HEAVIER ALKALI METALS

ANNUAL SURVEY COVERING THE YEAR 1979 *

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1. PHYSICAL ORGANIC CHEMISTRY

A review article described the current status of research in the field of anions of sodium, potassium, rubidium, and cesium [1]. The author speculates on possible future developments with these highly reduced species in the -1 oxidation state.

X-Ray diffraction of sodiotriphenylmethane complexed with tetramethylethylenediamine (TMEDA) has revealed that the carbanion is planar in its central part but has twisted and elongated phenyl groups [2]. The structure, shown in Fig. 1, can be described as a contact ion pair of the carbanion and a sodium ion coordinated to the bidentate ligand.

As part of a larger study involving adducts of cyclopentadienylsodium with oxygen and nitrogen bases, full details have been provided on the X-ray data for the above carbanion complexed with TMEDA [3]. In the crystal, the adduct adopts a puckered chain structure as shown in Fig. 2. Elemental analysis data and NMR and IR spectra for each of the adducts are listed.

Another X-ray study on potassium-, rubidium-, and cesium isopropoxides has shown these compounds to be isostructural where the metal atoms lie in one plane and are coordinated on both sides by oxygen atoms [4]. The

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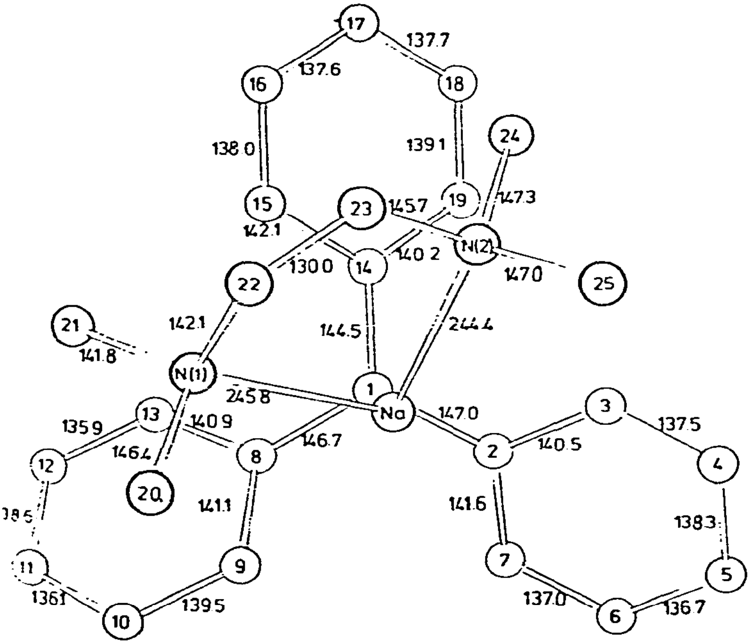
* Heavier alkali metals, Annual Survey covering the year 1978 see J. Organometal. Chem., 183 (1979) p. 107 - 140.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137.7
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23
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N(2)
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25
22
21
142.1
144.5
244.4
141.8
N(1)
1
245.8
Na
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3
13
140.9
135.9
146.7
2
140.5
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12
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20
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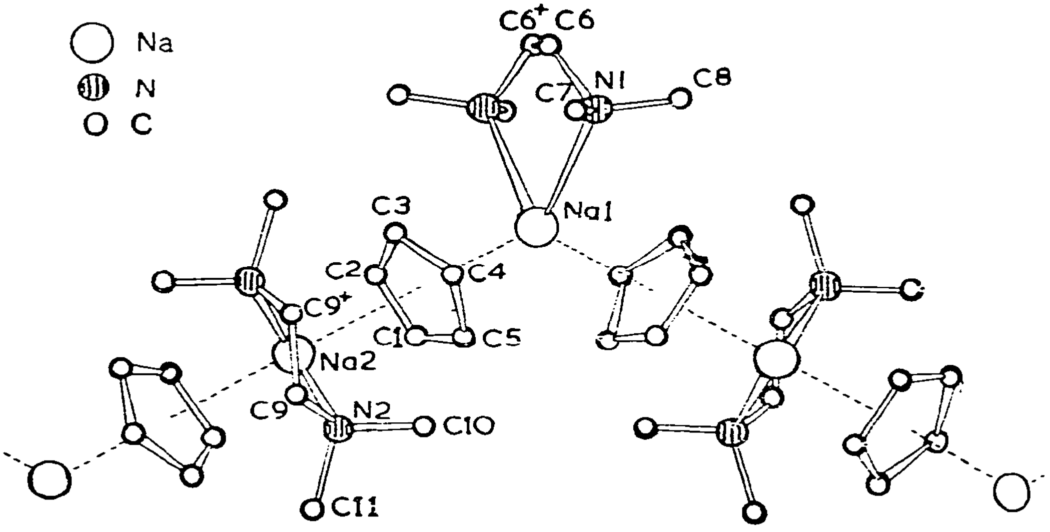
Fig. 1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N
C
C6+ C6
C7
N1
C8
Na1
C3
C2
C4
C9+
C1
C5
Na2
C9
N2
C10
C11

Fig. 2

structure of cesium isopropoxide is shown in Fig. 3 (reproduced by permission of Chem. Ber.).

Carbon-hydrogen bond energies for H_3COH, H_3CONa, H_3COK, and H_3CO^- have been calculated using ab initio generalized valence bond and configuration interaction theoretical methods in an effort to explain oxy anionic substituent effects in reactions such as the oxy-Cope rearrangement [5]. The values determined for the above compounds were 90.7, 80.6, 79.0, and 74.2 kcal/mol, respectively.

