounds resulted in ring-opening reactions as shown below [199]:



PMR spectra of the intermediate triphenylallyl anions were also reported.

The reaction of 1,5-dihalonaphthalenes with PhLi does not lead to the desired didehydro[10]annulene derivatives [200], rather the major products are diphenylnaphthalenes formed by conventional routes.



New insertion reactions of carbon monoxide with C-Li and N-Li bonds have been reported [201, AS 70; 282].

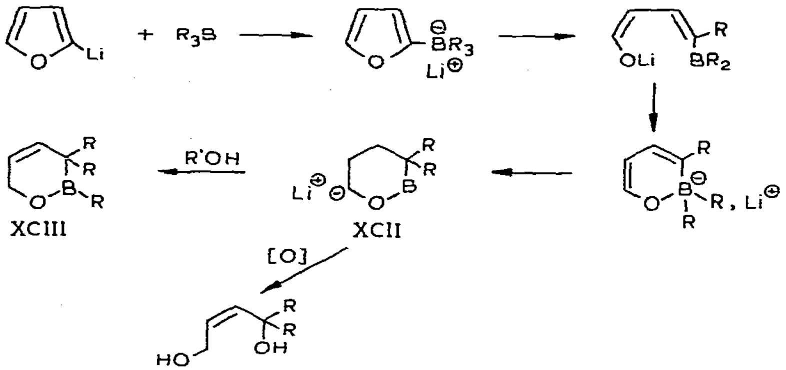
Finally, we note that the use of hydroxyl-tritiated propanol for the quenching of organolithium reagents has been utilized as a method for the analysis of these reagents [202].

IX. Reactions with Inorganic and Organometallic Compounds

Reichle has reviewed the preparation of many σ-bonded organometallics including some reactions involving lithium reagents [203].

Coupling of CH_2Li_2 with a series of boron compounds (BCl_3, BBr_3, $B(OMe)_3$, Me_2BBr, $(Me_2N)_2BCl$ and Me_2NBCl_2) and Me_3SiCl has been reported [204]. The latter gives $Me_3Si\text{-}CH_2\text{-}SiMe_3$ in good yield, but the corresponding boron compounds are generally unstable.

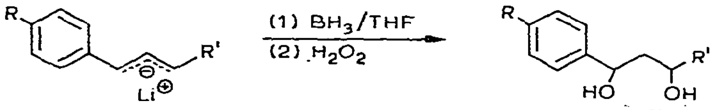
Reaction of trialkylboranes with α-lithiofuran proceeds by the following (proposed) route [205]:



XCIII XCII XCIV

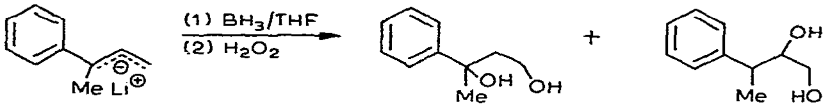
The intermediate XCII may be quenched with alcohol to yield the cyclic borate or oxidized to the unsaturated glycol XCIV.

Similarly, reaction of BH_3/THF with several phenyl-allyl-lithiums gives, after oxidation, the diols XCV [206].



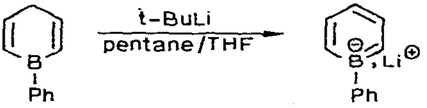
(R=H,Me; R'=Me) XCV

With the anion of 3-phenyl-1-butene a mixture of products was observed:

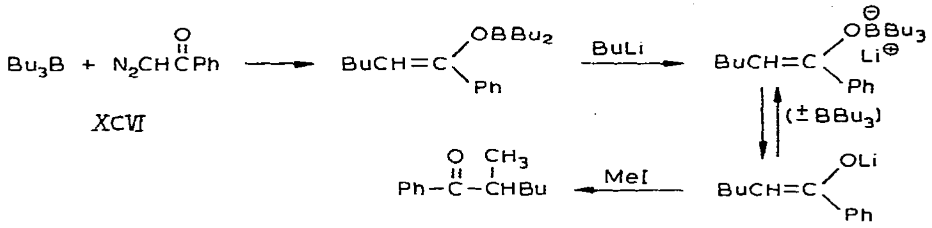


(Mixture of diastereomers)

t-BuLi has been utilized to prepare the anion of 1-phenylborabenzene [207]:



