ZINC AND CADMIUM

LITERATURE SURVEY COVERING THE YEAR 1976

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Contents

Zinc and cadmium, Literature Survey covering the year 1975 see J. Organometal Chem., Vol. 130(1977)157-172.

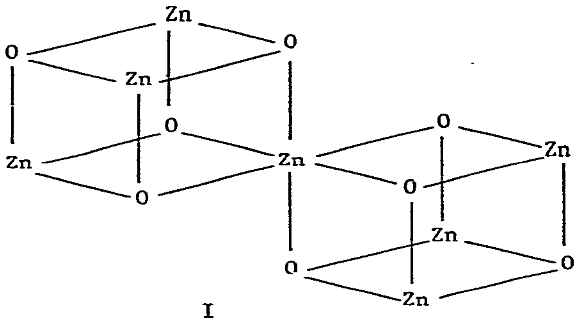
Introduction

The yearly number of papers dealing with organozinc- and organocadmium chemistry appears to have stabilized in the last few years. Although there is a continuing interest in the application of - mainly - organozinc compounds in organic synthesis and polymer chemistry, relatively few new organozinc compounds are published. There is a slowly growing interest in the use of organozinc- and organocadmium compounds in the synthesis of organo-transition metal compounds.

A review dealing with the reactions of, among others, organozinc- and organocadmium compounds with epoxides has appeared this year [1].

I Preparation of organozinc and organocadmium compounds

Tsuruta and co-workers investigated the molecular structure and reactivity of the zinc dimethoxide complex $[(EtZnOMe)_6 \cdot Zn(OMe)_2]$ [2]. This complex appeared to be an enantromorphic catalyst for the polymerizations of methyloxirane (propylene oxide). The molecular structure was determined by x-ray diffraction. The compound consists of two distorted Zn_4O_4 cubes sharing a zinc atom (I):



I

The structure of I is similar to that of $Me_6Zn_7(Ome)_8$[3], but in this case no dissociation equilibria involving Et_2Zn, $(EtZnOMe)_4$, $[(EtZnOMe)_6 \cdot Zn(OMe)_2]$ etc. occurred. The authors propose that the catalytically active species is formed by dissociation of two zinc-oxygen bonds at the shared zinc atom, leaving two vacant sites at that atom.

Murdock and Klabunde prepared highly reactive zinc and cadmium slurries by cocondensation of the metal vapors and excess solvent at 77K, followed by warming to ambient temperatures [4]. These slurries react with alkyl halides in all types of both polar and non-polar solvents. Although the dialkyl-metal compounds were isolated only in a few cases, generally good to excellent yields of alkylzinc- and alkylcadmium compounds were obtained. Diglyme and dioxane appeared to be the best solvents, but toluene and hexane were also acceptable.

Organozinc- and organocadmium alkoxides and germanoxides have been obtained by heating the zinc- or cadmium alkyls with carbinols or germanium hydroxides [5]:

$$R^1_2\,M^1 + Ph_2\,RMOH \xrightarrow[\text{2 hrs}]{80^o} \underset{\text{II}}{R^1M^1OMRPh_2}$$

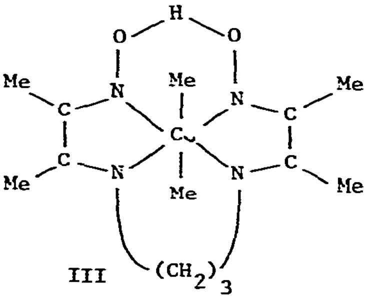
$R = Me, Ph;\ R^1 = Me, Et;\ M = C, Ge;\ M^1 = Zn, Cd$

When II ($M = Ge$) were thermolyzed, the M^1–O bond was cleaved and one phenylgroup was transferred from the germanium to the zinc or cadmium, analogous to the corresponding siloxy derivatives [6]:

$$R^1M^1OMRPh_2 \longrightarrow R^1M^1Ph + [PhRM = O].$$

Thermolysis of II ($M = C$) gave hexaphenylethane and zinc oxide or cadmium oxide.

