AMIDO DERIVATIVES OF METALS AND METALLOIDS
XII*. FURTHER REACTIONS WITH PROTIC COMPOUNDS

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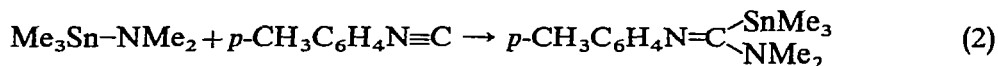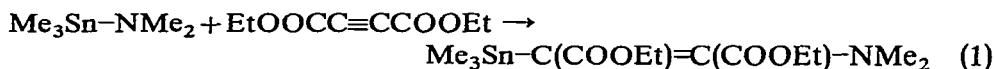*School of Molecular Sciences, University of Sussex, Brighton BN1 9QJ (Great Britain)*

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SUMMARY
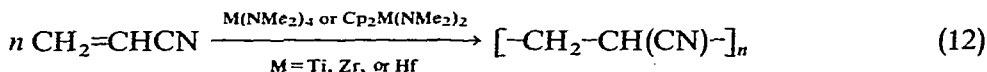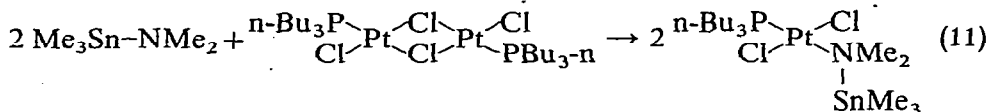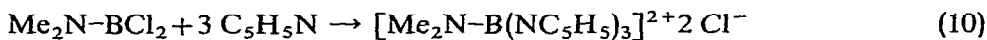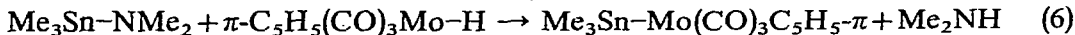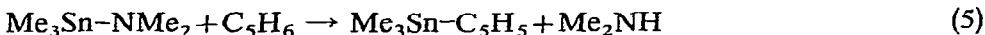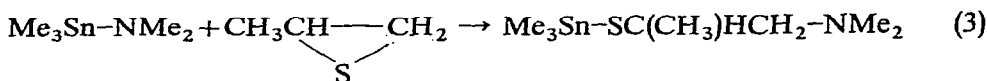
(Dimethylamido)trimethyltin, Me_3SnNMe_2 reacts with pentafluorobenzene in refluxing benzene to give trimethyl(pentafluorophenyl)tin, $Me_3SnC_6F_5$. This is the first example of such an amine elimination reaction involving an aromatic hydrocarbon. Amides of titanium(IV) [*e.g.* $Ti(NMe_2)_4$] or zirconium(IV) were found not to react with C_6F_5H; but the amine-elimination reaction furnished the following derivatives from appropriate organometallic dimethylamides: $Ti(\overline{NCH_2CH_2})_4$, Cp_2-$Zr(\overline{NCH_2CH_2})_2$ ($Cp = \pi\text{-}C_5H_5$), $\pi\text{-}(MeC_5H_4)Ti(C\equiv CPh)(NMe_2)_2$, $Cp_2M(C\equiv CPh)_2$ ($M = Zr$, or Hf), $[\pi\text{-}(MeC_5H_4)]_2Zr(C\equiv CPh)_2$, $CpNb(NMe_2)_3$, $Me_3SnC\equiv C(CH_2)_2$-$Pr\text{-}i$, $Me_3SnC\equiv CC(Me)=CH_2$, and $Me_3SnC\equiv C(CH_2)_3C\equiv CSnMe_3$.

INTRODUCTION

Organometallic amides (*e.g.*, $LMNMe_2$, where L represents the sum of all ligands other than one NMe_2 group attached to the metal or metalloid M) have proved to be remarkably reactive compounds. The principal reaction types may be classified as follows: (*i*) 1,2-insertion reactions[1-3], (*ii*) 1,1-addition[1,3], (*iii*) 1,3-insertion[1,4], (*iv*) 1,4-insertion[1,4], (*v*) reactions with protic compounds[5], (*vi*) reactions with metal hydrides[6], (*vii*) metathetical exchange reactions with inorganic complexes[7], (*viii*) metathetical exchange reactions with organic compounds[8], (*ix*) dehydrochlorination reactions[9], (*x*) reactions with bases[10], (*xi*) reactions with Lewis acids[11], and (*xii*) polymerisation initiation reactions[12]. These are examplified by eqns. (1)–(12), for the sake of simplicity for the case of the tin amide Me_3SnNMe_2 [unless this is unreactive, *i.e.* eqns. (10) and (12)].

