LITHIUM

ANNUAL SURVEY COVERING THE YEAR 1972

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CONTENTS

I. Structure and Bonding Studies

Stucky and coworkers have continued their investigation of delocalized carbanion - lithium - diamine complexes. Three papers have appeared this year which describe the crystal structures of triphenylmethyllithium·$(TMEDA)_2$ [1], fluorenyllithium·bisquinuclidine [2], and naphthalene dilithium·$(TMEDA)_2$ [3]. The molecular geometries of these three complexes are shown below in Figures 1-3 respectively. As in the case of benzyllithium·

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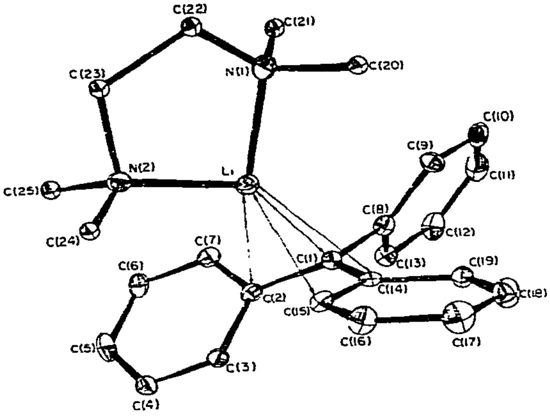


Figure 1

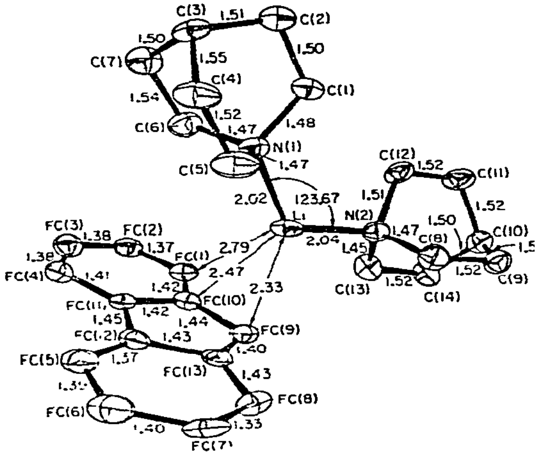


Figure 2

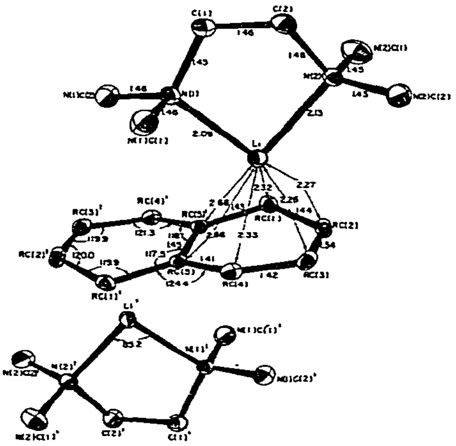
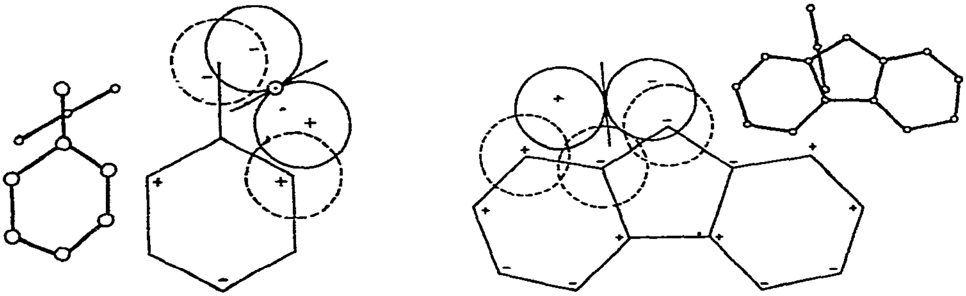


Figure 3

(TMEDA)$_2$ [AS 70; 254], the lithium atom in the triphenylmethyl complex is coordinated equally to the two nitrogen atoms, but its bonding with the π-group cannot be explained either by simple electrostatic considerations or by the assumption of a two-center σ bond with the central carbon atom (C(1) in Figure 1). The authors conclude that a substantial contribution to the bonding is made by a covalent three-center interaction. In valence bond language the lithium atom is pictured as sp^2 hybridized with two hybrid orbitals directed to the nitrogen atoms and the third directed to the π-system of the carbanion. In addition, the remaining lithium p orbital may interact with the highest occupied MO of the carbanion, as shown in Figure 4. In the case of the fluorenyl complex, Figure 2, the lithium atom interacts with only one side of the ring system in the solid state, but presumably would oscillate between the FC(1) and FC(8) sides of



Benzyllithium

Fluorenyllithium

Figure 4

FC(9). The naphthalene dianion structure is also interpreted in terms of this bonding concept.

Semi-empirical and *ab initio* MO calculations have been reported for methyllithium monomer and dimer [4]. Unlike previous calculations of the same systems [AS 69;2], the results indicate the carbon atom in the monomer to be *less* ionic than in the tetramer. Oliver *et al*. have reexamined the ultraviolet absorption spectra of ethyl, *n*-butyl-, *sec*-butyl and *tert*-butyllithium [5]. The results were compared with CNDO/2 calculations on the idealized tetrameric and hexameric methyllithium. The results shown in the table indicate that the absorption energy is effected both by the degree of aggregation and by the nature of the alkyl group.

CNDO/2 calculations also have been reported for some α-lithio carboxylic acid esters [6]. There is no good correlation between carbonyl bond order of the carbanions with the experimental carbonyl stretching frequencies. The authors conclude that the C-Li bond is not entirely dissociated in these compounds under the conditions used.

Ultraviolet Spectral Parameters of RLi

Compound	Solvent	Aggregation	λmax (nm)
R=Methyl		Tetramer	124 (calcd)
		Hexamer	171 (calcd)
Ethyl	Isooctane	Hexamer	210
	Vapor	Hexamer	215
n-Butyl	Hexane	Tetramer	203
		Hexamer	210
sec-Butyl	Vapor	Tetramer	206
tert-Butyl	Vapor	Tetramer	∿190

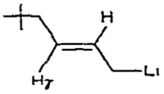
Ladd and Parker have reported further on the NMR spectra of substituted phenyllithium compounds [7, AS 69; 1]. In most cases, inductive and resonance effects may be used to explain ^1H and ^7Li shifts, although there are some discrepancies which are accounted for in terms of the anisotropy of the carbon bonded to lithium. ^7Li and ^1H NMR spectra have also been reported for methyllithium complexes with LiI and LiBr in diethyl ether [8]. Li_4Me_4, Li_4Me_3Br, $Li_4Me_2Br_2$ and $(LiBr)_n$ species are observed with the bromide, while the iodide shows only Li_4Me_4, Li_4Me_3I and $(LiI)_n$. Of particular interest is the observation that intramolecular lithium exchange within the Li_3R_3I species is slow at -70°, ruling out the importance of ionic equilibria such as

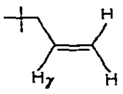
$$Li_4R_3X \rightleftarrows Li_4R_3^+ + X^-$$

Several papers have appeared the past year relating to the nature of allylic lithium compounds in various media. Young and coworkers have continued their investigations of phenylallylmetal compounds [AS, 70; 256]. UV absorption spectra of salts of 1,3-

diphenylpropene have been interpreted in terms of the formation of the solvent-separated ion-pair and two contact ion pairs which differ in the degree of solvation [9]. PMR results confirm that the lithium salt is predominately in the solvent-separated form [10]. Magnetic resonance and uv data are also reported for 1,3-diphenyl-2-methylallyllithium and 1,3-diphenyl-1-methylallyllithium [11]. The former exists as a dynamic equilibrium between *cis*-*trans* forms:

Neopentylallyllithium, the 1,4-addition product of t-butyllithium and 1,3-butadiene, has been investigated by two groups [12,13]. In non-donor solvents, the compound apparently exists as highly localized species of the *cis* and *trans* forms





in a ratio of approximately 1:3 [13]. The upfield shift of the γ-proton may be interpreted to indicate some ionic delocalization even in non-donor solvents. Further discussion of reference [13] may be found in Section V below, along with the discussion of other papers on oligomeric polydienyllithiums.

In a new monograph "Ions and Ion Pairs in Organic Reactions", considerable attention is given to the nature of organolithium compounds [14]. West has also reviewed the structures and reactions of alkali metal organics, especially those of lithium [15]. Smid has reviewed the structure of ion pair complexes, particularly giving attention to fluorenyllithium and to the effects of polyethers [16].

New uv absorption and conductivity data has appeared on ion pair complexes of 1,1-diphenylbutyllithium, 1,1-diphenylpolyisoprenyllithium, and 9-lithio-10-alkyl-9,10-dihydroanthracene [17], and on the lithium salts of fluorene, indene and triphenylmethane in amine solvents [18]. In the latter paper, constants for the contact-solvent separated ion pair equilibria are reported. As expected, the lithium salts are largely solvent-separated.

PMR spectra of indenyllithium in 1,2-dimethoxyethane have been interpreted to indicate that the geometry of the contact ion pair is different for Li than for Na and K salts [19,20]. More specifically, it is postulated that the lithium atom is located over the six-membered ring rather than the five-membered ring. More extensive data on the fluorenide and triphenylmethide systems [21] have also been interpreted in terms of ion pair equilibria [see Sodium and Potassium Annual Survey].

Sandel and coworkers [22,23] have proposed that the fraction $[1-(1/3.5R_{M^+})]$, where R_{M^+} is the radius of the cation, may be taken as the fractional delocalization of the carbon-metal bond in the contact ion pairs of 1- and 2-naphthylmethyl metal compounds. Thus, the chemical shifts of the ring protons were found to obey the relationship $\delta_N = \delta_{N\infty}(1-1/3.5R_{M^+})$ where $\delta_{N\infty}$ is the extrapolated chemical shift of the contact ion pair. For lithium, the above equation suggests that the C-Li bond is 58% delocalized.

The effect of dimethyl ether and THF on the infrared spectrum of n-BuLi has been reported [24]. The limiting effect was observed at a ratio n-BuLi/ether = 1, and the effect of THF is stronger than that of Me_2O.

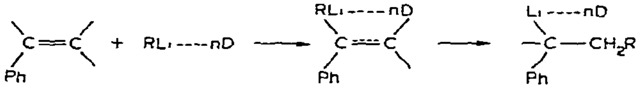
II. Kinetics and Mechanisms

The reaction of p-tert-amylbenzyllithium with 1,1-diphenylethylene has been found to be 0.5 order in toluene, a solvent in which the benzyllithium is quite soluble [25]. The effect of lithium (-)-(IR)-menthoxide, tert-BuOLi, and n-BuOLi on the rate of addition of n-BuLi to 1,1-diphenylmethane has been reported [26].

Dobson and coworkers have reported stopped-flow kinetics for the reactions of MeLi with $Cr(CO)_6$, $Mo(CO)_6$, $W(CO_6)$, $W(CO)_5[P(OC_6H_5)_3]$, $W(CO)_5[As(C_6H_5)_3]$ and $W(CO)_5[P(C_6H_5)_3]$ [27]. The reactions are one-quarter order in MeLi. The stopped-flow method was also used, in part, by Evans et al. who investigated the reactions of n-BuLi with triphenylmethane and triphenylethylene, and of triphenylmethyllithium with THF [28]. In THF the n-BuLi-Ph_3CH and n-BuLi-$\phi_2C{=}CH\phi$ reactions are 0.5 and 1.0 order in n-BuLi and substrate respectively. In benzene the former reaction was too slow to allow determination of order. In the reaction of Ph_3CLi with THF, the order is 1.0 in the lithium compound and the half-life is approximately 200 min. at 95° C. Russian workers have reporter further data on the decomposition of THF by n-BuLi [29, AS 71; 9]. Also reported were further details of the reactions of alkyllithiums with ethers in which a negative temperature coefficient has been reported [30, AS 69; 259].

The kinetics of the reaction of MeLi with 2,4-dimethyl-4'-methylmercaptobenzophenone, also investigated by stopped-flow methods, has been found to be one-fourth order in MeLi and first order in ketone in Et_2O [31]. The effect of LiBr or LiI on the rate is discussed in terms of the magnetic resonance data referred to above (reference 8).

The proton abstraction reaction between polystyryl carbanion salts and triphenylmethane has been investigated by Chan and Smid [32]. Their results are discussed in the Sodium and Potassium Annual Survey which follows. Also discussed there is the study of Ise et al. on the effect of electric fields on the anionic polymerization of o-methylstyrene in 2-MeTHF and THF [33, AS 70; 260] with alkali metals as gegenions. Russian workers have utilized ^{14}C-labelled n-BuLi to assist in the study of the kinetics of polymerization of α-methylstyrene in THF [34]. The results are interpreted in terms of the "coordinative" mechanism [AS, 71; 7] in which the polymer chain is assumed to grow on active centers consisting of complexes (I) of an organometallic compound

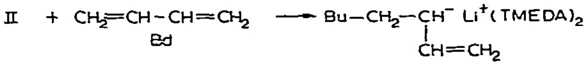


I

and the monomer. In this view, the rate of the polymerization process is effected by the degree of coordinative unsaturation of the organometallic compound, i.e., the value of n.