Reaction of alkyllithiums with vinyloxyboranes such as XCVI is a route to dialkylated ketones from diazoketones [208]:



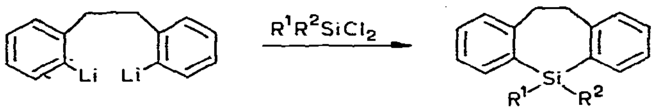
XCVI

Several papers have appeared which describe the use of alkyllithium reagents for the lithiation of o-, p-, and m-carboranes. The following reactions of lithiated carboranes have been described: methylation with MeI [209]; oxidation with benzoyl peroxide [210] and with air [211]; chlorination with Cl_2 [212] and CCl_4 [213]; addition to nitroalkenes [214]; addition to quinolines and other heterocycles [215]; and reaction with transition metal halides [216, 217].

Reactions of alkyllithium compounds with organosilanes is an often used method, of course. In addition to the many applications referred to above, the following papers have appeared. Metalation of the methyl groups and coupling are competing processes when t-BuLi/TMEDA is reacted with Me_3SiX compounds (X = F, Cl, Br, OMe, OEt, $OSiMe_3$). Difunctional silanes Me_2SiCl_2 and $Me_2Si(OEt)_2$ gave only the coupling products [218]. 9-Lithiophenanthrene has also been derivatized with several silyl substituents by the same coupling technique [219]. A series of trimethylsilyl, trimethylgermanyl, and trimethylstannyl derivatives of cypentadiene has been prepared by coupling with lithiated cyclopentadienes [220].

Silyated allenes may be prepared from BuLi and silylated propargylic ethers and thioethers [221, 222, 223, see 109 above]. Anions of the type $R\text{-}\bar{C}{=}C{=}CH\text{-}XR'_n \longleftrightarrow R\text{-}C{\equiv}C\text{-}\bar{C}H\text{-}XR'_n$ [where R = Ph, $SiMe_3$ and XR'_n = OMe, O^tBu, S^tBu] and dianions of type type $R\text{-}\bar{C}{=}C{=}\bar{C}\text{-}XR'_n$ have been utilized by Mantione and coworkers for the preparation of a large number of silylated allene derivatives, as well as some complex furans and dihydrofurans.

Two groups have synthesized 10,11-dihydro-5H-dibenzo[b,f]-silepin by the reaction of o,o'-dilithiobibenzyl with dichlorosilanes [224, 225]:



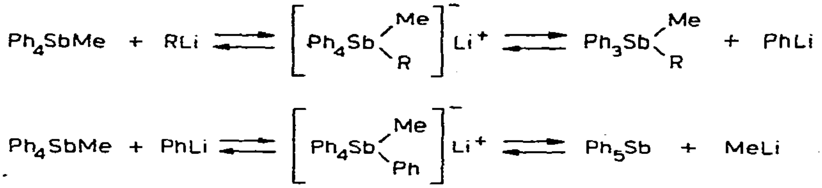
Perchlorinated 1,3,5-trisilacyclohexane is cleaved by MeLi to a variety of products [226]. The coupling reactions of $(CF_3)_2SiCl_2$ and $(CCl_3)_2SiCl_2$ were also reported by the same group [227].

N,N-Disubstituted aminomethyllithium compounds have been prepared by a transmetalation reaction [228]:

$$RR'NCH_2Sn(n\text{-}Bu)_3 + n\text{-}BuLi \xrightarrow[\text{or THF}]{\text{hexane}} RR'NCH_2Li + (n\text{-}Bu)_4Sn$$

The lithiated products (R,R' = alkyl, phenyl and $-CH_2CH_2OCH_2CH_2-$) are readily formed and derivatized in high yield, with the exception of the morpholine derivative which is apparently unstable.