$$Me_3Sn-NMe_2 + EtOOCC\equiv CCOOEt \rightarrow$$
$$Me_3Sn-C(COOEt)=C(COOEt)-NMe_2 \quad (1)$$

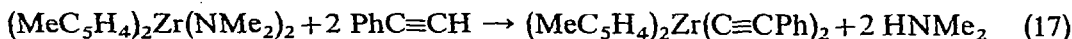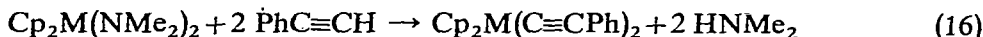$$Me_3Sn-NMe_2 + p\text{-}CH_3C_6H_4N\equiv C \rightarrow p\text{-}CH_3C_6H_4N=C\begin{smallmatrix}\diagup SnMe_3 \\ \diagdown NMe_2\end{smallmatrix} \quad (2)$$

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* For Part XI see ref. 22.

$$Me_3Sn-NMe_2 + CH_3CH\overbrace{}CH_2 \rightarrow Me_3Sn-SC(CH_3)HCH_2-NMe_2 \quad (3)$$
$$\overset{}{\underset{S}{}}$$

$$Me_3Sn-NMe_2 + \overline{CH_2CH_2OCO} \rightarrow Me_3Sn-OCH_2CH_2C(O)-NMe_2 \quad (4)$$

$$Me_3Sn-NMe_2 + C_5H_6 \rightarrow Me_3Sn-C_5H_5 + Me_2NH \quad (5)$$

$$Me_3Sn-NMe_2 + \pi\text{-}C_5H_5(CO)_3Mo-H \rightarrow Me_3Sn-Mo(CO)_3C_5H_5\text{-}\pi + Me_2NH \quad (6)$$

$$3\,Me_3Sn-NMe_2 + BF_3 \cdot OEt_2 \rightarrow B(NMe_2)_3 + 3\,Me_3Sn-F + Et_2O \quad (7)$$

$$Me_3Sn-NMe_2 + (CH_3CO)_2O \rightarrow Me_3Sn-OCOCH_3 + CH_3CONMe_2 \quad (8)$$

$$Me_3Sn-NMe_2 + (Ph_3P)_3IrCl_2H \rightarrow Me_3Sn-Cl \cdot HNMe_2 + (Ph_3P)_3IrCl \quad (9)$$

$$Me_2N-BCl_2 + 3\,C_5H_5N \rightarrow [Me_2N-B(NC_5H_5)_3]^{2+}2\,Cl^- \quad (10)$$

$$2\,Me_3Sn-NMe_2 + \underset{Cl}{\overset{n\text{-}Bu_3P}{}}{>}Pt\underset{Cl}{\overset{Cl}{>}}Pt\underset{PBu_3\text{-}n}{\overset{Cl}{<}} \rightarrow 2\,\underset{Cl}{\overset{n\text{-}Bu_3P}{}}{>}Pt\underset{NMe_2}{\overset{Cl}{<}} \quad (11)$$
$$\underset{SnMe_3}{\overset{|}{}}$$

$$n\,CH_2{=}CHCN \xrightarrow[\text{M = Ti, Zr, or Hf}]{M(NMe_2)_4 \text{ or } Cp_2M(NMe_2)_2} [-CH_2-CH(CN)-]_n \quad (12)$$
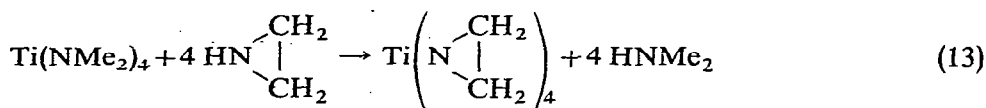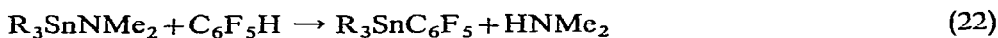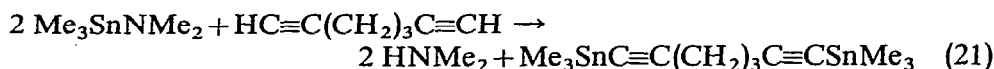
With regard to reaction type (v), hydrocarbon acids which have previously proved reactive in the sense of eqn. (5), other than cyclopentadiene, have been: $RC{\equiv}CH^5$, $HC{\equiv}CH$, indene, CX_3H $(X = Cl, Br)^{13}, CH_2N_2{}^{14}, MeNO_2{}^5, Me_2CO,$ and $RR'CHCN$.