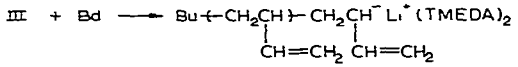
Also appearing the past year were studies on the polymerization kinetics of isoprene initiated by 1,1-diphenyl-n-hexyllithium [35] and two papers on the kinetics of reactions of N-chelated n-BuLi [36,37]. In the former paper, the kinetic order with respect to the initiator is 0.25 in both hexane and benzene solvents. In the latter, Hay and coworkers have shown that the initiation rate for butadiene polymerization is second order in monomer, and first order both in n-BuLi and TMEDA. A complex (n-BuLi•2TMEDA) was assumed to be the primary initiating agent and the following kinetic scheme was postulated:

$$\mathrm{BuLi\cdot TMEDA} + \mathrm{TMEDA} \rightleftharpoons \underset{\mathrm{II}}{\mathrm{Bu^-\,Li^+(TMEDA)_2}}$$



$$\mathrm{II} + \underset{\mathrm{Bd}}{\mathrm{CH_2{=}CH{-}CH{=}CH_2}} \longrightarrow \mathrm{Bu{-}CH_2{-}\underset{\underset{\displaystyle CH{=}CH_2}{|}}{CH^-}\,Li^+(TMEDA)_2}$$

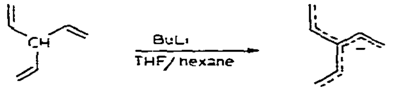
III



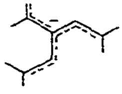
$$\mathrm{III} + \mathrm{Bd} \longrightarrow \mathrm{Bu\!\left(CH_2\underset{\underset{\displaystyle CH{=}CH_2}{|}}{CH}\right)\!CH_2\underset{\underset{\displaystyle CH{=}CH_2}{|}}{CH^-}\,Li^+(TMEDA)_2}$$

n-BuLi/TMEDA also polymerizes ethylene [36] yielding a wide range of oligomers, the rate of formation of which has been reported.

Several papers have appeared during the past year concerning carbanion cyclization and rearrangements. Bates and coworkers have continued their investigation of anions of vinyl homologs by the preparation of IV-VIII using reactions such as (1) [38]:

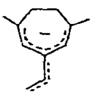


IV (1)

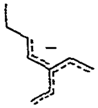


V

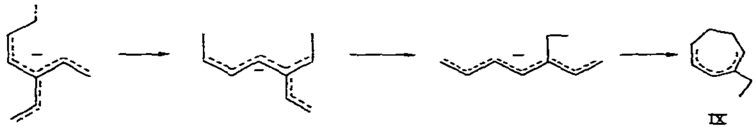
VI



VII



VIII trans

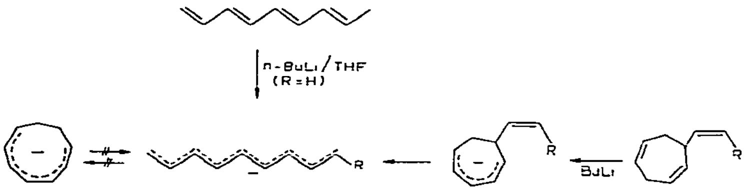


VIII cis

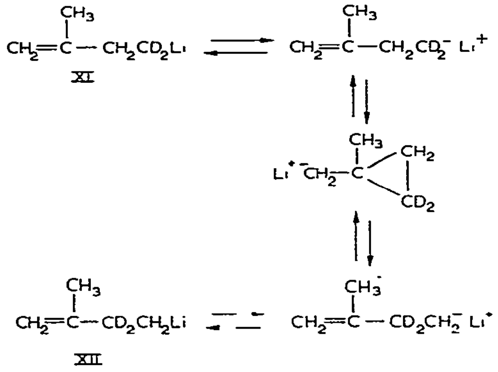
IX

The cis isomer of VIII is smoothly transformed to IX presumably by two sigmatropic proton shifts followed by a heptatrienyl-cycloheptadienyl rearrangement.

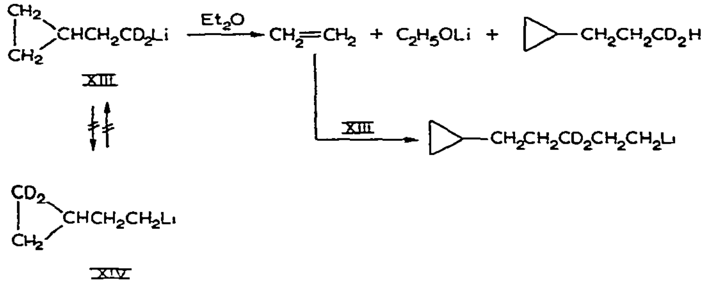
The nonatetraenyl anion X has been prepared by the same group and shown by NMR to exist in the extended planar form [39]. Unlike the heptatrienyl anion, X does not cyclize readily; in fact X may be prepared by a ring opening reaction of the 6-vinylcycloheptadienyl anion:



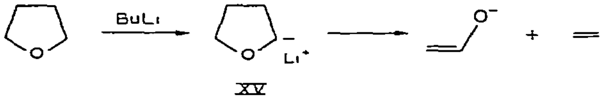
The rearrangement of XI $\rightleftarrows$ XII has been investigated by Maercker and Weber [40]. Kinetic results indicate that the rearrangement proceeds by a dissociation-recombination process. The Grignard analog apparently reacts by a different mechanism.



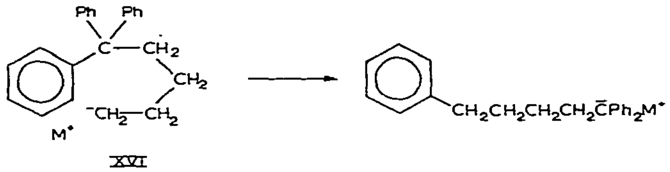
2-Cyclopropylethyllithium XIII does not rearrange to XIV; rather, cleavage of ether solvent and subsequent addition of ethylene is the principal process observed [41]:

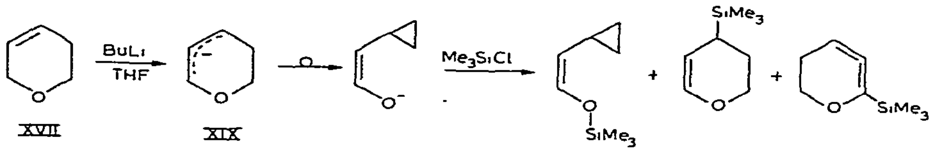


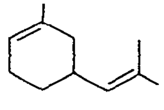
Similar cleavage of THF by n-BuLi yields lithium enclates of aldehydes. This reaction has been explored by Bates *et al.* who show it to be of limited utility [42]. Evidence is cited for the intermediary of the α-anion XV which opens by an allowed $-[_{\pi}4_s + {}_{\pi}2_s]$ cycloreversion process:



Grovenstein and coworkers have searched for evidence of 1,2, 1,4 and 1,5-aryl migrations in alkali metal compounds such as XVI [43-45]. These papers are discussed in the Sodium and Potassium Annual Survey which follows. Rearrangement of dihydropyranyllithium XVII and the anion of nerol oxide XVIII have been reported [46]. Reaction of the metalated product from XVII with Me_3Si gives a mixture of products as shown above. The cyclopropane-containing product is postulated to form *via* a [1,4]

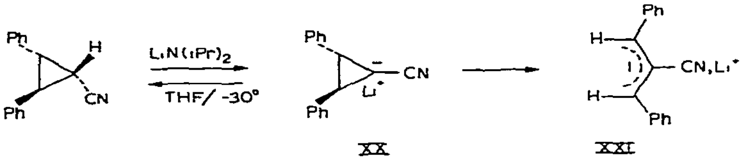


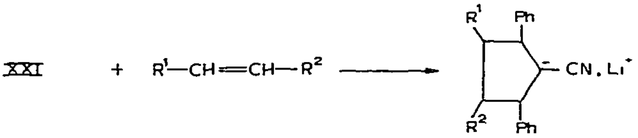




XVIII

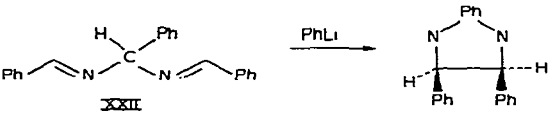
sigmatropic shift involving the intermediate XIX. Also reported was the effect of various solvents on the yield of rearranged products from allylic alcohols with allyllithium [47]. The cyclopropyl anion XX undergoes a ring opening to the allyl anion XXI which has been shown to undergo $[_{\pi}2s + {}_{\pi}4s]$ cycloaddition reactions with styrene, trans-stilbene, 1,1-diphenylethylene and acenaphthylene [48].



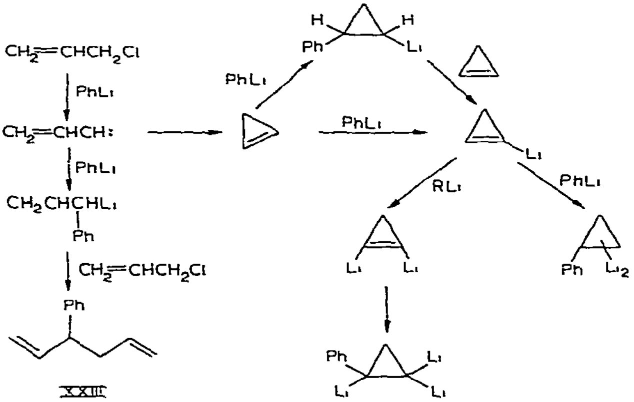


Cyclization of the anion formed from XXII has been reported [49].

The intermediate carbanion is assumed to be W-shaped and shows a H/D kinetic isotope effect of 2 when quenched with MeOH/MeOD.

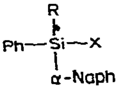


Further work on the reaction of phenyllithium with allyl chloride [AS 71; 12] has shown that cyclopropene is the source of the proton in the final step [50]. The following reaction scheme was proposed:

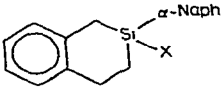


The small but significant yield of XXIII is explained by the trapping of the carbene intermediate by PhLi and subsequent coupling with allyl chloride.

Further evidence on the stereochemistry of the coupling reactions of the optically active silanes XXIV and the cyclosilanes XXV with organolithium reagents has been published [51,52, AS 70; 263]. The authors suggest that their results, particularly the observation of retention processes with EtLi/TMEDA, may not be explained satisfactorily by a $S_{N}i$-S_i mechanism involving metallic assistance.

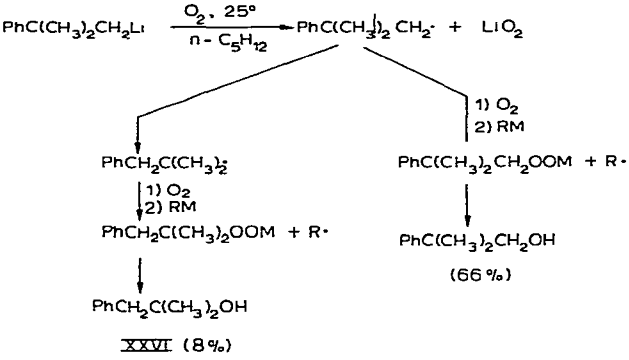


XXIV



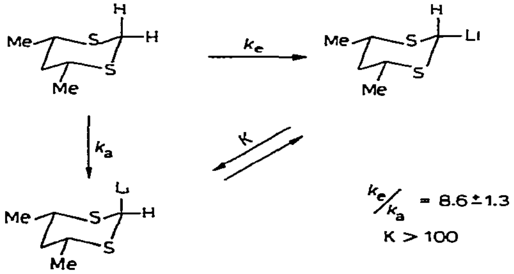
XXV

The observation that some of the reaction products from the autoxidation of neophyllithium contain the benzyldimethylcarbinyl moiety (e.g. XXVI) has led Panek and Whitesides to propose the following radical mechanism for the process [53]:

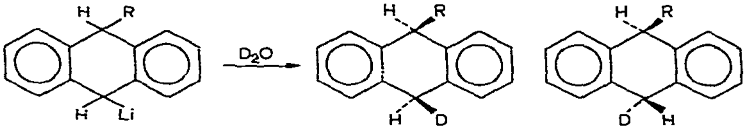


The effect of large magnetic fields on the product distribution from pentafluorophenylmethylchloride with n-BuLi has been interpreted in terms of a radical pair process [54].

Further work on the metalation of conformationally fixed 1,3-dithianes has shown that the preference of lithium for the equatorial position [AS 71; 9] is primarily a thermodynamic phenomenon, although a small kinetic preference is also observed (55). Evidently, the equatorial lithium compound may be formed from the axial isomer by a carbanion inversion (in THF or DME).