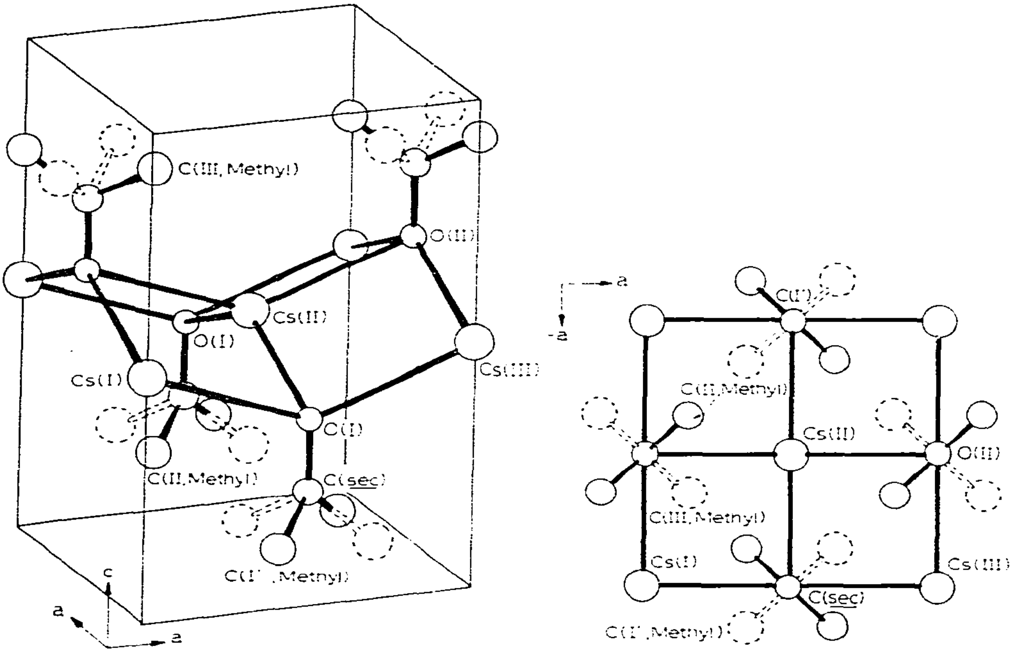
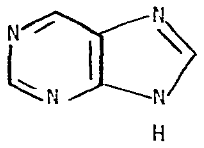


Fig. 3

Turning to the area of spectroscopy, ^1H and ^{13}C-NMR studies have been employed to demonstrate that the conversion of purine (1) to adenine (2) by potassium amide in liquid ammonia occurs via anion 3, then σ-adduct 4 [6]. The process is completed by loss of potassium hydride in a classic Chichibabin reaction.



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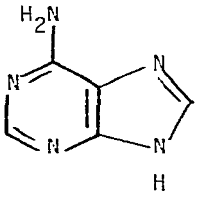

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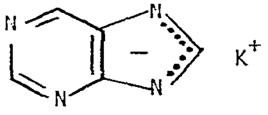

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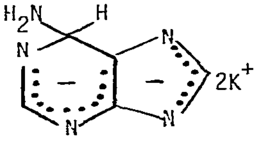

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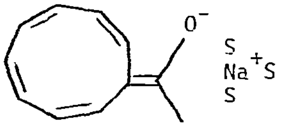
A dynamical equilibrium between anions 5 and 6 in THF has been studied by ^{1}H-NMR spectroscopy as a function of temperature [7]. While the equilibrium is shifted towards 5, a contact ion-pair, at higher temperatures, it is shifted towards 6, a solvent-separated ion-pair, at lower ones. The authors estimate a rate constant $\geq 330\ s^{-1}$ for the process corresponding to $\Delta G^{\ddagger}$ (-52°C) ≤ 10.2 kcal/mol. ^{13}C-NMR chemical shifts of disodiodiphenylfulvene were also reported in 1979 [8].



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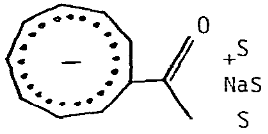

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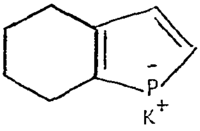
That the phosphorus nucleus in phospholides 7 and 8 is deshielded about 80 p.p.m. downfield from phosphoric acid is consistent with considerable double-bond character for the phosphorus atom [9]. Reagents 7 and 8 were prepared by cleavage of the corresponding p-phenyl systems with potassium metal in THF.



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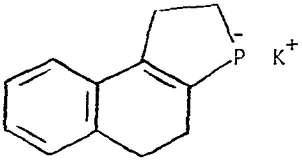

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^{23}Na-NMR spectroscopy has been employed to study preferential solvation of sodium cations by diethylenetriamine-THF mixtures [10] and in the competition of biogenic amines with sodium cation for binding to lasalocid [11].

ESR spectra of solutions of potassium in ethylenediamine and ethylamine as well as calculated values for all alkali metals except lithium in HMPA, ammonia, methylamine, and other solvents have allowed the authors to conclude that "despite the unique nature of e^-_{solv} and its 'tight' and 'loose' ion-pairs with solvated alkali metal cations, a standard ion-pairing treatment of equation 1 is capable of describing the variety of ESR spectra observed in these solutions" [12]. It is suggested that the "ESR spectra always consist of either a time-averaged singlet of all paramagnetic species or a singlet arising from e^-_{solv} alone, together with a multiplet which is a time-average of all species of stoichiometry M."

$$e^-_{solv} + M^+_{solv} \rightleftarrows M^+_{solv}e^-_{solv} \qquad (1)$$

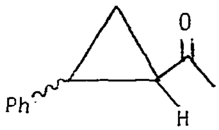
ESR spectra were reported for a variety of compounds including the radical-anions of several substituted [2.2]paracyclophanes [13], the radical anion of 1,2-diphenylcyclopentene [14], the rearrangement products from reduction of 1,6-methano[10]annulene and its 11-halogen derivatives [15], the products from 1,2-dibromo- and 1,2-diiodoadamantane with alkali metal vapors as part of a preparation of adamantene [16], of ketyls of cyclic α,β-unsaturated ketones obtained by electron transfer using trimethylsilylsodium or potassium t-butoxide in DMSO [17], of certain 1,2- and 1,4-semidiones obtained from polymeric peroxides and hydroperoxides [18], of cyclononane-1,2-semidione and certain derivatives [19], of substituted diazacyclopentadienone radical anions [20], of radical anions of perphenylcyclopolysilanes [21], of intermediates obtained in the decomposition of diazoalkanes induced by electron transfer from fluoren-9-yl anions [22], and of radical anions derived from reduction by potassium or electrolytically of bis(cyclopentadienyl)dialkyltitanium(IV) derivatives containing β-silylalkyl groups [23].

Other papers concerned with spectroscopy discussed the flash photolysis of alkali metal anions in THF and DME [24], absorption spectroscopy of alkali metal anions and trapped electrons in the presence of certain cryptands [25], the determination of the acidity of enols directly by flash spectroscopy [26], and Raman data on $Na_2W_2(CO)_{10}$ [27].

