observed in the conversions of $[Ru_6(CO)_{16}N]^-$ to $[Ru_5(CO)_{14}N]^-$ (quantitative with 1 atm of CO at 25 °C)[9] and $Ru_6(CO)_{17}C$ to $Ru_5(CO)_{15}C$ (quantitative with 80 atm of CO at 70 °C0.[6] We are currently studying methods of forming the anionic cluster $[Ru_5(CO)_{15}B]^-$, initially by removing the gold(I) phosphine group from 2.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research, to the SERC for a studentship (D.M.M.), and to the NSF for a grant (CHE9007852) toward the purchase of a diffractometer at the University of Delaware. Johnson-Matthey is thanked for loans of $RuCl_3$. We acknowledge preliminary work by Ann K. Keep and Xuejing Song on the preparation of 1.

Supplementary Material Available: Tables of crystal and refinement data, atomic coordinates, bond distances and angles, thermal parameters, and H atom fractional coordinates (8 pages). Ordering information is given on any current masthead page.

OM920309U

New Simple Route to Allylstannanes by Zinc-Mediated Coupling of Allyl Bromides with Bu_3SnCl or Bu_2SnCl_2 in an H_2O (NH_4Cl)/THF Medium

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Received June 4, 1992

Summary: A new zinc-mediated coupling reaction of allyl and allyl-like bromides with Bu_3SnCl (1) or Bu_2SnCl_2 (2), performed in H_2O (NH_4Cl)/THF medium, represents an easy route to the following organostannanes: $Bu_3SnCH_2CH{=}CH_2$ (3) and $Bu_2Sn(CH_2CH{=}CH_2)_2$ (4), $Bu_3Sn(C_4H_7)$ (5) and $Bu_2Sn(C_4H_7)_2$ (6) (where C_4H_7 stands for *trans*-crotyl, *cis*-crotyl, or α-methylallyl), and $Bu_3SnCH{=}C{=}CH_2$ (7). This striking one-pot procedure successfully improves the already general routes where Grignard or other conventional organometallic reagents are used.

Allylstannanes have been shown to be versatile reagents in organic synthesis[1] in (i) *allylstannation*[2] of carbonyl compounds, (ii) allylations promoted by $BF_3{\cdot}Et_2O$[3] or by scrambling reagents[4] such as Bu_2SnCl_2, Bu_3SnCl, $SnCl_4$, BCl_3, BBr_3, $TiCl_4$, and Cp_2TiCl_2, and (iii) catalyzed cross-coupling reactions of allyl halides with triorganoallylstannanes.[5]

The various approaches provide convenient methods for C–C bond formation via stoichiometric or catalyzed reactions. Even reactions leading to C–O–C formation, by means of a thermal condensation of two molecules of aldehydes with allyltin substrates, have been considered.[6] Furthermore, it has been demonstrated that allylstannations by means of allyltin halides occur under mild conditions, with very short reaction times and without the use of organic solvents and an inert-gas atmosphere.[2,6] In fact, allylstannations leading to C–C bond formation can be performed in the presence of water[7] and even in aqueous acid media.[8]

Synthetic routes to allylstannanes are based on the following procedures: (a) preparation of a conventional organometallic compound (Grignard, RLi, etc.) which will react with an organotin halide or oxide,[9] (b) reactions of

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Table I. Preparation of Allyl- and Allenylstannanes

entry no.	procedure	reagent					amt of H_2O^c/THF, mL	product	product amt, g (yield, %)
		organotin	amt, g (mmol)	organic bromide[a]	amt, g (mmol)	Zn amt,[b] g			
1	I	Bu_3SnCl	24.0 (73.7)	$CH_2{=}CHCH_2Br$	26.7 (220.7)	14.5	50/50	$Bu_3SnCH_2CH{=}CH_2$ (3)	15.6 (64)
2	II		60.0 (184.3)		28.9 (238.9)	15.3	200/200	3	46.5 (77)
3	I	Bu_2SnCl_2	20.0 (65.8)	$CH_2{=}CHCH_2Br$	47.2 (390.2)	25.8	100/100	$Bu_2Sn(CH_2CH{=}CH_2)_2$ (4)	16.0 (78)
4	I	Bu_3SnCl	24.0 (73.7)	$CH_3CH{=}CHCH_2Br$[d]	29.9 (221.5)	14.5	50/50	$Bu_3Sn(C_4H_7)$[e] (5)	20.8 (82)
5	II		24.0 (73.7)		12.9 (95.5)	6.3	100/100	5[f]	20.1 (79)
6	I	Bu_2SnCl_2	20.0 (65.8)	$CH_3CH{=}CHCH_2Br$[d]	53.2 (394.0)	25.8	150/150	$Bu_2Sn(C_4H_7)_2$[g] (6)	20.1 (89)
7	II		20.0 (65.8)		23.1 (171.1)	11.2	100/100	6[h]	19.4 (87)
8	II	Bu_3SnCl	24.0 (73.7)	$CH{\equiv}CCH_2Br$	11.4 (95.8)	6.3	100/100	$Bu_3SnCH{=}C{=}CH_2$ (7)	19.5 (80)

