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Development of chromatographic methods to monitor the synthesis of (tributylstannyl)methanol through unstable lithiated intermediates

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Abstract

Simple and robust derivatization methods for monitoring the formation of (tributylstannyl)methanol from tributyltin hydride via unstable lithiated intermediates have been developed and validated. These analytes present both chromatographic and detection difficulties in their native states due to low volatility, poor aqueous solubility/stability, and lack of a chromophore. Derivatization of these analytes to trimethylsilyl analogues for gas chromatographic analysis or to benzenesulfonyl urethane analogues for reversed-phase liquid chromatographic analysis was evaluated. The derivatization/gas chromatographic methods developed were demonstrated to be more specific and sensitive for impurity monitoring during (tributylstannyl)methanol preparation.

Keywords: Derivatization, GC; Derivatization, LC; (Tributylstannyl)methanol; Organotin compounds

1. Introduction

Numerous analytical methods have been reported for the analysis of alkyltin species, however these methods may generally be placed into one of two categories: direct and indirect methods [1]. Direct methods of analysis are those which permit speciation and quantitation of tin compounds without pre-analysis chemical transformations. Gas chromatographic methods for speciating alkyltin halides have been reported, but at the expense of on-column degradation, analyte adsorption/reactivity with the stationary phase, and analyte rearrangement at the labile tin–halide bond [2–4]. To avoid these potential thermal effects, ambient liquid chromatographic methods have been developed in normal-phase [5], ion-pair reversed-phase [6], ion-exchange [7], and micellar reversed-phase modes [8,9]. When no UV chromophore is present in the alkyltin analyte, alternate detection schemes have been attempted such as refractive index [10], graphite furnace atomic absorption [11–16], flame atomic absorption [17], laser-induced ionization [18], laser-induced atomic fluorescence [19], and inductively coupled plasma MS [6,9,20–23]. However, refractive index detection is not adequately sensitive to enable trace impurity analysis and the required interfaces with atomic spectroscopy detectors are not commonplace in many industrial laboratories. Post-column flourescence complexation of mono- and dialkyltin species with morin hydrate has been reported to enhance analyte detectivity [24–26].

Indirect methods of alkyltin analysis are those which require a pre-analysis chemical transformation

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of the analyte. Such an approach is required in circumstances where the alkyltin species is either unstable to analysis, not amenable to any available methods of detection, or species cannot be chromatographically resolved in their native states. One common derivatization technique is Grignard alkylation to the respective methyl [27–31], ethyl [32–36], propyl [37,38], butyl [39], pentyl [40–48], and hexyl analogues [49–51] for GC analysis ($n=1$, 2, or 3; R and R′ are organic groups):

$$R_nSn^{(4-n)+} + R'MgX \rightarrow R_nSnR'_{(4-n)} \quad (1)$$

Another frequently used technique is to convert alkyltin species to the corresponding volatile hydride [52] under acidic conditions for GC analysis:

$$R_nSn^{(4-n)+} + NaBH_4, H^+ \rightarrow R_nSnH_{(4-n)} + H_2\uparrow \quad (2)$$

(Tributylstannyl)methanol is an organometallic reagent which was employed in a palladium-catalyzed cross coupling reaction used in the preparation of a novel carbapenem antibiotic [53]. Many of these methods of alkyltin separation and detection were considered during the development of analytical methods for monitoring the synthesis [54,55] of a (tributylstannyl)methanol reagent (Bu_3SnCH_2OH) from the corresponding hydride (Bu_3SnH):

$$Bu_3SnH \xrightarrow{LDA} Bu_3SnLi \xrightarrow{[HCHO]_n} Bu_3SnCH_2OLi \xrightarrow[\text{aqueous work-up}]{} Bu_3SnCH_2OH \quad (3)$$

While the starting material (Bu_3SnH) is suitably volatile to permit GC analysis with flame ionization detection (FID), the reactivity of the lithiated intermediates and high boiling point (>300°C) of the product alcohol (Bu_3SnCH_2OH) present difficulties for the direct analysis via gas chromatography. Derivatization of the reaction species by Grignard alkylation or hydride formation results in the loss of speciation information due to conversion into common products. Impediments to the direct monitoring of the reaction by reversed-phase HPLC include lack of a chromophore, low aqueous solubility, and high reactivity of the lithiated species in protic media. Post-column chelation of the trialkyltin product alcohol with morin to permit fluorescence detection was unsuccessful due to steric effects, consistent with literature reports [26]. Normal-phase HPLC methods would therefore require alternate tin-selective detectors, such as atomic or mass spectrometers, however such systems were not available for methods development or routine in-process use. In light of these limitations, derivatization methods were investigated in this paper to monitor the preparation of (tributylstannyl)methanol through its unstable lithiated intermediates.