The methylation of Zn(II) and Cd(II) ions by the trans-dimethylcobalt chelate $Me_2Co(BDM\ 1{,}3pn)$(III):



III

was reported by Witman and Weber [7].UV – visible spectral titrations showed that III reacts with Zn(II) and Cd(II) ions in a 1 : 1 reaction in isopropanol:

$$Me_2Co(BDM\ 1,3pn) + M^{2+} \xrightarrow{fast} MeCo(BDM1,\ 3pn) + MeM^+$$

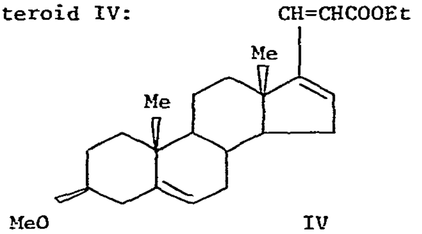
Surprisingly, the MeM^+ cations appeared to decompose only slowly in this protic solvent. Kinetic studies were carried out on the decomposition of these cations resulting in the relative rates $MeZn^+ \gg MeCd^+$ with half-lives of 1.5 and 52.6 hrs, respectively. The demethylation of III was pseudo-first order and revealed the presence of binuclear intermediates of the type M^{2+}. III. The authors suggested that solvation of the MeM+ species by isopropanol contributes to their stabilization.

II Reactions of organozinc and organocadmium compounds

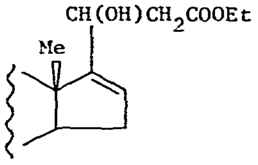
A. The Reformatsky reaction and related reactions

The Reformatsky reaction continues to find numerous applications in synthetic organic chemistry especially in the field of steroid synthesis.

The steroid IV:

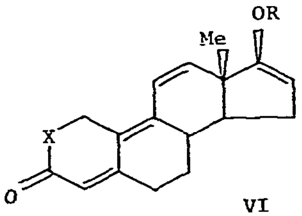


IV



V

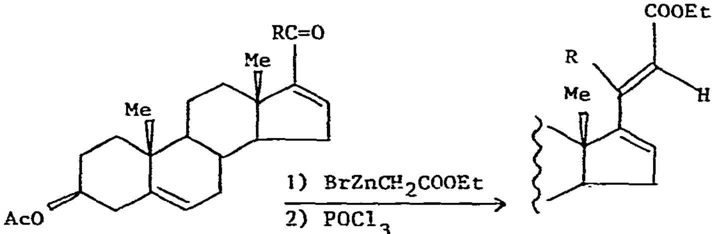
was prepared by the Reformatsky reaction of 3 β-acetoxy-androsta-5, 16-diene -17- carboxaldehyde with ethylbromoacetate and $POCl_3$-dehydration of the resulting pregnadienecarboxylate V [8].
The Reformatsky reaction was used as a key step in the synthesis of heterocyclic 19- norsteroids VI [9], [10]:



VI

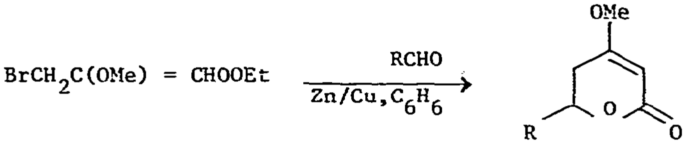
X = O,N,S.

The heterocyclic rings were built up in multistep reactions using the $BrZnCH_2COOEt$ reagent in the first step.
Nor- and dinorchola- 5,16,20 (22) trienoic acids VII were prepared by the Reformatsky reaction of the dehydropregnenolones VIII, followed by dehydration [11]:



VIII VII

A modification of the Reformatsky reaction, involving the use of zinc-copper couple instead of zinc powder alone, was used to prepare kavain (IX) and dihydrokavain (X) in much higher yields than reported earlier [12]:



IX : R = PhCH=CH- 80%
X : R = $PhCH_2CH_2$- 50%

A series of twelve α-ketosulfides (XI) were prepared by reacting $RCOCR^1R^2ZnBr$ with R^3SCl [13]:

$$RCOCR^1R^2Br + R^3SCl \xrightarrow{Zn} \underset{XI}{RCOCR^1R^2SR^3}$$

(R = Ph,Me,Et,Pr,Bu; R^1= Me,Et,Pr; R^2= H,Me; R^3= Ph,p-ClC_6H_4, p-BuC_6H_4)

Similarly, eleven β-alkylthiocarboxylates (XII) were made by the Reformatsky reaction of $BrZnCR^1R^2COOR^3$ with $RSCH_2Cl$[14]:

$$BrCR^1R^2COOR^3 + RSCH_2Cl \xrightarrow[C_6H_6/Et_2O]{Zn} \underset{XII}{RSCH_2CR^1R^2COOR^1}$$