[a] Organotin chlorides and organic bromides (commercially available from Aldrich) were purified before use. [b] Zinc powder 325 mesh (commercially available from Aldrich) was used. [c] Deionized water saturated with NH_4Cl was used. [d] Crotyl bromide consists of a mixture of trans (76%), cis (9%), and α-methylallyl forms. (15%). [e] Isomeric composition: *trans*-crotyl 59% and *cis*-crotyl 41%. [f] Isomeric composition: trans 34%, cis 52%, and α-methylallyl 14%. [g] Isomeric composition: trans,trans 6%, cis,trans 42%, and cis,cis 52%. [h] Isomeric composition: trans,trans 20%, cis,trans 51%, and cis,cis 29%.

stannyl anions with allylic or organic substrates,[10] (c) 1,4-hydrostannation of 1,3-dienes,[11] (d) preparation of organotin phosphorus ylides as intermediates,[12] and (e) thermal rearrangement of mixed (allylalkoxy)organostannanes.[13] Both procedures (b) and (c) are also applicable to the formation of allenylstannanes.[14] However, all these procedures require two or more steps and all operations must be carried out under a dry, oxygen-free atmosphere.

Thus, we have devoted our efforts to find a simple and direct synthetic procedure, where triorgano- and diorganoallylstannanes can be produced in a one-pot reaction and in the presence of air and water. Our best procedure to date is depicted in eq 1. A Wurtz-type reductive

$$n\mathrm{RBr} + \mathrm{Bu}_{4-n}\mathrm{SnCl}_n \xrightarrow{\mathrm{Zn/H_2O\ (NH_4Cl)/THF}} \mathrm{Bu}_{4-n}\mathrm{SnR}_n \qquad (1)$$

$$\mathrm{R} = \text{allyl or allyl-like group};\ n = 1,2$$

coupling reaction mediated by zinc powder forms the basis of this reaction, which has some analogies with those performed in H_2O (NH_4Cl)/THF medium between allyl bromides, aldehydes, and zinc to give homoallylic alcohols.[15] Zinc powder is known to be active either in water or in saturated NH_4Cl aqueous medium.[15] In the latter case, the activation process is accelerated, since the zinc particle surface is continuously cleaned from oxides by the acidity of the ammonium salt solution.

Table I summarizes the experimental data and results for the following organostannanes: $Bu_3SnCH_2CH{=}CH_2$ (3), $Bu_2Sn(CH_2CH{=}CH_2)_2$ (4), $Bu_3Sn(C_4H_7)$ (5), $Bu_2Sn(C_4H_7)_2$ (6) (where C_4H_7 stands for *trans*-crotyl, *cis*-crotyl, or α-methylallyl), and $Bu_3SnCH{=}C{=}CH_2$ (7). Two different procedures, I and II, with differing stoichiometries have been adopted.[16] Both are equivalent if one considers that yields are practically identical for a given compound. Procedure II is more economical than procedure I when expensive allyl bromides are employed. In addition, if we take into account that the reaction is highly exothermic, procedure II allows a more accurate control of the temperature when large amounts of reactants are employed.

We must emphasize the particular facility of this coupling reaction, which is performed in the presence of water and air. This is a further example of the use of aqueous media for organic synthetic purposes.[7,8,15,17] In the present cases, only short times are necessary to isolate the pure products. For example, preparation of 46.5 g of compound 3 (see Table I, entry 2) requires only 4 h, which is much faster than other procedures.

Organostannanes 5 and 6 are isolated as isomeric mixtures, the compositions of which are strongly dependent on the workup, analogous to results obtained when Grignard procedures are used.[9] The compound $Bu_3Sn(C_4H_7)$ (5), synthesized by procedure I (entry 4), consists of a 59:41 mixture[18] of *trans*- and *cis*-C_4H_7 groups. On the other hand, by means of procedure II (entry 5) a mixture[18] containing three isomeric species in the ratio trans:cis:α-methylallyl = 34:52:14 is isolated. Neither of these com-

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(16) Procedure I: In a round-bottom two-necked flask (250–1000 mL) equipped with a condenser, the appropriate butyltin chloride and a 3-fold excess of allyl bromide were suspended in a H_2O (NH_4Cl saturated)/THF mixture. Then, zinc powder (in a 1:1 stoichiometric ratio with respect to allyl bromide) was carefully added with vigorous stirring over about 20 min (the reaction is almost instantaneous and highly exothermic). After addition, the mixture was stirred a further 15 min to ensure completion of the reaction. Extraction with Et_2O was performed; the separated organic layer was washed with saturated aqueous NaCl and dried over $MgSO_4$. Removal of solvent left an oil, which was distilled under vacuum to afford the pure allyltin compound. Procedure II: In a round-bottom two-necked flask equipped with a condenser and a dropping funnel, the appropriate butyltin chloride and a 30% excess of zinc powder were suspended in H_2O (NH_4Cl saturated)/THF mixture. With vigorous stirring, the allyl bromide (in a 1:1 stoichiometric ratio with respect to zinc) was added dropwise at a rate sufficient to maintain a gentle reflux. The addition took about 30 min. The heterogeneous mixture was stirred for a further 30 min and then extracted with petroleum ether (35–60 °C). The organic layer was separated, and workup was performed as described above to isolate the pure allyltin compound. See Table I for reactant and solvent quantities used.