2. Experimental

2.1. Materials used

Chlorotrimethylsilane (TMSCl), *N*,*O*-bis-(trimethylsilyl)trifluoroacetamide (BSTFA), methyl iodide (CH_3I), benzenesulfonylisocyanate ($C_6H_5SO_2NCO$), tributyltin hydride (Bu_3SnH), and paraformaldehyde [$(HCHO)_n$] were all purchased from Aldrich (Milwaukee, WI, USA). Lithium diisopropylamine (LDA) was prepared in-house from diisopropylamine (Air Products, Allentown, PA, USA) and 1.6 *M* *n*-butyllithium in hexanes (Cyprus Foote, Kings Mountain, TN, USA).

2.2. Development of an HPLC procedure

A Shimadzu Scientific Instruments HPLC (Columbia, MD, USA), which included an SIL-10A autoinjector, an SCL-10A system controller, LC-10AS gradient pumps, and an SPD-10AV UV–Vis detector, was used for all liquid chromatographic experiments. A 25 cm×4.6 mm I.D. (5 μm particles) YMC-ODS AQ column (Wilmington, NC, USA) was used with an isocratic 0.1% (v/v) aqueous H_3PO_4–acetonitrile (15:85, v/v) mobile phase at ambient column temperature. Ultraviolet (UV) detection was performed at 210 nm. The method flow-rate was set at 2.0 ml/min.

2.3. Development of a GC procedure

A Hewlett-Packard 6890 (Piscataway, NJ, USA) GC system, equipped with an RTX-1 column (100% dimethylpolysiloxane, 30 m×0.32 mm I.D., 1.0 μm;

Restek, Bellefonte, PA, USA), was used for all gas chromatographic experiments described in this paper unless otherwise noted. The chromatographic conditions used include a temperature program which began isothermally at 200°C for 30 min, followed by a 100°C/min ramp to 300°C and an isothermal hold at 300°C to ensure elution of any highly retained components. A volume of 0.1 μl was injected with a 50:1 split injection at 250°C. A flame ionization detector (FID) was used at 250°C. The He carrier gas was controlled in pressure control mode at 10 p.s.i. (1 p.s.i. = 6894.76 Pa).

2.4. Derivatizations of (tributylstannyl)methanol for HPLC and GC

2.4.1. HPLC

Approximately 25 mg of isolated (tributylstannyl)methanol was weighed into a tared 50 ml volumetric flask, then dissolved in approximately 15–20 ml of HPLC-grade acetonitrile. Approximately 2–3 drops of neat benzenesulfonylisocyanate was added to the solution and the reaction was vigorously swirled for approximately 30 s to ensure complete derivatization. The solution was brought to volume with HPLC-grade acetonitrile to make an approximate 0.5 mg/ml sample which was directly injected into the HPLC for analysis.

2.4.2. GC

A 400 μl aliquot of BSTFA and a 50 μl aliquot of a (tributylstannyl)methanol sample were pipetted into a GC autosampler vial (8:1, v/v). The vial was crimped and briefly shaken to mix, then heated at 40°C for ca. 5 min to ensure complete derivatization. The final solution prepared in the manner described contains approximately 125 mg/ml of (tributylstannyl)methanol. The sample was directly injected into the GC for analysis.

2.5. Derivatizations of lithiated tributylstannyl intermediates for GC

2.5.1. Iodomethane derivatizations

A 250 μl aliquot of iodomethane was mixed with 500 μl dry tetrahydrofuran (THF) in a nitrogen-purged, septum-sealed vial and maintained at ambient temperature. A 500 μl aliquot of a cold reaction solution containing the active lithium intermediate (~0.35 *M*) was added via a gas-tight syringe to the vial, which represents an approximate 20× molar excess of iodomethane. The reaction was warmed to room temperature over 15 min before the resulting solution was directly analyzed by GC.