Disproportionation enthalpies of certain radical anions have been determined using calorimetric determination of the heats of reaction of the radical anions and the corresponding dianions with water in a solution calorimeter [28]. For example, such enthalpies for the radical anions of anthracene and tetracene were found to be +7.1 and +27.7 kcal/mol, respectively. Calorimetry has also been employed to provide the first reported solvation enthalpies of radical anions [29,30] and crystal lattice energy measurements of sodium anthracenide [31]. In contrast to earlier beliefs, the solvation study provided evidence that sodium cations are solvated by HMPA [29,30]. A paper unrelated to those above described the photoinduction of current (~0.1-0.5 $\mu A/cm^2$) when disodio- or dipotassiocyclooctatetraene was irradiated at single crystal or polylcrystalline n-TiO_2 in ammonia [32].

In a study of the equilibrium acidities of several fluorene derivatives and 1,3-diphenylindene, the active hydrogen compounds were found to be more completely ionized in methanol by tetramethylammonium methoxide than by potassium methoxide [33]. The authors conclude that "carbanion-cation interaction can be an important stabilizing influence in methanol but that the magnitude of this effect varies considerably with carbanion structure". The establishment of the acidities of several aniline indicators and the equilibrium acidity of phenylacetylene were also described [34].

Deuterium exchange of cis- and trans-9 with sodium deuteroxide in perdeuteromethanol/deuterium oxide has revealed that the acidity of 1-H of the cis-isomer is much higher than that of the trans-isomer [35]. The results are supported by CNDO/2 calculations. Similar exchange studies have revealed that k_{eq}/k_{ax} of 10 is 3.8:1, a result of steric rather than stereoelectronic factors [36], that the 5'-hydrogen of 11 is slowly replaced by deuterium [37], and that the carbonyl group is more effective than the imino moiety in activating the 4-methyl group of 12 and 13 [38]. The latter paper describes rate-acidity profiles for the exchange of such 4-methyl protons.



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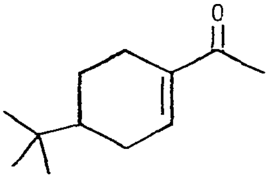

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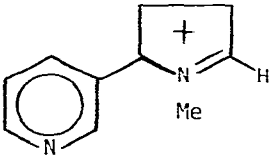

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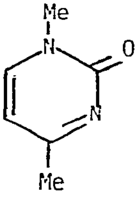

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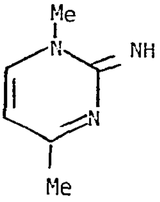

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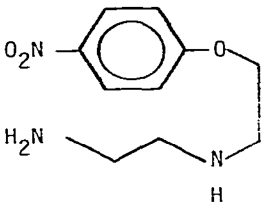
Other papers concerned with acidities discussed the rates of deprotonation and pKa values of transition metal carbonyl hydrides including several cluster compounds [39], a new semi-quantitative method for measuring the relative acidities of aliphatic alcohols involving reactions with n-butyl bromide [40], and a full paper on the cleavage of picolyl- and quinolylmethyl trimethylsilanes by sodium methoxide in methanol [41].

The rearrangement of 14 to 15 has been found to occur via a specific base catalyzed mechanism [42]. The neighboring amino group promotes rate determining deprotonation of a spiro-Meisenheimer intermediate. A large number of similar compounds with O,O'-substituents have also been studied [43].



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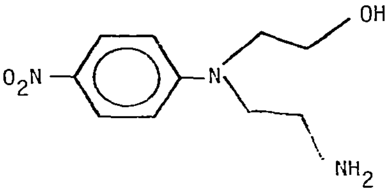

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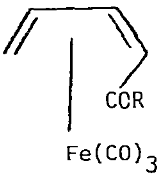
Other kinetic studies were concerned with anti to syn isomerization of (dienone)tricarbonyliron compounds illustrated by 16 and 17 [44], the isomerization of acrylonitriles 18 (Ar = p-substituted phenyls, furan, thiophene, selenophene)[45], the mechanism of the Cannizzaro reaction [46], the reduction of benzaldehyde by methoxide ion in aqueous methanol [47], regiospecific eliminations of N-chlorobenzylmethylamines by sodium methoxide and potassium t-butoxide to afford imines [48], general acid catalysis of the cleavage of the anions of 1-phenylcyclopropanol and 1-phenyl-2-arylcyclopropanol [49], and the reductions of N,N-dimethylformamide and -acetamide [50].



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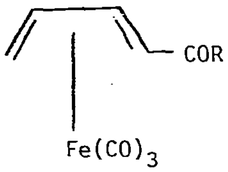

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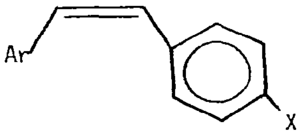

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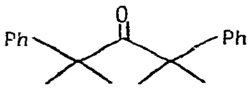
A 1,3-shift of an acyl group has been observed in the conversion of 19 to 20 and 21 effected by potassium t-butoxide [51]. The reaction is postulated to proceed via 22.



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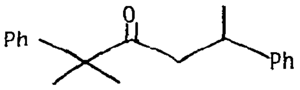

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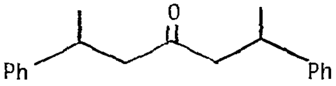

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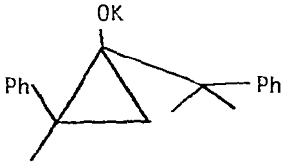

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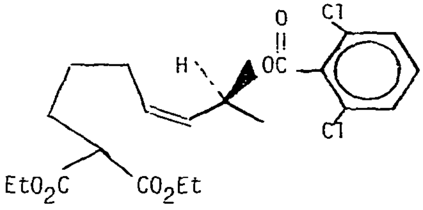
Cyclization of 23 to afford 24 has been achieved by sodium hydride [52]. Such an internal displacement with rearrangement (ScN') occurred anti to the departing carboxylate moiety.



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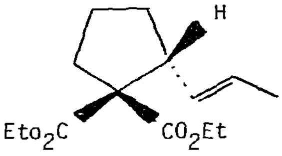

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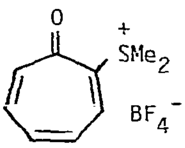
Tropone 25 has been found to react with sodium methanethiolate to give an equilibrating mixture of 26 and 27 as evidenced by NMR spectroscopy [53].