DISCUSSION

Compounds containing protic hydrogen often undergo amine elimination when treated with a metal amide (see above). Such reactions are now extended [eqns. (13)–(22)] to a cyclic amine, monosubstituted acetylenes, a diacetylene, and pentafluorobenzene.

$$Ti(NMe_2)_4 + 4\,HN{\overset{\diagup CH_2}{\underset{\diagdown CH_2}{|}}} \rightarrow Ti\left(N{\overset{\diagup CH_2}{\underset{\diagdown CH_2}{|}}}\right)_4 + 4\,HNMe_2 \quad (13)$$

$$Cp_2Zr(NMe_2)_2 + 2\,HN{\overset{\diagup CH_2}{\underset{\diagdown CH_2}{|}}} \rightarrow Cp_2Zr\left(N{\overset{\diagup CH_2}{\underset{\diagdown CH_2}{|}}}\right)_2 + 2\,HNMe_2 \quad (14)$$

$$(MeC_5H_4)Ti(NMe_2)_3 + PhC{\equiv}CH \rightarrow (MeC_5H_4)Ti(C{\equiv}CPh)(NMe_2)_2 + HNMe_2 \quad (15)$$

$$Cp_2M(NMe_2)_2 + 2\,PhC{\equiv}CH \rightarrow Cp_2M(C{\equiv}CPh)_2 + 2\,HNMe_2 \quad (16)$$

$$(MeC_5H_4)_2Zr(NMe_2)_2 + 2\,PhC{\equiv}CH \rightarrow (MeC_5H_4)_2Zr(C{\equiv}CPh)_2 + 2\,HNMe_2 \quad (17)$$

$$Nb(NMe_2)_5 + CpH \rightarrow CpNb(NMe_2)_3 + HNMe_2 \quad (18)$$

$$Me_3SnNMe_2 + HC{\equiv}C(CH_2)_2CHMe_2 \rightarrow Me_3SnC{\equiv}C(CH_2)_2CHMe_2 + HNMe_2 \quad (19)$$

$$Me_3SnNMe_2 + HC{\equiv}CC(Me){=}CH_2 \rightarrow Me_3SnC{\equiv}CC(Me){=}CH_2 + HNMe_2 \quad (20)$$

$$2 \; Me_3SnNMe_2 + HC{\equiv}C(CH_2)_3C{\equiv}CH \; \rightarrow$$
$$2 \; HNMe_2 + Me_3SnC{\equiv}C(CH_2)_3C{\equiv}CSnMe_3 \quad (21)$$

$$R_3SnNMe_2 + C_6F_5H \; \rightarrow \; R_3SnC_6F_5 + HNMe_2 \qquad (22)$$

These reactions proceed under mild conditions and are of preparative value for obtaining a wide range of functionally-substituted organometallic compounds.

Upon adding excess of ethyleneimine to tetrakis(dimethylamido)titanium [eqn. (13)] or dicyclopentadienylbis(dimethylamido)zirconium [eqn. (14)], the corresponding aziridides were obtained. By contrast, similar reactions with acyclic secondary amines gave[15] mixed compounds of the type $(Me_2N)_nTi(NR_2)_{4-n}$; the difference in the two systems no doubt has a steric origin. It was thought possible that ethyleneimine might be polymerised by the amides, by analogy with their effect on alkylene sulphides[12], although epoxides do not react.