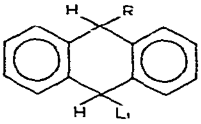


Further work on the protonation of lithiated dihydroanthracenes has shown that the stereochemistry of the process is dependent on the nature of the solvent [56]. In the presence of HMPA, only one product cis-XXVII is observed while in THF one obtains approximately equal amounts of the two isomers. The

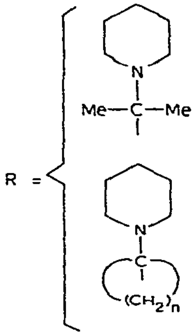


R = Et, i-Pr, t-Bu   cis-XXVII   trans-XXVII

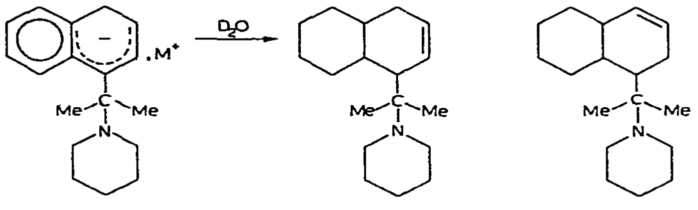
authors conclude that the solvent-separated and contact ion pairs are the reactive species in HMPA and THF respectively. Similarly, Fabre et al. have found that the lithium derivatives XXVIII give a mixture of cis and trans products upon reaction with D_2O in diethyl ether [57].



XXVIII



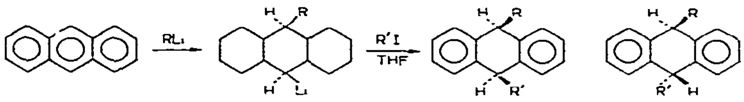
It is interesting to note that the sodium compound gives only the trans-isomer of XXIX. The same group has studied the isomers obtained from the protonation of XXX [58]. Again, the reaction is stereospecific in the case of M=Na. Only the trans-isomers of



XXX XXXI XXXII

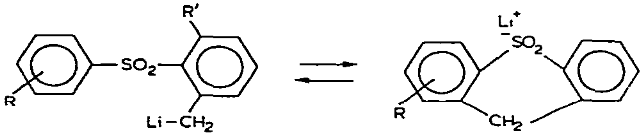
XXXI and XXXII are found with the latter in predominance. With M=Li, XXXI is the major product and both cis and trans isomers of both products were found in nearly equal quantities.

Alkylation of 9-lithio-10-alkyl-9,10-dihydroanthracenes has been reexamined [49]. Most interesting is the observation that isopropyllithium addition to anthracene followed by methylation with MeI gives a 90:10 ratio of the cis and trans adducts XXXIII whereas addition of MeLi followed by i-PrI gives a 40:60 product ratio.



cis-XXXIII *trans*-XXXIII

The equilibrium between the open chain and cyclic forms of lithiated diaryl sulfones has been investigated by uv spectroscopy for a variety of sulfones and in several solvents [60, AS 69; 29]. However, no quantitative values for the equilibrium constants were obtained.



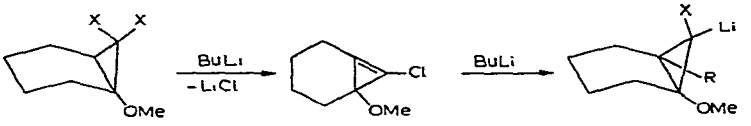
III. Lithium Carbenoids and Other Halogen-Substituted Organolithiums

The reaction of dichloromethyllithium with $ClSiMe_3$ has been reinvestigated and shown to give a variety of products, not simply the expected $Me_3SiCHCl_2$ and $Me_3SiCCl_2SiMe_3$ as previously reported [61]. Seyferth et al. have reported the preparation of $(Me_3Sn)CCl_2Li$ and $(Me_3Sn)CBr_2Li$ [62], most successfully by the transmetalation reaction:

$$(Me_3Sn)_2CX_2 + BuLi \xrightarrow[-130°]{THF} Me_3SnCX_2Li + BuSnMe_3$$

However, reactions with either Me_3SiCl or MeI are complicated by side reactions which decrease the ultimate yield of derivatives.

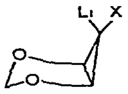
Taylor and coworkers have continued to examine carbenoids stabilized by neighboring heteroatoms such as oxygen [63, AS 71; 14]. Carbenoid XXXVa results from the treatment of the dichlorocyclopane XXXIVa and the proposed mechanism of its formation is shown below. The thermolysis products of XXXVa and XXXVb were investigated.



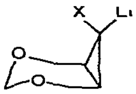
XXXIV
a (X = Cl)

XXXV
a (X = Cl, R = n-Bu)
b (X = Cl, R = H)

Also, this group has reported that the C-H insertion reaction of carbenoids depends on the configuration of substituents at the carbenoid carbon atom. Thus, carbenoid XXXVII, which is substantially less stable than XXXVI, gives products resulting

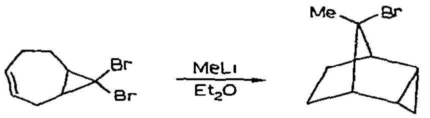


XXXVI



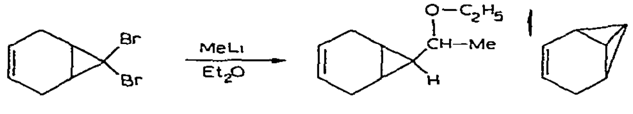
XXXVII

from intermolecular reactions while XXXVI gives C-H insertion products.



XXXVIII [64]

95%

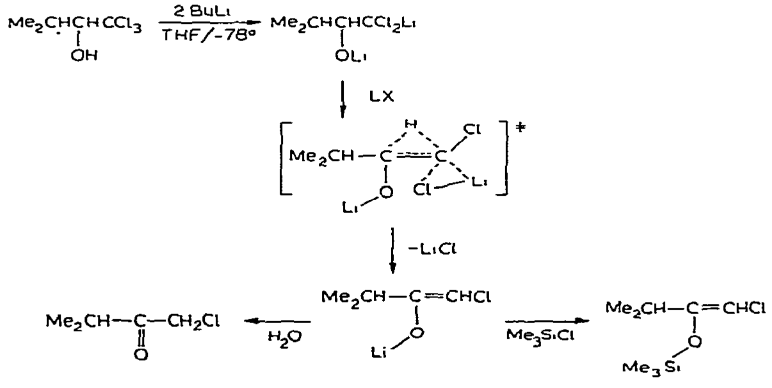


XXXIX [65]

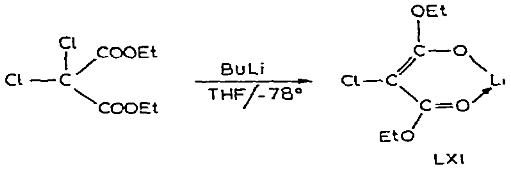
(MAJOR) (MINOR)

The reaction of two dibromocyclopropanes XXXVIII and XXXIX with MeLi has been reported in which neither gives an allene as the principal product.

The carbenoid LX yields hydrolysis and silylated products which have been interpreted in terms of an α-elimination process with simultaneous hydrogen migration [66]:



Also reported by Villieras was the chlorocarbenoid LXI [67]. A chelate structure was proposed to rationalize the greater stability and lower reactivity of LXI as opposed to other chlorocarbenoids. Also reported during the past year was the formation of low molecular weight, cyclopropane-containing polymers by the decomposition of lithium carbenoids from gem-dihalocyclopropanes [68].

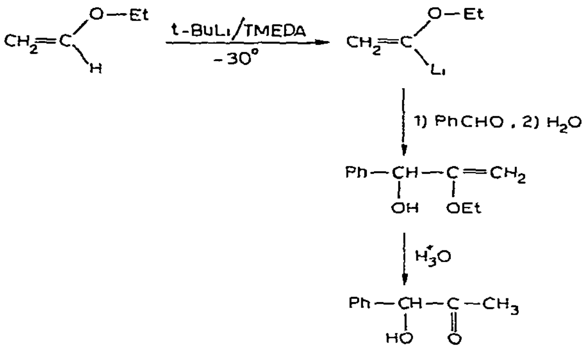


Non-halogen containing carbenoids continue to be of interest The monolithium derivatives of CH_3CN and $PhCH_2CN$ have been prepared and isolated [69]. $LiCH_2CN$ is tetrameric in DMSO and PhCH(Li)CN is a dimer. IR spectra are reported along with conductivity data in DMSO. A rather high conductance value implies that the aggregate may not have the same structure as $(MeLi)_4$.

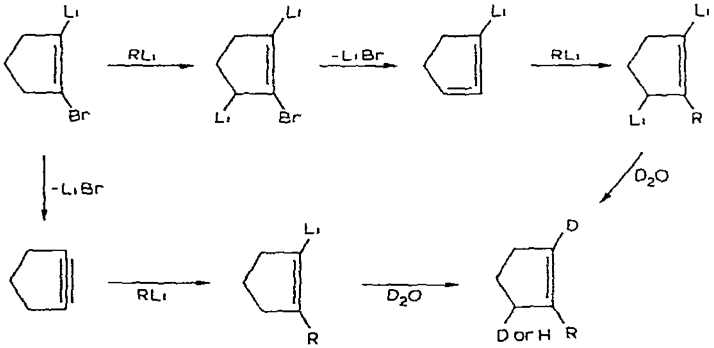
Also reported were the new carbenoids $LiCH_2N(CH_3)NO$ [70] and $(C_6H_5S)_3CLi$ [71]. Several reactions of the latter are described including the incorporation of p-thiocresolate and complete scrambling with tris(p-tolythio)methyllithium.

α-Lithiation of carboxylic acids in THF and THF/HMPA has been described in more detail [72, AS 70; 271]. The primary benefit derived from the use of HMPA is the solubilization of the dianions and the acceleration of their alkylation reactions.

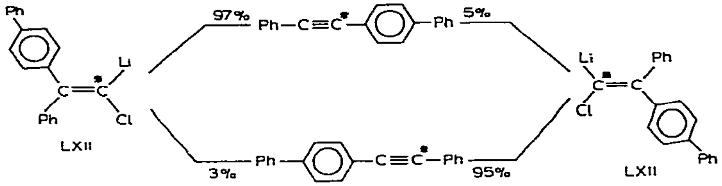
1-Ethoxyvinyllithium has been prepared from t-BuLi and vinyl ethyl ether in TMEDA [73].



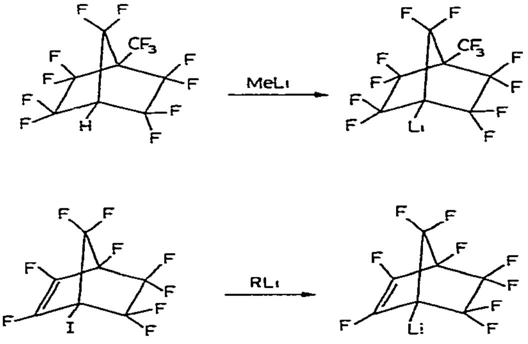
Wittig and Heyn have published further on the properties of 1-lithio-2-bromocyclopentene which is stable at room temperature [74]. Reaction with PhLi and n-BuLi yields 1-phenyl- and 1-butylcyclopropene. Evidence is presented for the occurrence of both allene and cyclopentyne intermediates.



Kobrich and coworkers have examined the stereochemistry of the thermolysis of the carbenoids LXII using ^{14}C-labels [75]. The process is postulated to occur by a rearrangement in which the aryl group trans to the halogen migrates.

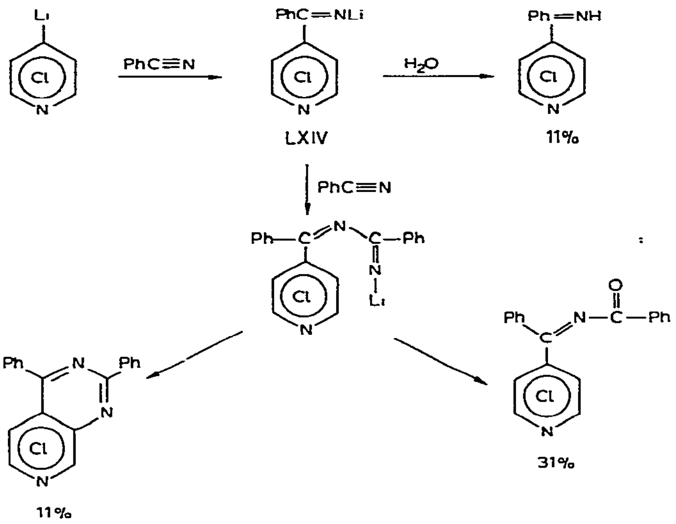


Further work on the lithium derivatives of perfluoro-bicycloheptanes has appeared [76,77]. LXIII is unusually stable.