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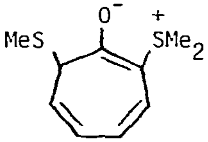

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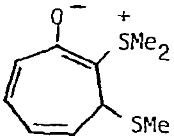

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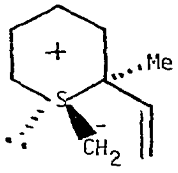
The stereochemistry of ring enlargement by [2,3]sigmatropic rearrangement of cyclic sulfonium ylides has been studied [54]. Specifically, "cis" ylides such as 28 rearrange faster than "trans" ylides such as 29. While both 28 and 29 react completely stereoselectively to afford ring-expanded olefin of E-configuration, the five-membered "cis" methylide reacts less stereoselectively to give the Z-olefin. Vinylsulfilimines have similarly been converted N-allyltosylamides by sodium ethoxide in ethanol as illustrated by the transformation of 30 to 31 [55].



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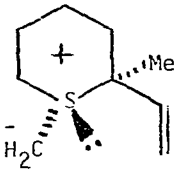

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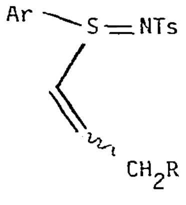

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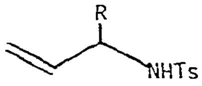

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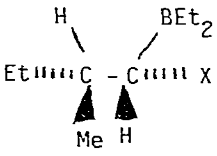
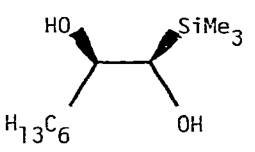
Base-induced rearrangements of α-haloorganoboranes have been found to be stereospecific in the absence of THF or dimethyl sulfide [56]. That the RS, SR pair rather than the RR, SS pair of 4-methylhexan-3-ols was obtained in the reactions of 32 (X = Br, I) and its enantiomer with sodium hydroxide and hydrogen peroxide demonstrates that such migrations occur with inversion at the migration terminus.



32

Acyclic α,β-dihydroxysilanes such as 33 have been converted to mostly 34 by potassium hydride in ether [57]. The results are consistent with anti elimination via α-oxidosilanes. Cyclic α,β-dihydroxysilanes react similarly provided the silicon and the β-hydroxy group can be anti to one another [58].



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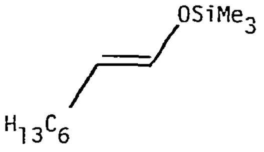

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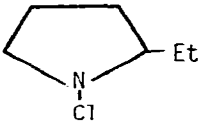
Deuterium labeling studies in the elimination of cyclooctylammonium iodides have been employed to determine that the effective base in potassium t-butoxide/DMSO is the alkoxide rather than the potassium salt of DMSO [59]. Unrelated studies have revealed that the "poorer" leaving group can be eliminated in dehydrohalogenations of 1,2-dihalocycloalkanes effected by sodium amide-sodium t-butoxide in THF [60]. For example, trans-1-bromo-2-fluorocyclohexane and this base pair afford 1-bromocyclohexene (85%). Dehydrochlorinations of 35 to give 36 and the previously undescribed 37 were also disclosed [61].



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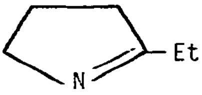

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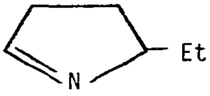

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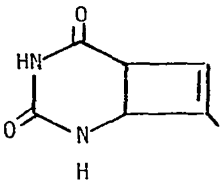
Other papers dealing with stereochemical aspects were concerned with the directing effect of remote hydroxyl groups in intramolecular additions of alkoxide to conjugated double bonds [62], the mechanism of Michael additions to enol esters [63], asymmetric induction in Michael additions of α-sulfinyl esters to α,β-unsaturated esters [64], and reductive alkylation of anthracene and naphthalene [65].

Treatment of uracil-acetylene cycloadducts such as 38 with potassium t-butoxide has been shown to afford pyridones illustrated by 39 [66]. Such cycloreversions are thought to involve dianions.



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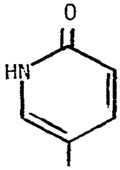

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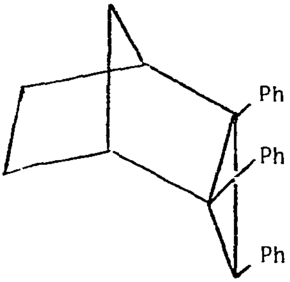
Deoxygenation of carboxylic esters by potassium in 18-crown-6/t-butylamine or lithium in ethylamine has been shown to proceed via alkyl-oxygen cleavage of the derived radical anion [67]. Cleavage of the cyclopropyl ring of 40 by potassium t-butoxide to give 41 has been postulated to proceed via a radical anion pathway, not by a disrotatory anion opening [68].



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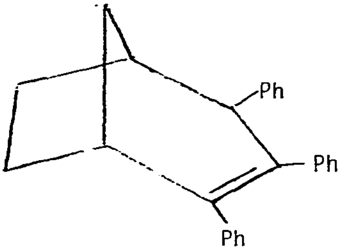
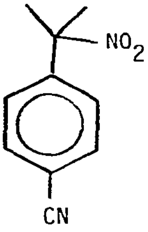


41

A synthetically useful replacement of aliphatic nitro groups by hydrogen can be achieved using sodiomethanethiol under exposure to ordinary fluorescent lights [69]. The reaction, proposed to proceed via a radical anion-free radical chain mechanism, is illustrated by the conversion of 42 to 43 in HMPA. Importantly, choice of solvent is critical since 44 is obtained from 42 in DMSO. β-Arylated nitroparaffins undergo rearrangement in such reactions, presumably via spiro free radical intermediates [70].



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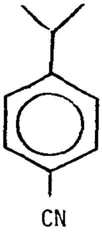

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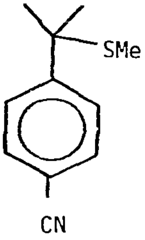


44

The area of substitutions on aromatic systems by the $S_{RN}1$ mechanism continues to be actively investigated. That the same relative reactivities of potassium diethylphosphite and potassiopinacolone were obtained with six halobenzenes and related compounds has been taken as evidence that a second order process ($S_{RN}2$) is not occurring in such reactions [71]. Full papers in this area discussed substituent effects on reactivity of aryl iodides with pinacolone enolate ion [72], condensations of halobenzenes with cyanomethyl anions [73], solvent effects in such reactions on quinoline systems [74], light and dark reactions of o-haloiodobenzenes with diethyl phosphite ion [75], condensations of halotoluenes with potassium diphenylphosphide [76], $S_{RN}1$ reactions of phosphorus nucleophiles with iodo- and bromobenzene [77], condensations of thiolate ions with unactivated aryl halides [78], similar reactions of phenyl selenide and phenyl telluride ions with halo- and dihaloarenes [79], combination of carbon nucleophiles with 4-phenyl- and 4-t-butyl-5-halogenopyrimidines [80], and the preparation of indoles from iodoanilines and potassium enolates [81].