The titanium aziridide is a non-volatile, hygroscopic, orange solid, whereas the white zirconium compound can be sublimed at 100°/0.005 mm. The NMR spectra of these compounds show a singlet for

$$-N{\overset{\textstyle CH_2}{\underset{\textstyle CH_2}{\big|}}} \quad \text{(Table 2)}.$$
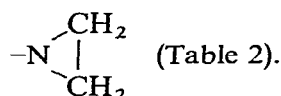
Reactions (15)–(17) provide a convenient route to the Group IVA transition metal acetylides; which are of interest as rare examples, for these elements, of complexes containing metal–carbon σ-bonds. Although the products of these reactions were not analytically pure, their formulation and structures were substantiated by

TABLE 1

IR DATA ON GROUP IVA METAL ACETYLIDES[a]

Compound	$\nu(C{\equiv}C)$ (cm^{-1})	$\nu(C{=}C)$ (cm^{-1})
$(MeC_5H_4)Ti(C{\equiv}CPh)(NMe_2)_2$	2080 m	1595 m
$Cp_2Zr(C{\equiv}CPh)_2$	2073 s	1592 s
$Cp_2Ti(C{\equiv}CPh)_2$ (see ref. 16)	2070 s	1590 s
$Cp_2Hf(C{\equiv}CPh)_2$	2083 s	1593 m
$(MeC_5H_4)_2Zr(C{\equiv}CPh)_2$	2078 s	1598 m

[a] m = medium; s = strong.

IR (Table 1), NMR (Table 2), and mass (Table 3) spectra, and by a cleavage reaction [eqn. (23)].

$$Cp_2M(C{\equiv}CPh)_2 + 2 \; HCl \; \rightarrow \; Cp_2MCl_2 + 2 \; HC{\equiv}CPh \qquad (23)$$
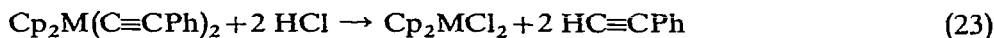
While this work was in progress, the preparation of similar titanium acetylides (by a sodium salt elimination) was reported[16].

The ^1H NMR spectra of the methylcyclopentadienyl derivatives were consistent with structures (I) and (II). Singlets corresponding to protons of the ring methyl and the phenyl group were present. The ring protons gave a group of 6 lines,

TABLE 2

CHEMICAL SHIFTS (τ) OF SOME NEW COMPOUNDS[a]

Compound	τ(Ph)	τ(Cp)	$\tau\left(-N\begin{smallmatrix}CH_2\\ \vert\\ CH_2\end{smallmatrix}\right)$ or τ(NMe$_2$)	τ(Me)
Ti(NC$_2$H$_4$)$_4$[b]			8.16(s)	
Cp$_2$Zr(NC$_2$H$_4$)$_2$[b]		3.95(s)	7.95(s)	
Cp$_2$Ti(C≡CPh)$_2$[c]	2.77(s)	3.62(s)		
Cp$_2$Zr(C≡CPh)$_2$[d]	2.73(s)	3.58(s)		
Cp$_2$Hf(C≡CPh)$_2$[d]	2.83(s)	3.77(s)		
(MeC$_5$H$_4$)Ti(C≡CPh)(NMe$_2$)$_2$[e]	2.75(m)	4.18(m)	6.9(s)	7.75(s)
(MeC$_5$H$_4$)$_2$Zr(C≡CPh)$_2$[d]	2.83(s)	3.88(m)		7.67(s)
CpNb(NMe$_2$)$_3$[b]		3.99(s)	6.86(s)	

[a] s = singlet; m = multiplet. [b] In C$_6$H$_6$. [c] See ref. 16. [d] In CS$_2$ (using TMS as standard). [e] In C$_6$D$_6$ (using TMS as standard).

TABLE 3

MASS SPECTROSCOPIC RESULTS FOR METAL ACETYLIDES[a]

Cp$_2$^{90}Zr-(C≡CPh)$_2$ (m/e)	Cp$_2$^{180}Hf-(C≡CPh)$_2$ (m/e)	Assignments of ions formed	(MeC$_5$H$_4$)$_2$^{90}Zr-(C≡CPh)$_2$ (m/e)	Assignments of ions formed
422	512	Cp$_2$M(C≡CPh)$_2^+$	450	(MeC$_5$H$_4$)$_2$-M(C≡CPh)$_2^+$
321	411	Cp$_2$M(C≡CPh)$^+$	349	(MeC$_5$H$_4$)$_2$-M(C≡CPh)$^+$
320		Cp(C$_5$H$_4$)M(C≡CPh)$^+$		
220[b]	310[b]	Cp$_2$M$^+$	248[b]	(MeC$_5$H$_4$)$_2$M$^+$
194		CpMC$_3$H$_3^+$		
			168	(C$_6$H$_6$)M$^+$