(R = Me,Pr,Bu,i-Bu,C_5H_4; R^1= Me,Pr,Bu; R^2= Me,Et,Pr,i-Pr,H; R^3= Me,Et,Pr)

The Reformatsky reaction of RCH=O (R = 2-thienyl, 5-bromo-2-

thienyl, and 3-methyl-2-thienyl) with $BrCR^1R^2COOEt$ (R^1=Me, R^2= H, Me; R^1 = Et, R^2 = H) yielded β-(a-thienyl)-β-hydroxycarboxylates $HOCHRCR^1R^2COOEt$ (XIII) in 54 – 82% yields [15].

When phosphorus trichloride and diethoxyphosphorus chloride were treated with the Reformatsky reagent derived from $BrCHR^1COOR^2$ (R^1 = H, Me, COOEt; R^2 = Me, Et), six C-phosphorylated esters of carbonylic acids (XIV) were obtained in 19 – 48% yields [16]:

$$R_2PCHR^1COOR^2 \qquad \text{XIV}$$

(R = Cl, EtO; R^1= H,Me,COOEt; R^2= Me,Et)

Only one example of a non-classical Reformatsky reagent has been reported. Dardoize and Gaudemar investigated the stereo chemistry of the addition of the Reformatsky reagents derived from α-bromoamides to aldimines [17]:

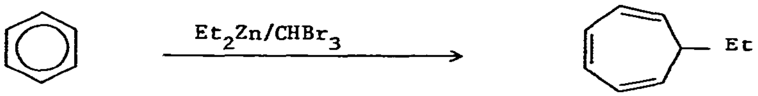
$$EtCH(ZnBr)CONR_2 \xrightarrow[\text{2) } H_2O,OH^-]{\text{1) } Ph\text{-}CH = NR^1} PhCH(NHR^1)CH(Et)CONR_2$$

(R = Me,Et,iPr; R^1= Me,Et,iPr,Ph)

The addition to $PhCH{=}NR^1$ (R^1=aliphatic) was kinetically controlled, giving mainly threo-products. Under the same conditions, PhCH=NPh gives a mixture of both erythro- and threo-products, although also here the threo-products are formed in excess. The basicity of the imine appeared to be the main factor determining the stereochemical course of the reaction, the more basic imines giving higher yields of threo-isomers.

B. Carbenoid reactions

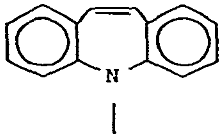
Miyano, Hashimoto and co-workers extended their work on oxygen-accelerated zinc-halocarbenoid reagents to the diethylzinc / bromoform system. This system reacted with benzene to give a 58% yield of 7-ethyltropilidene [18]:



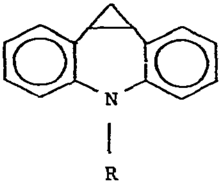
Toluene and cumene gave the corresponding alkyl-7-ethyltropilidenes.

The isomer distributions in these reactions were closely similar to those in reactions of the diethylzinc/iodoform system. Bromo- and chlorobenzene gave the halo-7-tropilidenes in lower yields along with alkylation - and dihalogenation products like $PhCHEt_2$, propylbenzene and traces of toluene.

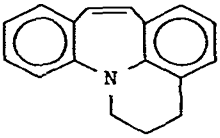
The Simmons-Smith reaction was used to introduce a cyclopropane ring into the dibenz [b,f]azepine XVI (R = Me)



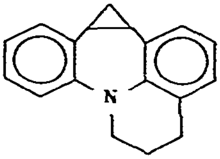
XVI



as a first step in the synthesis of potential anticonvulsants and antidepressants [19]. When R was a 3-chloro- or 3-bromopropyl group, however, reaction with the Simmons-Smith reagent gave only XVI (R = alkyl, cyclopropylmethyl) and cyclization products XVII and XVIII.