2.5.2. Chlorotrimethylsilane derivatizations

A 250 μl aliquot of chlorotrimethylsilane was mixed with 500 μl dry THF in a nitrogen-purged, septum-sealed vial and maintained at ambient temperature. A 500 μl aliquot of a cold reaction solution containing the active lithium intermediate (~0.35 *M*) was added via gastight syringe to the vial, which represents an approximate 10× molar excess of chlorotrimethylsilane. The reaction was warmed to room temperature over 15 min and the resulting solution was directly analyzed by GC.

2.6. Identification of impurities in (tributylstannyl)methanol

Crude (tributylstannyl)methanol product was dissolved in acetonitrile and the solution was washed with hexanes to extract impurities from the product for structure elucidation. An aliquot of the hexanes layer was evaporated to dryness and reconstituted in $CDCl_3$ for 400 MHz ^{119}Sn NMR (Bruker Instruments, Billerica, MA, USA). Another aliquot of the hexanes layer was analyzed with an HP5890 gas chromatograph equipped with an electron impact mass spectrometry (EI-MS) instrument. The gas chromatographic separation was performed on a DB-1 column (100% dimethylpolysiloxane, 30 m×0.25 mm I.D., 1.0 μm film thickness; J&W Scientific, Folsom, CA, USA) with 1.5 ml/min He carrier gas flow-rate, 250°C injector temperature, 270°C transfer temperature, and a 200°C isothermal oven temperature program with a 10°C/min ramp to 270°C. A mass range of 40–600 amu was scanned.

3. Results and discussion

3.1. Derivatizations of (tributylstannyl)methanol for HPLC

Derivatization reactions with chromophoric acid

chlorides and isocyanates have been widely used to insert chromophores into non-chromophoric alcohols [56]. Specifically, phenylisocyanate and *p*-dimethylaminophenylisocyanate have been used to derivatize alcohols to the corresponding urethanes for HPLC analysis under mild reaction conditions, where R is a chromophore and R′ is non-chromophoric [57,58]:

$$R{-}N{=}C{=}O + R'OH \rightarrow R{-}NH{-}COOR' \quad (4)$$

Benzenesulfonylisocyanate was selected as a derivatizing agent because of its high reactivity [59]. Fig. 1 displays a typical chromatogram for the urethane derivative of an isolated (tributylstannyl)methanol sample. The peaks due to the derivatizing agent elute in the first 5 min of the chromatogram and there are no other observable chromatographic interferences with the benzenesulfonylurethane product at 210 nm. The benzenesulfonylisocyanate derivatization of Bu_3SnCH_2OH for HPLC analysis was demonstrated to be linear ($R>0.9999$), response factors precise ($<1.0\%$ RSD; $n=20$) for multiple sample preparations, and results robust for 2–4 drops of neat derivatizing agent. However, key potential impurities in (tributylstannyl)methanol such as the Bu_3SnH starting material are not derivatized and thus not detected by UV. For this reason, a derivatization procedure to generate GC-compatible analytes for FID was investigated.