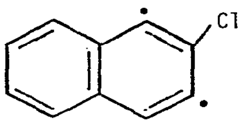
Other papers concerned with nucleophilic aromatic substitution discussed the Sn(ANRORC) mechanism in the Chichibabin amination of 4-phenylpyrimidine [82] and in the amino-dehalogenation of 4-substituted 6-halogenopyrimidines [83], mechanistic features of the reactions of methoxide ion with halogenobenzenes complexed with transition metals [84], tele- versus normal substitution in the reaction of 1,4-dialkyl-2,3-dinitronaphthalenes with sodium arenethioletes in DMSO [85], and direct **or** cine methoxydenitration of 1-alkyl-3,4-dinitropyrroles [86].

2. SYNTHETIC ASPECTS

Reviews appropriately included in this section discussed organic synthesis using supported reagents [87], chemistry of nitroacetic acid and its esters [88], addition reactions of esters of phosphorus(III) acids with unsaturated systems [89], and recent synthetic developments involving azetidines [90].

Certain dialkylamides of heavier alkali metals may be prepared conveniently by interaction of lithium dialkylamides and ROM (M-Na,K) [91]. The best results are obtained with t-butoxide. Several examples of the synthetic utility of these reagents are described.

The reactions of sodium vapor with several chloroalkanes followed by deuterolysis have been reported [92]. For example, carbon tetrachloride with these reagents affords C_2D_4 (80%) and C_2D_6 (15%). The absence of CD_4 is in contrast to the formation of this product in the related reactions with lithium vapor. Results with ethyl chloride, 1,2-dichloroethane, and others are also reported. Deuteration has also been employed to demonstrate the intermediacy of diyl 45 in the reaction of 46 with potassium t-butoxide [93].



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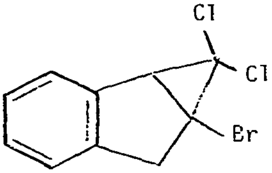
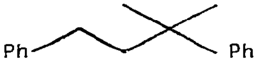


46

1,3-Diarylpropanes including unsymmetrical ones may be prepared from sodiotoluene derivatives and HMPA [94]. For example, toluene, cumene, sodium amide, and HMPA afford 47 (60%). Another paper listed UV data for triphenylmethylpotassium and -lithium in ethereal solvents and discussed the results in terms of ion pairing [95].



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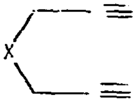
A full paper discussed the formation of ring compounds by base-catalyzed rearrangement of 4-heterohepta-1,6-diynes [96]. The process is illustrated by the conversion of 48 to 49 (X=S,O,NEt).



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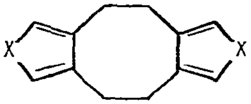
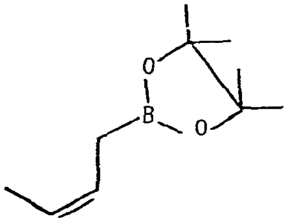


49

β-Methyl homoallyl alcohols may be obtained diastereoselectively from 50, itself prepared from 51 and chlorobis(dimethylamino)borane [97]. The stereochemistry of the double bond is not preserved when 51 is treated with stronger Lewis acids such as chloro(dialkoxy)boranes. Another paper described the conversion of 52 to 53 and 54 by sodium methoxide [98].



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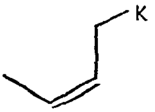

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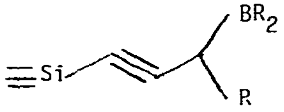

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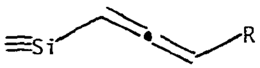

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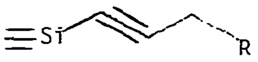
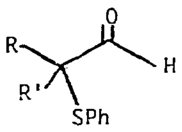


54

Other papers involving carbanions not stabilized by carbonyls or other heterofunctional groups have been concerned with condensations of 1,2:5,6-dibenzocyclooctatetraene dianion [99] and certain derivatives of cyclononatetraenes [100], and zwitterionic species from deprotonation of several arene-cyclopentadienyliron cations [101].

Additional papers appeared in 1979 which discussed enolates of aldehydes. Thus, such enolates, prepared by metalation of the parent compounds by potassium hydride, have been sulfenylated by diphenyldisulfide and iodinated by iodine to afford products like 55 and 56, respectively [102], and converted to enol acetates by acetyl chloride [103].



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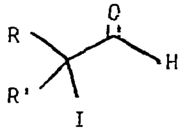
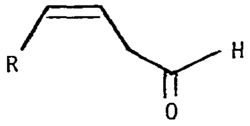


56

A stereospecific synthesis of Z-α,β-unsaturated aldehydes 57 has been achieved by the conversion of 58 to 59 by addition of sodium amide followed by the addition of ethanol [104]. The process is completed by the addition of RCHO. The ninth paper in a series discussed sodium derivatives of several haloglutaconaldehydes [105].



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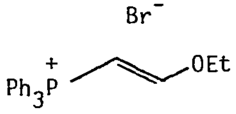

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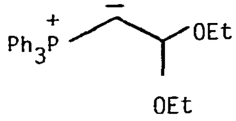
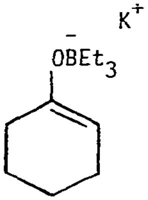


59

Ketones may be conveniently monoalkylated via boron enolates [106]. For example, cyclohexanone, potassium hydride, and triethylborane give 60 which, upon treatment with allyl bromide, yields 61 (90%). γ-Alkylation of 62 to afford 63 has been achieved by sodium hydroxide in DMSO-water in which the amount of water is critical [107]. Product 63 was accompanied by only small amounts of the α-alkylated isomer provided larger amounts of water were employed.