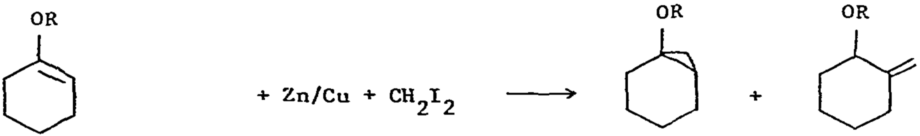


XVII



XVIII

A mixture of cyclopropylether XIX and allylic ether XX was formed when 1-methoxy- and 1-ethoxycyclohexene were reacted with zinc-copper couple and methylene iodide [20]:

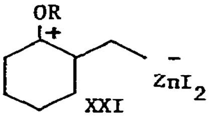


R = OMe, OEt

XIX

XX

The relative amount of XX was higher for R = OEt, which is in agreement with the occurrence of intermediate XXI, proposed earlier:



XXI

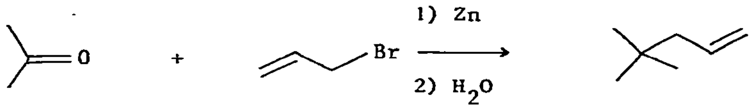
Gragg and Ryschkewitsch have shown that the Simmons-Smith reagent can insert a methylene group into a boron hydrogen bond [21].

A 25% yield of trimethylboron was obtained when the Simmons-Smith reagent was prepared in the presence of trimethylamine-borane (3 : 1 molar ratio). A comparison with the calculated yield for purely statistical methylene-transfer (22.2%) showed that the insertion was essentially non-selective. When the same reaction was carried out using a 1 : 1 ratio of reactants, no trimethylboron was formed, over 68% of the trimethylamine used being recovered as a zinciodide complex. Apparently, the boron-nitrogen coordinate bond was destroyed during the initial stages of the reaction. Surprisingly, only a 6.9% yield of trimethylboron was obtained when the Simmons-Smith reagent was prepared seperately. The authors suggested that the Simmons-Smith reagent is a methylene-transfer reagent that has a high percentage of reactive intermediate on initial formation and rearranges or decomposes with time into less-reactive species.

C. Reactions of alkenyl- and alkynyl-zinc compounds with carbon-carbon and carbon-heteroatom unsaturated bonds

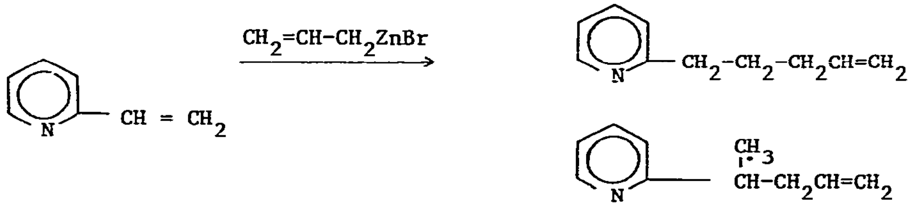
Most papers in this area of organozinc chemistry this year deal with various aspects of the stereo selectivity and regio selectivity of these reactions.

The highly efficient continuous-flow system developed by Ruppert and White for carrying out Reformatsky reactions with superior yields (cf. AS 115, 3) has been used successfully by these authors in addition reactions of allylic zincbromides with carbonyl compounds [22]:

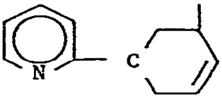


This technique afforded a large variety of homo-allylic alcohols in yields which were consistently higher than those obtained under conventional conditions. The method was uneffective with allylic chlorides and with saturated bromides.

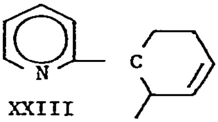
Allylzincbromide reacted with 2-vinylpyridine to give a mixture of two isomers, the allylic group adding to both sides of the vinyl-group [23]:



With methyl-substituted allylic zincbromides also mixtures were obtained. The cyclic compounds XXII and XXIII were formed when 2-vinylpyridine was reacted with 2,4-pentadienylzincbromide:



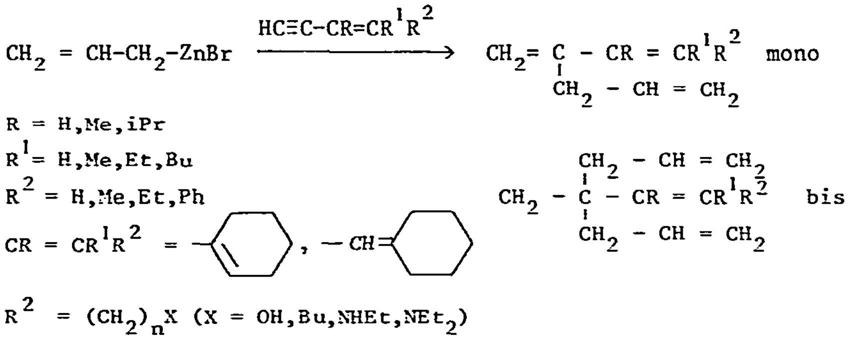
XXII



XXIII

These products were probably formed by cyclization of the intermediate organozinc-adducts.