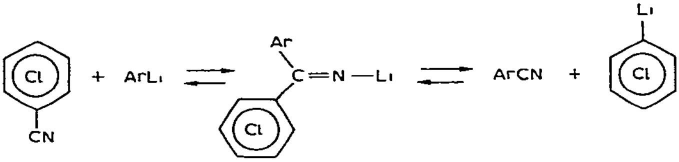


LXIII

Perhaloaryllithium compounds have been utilized to synthesize lithium pentakis (pentafluorophenyl)tungsten [78] and nonachlorobiphenyl copper complexes [79]. The reaction of chloropyridyllithium compounds with nitriles has been examined [80]. The products were not entirely those expected by analogy with the reactions of pentachlorophenyllithium.



The formation of adducts such as LXIV using C_6Cl_5Li has been reported before to be reversible [AS 70; 273]. Recently, such adducts have been prepared by an alternate route [81]. The



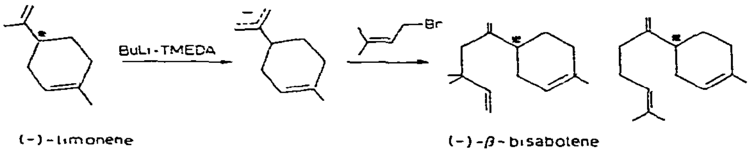
equilibrium may be displaced to the right under appropriate conditions. The net result is the formation of C_6Cl_5Li and aryl cyanide.

Tris(pentafluorophenyl)methyllithium has been prepared by the reaction of the corresponding H-compound with LiH in HMPA. The ^{19}F-spectrum of the lithium compound was reported [82].

Decafluoro- and decachloro-benzil have been prepared and reacted with alkyl- and aryllithium compounds [83]. The reactions are complex and the products depend on the lithium compound used and the reaction conditions.

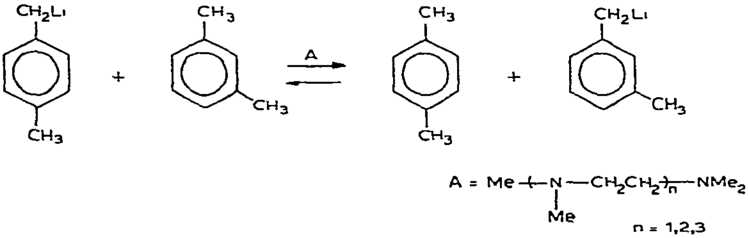
IV. Lithiations (Hydrogen-Lithium Exchange Reactions)

Crawford and coworkers have utilized n-BuLi-TMEDA metalation of limonene, followed by coupling with allyl bromides to obtain a series of bisabolene derivatives [84]. The ready availability of



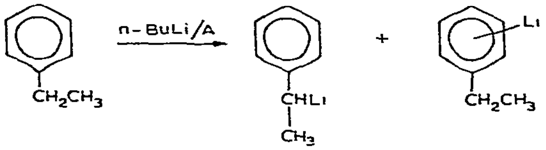
both enantiomeric forms of limonene makes this synthesis an extremely attractive method for the preparation of enantiomer β-bisabolene derivatives.

Gau has investigated the effect of di-, tri-, and tetramines on the rate of metalation reactions (2) and (3) [85]:



$$A = Me\text{-}(N(Me)\text{-}CH_2CH_2)_n\text{-}NMe_2 \quad n = 1,2,3$$

(2)

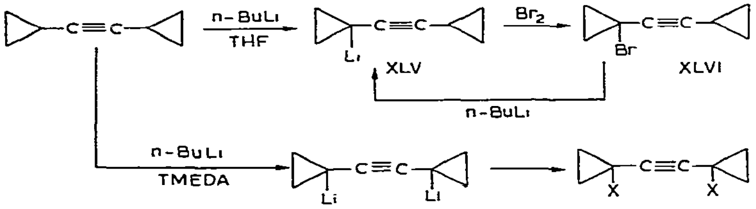


(3)

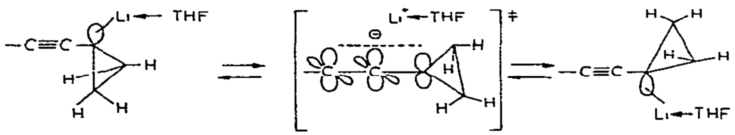
The amines were found to accelerate the reactions in the order n=1<2<3.

Klein and coworkers have found that MeLi may be used to obtain monometalation products of acetylenes which would di- or polymetalate with BuLi [85]. Thus, 1-phenyl-1-butyne gives only 37% yield of metalated product after 232 hr with MeLi/Et_2O but it is entirely the mono-lithium derivative.

The lithiation of dicyclopropylacetylene may be controlled to give either the mono- or di-lithium compounds [86]. Several derivatives of each were prepared including the bromide XLVI

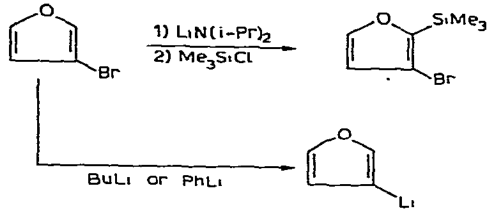


which may be used to prepare solid XLV by halogen/lithium exchange in petroleum ether. The IR spectrum of XLV is that of an acetylene rather than allene. The PMR spectrum indicates a rapid inversion of the cyclopropyl ring, perhaps due to stabilization of the transition state.



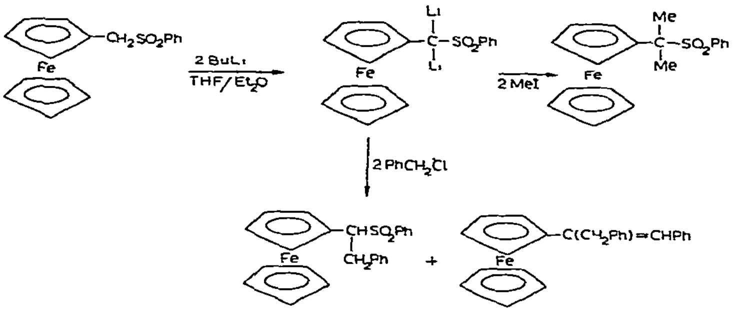
Lithiation of several heterocyclic compounds with lithium diisopropylamide has been shown to be much more specific than with organolithium compounds [87]. The method appears to be particularly useful for preparing lithium intermediates when halogen atoms are present or have been added as blocking groups.

Slocum and coworkers have shown by alternate syntheses that



chloro- and methoxy-ferrocenes are lithiated at the 2-position [88] In addition, a method has been demonstrated for the determination of the site of lithiation in anisoles [89]. After condensation with benzophenone, it was found that the chemical shifts of the ring protons were effected by the diphenylmethanol substituent in such a way that the site of attachment could be determined.

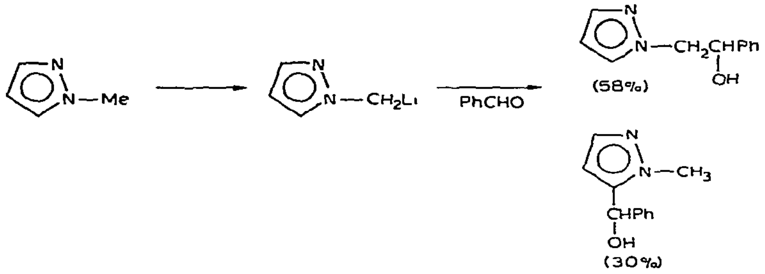
The dilithiated derivative of ferrocenylmethyl phenyl sulphone may be prepared with ease [90]. Attempts to prepare the



XLVII

dibenzyl derivative resulted in cleavage of the sulphone to the olefin XLVII.

Lithiation of n-alkyl-substituted pyrazoles has been reported [91]. Two generalizations from the work are given: (1) a 1-methyl substituent will undergo lithiation to some extent, and (2) a pyrazole with an unactivated 1-substituted will metalate at the 5-position if a 5-H is available. The following reaction is illustrative:



The lithiation and cleavage of aryl ethers by paramagnetic lithium arenes has been followed by ESR [92]. The rate constants for lithiation of anisole decrease as the electron affinity of the arene increases, suggesting that electron transfer from the lithium arene to the ether is rate-limiting. The following mechanism is visualized to account for the kinetic data and for the observed reaction products:

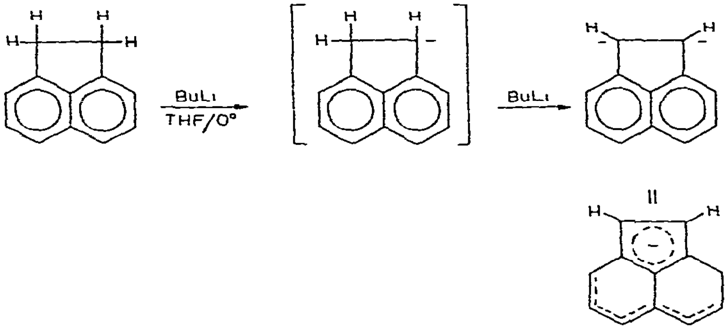
$$\mathrm{ArH}^{\dot{-}},\mathrm{Li}^{+} + \mathrm{PhOR} \xrightarrow{\text{slow}} \mathrm{ArH} + (\mathrm{PhOR})^{\dot{-}},\mathrm{Li}^{+}$$

$$(\mathrm{PhOR})^{\dot{-}} \longrightarrow \mathrm{PhO}^{-} + \mathrm{R}^{\bullet}$$

$$\mathrm{R}\bullet + \mathrm{ArH}^{\dot{-}} \longrightarrow \mathrm{R}^{-}$$

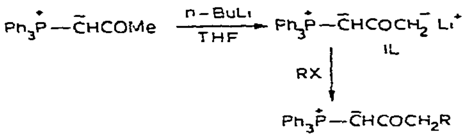
$$\mathrm{R}^{-} + \mathrm{PhOR} \longrightarrow \mathrm{RH} + {}^{-}\mathrm{PhOR} \quad \text{(o-metalation)}$$

Freedman and coworkers have discussed the factors which favor or disfavor dideprotonation of olefinic and aromatic molecules [93]. Processes such as $\mathrm{RH}^{-} + \mathrm{R'M} \longrightarrow \mathrm{R}^{-2} + \mathrm{R'H}$ usually have sizeable kinetic barriers unless the proton is far removed from the site of negative charge. Surprisingly, acenaphthene is converted to the dianion XLVIII even in the presence of one-half of an equivalent of BuLi. The authors rationalize their observations by noting that XLVIII is stabilized by cyclopentanoid character as shown in the delocalized form of XLVIII above.

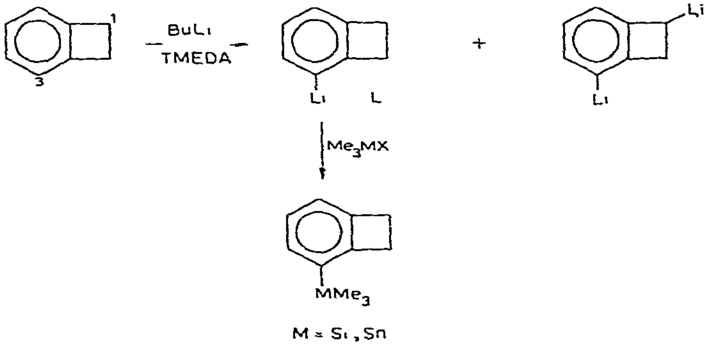


XLVIII

Also appearing during the past year were papers which describe the relative rates of ring vs methyl metalation of toluene by n-BuLi [94], and the lithiation of triphenylphosphinio acetanide to yield the new 1,3-dianion IL [95].



Eaborn and coworkers have found that 3-derivatives of 1,2-dihydrobenzocyclobutene can be prepared by lithiation with BuLi/TMEDA [96]. Some of the 1-lithio-derivative is also formed but long exposure to BuLi results in increasing yields of L.



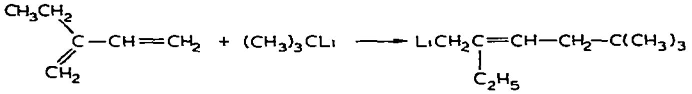
V. Polymerization Reactions

Several interesting papers have appeared the past year which relate to the nature of anionic polymerization processes. These include reviews by Fetters on the synthesis of block and graft copolymers [97], by Kennedy and Otsu on hydrogen transfer polymerization of N-substituted acrylamides [98], by Yuki [99] on alternating anionic copolymerization, and a review by Adams *et al.* on the impact of lithium initiators on the synthesis of rubbers [100]. Also published was a brief discussion of anionic polymerization by Morton [101].