[a] An intense peak at m/e 202, due to (C≡CPh)$_2^+$, was observed in the spectra of all these compounds. [b] Most abundant peak, excluding (HC≡CPh)$^+$.

the pattern of which was in agreement with an A$_2$B$_2$ coupling system. A similar pattern has recently been observed for (MeC$_5$H$_4$)$_2$ZrCl$_2$[17].
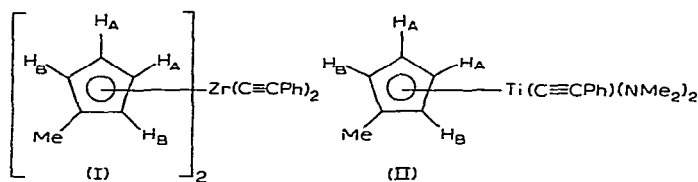
Mass spectra showed the monomeric parent molecular ion; the major metal-containing fragments are due to the loss of (PhC≡C).

Pentakis(dimethylamino)niobium and cyclopentadiene gave dimethylamine and a black intractable mass [eqn. (18)]. Attempts to sublime or distil the residue

resulted in its decomposition. However, the ^{1}H NMR spectrum (Table 2) of the residue in benzene is consistent with the formula CpNb(NMe$_2$)$_3$. Reactions involving the reduction of Nb^V → Nb^{IV} in other displacement reactions have been reported[18].

Reactions of aminostannanes with acetylenes proceeded smoothly in good yield. In reaction (20), amine elimination was evidently preferred over the potentially competing 1,2-addition at C=C. The preparation of (3-methyl-3-buten-1-ynyl)tri-methyltin (the product of reaction 20) (by a Grignard method in 37% yield) has previously been reported[19]. Reaction (21) (like that with acetylene[5]) proceeds directly to the second stage. A strong IR band at $\sim$2140 cm^{-1} in the spectra of these compounds is assigned to ν(C≡C).

Aminosilanes and -germanes did not react with 5-methyl-1-hexyne. This is attributed to a combination of the lower basicity of the Si–N or Ge–N compounds (compared with Sn–N), combined with the relatively strong M–N bonds.

The formation of the trialkyl(pentafluorophenyl)tin R$_3$SnC$_6$F$_5$ (R = Me, Et) by reaction (22) is surprising as (i) C$_6$F$_5$H is an aromatic hydrocarbon, (ii) C$_6$Cl$_5$H does not react[5], and (iii) amine elimination [eqn. (22)] is favoured over nucleophilic substitution of fluoride by an –NMe$_2$ group (see ref. 4). The compound Me$_3$SnC$_6$F$_5$ has been independently prepared from the chloride Me$_3$SnCl and the appropriate Grignard reagent[20]. Previously we have taken the view that a metal amide, such as LM–NMe$_2$, will react with a protic compound HA if the latter has high acidity and low pK_a[5]. However, we had not expected pentafluorobenzene to be particularly acidic, in this sense[21] (e.g. not comparable with C$_5$H$_6$), because the conjugate base C$_6$F$_5^-$ has no special stability (say compared with C$_5$H$_5^-$). The electron-attraction of the fluorine substituents is purely inductive. The present experiment suggests that the appropriate criterion for the reactivity of HA is its *kinetic* rather *thermodynamic* acidity. Pentafluorobenzene certainly is kinetically a strong acid, as shown by the high rate of hydrogen/deuterium exchange in basic media[21].

Unexpectedly the Group IVA amides did not react with pentafluorobenzene. This may be due to the fact that the preparation of compounds having a Ti–C (or Zr–C) σ-bond is not likely to be thermodynamically favourable. However, the compounds Cp$_n$M(C$_6$F$_5$)$_{4-n}$ (n = 2, 3) are known.