3.2. Derivatizations of (tributylstannyl)methanol for GC

Trimethylsilylating agents are commonly used to

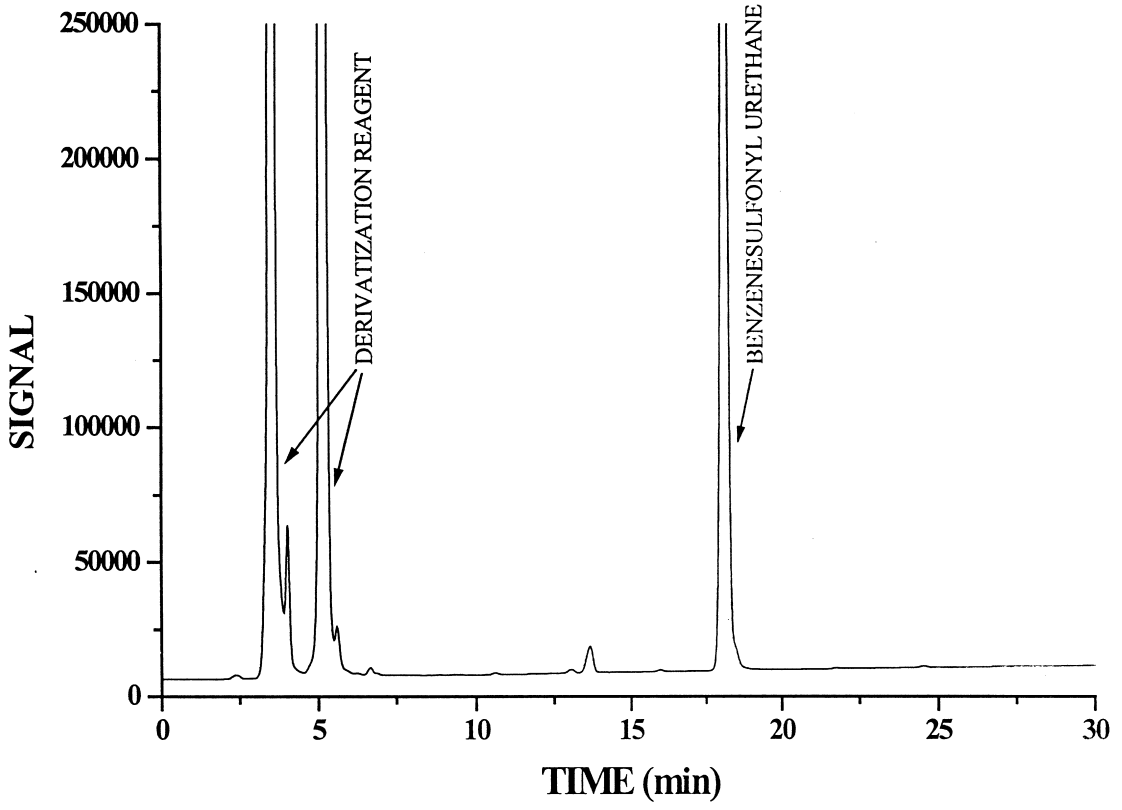

Fig. 1. Typical HPLC chromatogram of benzenesulfonylisocyanate-derivatized (tributylstannyl)methanol. Separation conditions: YMC-ODS AQ column (25 cm×4.6 mm I.D., 5 μm particles); isocratic (0.1%, v/v, aqueous H_3PO_4)–acetonitrile (15:85, v/v), mobile phase; ambient column temperature; UV detection at 210 nm; flow-rate of 2.0 ml/min.

form volatile derivatives for GC analysis from non-volatile alcohols, amines, and carboxylic acids; the ability of trimethylsilylating agents to ''donate'' a silyl group has been reviewed [56], as has the ability of various molecules to ''accept'' a silyl group:

$$R–OH + BSTFA \rightarrow ROTMS + MSTFA \quad (5)$$

BSTFA is reported to be one of the most active silyl donors and alcohols are the greatest silyl acceptors. Therefore, BSTFA was chosen to afford the trimethylsilyl derivative of the (tributylstannyl)methanol product (Bu_3SnCH_2OTMS) for gas chromatography as seen in Fig. 2. No chromatographic interferences between the derivatization blank and the analyte were observed and the method was found to be specific for Bu_3SnH (Eq. (3) starting material) at relative retention time of 0.51 (~6.5 min) as well as three significant process impurities. Impurity #1 was identified as tetrabutyltin (Bu_4Sn) by GC-MS, ^{119}Sn NMR ($\delta = -12.84$ ppm), and co-chromatography of an authentic impurity sample. Impurity 2 was identified by co-chromatography of an authentic impurity sample and GC-MS as $Bu_3SnCH_2OCH_2OTMS$. Impurity 3 was identified as the tributylstannane dimer ($Bu_3SnSnBu_3$) via co-chromatography of an authentic impurity sample, ^{119}Sn NMR ($\delta_{ppm} = -84.36$ and $^1J_{^{117}Sn-^{119}Sn} = 2583.6$ Hz calculated from $\delta_{ppm} = -75.69$ and -93.02), and GC-MS.