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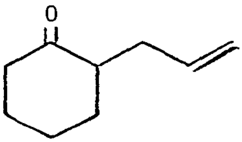

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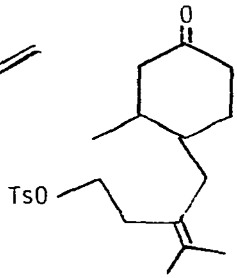

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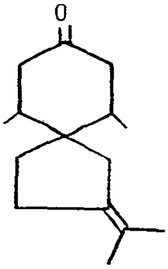
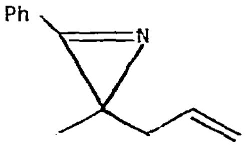


63

Pyrrole derivatives have been obtained in the reaction of azirines with ketone enolates [108,109]. For example, 64 and sodioacetophenone yield 65 [108]. o-Derivatives 66 [110] and 67 [111] have been found to undergo cyclization to 68 and 69, respectively.



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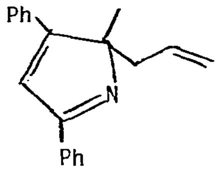

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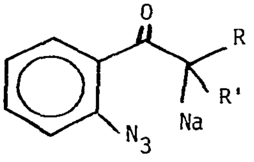

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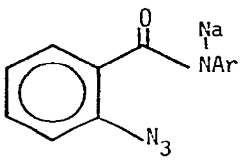

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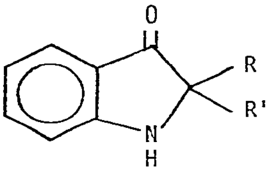

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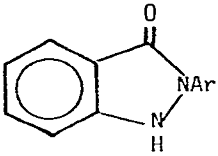
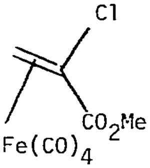


69

Iron complex 70 has been treated with sodiodimethyl malonate and related dicarbonyl-containing materials to afford adducts illustrated by 71 [112]. Such compounds apparently arise via iron-carbene complexes rather than via a Michael-type reaction. 3-Sodiopentan-2,4-dione has been added to 72 in a trans-fashion to give 73 [113].



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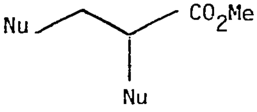


71

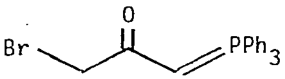
$$[Pd(C_5H_5)(PPh_3)(H_2C{=}CH_2)]^+ \; ClO_4^-$$

72

$$Pd(C_5H_5)(PPh_3)CH_2CH_2CH(COMe)_2$$

73

Phosphorane 74 has been shown to constitute a novel cyclopentannelating agent. For example, 74 and sodioethyl acetoacetate yield 75 (39%) [114]. Annelations using β-vinylbutenolide (76) were also reported [115]. Thus, 76 and sodioethyl α-methylacetoacetate afford 77 (42%).



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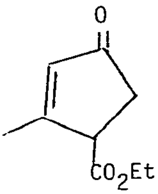

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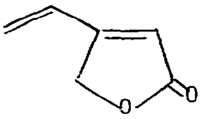

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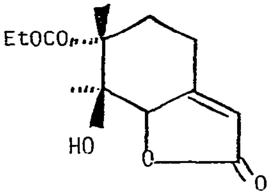
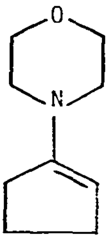


77

Sodioethyl acetoacetate and similar compounds have been added to enamines such as 78 under electrochemical oxidation conditions to give, after hydrolysis, products such as 79 [116]. Such carbanions have also been sulfenylated with phenyl- and methylsulfenyl chloride [117].



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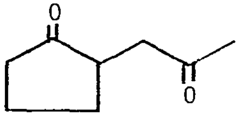
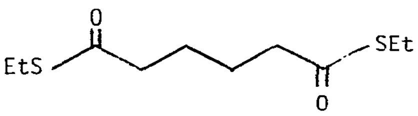


79

The Deckmann condensation has been effected on dithiol esters using sodium hydride [118]. For example, 80 yields 81 (91%).



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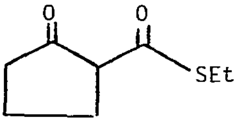
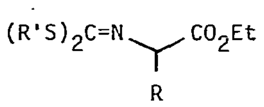


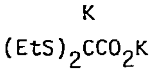
81

A full paper disclosed that esters such as 82 (R=H , R'=Me or $-CH_2CH_2-$) can be deprotonated by potassium t-butoxide, then alkylated [119]. Alkylations of 83 [120] and formation of 84 from disodio- or dilithiomethylpropanoate and styrene [121] have also been reported.



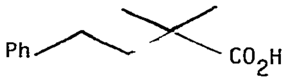
$(R'S)_2C{=}N\underset{R}{CH}CO_2Et$

82



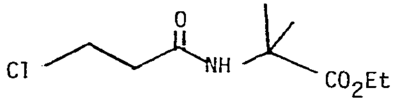
$(EtS)_2\overset{K}{C}CO_2K$

83



84

Additional examples of the preparation of β-lactams by cyclization of β-halopropionamides using sodium hydride have been presented [122]. The process is illustrated by the conversion of 85 to 86 (86%). 2,6-Dihydroxypyridines 87 ($R'=CONH_2,CN,COCH_3,Ph$) have been obtained from $RCHNaCONH_2$ and 88 [123].



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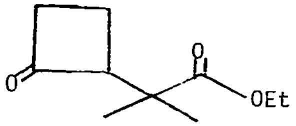

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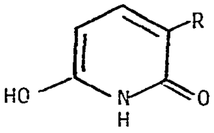

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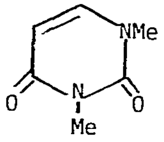
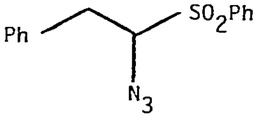


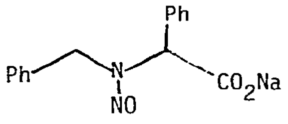
88

A full paper described the direct synthesis of α,β-unsaturated nitriles from acetonitrile and carbonyl compounds effected by alkali hydroxides [124]. Nitriles themselves are obtained from α-azidosulfones such as 89 and alkali alkoxides [125]. Thus, 89 and sodium methoxide give phenylacetonitrile (85%).



89

N-Nitrosamines have been obtained by decarboxylation of the sodium salts of α-nitrosamino acids [126]. For example, 90 affords N-nitrosodibenzylamine. Vinylic nitrosamines such as 91 have been synthesized from the corresponding β-tosyloxy systems 92 and potassium hydroxide [127].