Bywater and coworkers have published a very significant paper on the structure of oligomeric polybutadienyllithium [102]. Most significant were the following observations: (a) the percentage of 1,2-units in low molecular weights polybutadiene depends on the concentration of the initiator used and (b) the PMR frequencies of the γ-proton region in specifically deuterated polybutadienyllithium are determined by penultimate structure. That is to say, 1,2-units in the butadiene chain immediately back of the "living end" will effect the chemical shift of the protons in the last unit containing the lithium atom. Although these studies do not yet elucidate the mechanism of diene polymerizations initiated by RLi, it is clear that the penultimate effect must be taken into account in any final mechanistic formulation [AS 70; 265].

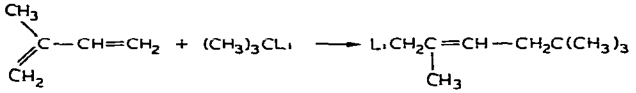
This group has also reinvestigated the degree of association of polybutadienyl-, polyisoprenyl-, and polystyryllithium in cyclohexane [103]. The results indicate the first two living polymers are tetrameric while the latter is dimeric. The authors point out the difficulties inherent in the use of inherent viscosities to determine molecular weights of living polymers.

Schue' and coworkers have examined the living and/or terminated oligomers from the following dienes: 2-ethyl-1,3-butadiene LI [104], *trans*-2-methyl-1,3-pentadiene [105,106], and 2-phenylbutadiene [107]. The PMR spectrum of the 1:1 adduct of LI with t-butyllithium indicates predominant 4,1-addition with no evidence for any living 4,3-unit. Similarly, 2-methyl-1,3-butadiene gives a 1:1 adduct whose PMR spectrum indicates only 4,1-addition:



LI

cis . *trans* = 52 : 38

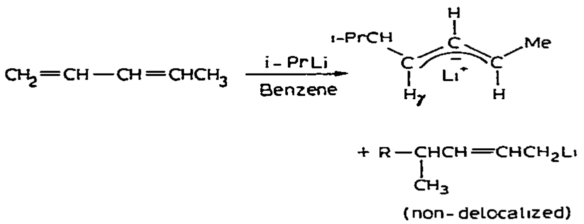


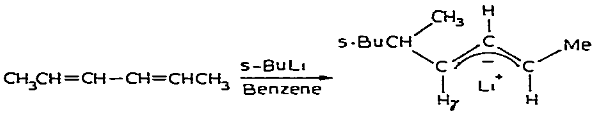
In both of these cases, the terminated oligomers have 4,3-content the mechanism of the formation of which still is not established.

Morton and coworkers have studied the living oligomers of 1,3-pentadiene and *trans*-*trans*-2,4-hexadiene [108]. The γ-proton of both species was found upfield of its usual position indicating to the authors that the living end is delocalized even in benzene.

Essel and Pham have examined the microstructure of polyisoprene obtained with lithium naphthalene radical anion initiator [109]. The authors contend that free ions and contact ion pairs are the responsible active species in DME and dioxane respectively.

The relative rates of polymerization of methyl-, ethyl-, and n-propyl-isoprene carboxylates have been studied using n-BuLi/toluene initiator at -52° [110]. $(CF_3)_2C{=}CH{-}CH{=}CH_2$ has been



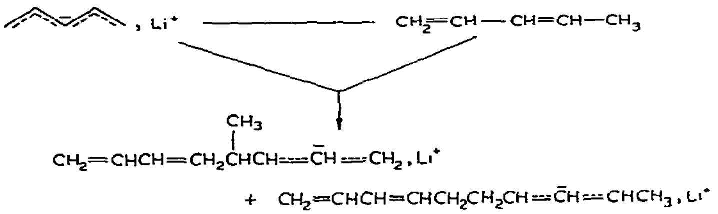


polymerized with butyllithium catalysts in THF to yield a crystalline polymer of unknown microstructure [111]. Likewise, trans-CNCH=CH-CH=CH_2 is rapidly polymerized by n-BuLi in toluene or THF to give a polymer with predominantly trans-1,4 content [112]. Copolymers of this material was also reported. The synthesis and properties of butadiene rubber using n-BuLi, dilithionaphthalene and lithiopolybutadiene catalysts has also been described [113]. Other papers describe the effect of prenyl chloride on the molecular weights and conversions of lithium initiated isoprene [114]; the effect of temperature [115], t-BuOK [116], and Et_4Sn [117] on the reactivity ratios of styrene and butadiene; the formation of alternating copolymers of styrene and 2,3-dimethylbutadiene in THF and benzene [118]; and a similar paper on the copolymers formed from styrene and α-methylstyrene in the presence of polyethers and HMPA [119]. Also, papers have appeared on the formation of ABA block copolymers of styrene and butadiene/isoprene with sec-BuLi [120], on the polymerization of methacrylic esters with alkali metal biphenyl complexes [121], on the polymerization of 1-vinylpyrene [122, see Sodium and Potassiu Annual Survey], on the block copolymerization of isoprene with p-t-butylstyrene, 2,4-dimethylstyrene and 2,5-dimethylstyrene

[123], on the use of aryl dilithium initiators [124], and on the effect of 2,3-dimethoxybutane on the content of polyacrylonitrile-methyl methacrylate initiated by BuLi/toluene [125].

As many of these papers indicate, there is great interest in the use of polar modifiers such as HMPA, amines and polyethers in lithium-initiated polymerizations. Tate and coworkers have examined the effect of temperature on the vinyl content of polybutadiene in the presence of such modifiers, and have developed a method to ascertain the proper conditions for the preparation of a polymer of prescribed composition [126]. Other workers have examined the effects of polydentate amines on the polymerization of methyl and cyclohexyl methacrylates [127]. Also studied were the effects of TMEDA and the lithium salt of 2-ethoxyethanol on the telomization of butadiene in the presence of diphenylmethane [128]. The principal products in the latter case are 5,5-diphenyl 1-pentene.

Newcomb and Ford have used a novel technique to determine the stereochemistry of the addition of pentadienyllithium and benzyllithium to 1,3-pentadiene in THF [129]. Weak acids such as furan and toluene compete with addition. Thus, 1,3-pentadiene is generated by HA from pentadienyllithium and may react to form dimers or may add benzyllithium (in the case of HA = toluene). Dimers of pentadienyllithium with 1,3-pentadiene produced by this method contain approximately equal amounts of 1,4 and 4,1 addition. The following scheme illustrates the method (in fact, the dimers were isolated after closing to cyclohexyl derivatives).

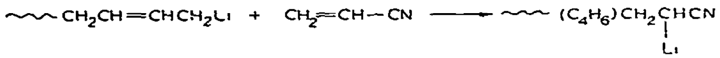


Japenese workers have studied the effect of polar modifiers on the anionic copolymerization of styrene and butadiene. Compounds studied include ethers and amines [130], pyridines, picolines, <u>etc</u>. [131], and group IIA alkoxides [132]. Incorporation of styrene is generally increased by such modifiers.

Mochel has investigated the ^{13}C-NMR spectra of n-BuLi catalyzed polybutadienes [133]. Surprisingly, the results indicate that the polymer consists of blocks of <u>trans</u>-1,4-polybutadiene and of <u>cis</u>-1,4-polybutadiene with intervening 1,2-units. A mechanistic explanation of this data has not been offered as yet.

The thermal decomposition of polybutadienyllithium in hexane is a rather complex process which includes elimination of lithium hydride, metalation and additions [134]. Reaction pathways are proposed to account for the observed effects.

Several other papers have appeared concerning the modification of polymers by post-treatment. These include the attachment of carboranyl and ferrocenyl groups to polyvinyl chloride by coupling with the corresponding lithium reagents [135], the coupling of polybutadienyllithium with toluene diisocyanate [136], the cleavage of polyphenyl ethers with lithium biphenyl complex [137], and the cleavage of polyethers, polysulfides and polyimines by n-BuLi and PhLi [138]. Also reported was the reaction of acrylonitrile with polybutadienyllithium:



$$\sim\sim CH_2CH{=}CHCH_2Li + CH_2{=}CH{-}CN \longrightarrow \sim\sim (C_4H_6)CH_2\underset{Li}{\underset{|}{C}}HCN$$

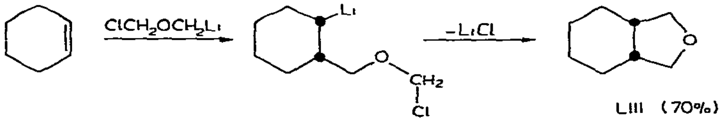
LII

The uv and conductimetric behavior of species such as LII was reported [139].

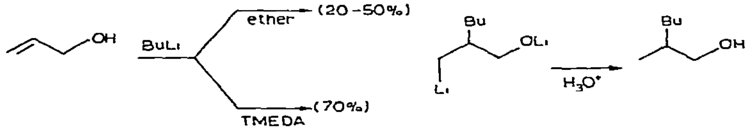
Also, crosslinked microgel particles (polystyrene with divinylbenzene) were reacted with BuLi to give highly colored particles [140].

VI. Addition Reactions to Carbonyls, Olefin and Similar Substrates

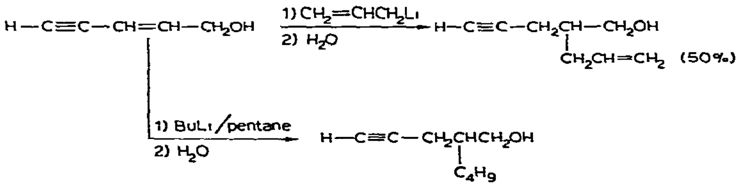
This section lists papers which describe addition reactions other than polymerizations. Groen and Jacobs have found that chloromethoxymethyllithium adds to the double bond of cyclohexene in a cis fashion [141]. The adduct then closes to form LIII:



The addition of organolithiums to allyl alcohols has been described using a number of different RLi, alcohols, solvents, and temperatures [142]. Yields were increased markedly by the addition of TMEDA.

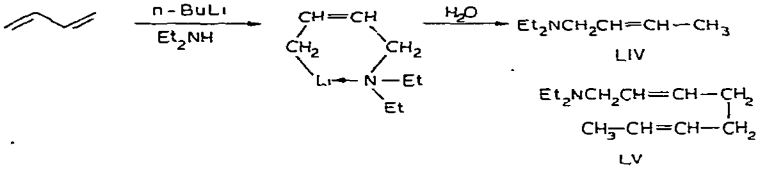


Similarly allyllithium and BuLi add to pent-2-ene-4-yne-1-o-1 as follows [143]:



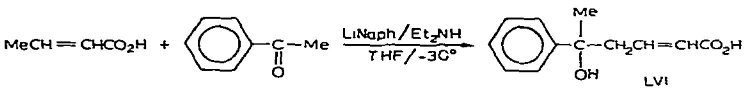
Bal yan and coworkers have continued to exploit the addition of organolithiums to enynes [144, AS, 69; 35]. Pearce and coworkers have described conditions for the addition of alkyl- and aryl-lithiums to carbonyl compounds [145]. The organolithium compounds may be generated from the appropriate halide in THF in the presence of the carbonyl compound, thus allowing a one-step procedure. In most cases yields are quite good.

1,3-Butadiene, n-BuLi and diethylamine react to give, after ethanolysis, the amine derivatives LIV and LV [146]. Similar telomers were formed with isoprene and with Me_2NH.

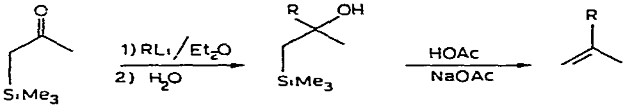


Watanabe and coworkers have reviewed their recent work on the synthesis of terpene compounds using lithium naphthalene and isoprene [147]. Using similar methods, French workers have synthesized oligomers of isoprene and butadiene [148].

This technique has now been extended to include the dimerization of indene, styrene and α-methylstyrene with lithium naphthalene in the presence of ethylene glycol monobutyl ether [149]. Also, in the presence of lithium naphthalene and diethylamine, crotonic acid is caused to add to ketones to give the corresponding hydroxy acids [149,150]. Thus, for example, acetophenone gives 53% yield of LVI:

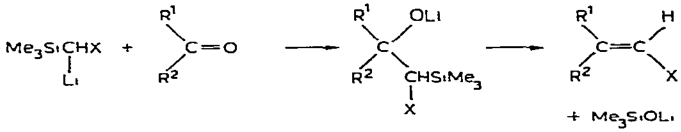


The addition of organolithiums to β-ketosilanes has been used to prepare the corresponding β-hydroxysilanes or olefins under appropriate conditions [151]:



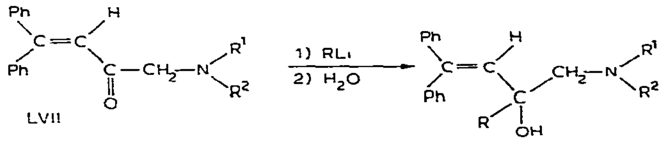
R = n-Bu, n-C_5H_{11}, Ph

Also reported was the reaction of new trimethylsilyl-substituted alkyllithiums with ketones to yield olefins [152]:



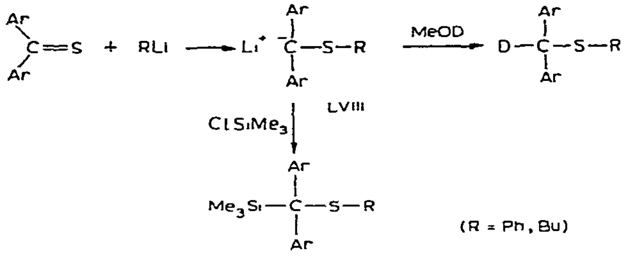
X = SPh, P(O)(OEt)$_2$

Others have examined the addition of RLi to benzophenone [153] and to LVII in which 1,2-addition is the primary process [154]:



LVII

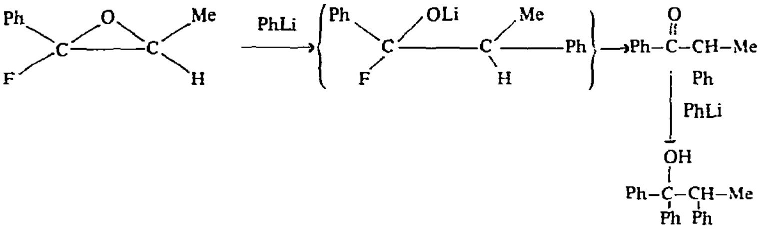
Further information on the thiophilic addition of RLi to thioketones has appeared [155, AS 70; 291]. The intermediate LVIII was intercepted with several reagents.