L. Miginiac and her group have extended their studies on the reactions of alkenylzinc compounds to both normal and functionally-substituted enynes [24], [25]. With both types of compound, mono- and bis-3,4-addition was observed upon reaction with allylzinc bromide:



$$CH_2 = CH-CH_2-ZnBr \xrightarrow{HC\equiv C-CR=CR^1R^2} CH_2=\underset{CH_2-CH=CH_2}{C}-CR=CR^1R^2 \quad \text{mono}$$

$$CH_2-\overset{CH_2-CH=CH_2}{\underset{CH_2-CH=CH_2}{C}}-CR=CR^1R^2 \quad \text{bis}$$

R = H,Me,iPr

R^1 = H,Me,Et,Bu

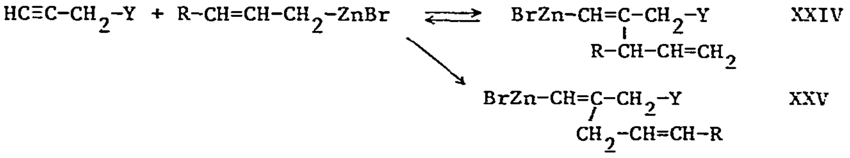
R^2 = H,Me,Et,Ph

$CR = CR^1R^2$ =

$R^2 = (CH_2)_nX$ (X = OH,Bu,NHEt,NEt_2)

The reactivity decreased when bulky groups were present around the double bond.

New examples of the reversibility of the additions of α-unsaturated organozinc compounds to terminal acetylenes were found [26]:

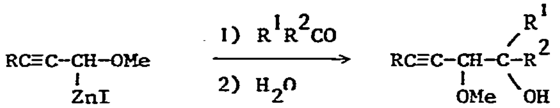


$$HC\equiv C-CH_2-Y + R-CH=CH-CH_2-ZnBr \rightleftarrows BrZn-CH=\underset{R-CH-CH=CH_2}{C}-CH_2-Y \quad \text{XXIV}$$

$$\searrow \; BrZn-CH=\underset{CH_2-CH=CH-R}{C}-CH_2-Y \quad \text{XXV}$$

It was shown that product XXIV was transformed into product XXV, particularly when an excess of organozinc reagent was used. The authors interpreted this result in terms of the reversibility of the reaction leading to XXIV.

The stereochemistry of the addition of the zinc derivative of the propargylic ether $PhC\equiv CCH_2OMe$ to saturated, aromatic, ethylenic and acetylenic carbonyl compounds was investigated by Mercier and Epsztein [27]:

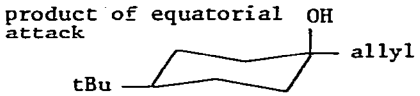


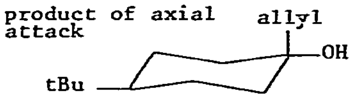
$$RC\equiv C-\underset{\displaystyle ZnI}{CH}-OMe \xrightarrow[2)\ H_2O]{1)\ R^1R^2CO} RC\equiv C-\underset{\displaystyle OMe}{CH}-C(R^1)(R^2)OH$$

They found, that with aldehydes in all cases the erythro-diastereoisomer was formed preferentially in yields which could be correlated with the size of the substituents R^1,R^2 , i.e. the bulkiest groups gave the highest yields.

Also for fully saturated methylketones the same trends were found. When phenyl, vinyl or alkynyl groups were present in the α position, the erythro- threo product ratio's could not be explained on the basis of steric considerations alone, but conformational and electronic factors had to be considered as well.

The amount of axial attack by allylic compounds of zinc, aluminium, magnesium, potassium sodium and lithium on 4-tert.butylcyclohexanone increased on passing from zinc to the alkalimetals [28]:





The hard or soft character of the allylic nucleophile, which varies according to the hardness or softness of the accompanying cation, appeared to be the domminating factor in the stereochemistry of this reaction.

