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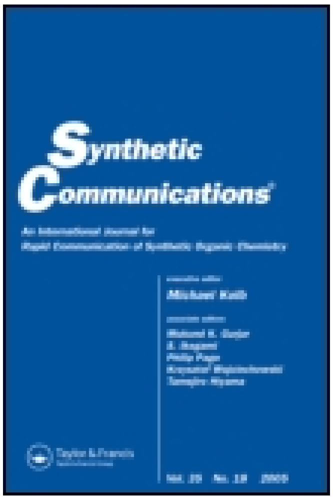

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RADICAL CYCLISATION OF 1-BROMO-2-(PROP-2-ENYLOXY)BENZENE USING A POLYMER-SUPPORTED ORGANOTIN HYDRIDE

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RADICAL CYCLISATION OF 1-BROMO-2-(PROP-2-ENYLOXY)BENZENE USING A POLYMER-SUPPORTED ORGANOTIN HYDRIDE

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ABSTRACT

The use of polymer-supported organotin hydride for the cyclisation 1-bromo-2-(prop-2-enyloxy)benzene **I** to give 2,3-dihydro-3-methylbenzofuran **II** has been studied. The supports were prepared by suspension copolymerisation of an organotin-functionalised monomer. Two approaches have been investigated either using a stoichiometric or a catalytic amount of organotin support.

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1. INTRODUCTION

Tributyl tin hydride plays a important role as reagent for reductive cyclisations.[1] However, the problem of separation, toxicity and disposal of trialkyltin derivatives are now broadly acknowledged.[2] Two kinds of solution have been proposed to solve this important problem. The first approach includes an assortment of workup procedures in order to remove the trialkyl tin derivatives from the reaction mixture.[3] In the second approach, attempts have been made to modify the structure of the tin hydride itself in order to facilitate separation and reduce toxicity.[4] Examples of this approach include the fluorous tin hydrides[5] and the polymer-bound tin hydrides.[6]

In the course of our work on the synthesis and applications of polymer-supported organotin reagents and catalysts,[7] our results on the radical cyclisation of 1-bromo-2-(prop-2-enyloxy)benzene using a polymer-supported organotin hydride are described below.

2. RESULTS AND DISCUSSION

2.1. Radical Cyclisation of 1-Bromo-2-(prop-2-enyloxy)-benzene Using Support P_1Cl

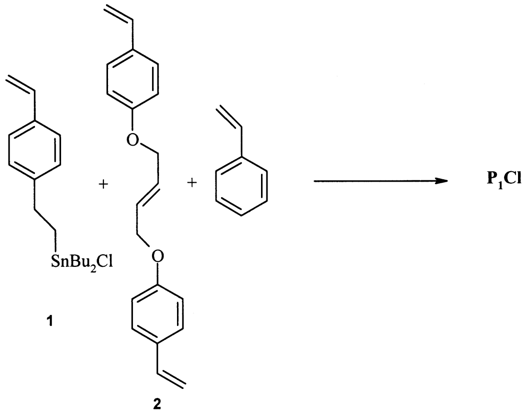
2.1.1. Synthesis of Support P_1Cl

Support $\mathbf{P_1Cl}$ was obtained by suspension copolymerisation of dibutyl-2-[(4-vinyl)phenylethyl]tin chloride **1** with *bis*-1,4-(4-vinylphenoxy)but-2*E*-ene **2** and styrene as previously reported[8] (Scheme 1).

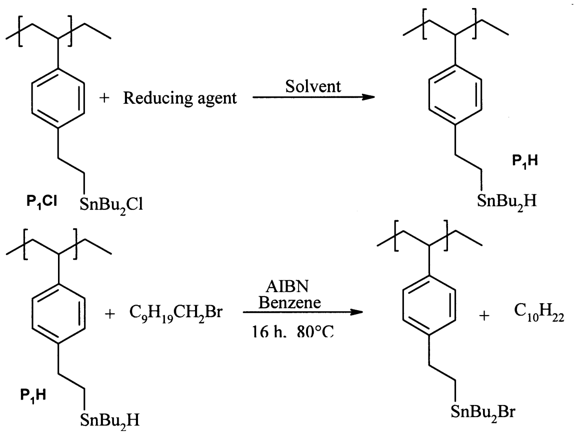
2.1.2. Reactivity of Organotin Polymer P_1Cl

Having the polymer-supported organotin chloride $\mathbf{P_1Cl}$ in hand, we first decided to test the ability of different reducing agents to convert the grafted Sn-Cl functions into their Sn-H counterpart to obtain support $\mathbf{P_1H}$. The polymer being insoluble, the reaction medium is heterogeneous, so we had to find a compromise between the compatibility of the solvent with the polymer and the solubility of the reducing agent. The Sn–Cl bonds were reduced into Sn-H using an excess of reducing agent. After washing of the beads by water, ethanol and ether, the efficiency of the reduction was estimated by a titration of the grafted Sn–H bonds by reduction of bromodecane into decane followed by GC (Scheme 2). The principal results of this investigation are reported on Table 1.