LVIII

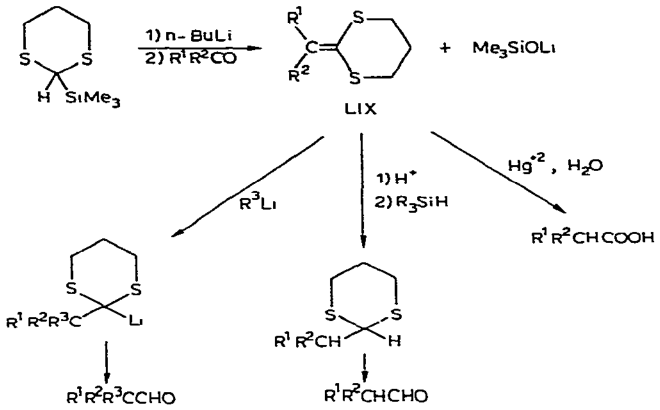
(R = Ph, Bu)

New data on the reaction of PhLi with halogenated epoxides has also appeared [156, AS 71; 37]. Fluoroepoxides were shown to react as shown below.



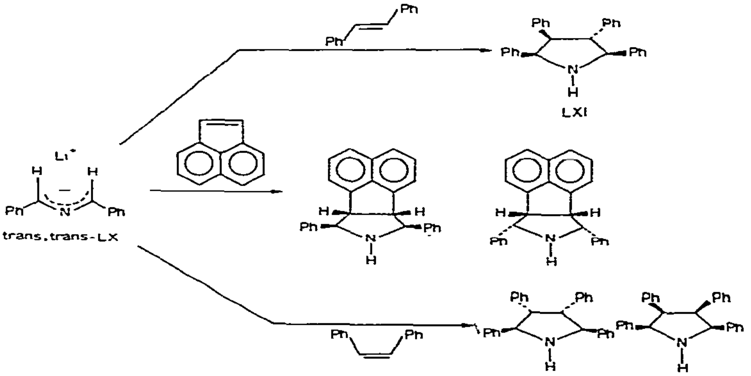
The reaction is rapid and clean in the case of the cis-isomer, but is accompanied by other side-reactions if the more sluggish trans-isomer is used.

Several papers have appeared on the use of lithio-1,3-dithianes for synthetic purposes. Carey and Court [157] and Jones and Lappert [158] have utilized 2-lithio-2-trimethylsilyl-1-3-dithianes for the preparation of ketene thioacetals by reaction with carbonyl compounds. The synthetic utility of LIX is considerable as shown below:



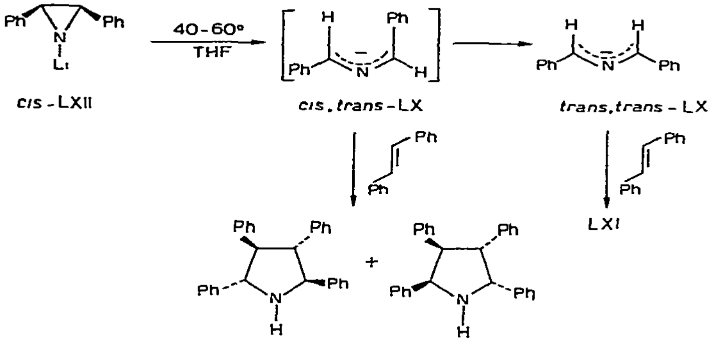
Other workers have utilized lithio-dithianes in model steroid syntheses [159,160].

Kauffmann and coworkers have continued their study of the cycloaddition reactions of 1,3-diphenyl-2-azaallyllithium [AS 71;11]. The reaction of LX with three olefins was shown to be a stereospecific cis-addition [161].

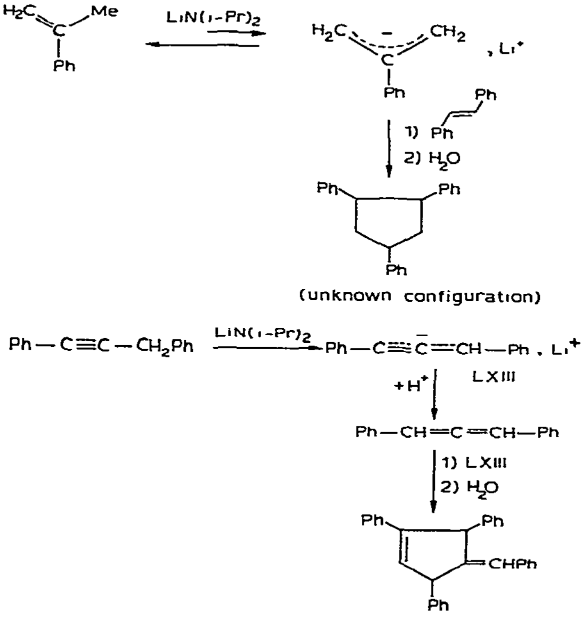


The most stable conformation of LX is the trans-trans structure as shown. However, the cis,trans form may be trapped if generated at -60° from N-benzylidenebenzylamine. cis , trans-LX may also be generated by ring opening of N-lithio-2,3(cis)-diphenylaziridine

LXII, but isomerization to the trans,trans isomer occurs in the absence of a trapping agent [162].

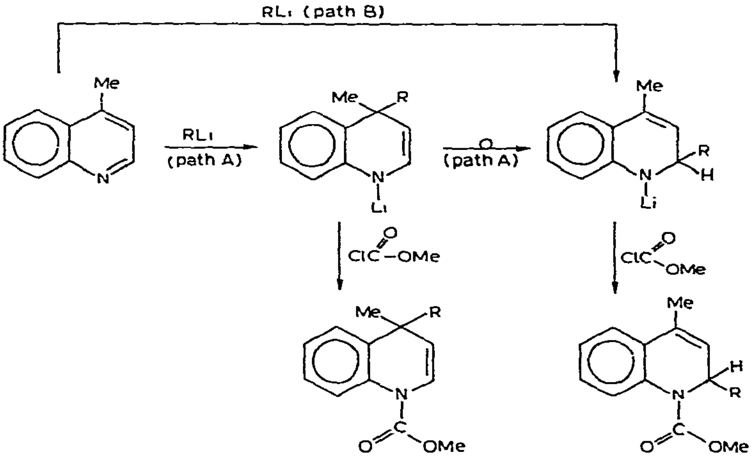


1,3-Cycloadducts of the 2-phenylallyl anion may also be prepared using a similar method [163]. Likewise, cycloadducts of the 1,3-diphenylpropyne anion have been observed.

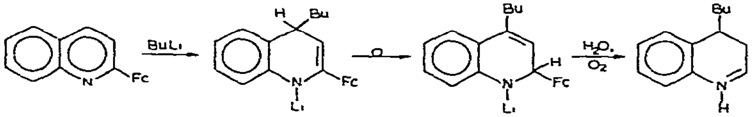


VII. Reactions with Heterocyclic Compounds

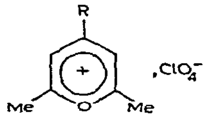
Two groups have reexamined the addition products of PhLi and BuLi to 4-methylquinoline [164]. They refute the recent claim of Japanese workers [AS 71; 30] that the reaction is actually a 1,4-addition process followed by rearrangement to the usually observed 1,2 product (path A). NMR spectra of the methyl chloroformate adducts of better resolution are reported in the latest papers which the authors contend can only be interpreted in terms of structure LXV rather than LXIV. On the other hand, other workers have claimed evidence for 1,4-adducts of BuLi with 2-ferrocenylquinoline [166]. A 4,2-hydrogen shift to the observed product is proposed.



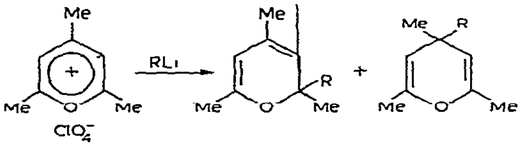
LXIV LXV



Other papers have described the synthesis of 2,6-dimethylpyrylium salts LXVI [167] by the treatment of 2,6-dimethyl-γ-pyrone

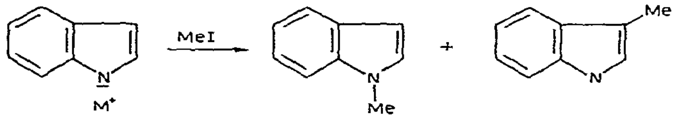


LXVI

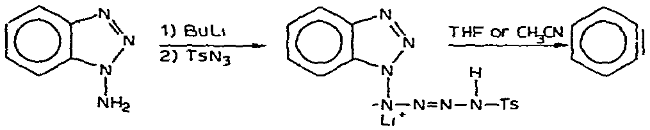


with RLi, and the action of various RLi on such salts [168]. 1,2- and 1,4-addition products are observed, the distribution of which is slightly effected by the presence of TMEDA. Also reported was the formation of imidazole derivatives by the action of oxygen on lithium aldimines [169]. Similarly, PhLi with Me$_3$CNC gave 30% of the dimer Me$_3$CN=CPhCPh=NCMe$_3$ upon oxidation.

Reinecke and coworkers have examined the effect of cation and solvent variation on the reaction of indole salts LXVII with MeI [170]. In the case of M=Li, the percentage of C-methylation changes from 44 to 85 to 91 as the solvent is changed from THF to Et$_2$O to toluene.

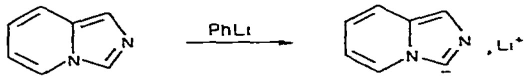


The lithium salt of 1-aminobenzotriazole (LXVIII) couples with toluene-p-sulphonyl azide to give LXIX which immediately forms benzyne when dissolved in THF or acetonitrile [171]:

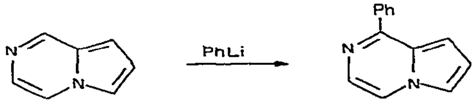


LXVII LXIX

Imidazo[1,5-a]pyridine LXX and pyrrolo[1,2-a] LXXI react differently with PhLi [172] as shown below:



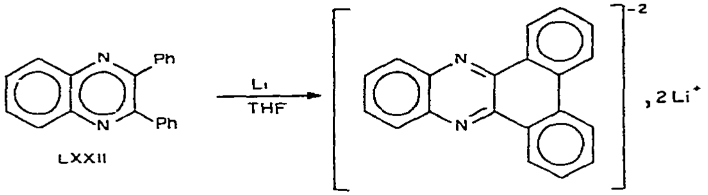
LXX



LXXI

Other examples of the reaction of PhLi with polyazaindenes are reported in the same paper.

Reaction of lithium metal with 2,3-diphenylquinoxaline LXXII results in the formation of the dibenzo[a,c]phenazine ring system several derivatives of which were prepared [173]:

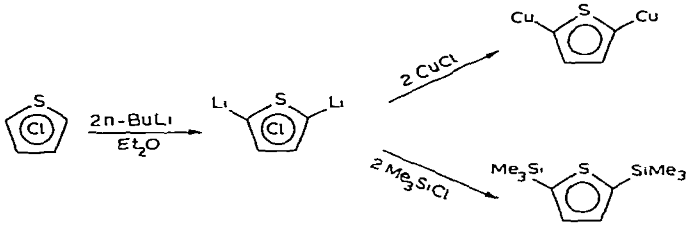


LXXII

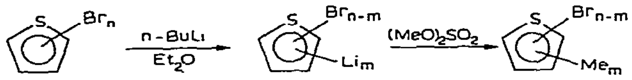
Also reported was the reductive cleavage of piperidinium salts with lithium-biphenyl adduct [174].