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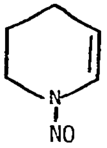

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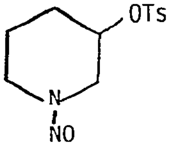
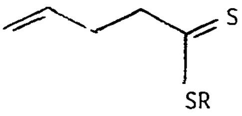


92

Metalation of 93 (R=Me,Et) with potassium hydride followed by s-butyllithium affords 94 which combines with aldehydes and ketones at the 5-position to give 95 (128].



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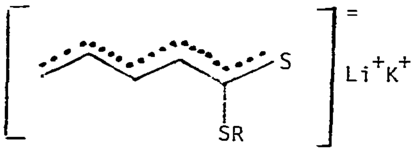

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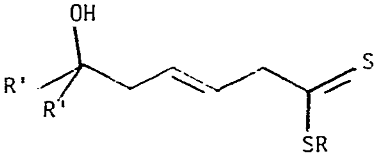
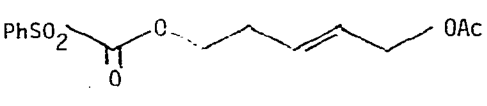
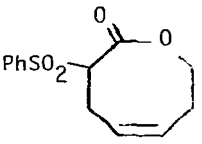


95

Several ring closures of acetoxysulfones effected by sodium hydride and palladium complexes have shown a preference for formation of eight- and nine-membered rings over competing six- and seven-membered ones [129]. For example, 96, NaH, and $(Ph_3P)_4Pd$ give 97 (94%) and 98 (6%). α-Sodiosulfone chemistry has also been employed in the cyclization of a C_{2V}-symmetric diepoxy tetraquinane disulfone [130].



96



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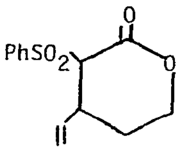
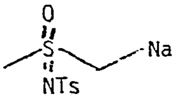
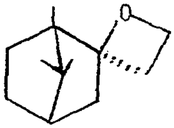


98

Combination of three equivalents of sulfoximine 99 with ketones has been found to afford 2,2-disubstituted oxetanes [131]. Thus, this reagent and camphor give 100 (59%).

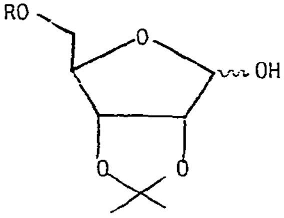


99



100

While treatment of 101 (R=H) with potassium t-butoxide affords 102, reaction of this ribofuranose (R=CPh_3,Me_2Sit-Bu) with sodium hydride gives 103 [132]. Condensations of the alkoxides are reported. Another paper reported the rearrangement of a variety of hydroxyacenaphthalenes 104 (R=CN,$CONH_2$,CO_2Et,Ar,Me) to 105 by means of certain alkoxides, sodium hydride, and DBU [133].



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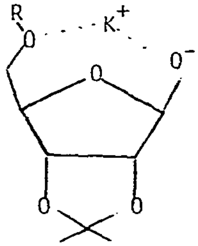

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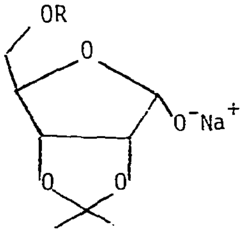
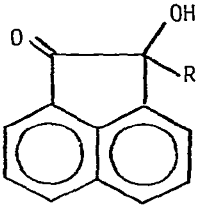


103



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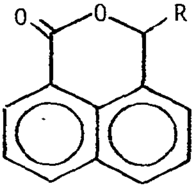
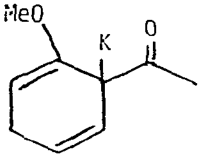


105

Ketones and terminal olefins have been reductively coupled by sodium/t-butyl alcohol in N-ethylpyrrolidone [134]. For example, acetone and ethylene yield 2-methyl-2-butanol. The use of this solvent and N,N-diethylacetamide for SN_2 displacements are also discussed. Other papers concerned with reductions discussed the formation of 106 as an equivalent of 107 [135], the sodium-ammonia reduction of flavonols to afford α-methoxydihydrochalcones [136], the reductive cleavage of 108 to give 109 [137], and the use of complex reducing agents such as $NaH\text{-}RONa\text{-}Co(OAc)_2\text{-}CO$ to effect carbonylation of aryl halides at atmospheric pressure [138] and to effect regioselective reduction of α,β-unsaturated ketones [139].



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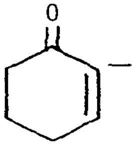

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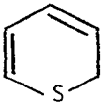

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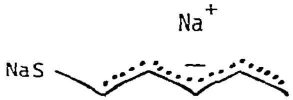


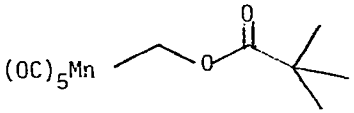
109

Finally, in this section, it has been shown that TMEDA and HMPA minimize chain transfer reactions in the polymerization of 1,3-butadiene effected by alkylsodium and -potassium reagents [140].

3. INORGANIC AND ORGANOMETALLIC COMPOUNDS

Highly reactive uranium metal has been prepared by reduction of uranium tetrachloride using NaK and 5-10% naphthalene [141]. Both this metal and similarly prepared copper metal were reacted with a variety of organic substrates. Reaction of sodium(hexamethyldisilyl)amide with appropriate uranium chlorides has been employed to synthesize tris(hexamethyldisilyl)amido)uranium(III) [142] and its hydrideo derivative [143] as well as bis((hexamethyldisilyl)amido)dioxouranium(VI) complexed with THF [144].

Other compounds prepared were binuclear bis(η^5-methylcyclopentadienyl) titanium(III) complexes bridged by the dianions of 2,4-dithiopyrimidine and related dianions [145], chlorotris(N,N-dialkylmonothiocarbamoto)titanium (IV) complexes [146], a dimeric naphthyl hydride derivative of zirconocene [147], a quadruply bonded dimolybdenum compound with bridging pyrimidinethiol ligands [148], alkyl(η^5-cyclopentadienyltricarbonylmolybdenum and -tungsten) complexes [149], pentacarbonyl[diethylamino(triphenylstannyl) carbene]chromium(0) [150], and bis(pentacarbonylmanganate)mercury(II)·THF [151]. The latter compound was prepared from $NaMn(CO)_5$ with chlorodiethylaluminum. This same manganese reagent has been combined with acetic [^{13}C] formic anhydride to give predominately ^{13}CO substituted pentacarbonylmanganese hydride [152], and with α-chloroesters to afford 110 as part of a study of the $-CH_2OH$ ligand in Fischer-Tropsch and related reactions [153]. Another paper discussed the reactions of $NaMn_2(CO)_9CN$ with alkylating agents to yield $Mn_2(CO)_9CNR$ (R=Et,Si≡,Ge≡,PPh_2, and COMe) [154]. Monohapto derivative 111 has been prepared from C_7H_7COCl and $NaRe(CO)_5$ followed by photolysis [155].