Propargylic organozinc compounds $RC\equiv CCH_2ZnBr$ were shown to add to acetylenes and their organomagnesium derivatives [29]:

$$RC\equiv CCH_2ZnBr \xrightarrow[2)\ H_2O]{1)\ R^1C\equiv CH} RC\equiv CCH_2CR^1=CH_2$$

R=Et,Pr; R^1= nonyl, Ph, CH_2OtBu

$$EtC\equiv CCH_2ZnBr \xrightarrow[2)\ H_2O]{1)\ R^1C\equiv CMgBr} EtC\equiv CCH_2-CR^1=CH_2 \qquad R^1=CH_2OH,\ CH_2OPh$$

D. Miscellaneous reactions of organozinc and organocadmium compounds

Thiele and co-workers have continued their study of the use of alkylzinc compounds in alkylating transition metals. When vanadium IV chloride was treated with dimethylzinc in diethylether, three different products were isolated, depending on the stoechiometry of the reactants [30]:

a) $2VCl_4 + Me_2Zn \longrightarrow 2\ VCl_3.MeZnCl + MeCl$

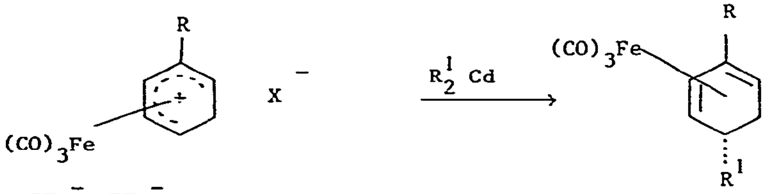
$\qquad\qquad\qquad\qquad\qquad\qquad \xrightarrow[0.5\ Me_2Zn]{} 2\ VCl_3.MeZnCl.0.5\,Me_2Zn$

b) $2\ VCl_4 + 2\ Me_2Zn \longrightarrow 2\ Me\ VCl_2.MeZnCl.ZnCl_2 + MeCl$

c) $2\ VCl_4 + 3\ Me_2Zn \longrightarrow \begin{cases} 2\ VCl_2.3\ MeZnCl + MeCl + Me. \\ \text{or} \\ 2\ MeVCl.MeZnCl.2\ ZnCl_2 + MeCl + Me. \end{cases}$

No choice could be made between the two reaction products in the case of reaction c.
The reaction of diethylzinc with VCl_4 in 1 : 1 molar ratio gave a mixture of ethylvanadiumdichloride, zinc chloride and ethylzinc chloride.

The scope of the alkylation of both cyclic and non cyclic tricarbonyl dienyliron salts by organocadmium reagents was investigated by Birch and Pearson [31] (cf. AS 130; 168):



$X = BF_4^-, PF_6^-$

R^1 = Ph, $PhCH_2$, iPr, allyl, methallyl, crotyl.

Alkylation of the cyclohexadienyl complexes mainly occurred regioselectively on the terminal carbon of the dienyl system and stereospecifically on the face opposite to the iron tri carbonyl group.
The regio selectivity was poor in the acyclic diene series, although the cisoïd geometry of the double bond was retained in the products.
In the reaction of bis(π-allyl) dipalladium dichloride with diethylcadmium in toluene at -15°, no alkylation of the palladium was observed. Instead, a mixture of hydrocarbons, cadmium, cadmium chloride, and palladium was obtained [32].
Mengoli and co-workers have coupled the cathodic formation - by electrocatalysis and electrosynthesis - of zinc alkyls to anodic processes which lead to the synthesis of organolead and organotin compounds by using the zinc alkyls as alkylating agents [33], [34], [35]. To this purpose, methyl iodide and methyl bromide solutions in DMF were electrolyzed between a zinc cathode and lead or tin anodes. High yields of lead- and tin alkyls were obtained.

The first silylcadmium compound isolated in substance, bis (tri-tert.butylsilyl) cadmium, was prepared by reacting tri-tert.butylsilane with diethylcadmium in 2 : 1 molar ratio at 90° [36]:

$$2(t.Bu)_3SiH + Et_2Cd \longrightarrow [(t.Bu)_3Si]_2Cd + 2\ EtH$$

The reactions of dimethylzinc and dimethylcadmium with nitric oxide have been reinvestigated [37]. In both cases, two moles of NO are taken up by each mole of dimethylzinc or dimethylcadmium. From IR, Raman and mass spectral data the authors conclude that the adduct should be formulated as $Me\ M[ON(NO)Me]$ (M = Zn,Cd) instead of $Me_2M.M[ON(NO)Me]_2$ as suggested by Frankland [38].Upon hydrolysis, the adducts disproportionate as follows:

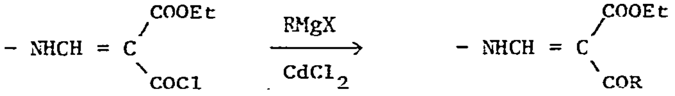
$$2\ MeM[ON(NO)Me] + H_2O \longrightarrow M[ON(NO)Me]_2 + M(OH)_2$$

The reaction of triphenylchloromethane with organocadmium compounds yielded alkylation products and/or reduction products, depending on the nature of the organic radical in the organometallic compound [39]:

$$Ph_3CCl + Me_2Cd \text{ —— } 98 \text{ mole \% } Ph_3CMe$$

$$Ph_3CCl + Et_2Cd \text{ —— } 75 \text{ mole \% } Ph_3CH + 20 \text{ mole \% } Ph_3CEt$$

Enamines XXVI have been prepared from the corresponding acid chlorides XXVII using the classical cadmium-ketone synthesis [40]:



$$-\ NHCH = C\begin{matrix} ^{/COOEt} \\ _{\backslash COCl} \end{matrix} \xrightarrow[CdCl_2]{RMgX} -\ NHCH = C\begin{matrix} ^{/COOEt} \\ _{\backslash COR} \end{matrix}$$

XXVII XXVI

III Organozinc compounds as polymerization catalysts

As in earlier years, only those papers in which organozinc compounds play a major role will be discussed.

Hsieh [41] has investigated the effects of diethylzinc on the alkyllithium - initiated polymerizations of butadiene and styrene. It appeared that diethylzinc complexed with the initiator and effectively increased the initiation rate. This was particularly true with initiators having low solubility due to their very high degree of association. Diethylzinc also lowered the apparent solution viscosity by complexation with aggregated polymer-lithium molecules. In the presence of bases like THF, diethylzinc served as chain-transfer agent by exchange between R-(monomer)n-Li and R^1_2Zn.

Diethylzinc has been used to polymerize organic thiocyanates into polymers having a high degree of purity [42].

The combination of diethylzinc and tertiary phosphines appeared to catalyze the co-polymerization of epoxy compounds with carbon disulfide [43].

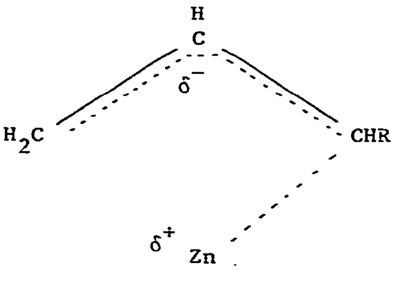
Pasienkiewicz and co-workers investigated the alternating copolymerization of propylene oxide with carbon dioxide in the presence of various organometallic catalysts including organozinc compounds [44].

IV Physical and spectroscopic studies

Mixing enthalpies for diethylzinc and diethylcadmium with triethylamine have been determined [45]. No evidence for electron donation by the d^{10} orbitals of Et_2Zn and Et_2Cd was found.

The thermal decomposition of di-tert. butylzinc in deuterated solvents was followed by 'H-CIDNP [46].The decomposition products included the polarized disproportionation products isobutane and isobutene and the unpolarized combination product 2, 2, 3, 2 - tetramethylbutane. The main decomposition process appeared to follow a radical mechanism in which uncorrelated tert. butyl pairs were involved.

The nuclear magnetic saturation transfer technique has been used to investigate the cis-trans isomerization of bis(5,5-dimethyl-2-hexenyl) zinc [47]. The secondary metalalkenyl XXVII was postulated as an intermediate in this isomerization:



XXVII

Both isomers behave like dynamic allylic systems.

The electrical field-gradient splitting of the cadmium 4d energy levels in dimethylcadmium was determined by high-resolution photoelectron spectroscopy in the gasphase [48].

A procedure for the indirect gaschromatographic determination of organic impurities in dialkylzinc and- cadmium compounds has been worked out [49]. The method is based on preliminary hydrolysis, extraction, concentration and subsequent glc-analysis. The sensitivity of the method appeared to be 10^{-4} to 10^{-3}%.

V Toxicology

The pathological changes in cat kidneys after intoxication with methylcadmium chloride has been studied [50]. Severe degenerative changes were observed. Methylcadmium chloride appeared to be even more nephrotoxic than inorganic cadmium compounds and it was suggested that more attention should be paid to its potential as an environmental hazard.

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