Lithiated benzofurans have been used as access to various functional derivatives [175]. Similarly, Thames and coworkers have utilized lithium derivatives to prepare a series of furan and thiophene derivatives containing both silicon, thiophosphate and thiophosphonate functionalities [176]. Mathey has studied the reactions of t-BuLi [177] and n-BuLi [178] with 1-phenylphospholes in which phenyl is displaced by the corresponding butyl group.

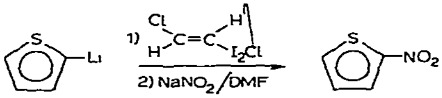
Several papers have appeared concerning the lithium derivatives of thiophenes. These include 2,5-dilithiotetrachlorothiophene which may be prepared in ether in good yield [179]. Similarly, polybromothiophenes have been metalated to form several di- and



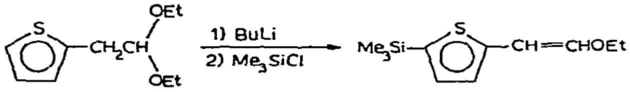
polylithiothiophenes which were subsequently methylated [180]:



Lithium derivatives have been used to prepare nitrothiophenes [181]:

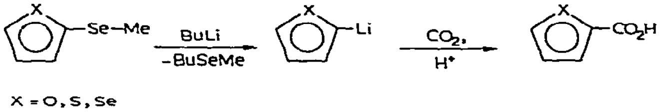


Treatment of the thiophene derivative LXXIII with BuLi results in lithiation at the 5-position as well as stereospecific elimination in the acetaldehyde diethylacetal functionality [182]:

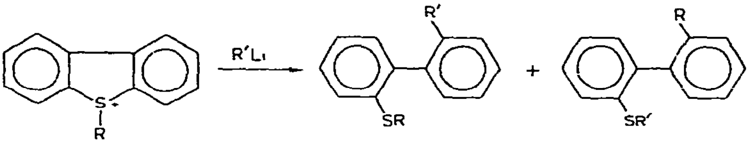


LXXIII

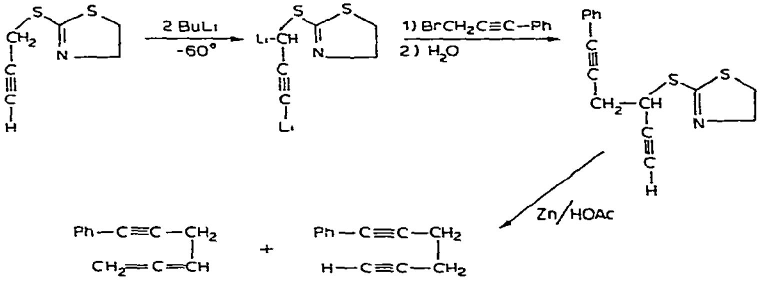
2-Methylselenyl-derivatives of furan, thiophene and selenophene are cleaved and lithiated simultaneously by BuLi [183]:



Also reported during the past year was the cleavage reaction of dibenzothiophenium salts with BuLi [184]:



A new 1,5-diyne synthesis using lithiated 2-propargylthiothiazoline has been described [185]:

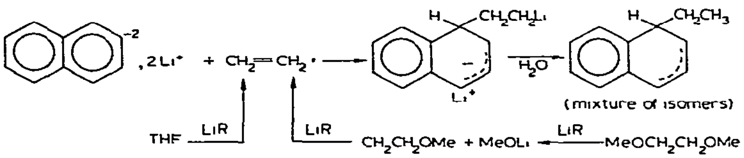


Hakansson [186] has discussed the racemization of optically active 2,2'- or 4,4'-dibromo-3,3'-bithienyls with alkyllithiums.

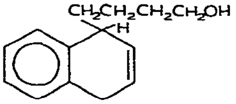
VIII. Miscellaneous Organic Reactions

J. M. Brown has reviewed the use of organolithium reagents in organic synthesis [187].

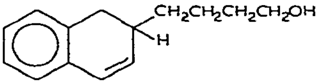
Two papers have appeared on the reaction of lithium naphthalene compounds with THF. In one paper the active species is identified as the naphthalene dianion [188] with the ethylene coming from the decomposition of THF [see reference 42 above]. In



fact, ethylene may be added directly or may also come from the cleavage of DME as shown above. Watanabe and coworkers have identified two different products LXXIV and LXXV from a similar system [189].

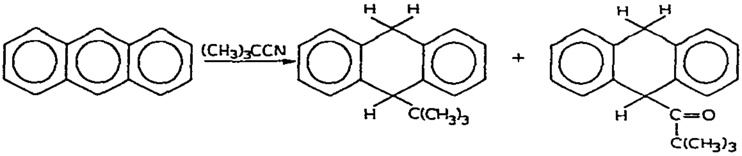


LXXIV



LXXV

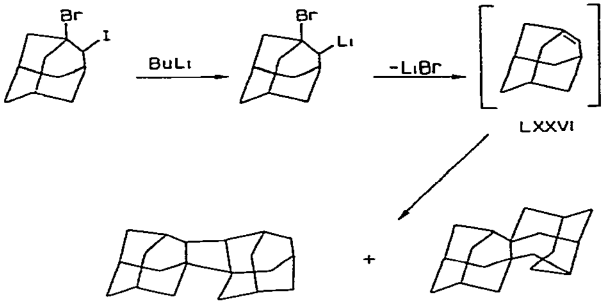
Also reported was the reaction of lithium anthracene with pivalo nitrile [190]. The ketone product presumably is formed by



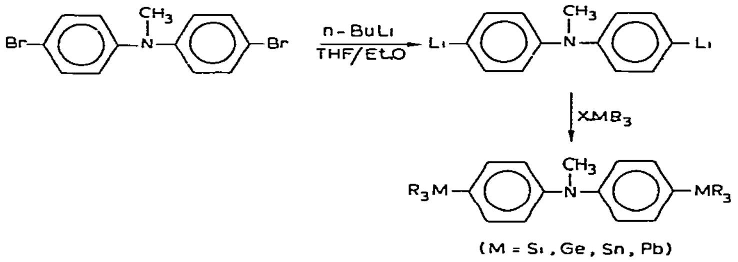
hydrolysis of the corresponding imine.

Smith and coworkers have reported further on the reaction of the benzophenone anil dianion with isopropyl halides [191, AS 70, 328].

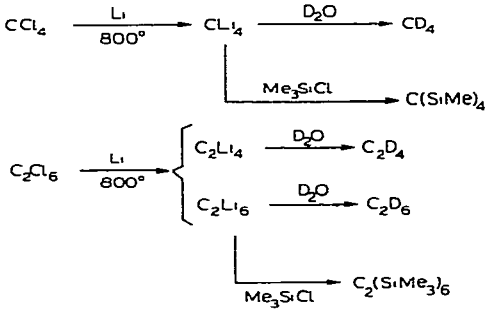
1-Lithioadamantane has been prepared from the corresponding iodide by halogen/lithium exchange [192]. Also, reaction of butyllithium with 1-bromo-2-iodoadamantane yields two dimeric hydrocarbons the formation of which was rationalized in terms of the strained olefin intermediate LXXVI [193].



Halogen/lithium exchange also has been utilized to prepare bis-(4-lithiophenyl)-N-methylamine and its various derivatives [194]:



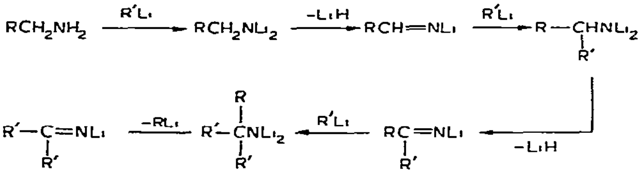
An interesting paper has appeared on the reaction of lithium atoms at 800° with chlorocarbons [195]. Evidence for CLi_4, C_2Li_4 and C_2Li_6 was given.



Screttas has shown that alkyl- and aryl-lithiums can be prepared by the reaction of the corresponding chloride (or fluoride in the case of PhLi) with lithium dihydronaphthylide [196]:

$$RX + 2Li^{+}ArH^{\overset{.}{-}} \xrightarrow[-100^{\circ}]{THF} RLi + LiX + 2ArH$$

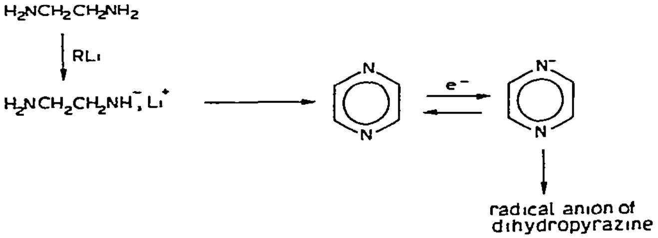
Non-hydrolytic work-up of the reaction mixture from alkyllithiums and primary amines has yielded imines [197, AS 71, 35].



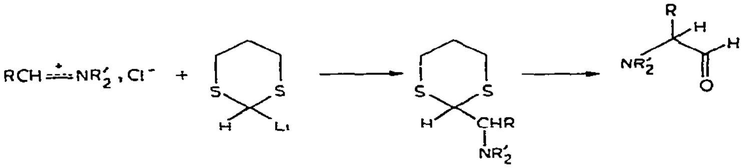
Thus, with R = n-C_5H_{11}, R' = n-Bu the yield of $CH_3(CH_2)_4\overset{\overset{NH}{\|}}{C}CH_2$-$(CH_2)_2CH_3$ is 33% with MeOH work-up. With R = $(CH_3)_3CCH_2$, R' = t-Bu, the yield of $(t\text{-}Bu)_2C{=}NH$ is 48%.

Bach and coworkers have reexamined the reaction of PhLi with trimethylcyclooctylammonium bromide reported earlier by Wittig [198]. Deuterium labeling experiments established that the reaction is an intramolecular syn α',β process.

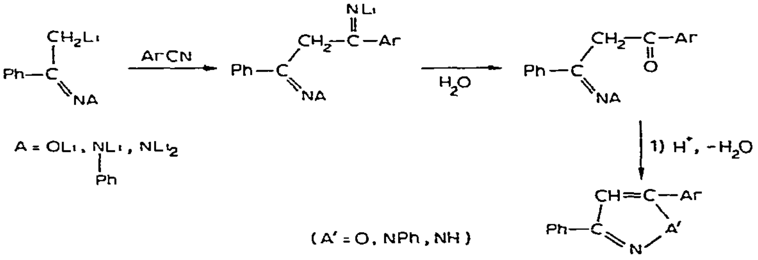
Wotiz and coworkers have examined the reaction of alkyllithiums or Li metal with ethylenediamine [199]. The pyrazine radical anion is formed which then goes to a dihydropyrazine radical.



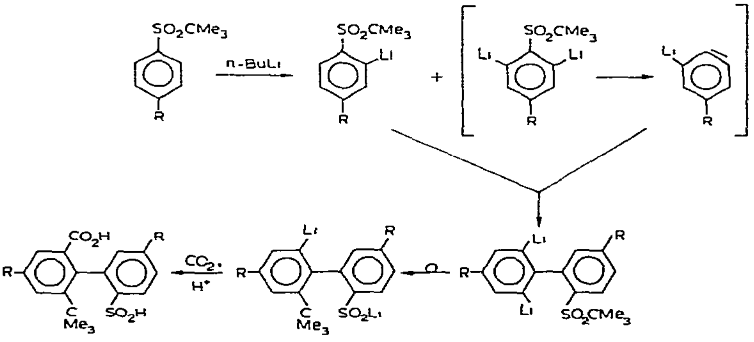
Immonium chlorides react with 2-lithio-1,3-dithiane to give α-aminothioacetals which are precursors of α-amino-aldehydes [200].