Sodium borohydride is well known to smoothly convert Bu_3SnCl in Bu_3SnH in polar solvents and complexing solvents such as ethyleneglycol dimethyl ether (glyme).[9] However, in our case, due to the poor compatibility of the glyme with the support, we had to use a large excess of reagent to obtain a satisfactory conversion level (Table 1, entries 1 and 2). Attempts to use reducing agents that were more compatible with the support, in solvents



Scheme 1. Preparation of support **P_1Cl**.



Scheme 2. Reduction of support **P_1Cl** and titration of the SnH functions.

Table 1. Reactivity of Support **P1Cl** Using Different Reducing Agents and Reduction Conditions

Entry	Reducing Agent (eq)	T (°C)	Reaction Time (h)	Solvent	Sn–Cl ($mmol.g^{-1}$)	Sn–H ($mmol.g^{-1}$)
1	$NaBH_4$ (1.5)	70	2	Glyme	0.6	0.11
2	$NaBH_4$ (20)	70	2	Glyme	0.6	0.30
3	$(C_8H_{17})_3CH_3NBH_4$ (1.5)	50	3.5	THF	0.6	0.17
4	$(C_8H_{17})_3CH_3NBH_4$ (20)	60	4	THF	0.6	0.28
5	TMDS/Bu_4NF (1.5)	50	3.5	THF	0.6	0.27
6	$NaBH_3CN$ (20)	60	2	THF	0.6	0.20
7	DIBAL-H (20)	70	2	Glyme	0.6	0.13

such as THF were made. Tricaprylmethylammonium borohydride readily soluble in THF was synthesised, its use does not significantly increase the yield of conversion of supported Sn-Cl into Sn-H functions (Table 1, entries 3 and 4). The use of the combination tertramethyldisiloxane/tetrabutylammonium fluoride (TMDS/Bu_4NF) was inspired by an analoguous published system: PMHS/KF.[10] The results obtained are similar with those observed using $NaBH_4$ (entry 5). Sodium cyanoborohydride, fairly soluble in THF, gave lower results than $NaBH_4$ (entry 6). Finally, the use of a large excess of DIBAL-H gave only a low conversion probably due to the presence of moisture into the polymer (entry 7).

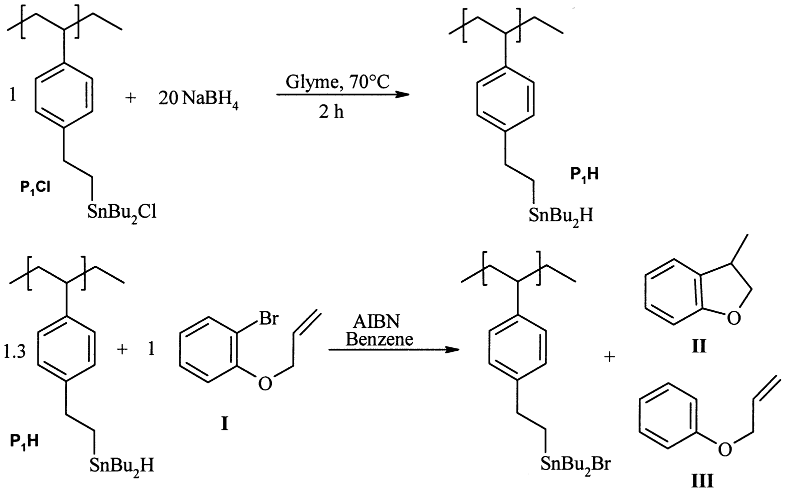
Therefore, we selected $NaBH_4$ as the reducing agent for the following of the study. This reagent is cheap, easy to handle and gives a reasonable Sn-H loading onto $\mathbf{P_1H}$.

2.1.3. Cyclisation Using a Stoichiometric Amount of Polymer-Supported Tin Hydride P_1H

The radical cyclisation of 1-bromo-2-(prop-2-enyloxy)benzene **I** is a good model for the research of a clean and effective synthesis of 2,3 dihydrobenzofurane derivatives.[11] The kinetics of the cyclisation of **I** has been studied by Beckwith et al.[12] They found that, whatever the concentration of Bu_3SnH used, the cyclisation arises almost exclusively by the exo mechanism, leading to the dihydrobenzofuran **II**.