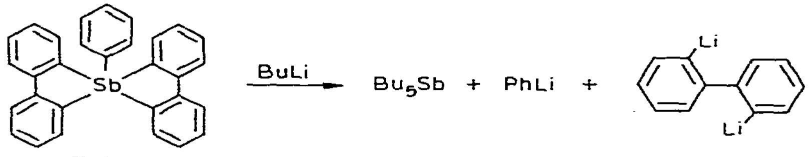
Pentaphenylantimony compounds are "symmetrized" by PhLi and n-BuLi, presumably through the formation of "ate" complexes [229]:



$$Ph_4SbMe + RLi \rightleftharpoons \left[Ph_4Sb\begin{smallmatrix}Me\\R\end{smallmatrix}\right]^- Li^+ \rightleftharpoons Ph_3Sb\begin{smallmatrix}Me\\R\end{smallmatrix} + PhLi$$

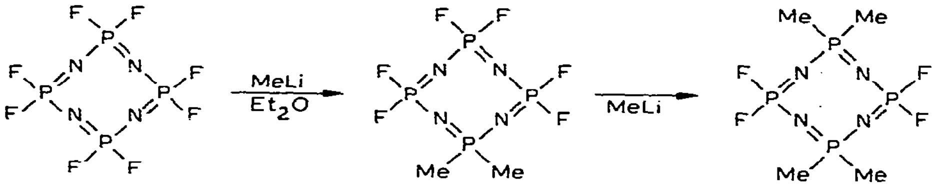
$$Ph_4SbMe + PhLi \rightleftharpoons \left[Ph_4Sb\begin{smallmatrix}Me\\Ph\end{smallmatrix}\right]^- Li^+ \rightleftharpoons Ph_5Sb + MeLi$$

Spirocylic antimony compounds such as XCVII are also cleaved by BuLi to produce the products shown [230]:

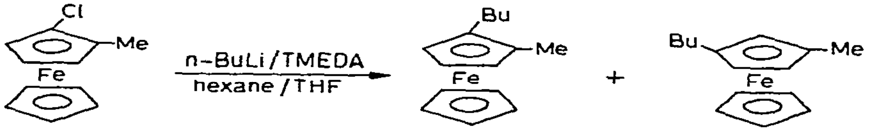


XCVII

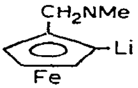
Cyclic polyphosphines such as $(C_2H_5P)_4$ and $(C_2H_5P)_5$ are also cleaved by PhLi to give compounds of the type $C_2H_5(Li)P-(PC_2H_5)_n-P(C_2H_5)Ph$ (n = 0-3) [231]. Phosphonitrilic fluorides are unexpectedly dimethylated by MeLi in a geminal fashion [232, AS 68; 208], after which the third methyl group enters antipodially.



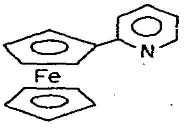
Several papers have appeared on the reactions of ferrocene derivatives with alkyllithium compounds. Evidence for ferrocyne has been obtained from a study of the reaction of 2-methylchloroferrocene and n-BuLi [233]. The two butylmethylferrocenes were



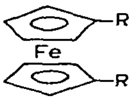
formed in approximately equal amounts (7-8%), but only in selected solvent/RLi systems. 1,2-Disubstituted ferrocene synthesis has been reported using the reagent XCVIII [234, AS 69, 31]. The



XCVIII



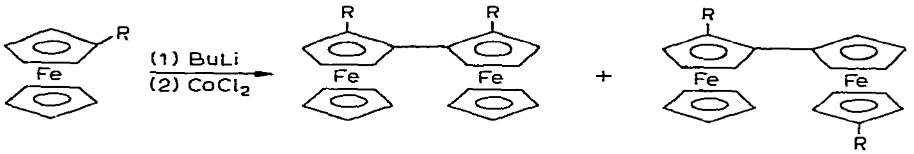
IC



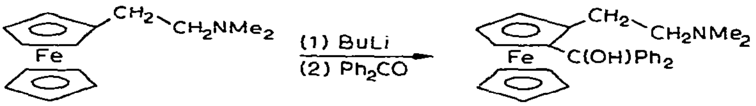
C

competition between addition and mono- and dimetalation of IC in a variety of solvent media and with t-BuLi, n-BuLi and MeLi has been explored [235; AS 68; 197]. 1,1'-dilithioferrocene, prepared from ferrocene and n-BuLi/TMEDA in high yield, has been used to prepare a number of symmetrically disubstituted ferrocene derivatives (R = $AsMe_2$; $AsPh_2$; PMe_2; PPh_2; SH) [236].