3.3. BSTFA Derivatization optimization

An ideal derivatization must be fast, complete, and insensitive to minor changes in reaction conditions. The composition of a (tributylstannyl)methanol–

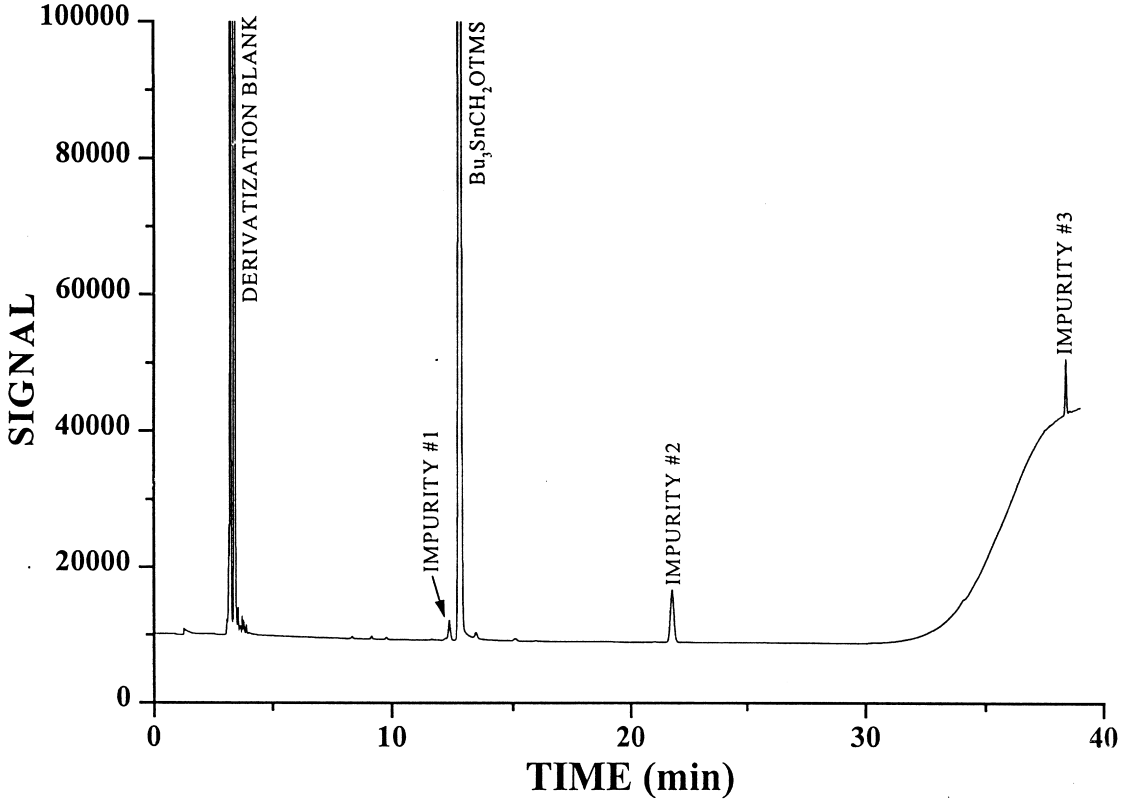

Fig. 2. Typical GC chromatogram of BSTFA-derivatized (tributylstannyl)methanol. Separation conditions: RTX-1 column (30 m×0.32 mm I.D., 1.0 μm); temperature program of 200°C for 30 min, 100°C/min to 300°C, and a 5 min hold at 300°C; 0.1 μl split injection (50:1) at 250°C; FID at 250°C; He carrier gas at 10 p.s.i. constant pressure.

BSTFA derivatization solution was varied from 1:1 to 10:1 (v/v) of BSTFA–(tributylstannyl)methanol to establish appropriate reaction conditions. The solutions were prepared at a constant volume of 450 μl in a GC vial, which is approximately the minimum vial fill required for GC analysis. These solutions were allowed to react at room temperature for 10 min and then directly analyzed by GC–FID. Injections of each solution were repeated until the area counts for Bu_3SnCH_2OTMS (derivatization product) reached a plateau, at which point the reaction was judged complete. The percent of Bu_3SnCH_2OH reacted was calculated as the ratio of Bu_3SnCH_2OTMS area counts at 10 min to the Bu_3SnCH_2OTMS area counts at reaction completion.

A monotonic increase in the extent of (tributylstannyl)methanol derivatization after 10 min was observed with increasing BSTFA–(tributylstannyl)methanol v/v ratio (see Fig. 3). However, the extent of derivatization is also a function of the initial (tributylstannyl)methanol concentration in each sample preparation which decreases exponentially with increasing BSTFA composition. As a compromise of these two effects, BSTFA:–(tributylstannyl)methanol (8:1, v/v) was arbitrarily chosen as the sample composition for further development. Since the derivatization only proceeds to 48% completion in the allotted 10 min time period at this composition, more rigorous reaction conditions were investigated.