A convenient new procedure for the preparation of isoxazoles and pyrazoles has been described [201]:

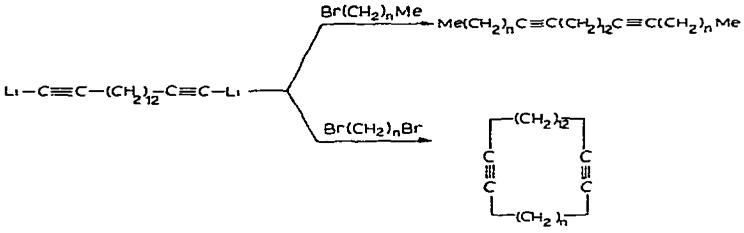


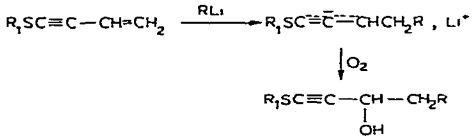
Also reported was the reaction of t-butylphenylsulfone with n-BuLi. The final product LXXVII was explained by postulating a lithio-benzyne intermediate [202]:



LXXVII

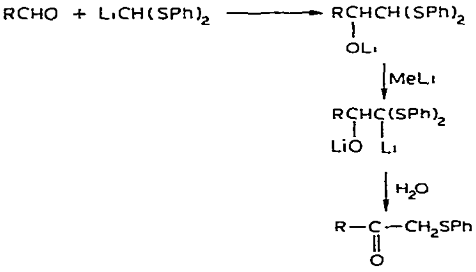
Also reported the past year were the following: alkylation of the dilithium salt of hexadeca-1,15-diyne with $Me(CH_2)_nBr$ and $Br(CH_2)_nBr$ [203], the oxidation (with O_2) of the adducts of lithium alkyls with thiovinyl acetylenes [204]; and the reaction of lithium trityl mercaptide with BuLi [205]:



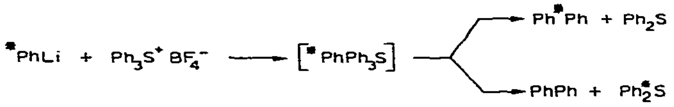


$$Ph_3CSLi + BuLi \longrightarrow Ph_3CLi + BuSLi$$

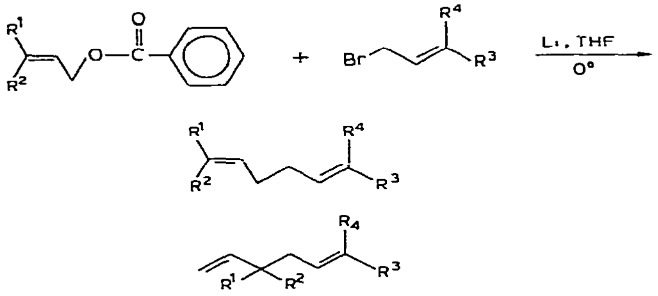
Also phenylthiomethyl ketones have been prepared by the following reaction [206]:



Sulphuranes (R_4S) have been identified as the principal intermediates in reaction of aryllithiums with sulphonium salts [207]. ^{14}C-labeling of the PhLi yielded products with equal radioactivity in its reaction with $Ph_3S^+BF_4^-$, as expected for a symmetrical intermediate.



Allyllithium compounds have been generated *in situ* by the lithium-cleavage of allyl mesitoates [208]. In the presence of allyl bromides, rather good yields of coupling products are observed; however, the method is not suitable for the preparation of allyllithiums since they are consumed by further reaction with the ester.

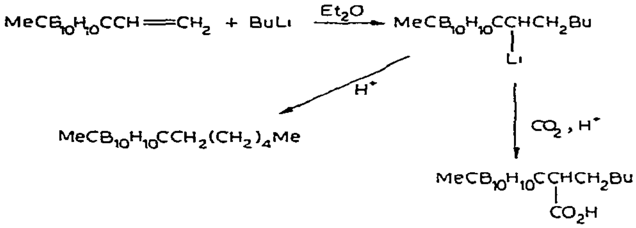


The reactions of neopentylallyllithium [see reference 12 above] have been studied in hydrocarbon media [209]. Allylic rearrangement is not observed to any significant extent in protolysis and silylation reactions. However, reaction with ketones and CO_2 does yield some rearranged product.

IX. Reactions with Inorganic and Organometallic Compounds

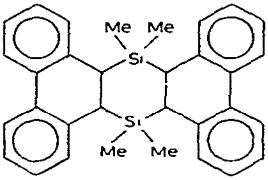
Zakharkin and coworkers have continued to study the chemistry of lithiated carboranes. Most recently, they have examined reactions with hexafluorobenzene and pentafluorochlorobenzene [210]. Whereas C_6F_6 caused lithio-1-methyl-o(and m)-carborane to form dimeric compounds, e.g. bis(methyl-o-carborane), C_6F_5Cl coupled to form (2,3,5,6-tetrafluoro-4-chlorophenyl)-substituted carboranes.

BuLi adds to vinyl- and isopropenyl-o-carboranes as follows [211]:



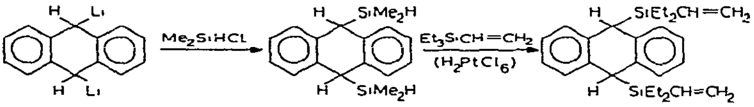
The use of organolithium compounds for the preparation of organosilicons is a well established method, of course, and all examples of the use of this method cannot be covered here. We note only the following papers which are illustrative.

Russian workers have prepared some interesting cylic compounds such as LXXVIII from 9,10-dithio-9,10-dihydrophenanthrene and Me_2SiCl_2 [212]:



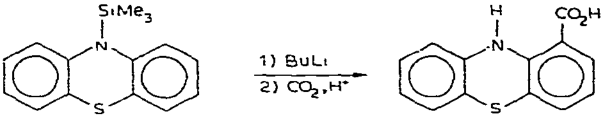
LXXVIII

Similarly, 9,10-dilithio-9,10-dihydroanthracene has been converted to the vinyl silane LXXIX by the following sequence [213]:



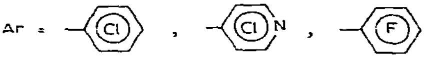
LXXIX

Metalation of organics containing silyl groups often causes cleavage of the Si-C or Si-N bond as the following examples illustrate:

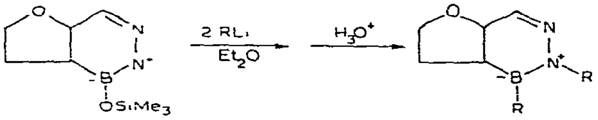


[214]

$$\mathrm{ArSiMe_2H + RLi \longrightarrow ArLi + RSiMe_2H} \qquad [215]$$



Cleavage of silyl ethers by alkyllithiums has been used as a route to B-substituted boron heterocyclics [216]:



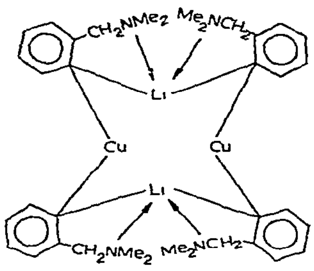
The reaction of arsonium salts with organolithium compounds yields a variety of products. [217]:

$$\mathrm{PhCH_2Li + Ph_4AsBr \xrightarrow{THF} Ph_2 + Ph_2(CH_2)_2 + AsPh_3 + PhH}$$

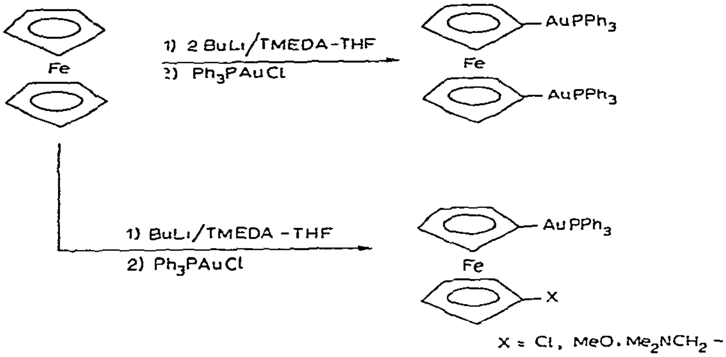
$$\mathrm{MeLi + Ph_4AsBr \xrightarrow{THF} CH_4 + AsMe_3 + Me_2PhAs + MePh_2As}$$

Two mechanistic schemes were postulated to account for these and other minor products.

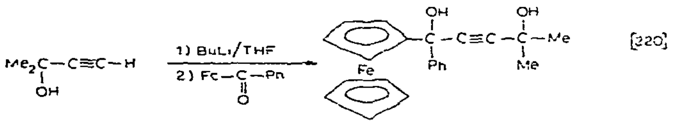
Although the chemistry of lithium-copper organic compounds is not covered in this section, we do note here the structure of the complex bis-{2-[(dimethylamino)methyl]phenyl}copper (I) lithium shown below, as determined by ^{13}C and ^{1}H NMR [218]:

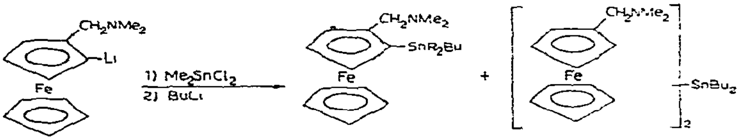


Several papers have appeared concerning the reactions of organolithium compounds with transition metal complexes. These include the preparation of ferrocene organogold compounds [219]:



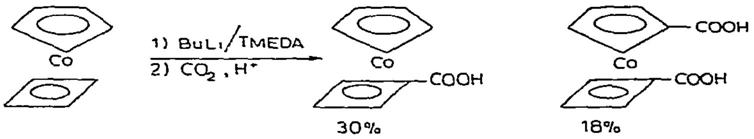
Other ferrocene derivatives were formed as shown below:



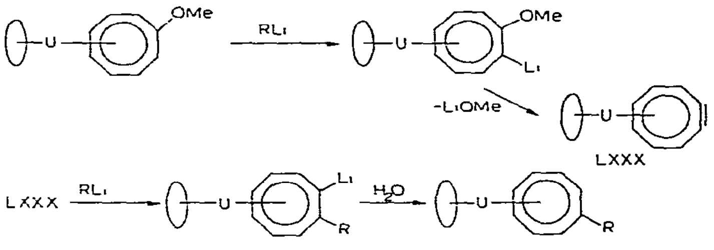


The latter reaction utilizes the dimethylaminomethalferrocenyllithium reported in earlier work by these authors [221].

Cyclobutadiene(cyclopentadienyl)cobalt is lithiated by BuLi/TMEDA on the cyclobutadiene ring in preference to the cyclopentadienyl ring and is much less reactive than ferrocene [222]:



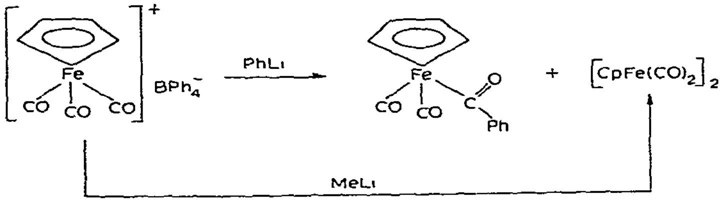
The reaction of 1,1'-dimethoxyuranocene with alkyllithiums has been reported as the first example of ring substitution of a uranocene compound [223]. A cyclooctatrienyne complex LXXX is proposed as the most reasonable intermediate in a metalation-elimination-addition mechanism:



(R = n, t-Bu, but not Ph, Me, H_2N)

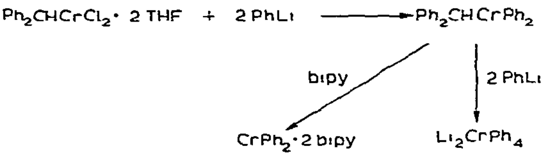
Similar substitution reactions of the bistrimethylammonium iodide, $U(C_8H_7NMe_3{}^+I^-)_2$ were reported to be more facile than those described above.

Also reported was the reaction of $V(C_5H_5)_2Ph$ and PhLi in Et_2O which gave a mixture of reduction products including Ph_2, C_6H_6 and complexes of the type $Ph_2V{\cdot}nPhLi{\cdot}mEt_2O$ [224]. Reactions of RLi with cyclopentadienyliron tricarbonyl cation also give a mixture of reduction and carbonyl addition products [225]. Two examples are shown below:



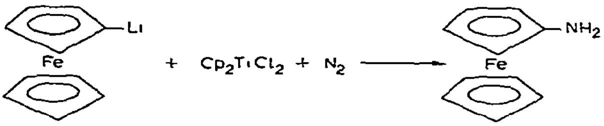
Dicyclopentadienyldibenzyltitanium with $PhCH_2Li$ gives C_5H_4Li, MePh, $(PhCH_2)_2$ and $(C_5H_5)Ti(PhCH_2)_2$ [226]. Similar reactions are reported with MeLi.

Diphenylmethylchromium dichloride $Ph_2CHCrCl_2 \cdot 2THF$ reacts with PhLi to give $Ph_2CHCrPh_2$ which has been used as a route to other aryl chromium compounds [227]:



Nitrogen fixation has been reported using two RLi-transition metal systems in the past year:

$$FeCl_3 + PhLi + N_2 \xrightarrow{Et_2O} \xrightarrow{HCl} N_2H_4$$ [228]



[229]

Metathesis of 2-pentene has been observed at room temperature with the system $BuLi-WCl_6$ [230].

Sarry has reported more data on the complexes formed between iron (III) chloride and PhLi in Et_2O. Under mild conditions the product is $Fe(LiPh)_5$, a benzene soluble, thermally unstable substance [231]. Several reactions of this product are given but its structure is not yet elucidated.

Finally, we note the appearance of two papers on the analysis of organolithium compounds. Reed and Urwin have described in more detail their method for the preparation and UV analysis of high purity n-BuLi, sec-BuLi, 1,4-dithio-1,1,4,4-tetraphenylbutane [232, AS 68; 209]. The paper is an excellent reference for those who wish to prepare and handle organolithium reagents under rigorously clean conditions.

Ellison and coworkers have described a direct titration of organolithiums with sec-BuOH at -78° [233, AS 67; 161]. The decomposition of THF and 1,2-dimethoxyethane was followed using this technique.

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