Coupling of ferrocenyllithium compounds with $CoCl_2$ results in the formation of a series of biferrocenes, as shown [237].

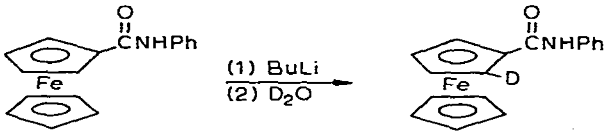


The lithiation of dimethylaminoethylferrocene gives good yields of the corresponding benzophenone derivative. Some dilithiated derivatives were observed if the metalation conditions were more rigorous [238].

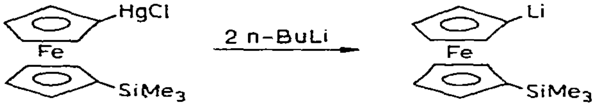


Lithiated N-phenylferrocenecarboxamide CI has been utilized to prepare the corresponding deuterio derivative [239]. 1-Lithio-

1'-(trimethylsilyl)ferrocene was prepared by a metal exchange



reaction [240] and used to prepare several derivatives:



Fluoroferrocene has been prepared for the first time by the reaction of lithioferrocene with perchloryl fluoride [241].

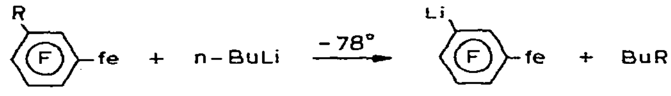
More stable carbene complexes with the following structures have been prepared by reaction of RLi with the appropriate transition metal carbonyl compounds: $(CO)_5Cr(OMe)R$ (R = substituted aryl [242]; R = pentachlorophenyl and ferrocenyl [243]).

Reaction of feHgCl (fe = $Fe(CO)_2\pi\text{-}C_5H_5$) with pentafluorophenyllithium results in the formation of bis(pentafluorophenyl)-mercury, not the desired $feHgC_6H_5$ [244]:

$$feHgCl + C_6F_5Li \longrightarrow feHgC_6F_5 + LiCl$$

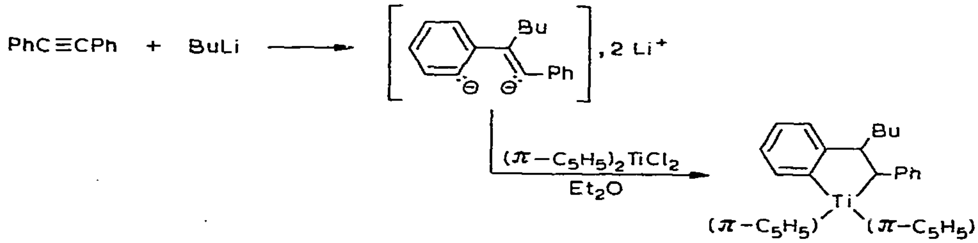
$$2\ feFgC_6H_5 \longrightarrow fe_2Hg + (C_6F_5)_2Hg$$

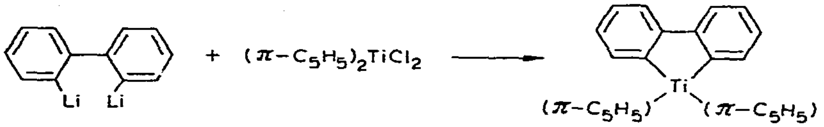
With CII, halogen-lithium (or hydrogen-lithium) rather than iron-lithium exchange was observed [245]:



(R= H,Br)
CII

Reaction of s-BuLi with Cu^{+2}, La^{+3} and Fe^{+3} yields stable organic radicals, presumably coordinated to the transition metal [246]. The product of the reaction of 2,2'-dilithiobephenyl and $CrCl_3$ yields various polyphenyl compounds when treated with transition metal halides [247; AS 67; 184]. Also reported was the preparation of two titanium metallocycles [248]:





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