The 8:1 (v/v) derivatization solution was heated for 5 min at 40°C in a crimped GC vial and then directly injected into the GC, resulting in a significant increase mass-based response of Bu_3SnCH_2OTMS vs. that obtained from the 10 min ambient age. Replicate preparations of the heated 8:1 (v/v) derivatization, as well as additional preparations heated for over an hour, produced response factors which varied within injector precision suggesting derivatization completion. No trace degradates were observed in any of the chromatograms resulting from this study, indicating acceptable product thermal stability during derivatization. The

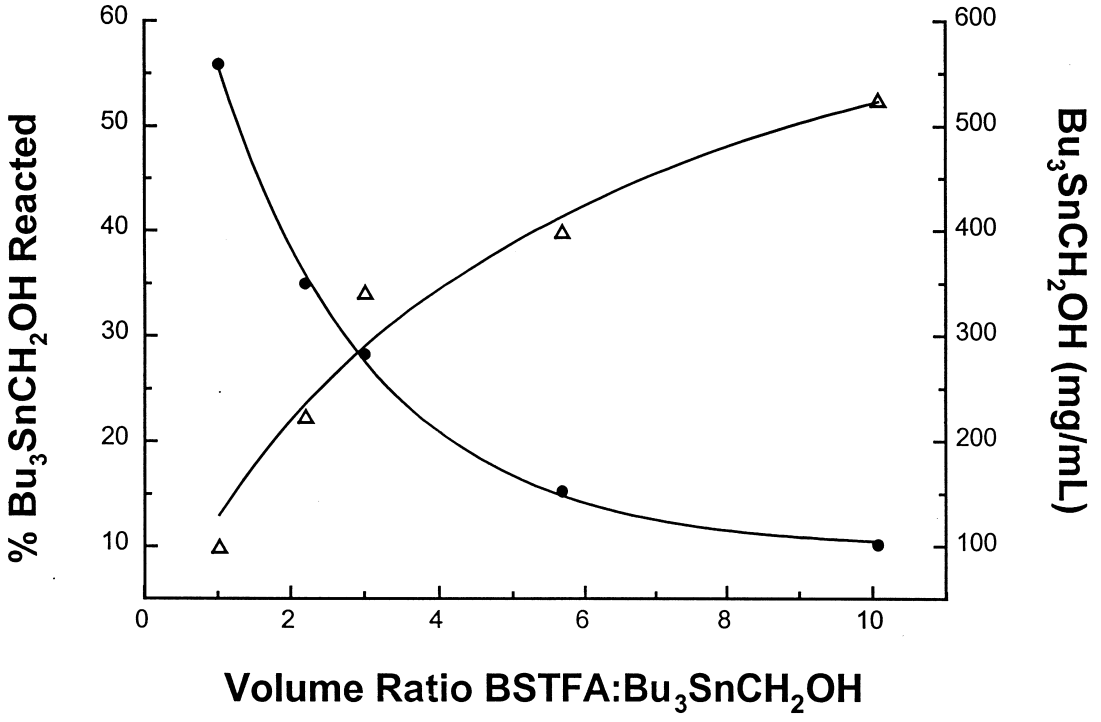

Fig. 3. Optimization of the ratio (v/v) of BSTFA/Bu_3SnCH_2OH for the TMS-derivatization of Bu_3SnCH_2OH. (△ = calculated %Bu_3SnCH_2OH reacted after 10 min; ● = Bu_3SnCH_2OH (mg/ml) in initial sample preparation.) Separation conditions: same as Fig. 2.