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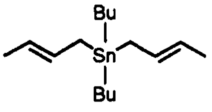
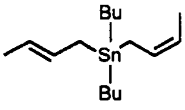
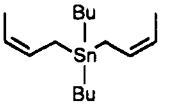
(18) The composition of the isolated isomeric mixture has been determined by ^{119}Sn NMR spectroscopy, by following the criteria adopted in ref 4c.

positions is related to the isomeric composition of the starting allyl bromide, which is trans:cis:α-methylallyl = 76:9:15.

The compound $Bu_2Sn(C_4H_7)_2$ (6) is obtained as an isomeric mixture in which the organic group C_4H_7 having the α-methylallyl configuration is completely absent. A mixture[18] of the isomers


trans,trans


trans,cis


cis,cis

is obtained with the following isomeric compositions: trans,trans:trans,cis:cis,cis = 6:42:52 (entry 6) and 20:51:29 (entry 7).

In the coupling reaction of Bu_3SnCl with propargyl bromide (entry 8) only the allenyl isomer 7 is obtained. This new route to allenylstannanes 7 has some advantages over the Grignard procedure, which requires a further step to isomerize the initial product mixture containing both isomers $Bu_3SnCH{=}C{=}CH_2$ and $Bu_3SnCH_2C{\equiv}CH$.[7b]

The present approach is analogous to the formation of Si—C and Ge—C bonds in organic solvents through coupling of organosilyl[19] and organogermyl chlorides[20] with α-bromo esters in the presence of zinc powder. These reactions presumably proceed via the intermediate formation of organozinc compounds. In our case, it is difficult to establish if organozinc or radical species are involved in the process. Zinc-mediated C–C bond-forming reactions in H_2O/THF medium are thought to follow a radical mechanistic pathway.[15]

These preliminary results clearly illustrate the potential of this Wurtz-type coupling reaction to form, in aqueous medium, simple allylstannanes. Further studies are now under way to apply this methodology to the preparation of more complex allyltin substrates.

Acknowledgment. We thank the Ministero dell'Università e della Ricerca Scientifica e Tecnologica (MURST), Rome, Italy, and the Consiglio Nazionale delle Ricerche, Rome, Italy, for financial support.

OM920316I

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Reactions of Palladium Amide Complexes with Dimethyl Acetylenedicarboxylate. Palladium-Mediated C–N Bond Formation

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Received June 17, 1992

Summary: Reaction of the complexes *trans*-$Pd(C_6H_4C(H){=}NPh)(NHPh)(PMe_3)_2$ (1) and *trans*-$Pd(C_6H_5)(NHPh)(PMe_3)_2$ (3) with dimethyl acetylenedicarboxylate (DMAD) results in the insertion of the acetylene into the Pd–N bond, forming *cis*- and *trans*-$Pd[(MeOOC)C{=}C(COOMe)(NHPh)](C_6H_4C(H){=}NPh)(PMe_3)_2$ (2) and *trans*-$Pd[(MeOOC)C{=}C(COOMe)(NHPh)](C_6H_5)(PMe_3)_2$ (4). Thermolysis of 2 at 110 °C gives a high yield (76%) of *N*-phenyl-1-anilino-3,4-bis(methylcarboxy)-1,2-dihydroisoquinoline (5).

The synthesis of late-transition-metal amide complexes (groups 8–10) continues to attract considerable interest from inorganic and organometallic chemists.[1-6] One reason for this interest is the potential use of these complexes to facilitate the formation of carbon–nitrogen bonds through the insertion of unsaturated organic molecules into the metal–nitrogen bond. Although examples of insertion reactions do exist for these complexes, relatively little is known about the factors which control these reactions.[2,3a,c,d,4,5a,b,6b,g,i] We now report that the reaction of two palladium–amido complexes with dimethyl acetylenedicarboxylate (DMAD) results in carbon–nitrogen bond formation.

When *trans*-$Pd(C_6H_4C(H){=}NPh)(NHPh)(PMe_3)_2$ (1)[7] was allowed to react with 1 equiv of DMAD in pentane at room temperature (eq 1), the immediate precipitation of an off-white solid was observed. The ^{1}H NMR spectrum

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