The cyclisation reaction was initially carried out using a stoichiometric amount of $\mathbf{P_1H}$. Starting from an excess of supported tin chloride $\mathbf{P_1Cl}$, the first step produces the Sn-H functions using $NaBH_4$. A part of the reduced support obtained $\mathbf{P_1H}$ was used for the titration of the Sn-H functions according to the method previously described. The Sn-H loading obtained was 0.3 mmol/g. The second step consists in the radical cyclisation of **I**. This reaction was performed using benzene in the presence of AIBN which is a good solvent for the support and for radical reactions (Scheme 3 and Table 2).

The results obtained showed that the five-membered ring product **II** can be obtained with the same selectivity and yield with the polymer-supported tin hydride in benzene as with Bu_3SnH.[12] For this cyclisation, we used 12 g of polymer supported organotin reagent (0.3 mmol.g^{-1} of tin hydride) to cyclise 0.589 g (2.8 mmol) of substrate. Tin residue analysis by ICP/AES of the product gave 120 ppm, which is a promising result



Scheme 3. Radical cyclisation of 1-bromo-2-(prop-2-enyloxy) benzene **I** by support $\mathbf{P_1H}$.

Table 2. Radical Cyclisation of **I** Using a Stoichiometric Amount of SnH Functions Supported on $\mathbf{P_1H}$

Solvent	T (°C)	Reaction Time (h)	Conversion of **I** (%)	Ratio **II** : **III**
Benzene	80	7	99	96 : 4

considering that no special precautions have been taken during the synthesis of **II** i.e. the glassware used was not washed in a particular manner in order to remove tin residues.[13] It was thought that decreasing the temperature of the reaction would probably reduce the level of residual tin as the polymer backbone would be less damaged and therefore tin leaching would be reduced. In that case, we would have to find another initiator, as AIBN is poorly efficient below a temperatures of 60°C.

The recycling of the polymer **P_1H** failed. The yield of recovered compound **II** was only 20% on the second use. Treatment of **P_1H** with I_2 was attempted in order to break the Sn–Sn bond which could have been formed during the cyclisation. We recovered some of the reactivity (42% of conversion of **I**) but not completely. This behaviour may be attributed to $NaBH_4$ remaining from the first step and obstructing the pores of the polymer.

2.1.4. Cyclisation Using a Catalytic Amounts of Polymer-Supported Tin Chloride P_1Cl

In order to reduce the large quantities of support needed by the stoichiometric reaction, we decided to explore the possibility of synthesising **II** using a catalytic amount of support. We also thought that, by using less support, the level of tin residue would be reduced. This approach has already been described using $Bu_3SnCl/NaBH_4$.[14]

Several reactions using different reducing agents and a catalytic amount (10% mol) of supported tin chloride were carried out. The results are reported in Table 3.

The nature of the solvent seems crucial for the reactivity of the support. $NaBH_4$ is significantly soluble only in polar and complexant solvents such as glyme. The reaction solvent must solubilise the product and at the same time, it must be compatible with the polymer. Therefore, the

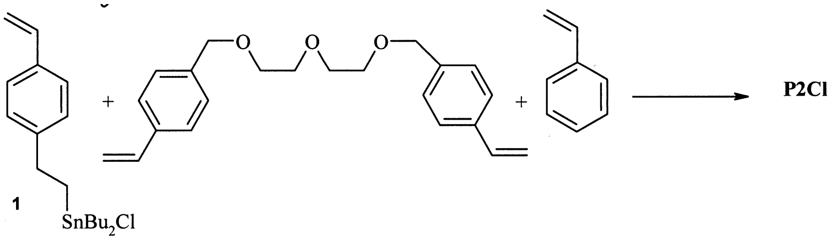
Table 3. Radical Cyclisation of **I** Using a Catalytic Amount (10%) of SnCl Functions Supported on **P_1Cl**

Reducing Agent (eq.)	Solvent	T (°C)	Reaction Time (h)	Conversion of **I** (%)	Ratio **II** : **III**
$NaBH_4$ (2)	Glyme	80	16	37	93 : 7
$NaBH_4$ (2)	Glyme/Benzene	80	16	10	91 : 9
$NaBH_4$ (20)	Glyme	80	16	89	93 : 7

reaction was performed in glyme using 2 equivalents of Sodium Borohydride and 10% molar (with respect to the substrate) of polymer-supported tin chloride **P_1Cl** (Table 3, entry 1). The conversion was rather low whereas the selectivity was good. This results is in agreement with the non recycling capacity of support **P_1Cl**. The use of a 1 : 1 mixture of glyme : benzene did not improve the conversion (entry 2). However, the use of a large excess of reducing agent (20 equivalents) gave an acceptable conversion level (entry 3). The tin residue analysis by ICP/AES of the product gave 15 ppm which is an acceptable level. For further work, it would be desirable to reduce this amount. One approach would be to increase the compatibility of the polymer with a polar solvent such as glyme, the solvent of choice for $NaBH_4$.