In the following systems, the reagents did not react under the stated conditions; essentially quantitative recovery of starting materials was achieved in: (a) Me$_3$Si-NMe$_2$ and Bu$_3$GeNMe$_2$ with NC≡C(CH$_2$)$_2$CMe$_2$, stirred (2–5 h) at 100°; (b) Me$_3$Sn-NMe$_2$ with C$_6$Cl$_5$H, reflux (2 h) in benzene; (c) Bu$_3$B with C$_6$F$_5$H, heated (2 h) at 100°; (d) Me$_3$SiNMe$_2$ with C$_6$F$_5$H, heated ($\sim$4 h) at 150°; (e) Bu$_3$SnOMe and Et$_3$SnH with C$_6$F$_5$H, stirred ($\sim$10 h) at 100°; (f) Ti(NMe$_2$)$_4$ and Cp$_2$Zr(NMe$_2$)$_2$ with C$_6$F$_5$H, heated (1 h) at 100°; and (g) (Et$_3$P)$_2$PtHCl with C$_6$F$_5$H, stirred ($\sim$5 h) in refluxing benzene.

EXPERIMENTAL

Reaction of tetrakis(dimethylamido)titanium with ethyleneimine

Ethyleneimine (0.70 g, 4.4 mol) in light petroleum (2 ml) was slowly added at 20° to a solution of tetrakis(dimethylamido)titanium (0.82 g, 1 mol) in the same solvent (5 ml). An orange solid immediately precipitated. After stirring (1 h), volatiles were removed under reduced pressure. The residue was washed with light petroleum

(5 ml) to afford orange tetraazirididotitanium (0.78 g, 98.7%), m.p. > 110° (decompn.). (Found: N, 25.7; Ti, 22.5. $C_8H_{16}N_4Ti$ calcd.: N, 25.9; Ti, 22.2%.) A sample of this product (0.3 g) decomposed when heated at > 150°/0.005 mm.

Reaction of dicyclopentadienylbis(dimethylamido)zirconium with ethyleneimine

Ethyleneimine (0.46 g, 4 mol) was slowly added to dicyclopentadienylbis(dimethylamino)zirconium (0.83 g, 1 mol) in benzene (5 ml) at 20°. The reaction mixture was stirred (1 h), and then freed from volatiles under reduced pressure. The residue was sublimed at 100°/0.001 mm, to give white dicyclopentadienyldiazirididozirconium (0.60 g, 73.3%). (Found: N, 9.0; Zr, 30.2. $C_{14}H_{18}N_2Zr$ calcd.: N, 9.2; Zr, 29.9%.)

Reaction of (methylcyclopentadienyl)tris(dimethylamido)titanium and phenylacetylene

The titanium amide (1.0 g, 1 mol), phenylacetylene (0.39 g, 1 mol), and benzene (3 ml) were heated (~3 h) at 100°. Dimethylamine (90%) was liberated. Other volatiles were removed under reduced pressure. The residual darkbrown, viscous liquid, believed to be (methylcyclopentadienyl)bis(dimethylamido)(phenylethynyl)-titanium (1.20 g, 98.4%) decomposed when heated.

Reaction of dicyclopentadienylbis(dimethylamido)zirconium with phenylacetylene

Dicyclopentadienylbis(dimethylamido)zirconium (1.40 g, 1 mol) and phenylacetylene (0.92 g, 2 mol) were stirred (~1 h) together in refluxing THF (5 ml). Volatiles were removed under reduced pressure, and the dark-brown residue was triturated with light petroleum (5 ml). The light-brown solid, believed to be dicyclopentadienylbis(phenylethynyl)zirconium (1.55 g, 80.7%) was filtered off, washed with light petroleum, and dried. (Found: C, 69.7; H, 4.9; mol.wt. mass spectrosc., 422. $C_{26}H_{20}Zr$ calcd.: C, 73.7; H, 4.8%; mol.wt., 423.7.) A portion (0.8 g) of this product was treated with hydrogen chloride (0.14 g, 2 mol) in ether (1 ml) at 20°. The reaction mixture was stirred ($\frac{1}{2}$ h); then light petroleum (10 ml) was added. Dicyclopentadienylzirconium dichloride (0.5 g, 90.6%) was filtered off. The filtrate was concentrated to give phenylacetylene (0.26 g, 67.4%) (authentic IR spectrum).

The hafnium analogue (70%) was similarly prepared and characterised.