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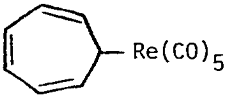
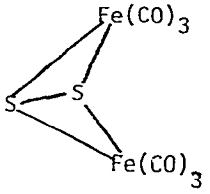


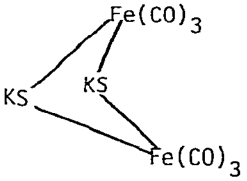
111

The preparation of $LFe(CO)_4$ (L=PPh_3,$P(OPh)_3$,$P(OPh)_3$,$P(OMe)_3$) has been achieved from $Fe(CO)_5$ and L provided the reactions are run in the presence of catalytic amounts of $Na_2Fe_2(CO)_8$ [156].

Cluster complex 112 has been converted to 113 by potassium hydride in THF, then treated with alkylating agents to afford dialkyl derivatives [157]. Alkylation of 112 has also been realized by addition of alkyllithiums to give a monoanion which can also be combined with electrophiles to yield the same dialkyl derivatives as above.



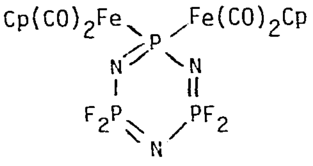
112



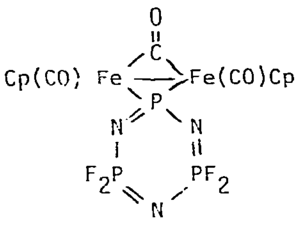
113

Other papers concerned with cluster complexes in this family discussed the synthesis of triiron carbonyl complexes containing isomeric triply bridging acimidoyl or alkylidenimido groups [158] and related nitrene complexes [159], the preparation of $Fe_2(CO)_6(\mu_2\text{-}PPh_2)_2Na_2$ [160], the synthesis of $Ru_3H(CO)_{11}Na$ [161], and stepwise deprotonation of $H_4Ru_4(CO)_{12}$ [162].

Hexafluorocyclotriphosphazene has been combined with $NaFe(CO)_2C_5H_5$ to give 114 which, under even weak laboratory lighting, is converted to the interesting spiro compound 115 [163]. The above sodioiron reagent has also been combined with iminium salts to produce dialkylaminoalkyl complexes [164].

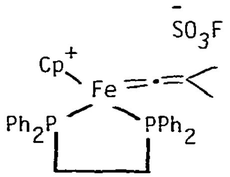


114

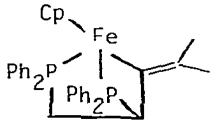


115

Cationic vinylidene complex 116 has been deprotonated by potassium hydroxide and sodium bis(trimethylsilyl)amide to yield 117, which contains a novel ring system [165]. Abstraction of an endo-proton from $(7\text{-}Ph_3GeC_7H_7)\text{-}Fe(CO)_3$ has also been reported [166].



116



117

Sodium tetracarbonylcobalt has been combined with certain germanium halides and imidoyl chlorides to afford products such as $MeGeCo_3(CO)_{11}$ [167] and η^1-iminoacylcobalt carbonyl complexes [168], respectively. Other papers dealing with cobalt were concerned with reductions of derivatives of cyclopentadienyldicarbonylcobalt [169] and the synthesis of $K[M_6N(\mu\text{-}CO)_9(CO)_6]$ (M=CO,Rh) [170].

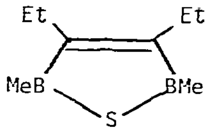
The preparation and reactions of highly reactive nickle [171], and palladium and platinum [172] have been discussed in separate papers.

The previously unknown potassium and cesium salts of $(CF_3)_2BF_2^-$ have been obtained from Me_3SnCF_3, BF_3, and KF or CsF [173]. The compounds have been characterized by ^{19}F NMR, IR, and Raman spectra.

A variety of interesting carboranes continued to be reported in 1979. For example, 118 has been synthesized from 119 and potassium metal [174]. Others included copper derivative $Cu(PPh_3)_2B_5H_8Fe(CO)_3$ from $K^+B_5H_8Fe(CO)_3^-$ and $Cu(PPh_3)_3Cl$ [175], polyhedral ferraboranes derived from NaB_5H_8 [176], $(C_5H_5Co)B_9H_9C{\cdot}NMe_3$ from $B_9H_{11}C{\cdot}NMe_3$, NaH, NaC_5H_5, and $CoCl_2$ [177], and polyhedral nickelboranes and nickelcarboranes from small borane and carborane anions [178].

$(EtC)_4(B\text{-}Me)_4$

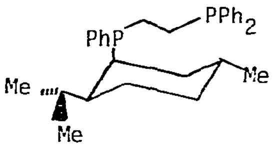
118



119

All of the permethylcyclosilanes $(Me_2Si)_n$ from n=5 through n=35 have now been obtained from Me_2SiCl_2 and Na/K by mixing the reagents over a ten hour period [179]. Novel compounds $P_{11}(SiMe_3)_3$ and $As_7(SiMe_3)_3$ have been prepared from chlorotrimethylsilane and M_3P_{11} (M=Na,Cs) and Rb_3As_7, respectively [180]. Similarly synthesized were $(Me_2SnTe)_3$ from Me_2SnCl_2 and NaHTe [181], and $PhSeSiMe_3$ from PhSNa and Me_3SiCl_3 [182].

Novel polyphospines illustrated by 120 have been obtained from vinylphosphines, neomenthylphenylphosphine, and potassium t-butoxide [183]. Other phosphorus reagents synthesized were $(RP)_3C_2H_4$ (R=Me,Et,t-Bu) from K(PR)nK and dihaloalkanes [184], and o-phenylenebis(methylphenylphosphine) from o-dichlorobenzene and NaPMePh [185].



120

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