2.2. Radical Cyclisation of 1-Bromo-2-(prop-2-enyoxy)-benzene Using Support P_2Cl

In order to make a polymer more compatible with glyme, we copolymerised the organotin monomer **1** with di(ethyleneglycol)*bis*-(4-vinylbenzyl) ether **3** and styrene to give support **P_2Cl** (Scheme 4). Compound **3** was already successfully used as crosslinking agent in the preparation of polymers containing a chiral Schiff base-Zinc complex.[15] The support obtained had a loading in tin of 1 mmol.g^{-1}.



Scheme 4. Preparation of support **P_2Cl**.

The results of two successive cyclisation reactions using the same catalytic amount (10% mol) of supported tin chloride **P_2Cl** are reported in Table 4. Using **P_2Cl** as the support, product **II** was obtained in a very good yield in 3 h using 10 equivalents of $NaBH_4$ in glyme at 80°C. So the reactivity of the support **P_2Cl** was increased compared with **P_1Cl**. Recycling of this polymer

Table 4. Radical Cyclisation of **I** Using a Catalytic Amount (10%) of SnCl Functions Supported on **P_2Cl**

Run	Reducing Agent (eq.)	Solvent	T (°C)	Reaction Time (h)	Conversion of **I** (%)	Ratio **II** : **III**
1	$NaBH_4$ (10)	Glyme	80	3	99	98 : 2
2	$NaBH_4$ (10)	Glyme	80	20	93	95 : 5

was also possible. After the first use, the polymer was washed successively with ether, water, ethanol and acetone, then extracted for one day in a soxhlet apparatus with THF. Finally the polymer was dried. In that case, the tin residue analysis by ICP/AES of the product gave 10 ppm which is an acceptable level. On the second run, a conversion of 93% for compound **I** was obtained but a longer period of time (20 h) was necessary to reach this value. The third run of the recycling has failed (22%). Between the second and the third run, the polymer was only washed with ether, water and ethanol, therefore, it seems that an exhaustive washing of the support is necessary to recycle the polymer.

The quantities of $NaBH_4$ used can probably be reduced by performing the reaction at lower temperature.

3. CONCLUSION

Two methods for the cyclisation of 1-bromo-2-(prop-2-enyloxy)benzene **I** have been carried out. The cyclisation of **I** using a stoichiometric amount of polymer-supported organotin reagent **P_1H** behaves similarly as the homogeneous one with Bu_3SnH. However the recycling of the support failed. This behaviour may be attributed to the obstruction of the pores of the beads by tetrahydroborates by-products. A possible solution to this problem could be to wash the beads more thoroughly.

The cyclisation of **I** using a catalytic amount of polymer-supported organotin reagent **P_1Cl** behaves similarly as the homogeneous reaction with Bu_3SnCl using at least a 20 fold excess of reducing agent with respect to the substrate. Reduction of this quantity was attempted by the development of a new polymer **P_2Cl** more compatible with glyme, a good solvent of $NaBH_4$. The amount of reducing agent could be lowered and the stability toward recycling of the support increased.

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4. EXPERIMENTAL PART

4.1. Preparation of Monomers and Reducing Agents

4.1.1. Preparation of Dibutyl-2-[(4-vinyl)phenyl]ethyl Tin Chloride 1

The synthesis of dibutyl-2-[(4-vinyl)phenyl]ethyl tin chloride have been published by our group.[7]

4.1.2. Preparation of *bis*-1,4-(4-vinylphenoxy)but-2E-ene 2

The synthesis of *bis*-1,4-(4-vinylphenoxy)but-2E-ene have also been published by our group.[8]

4.1.3. Preparation of Di(ethylene glycol) *bis*(4-vinylbenzyl)ether 3

Compound **3** was synthesised according to a published procedure.[15]

4.1.4. Preparation of Tricaprylmethylammonium Borohydride

In a 100 ml three-necked flask were introduced tricaprylmethylammonium chloride (10 g–24.7 mmol) and water (5 ml). The suspension was stirred at room temperature. An aqueous solution of 5 M NaOH (3 ml) was added dropwise. The mixture was cooled at room temperature, then sodium borohydride (1.05 g, 27.7 mmol) in water (5 ml) was added. The solution was stirred overnight, then extracted with dichloromethane (2 × 25 ml). The organic phase was washed to neutrality and dried over anhydrous K_2CO_3. CH_2Cl_2 was removed *in vacuo* to afford a pale yellow oil (9 g, 95%).