Reaction of bis(methylcyclopentadienyl)bis(dimethylamido)zirconium and phenylacetylene

Phenylacetylene (0.67 g, 2 mol) and bis(methylcyclopentadienyl)bis(dimethylamido)zirconium (1.10 g, 1 mol) were stirred (~3 h) together in refluxing diethyl ether (5 ml). Volatiles were removed under reduced pressure. The residue was triturated with light petroleum (15 ml), then filtered, to afford orange bis(methylcyclopentadienyl)bis(phenylethynyl)zirconium (1.2 g, 81.6%) m.p. 73–75°. (Found: Zr, 19.5%; mol.wt. mass spectrosc., 450. $C_{28}H_{24}Zr$ calcd.: Zr, 20.2%; mol.wt., 451.7.)

Reaction of pentakis(dimethylamido)niobium with cyclopentadiene

Pentakis(dimethylamido)niobium (1.5 g, 1 mol), cyclopentadiene (0.63 g, 2 mol), and benzene (5 ml) were stirred (1 h) together at 100°. Dimethylamine was evolved and other volatiles were removed *in vacuo*. The black residue decomposed when heated at > 150°/0.005 mm.

Reaction of (dimethylamido)trimethyltin with 5-methyl-1-hexyne

(Dimethylamido)trimethyltin (2.59 g, 1 mol) was stirred (2 h) with 5-methyl-1-hexyne (1.4 g, 1.17 mol) at 100°. Distillation yielded the colourless (5-methyl-1-hexynyl)trimethyltin (2.62 g, 81.1%) b.p. 84°/8 mm. (Found: C, 46.2; H, 7.9%; mol.wt., 239. $C_{10}H_{20}Sn$ calcd.: C, 46.4; H, 7.8%; mol.wt., 247.) IR: $v(C\equiv C)$ 2133 cm^{-1}.

Reaction of (dimethylamido)trimethyltin with isopropenylacetylene

Isopropenylacetylene (0.9 g, 1.2 mol) was heated (2 h) with (dimethylamido)-trimethyltin (2.38 g, 1 mol) at 40°. Dimethylamine was liberated. The residue afforded the colourless (3-methyl-3-buten-1-ynyl)trimethyltin (2.03 g, 77.5%), b.p. 57°/9 mm (lit.[19] b.p. 40–42°/5 mm). (Found: C, 41.8; H, 6.3. $C_8H_{14}Sn$ calcd.: C, 42.0; H, 6.2%.) IR: $v(C\equiv C)$ 2158 cm^{-1} and $v(C=C)$ 1608 cm^{-1}.

Reaction of (dimethylamido)trimethyltin with 1,7-heptadiyne

(Dimethylamido)trimethyltin (5.64 g, 2.1 mol) and 1,7-heptadiyne (1.2 g, 1 mol), when heated (2 h) at 90°, afforded dimethylamine (1.06 g, 90.6%) and the colourless 1,7-bis(trimethylstannyl)heptadiyne (4.65 g, 85.5%), b.p. 94–95°/0.02 mm. (Found: C, 37.6; H, 5.8; mol.wt., 409. $C_{13}H_{24}Sn_2$ calcd.: C, 37.4; H, 5.8%; mol.wt., 417.7.) IR: $v(C\equiv C)$ 2155 cm^{-1}.

Reactions of (dimethylamido)trialkyltins with pentafluorobenzene

Pentafluorobenzene (1.06 g, 1 mol) and (dimethylamido)trimethyltin (1.31 g, 1 mol) were stirred ($\frac{1}{2}$ h) together at 20° and then heated under reflux (1 h) with benzene (5 ml). Dimethylamine was liberated. Further volatiles were removed at 20°/10 mm, to yield the colourless trimethyl(pentafluorophenyl)tin (1.50 g, 71.9%) b.p. 80°/9 mm (lit.[20] b.p. 34–36°/0.01 mm).(Found: C, 32.9; H, 2.8. $C_9H_9F_5Sn$ calcd.: C, 32.7; H, 2.7%.)

Similarly, pentafluorobenzene (1.29 g, 1 mol) and (dimethylamido)triethyltin (1.92 g, 1 mol) were stirred (~3 h) together in refluxing benzene (5 ml). The residue was distilled twice to give a colourless liquid (1.8 g, 62.9%), b.p. 52–53°/0.1 mm, characterised as triethyl(pentafluorophenyl)tin. (Found: C, 38.6; H, 4.0; F, 25.5%; mol.wt., 365. $C_{12}H_{15}F_5Sn$ calcd.: C, 38.7; H, 4.1; F, 25.5%; mol.wt., 372.9.)

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