4.2. Preparation of the support P_1Cl and P_2Cl

The preparation of supports $\mathbf{P_1Cl}$ and $\mathbf{P_2Cl}$ was made by suspension polymerisation according to the procedure already published.[7] Both polymers have a nominal crosslink of 20% and a loading from 0.6 to 1 $mmol.g^{-1}$ of tin chloride.

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4.3. Preparation of the Support P_1H and P_2H and Titration of the SnH Functions

The reduction of supported SnCl and the titration of the obtained SnH functions supported on $\mathbf{P_1H}$ and $\mathbf{P_2H}$ have already been described.[7,8]

4.4. Preparation 1-Bromo-2-(prop-2-enyloxy)benzene 1

1-bromo-2-(prop-2-enyloxy)benzene has been synthesised according to a published procedure.[11]

4.5. Preparation of 2,3-Dihydro-3-methylbenzofurane II by the Stoichiometric Route

Reduction step: A 250 ml three-necked flask was charged with polymer supported organotin chloride $\mathbf{P_1Cl}$ (13.0 g, 0.6 mmol.g^{-1} of Sn-Cl) and diglyme (100 mL). The flask was purged with argon, then sodium borohydride (6.0 g, 158 mmol) was added. The mixture was gently stirred during 2 h at 70°C. The polymer was filtered and washed with water, ethanol then ether.

Cyclisation step: A 250 ml three-necked flask was charged with 1-bromo-2-(prop-2-enyloxy)benzene **I** (0.589 g, 2.8 mmol), decane (200 mg) as an internal reference and benzene (100 mL). The mixture was purged with argon. AIBN (20 mg) was added and 12/13 of the freshly reduced polymer was added (12 g about 4.8 mmol of Sn-H, according titration with bromoadamantane, (see below). The reaction mixture was stirred at 80°C for 16 h to obtain a quantitative conversion for **I**. The suspension was filtered and the filtrate concentrated under reduced pressure to give a colorless oil (314 mg, 85%). Mixture of **II** : **III** = 97 : 3 as determined by G.C analysis. Tin residue analysis by ICP/AES gave 120 ppm.

NMR: δ_H (200 MHz; $CDCl_3$); 7.25–6.75 (m, 4H, arom H); 4.75–4.6, 4.20–4.05 (ABX, 2H, $H_{A,B}$); 3.60–3.25 (ABX, 1H, H_C); 1.30 (d, 3H, CH_3, J = 6.8 Hz). δ_C (75 MHz; $CDCl_3$); 159.8 (C_{7a}); 132.1 (C_{3a}); 127.8 (C_4); 123.5 (C_6); 123.5 (C_5); 109.3 (C_7); 78.2 (C_2); 36.5 (C_3); 19.1 (CH_3). These results are in accordance with the full spectroscopic data previously published.[11]

The remaining 1/13 of the freshly reduced polymer $\mathbf{P_1H}$ was used to reduce bromodecane. The Sn-H loading of the polymer was estimated to 0.3 mmol.g^{-1}.

4.6. Preparation of 2,3-Dihydro-3-methylbenzofurane II by the Catalytic Route

A 250 ml three-necked flask was charged with polymer supported organotin chloride **P_2Cl** (1.4 g, 1 mmol.g^{-1} of Sn-Cl), **I** (1.5 g, 70 mmol) and glyme (100 mL). The solution was purged with argon, then AIBN (500 mg–3 mmol) and sodium borohydride (5.3 g, 140 mmol) were added. The suspension was gently stirred during 3 h at 80°C to obtain 99% of conversion of **I** by GC. The mixture was filtered and washed with ether (3 × 50 mL). 50 mL of water were added to remove $NaBH_4$ residue. Organic phase was extracted with ether (2 × 50 mL) and concentrated to afford 1.1 g of a mixture **II** and **III** as determined by G.C. analysis (**II** : **III** = 49 : 1) (94%). Tin residue analysis by ICP/AES gave 10 ppm.

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