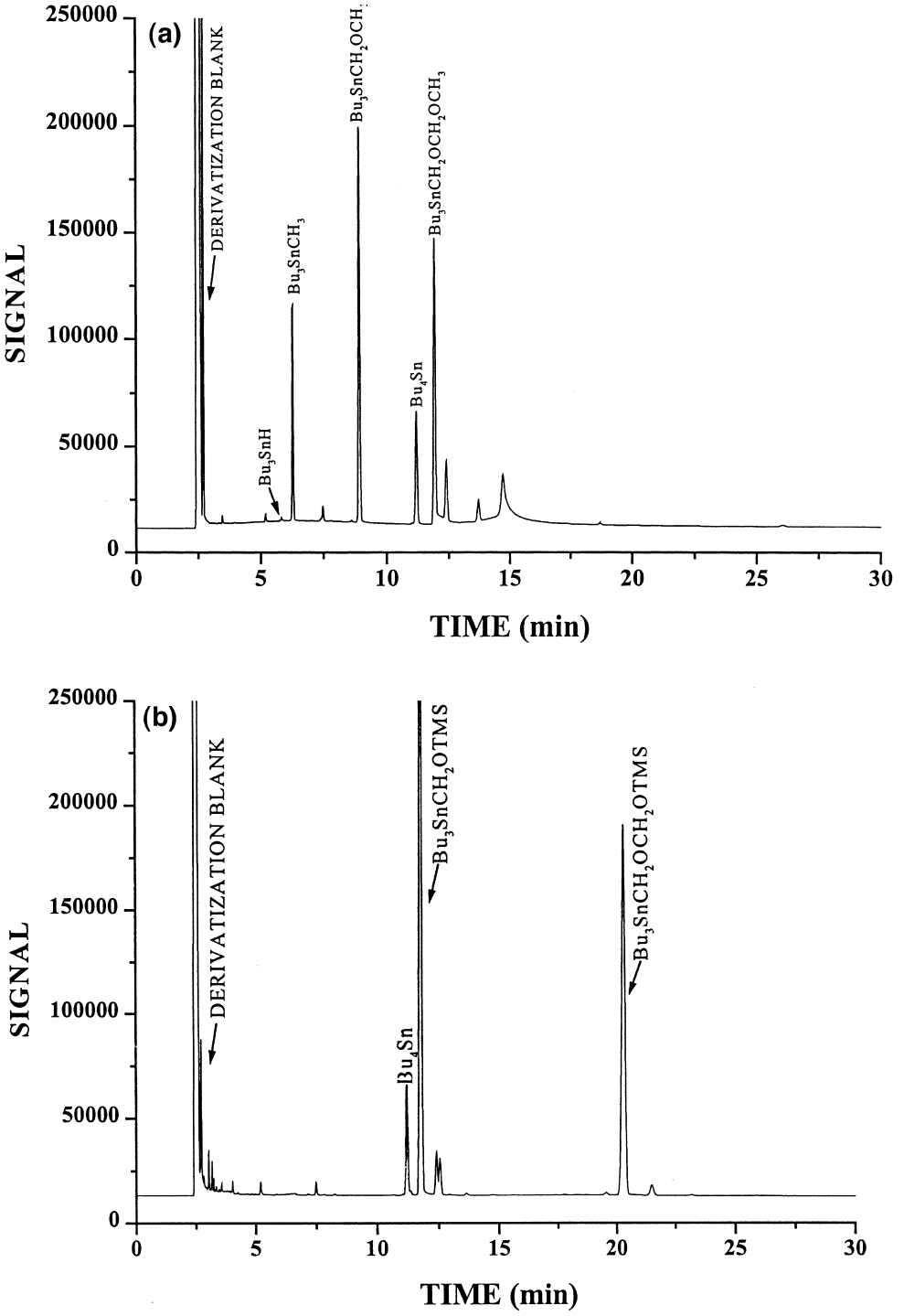

Fig. 4. (a) Typical GC chromatogram of an iodomethane quench of the cold reaction: $Bu_3SnLi \rightarrow Bu_3SnCH_2OLi$. Separation conditions: the sample was allowed to warm to ambient temperature and then directly analyzed by GC with the same conditions as in Fig. 2. (b) Typical GC chromatogram of a TMSCl quench of the cold reaction: $Bu_3SnLi \rightarrow Bu_3SnCH_2OLi$. Separation conditions: the sample was allowed to warm to ambient temperature and then directly analyzed by GC with the same conditions as in Fig. 2.

BSTFA derivatization of Bu_3SnCH_2OH for GC analysis was demonstrated to be linear ($R=0.9998$), quantitative for 0.02% impurities, precise (1.7% RSD) for the Bu_4Sn impurity at the 0.35A% level, and robust for several GC columns of the same type.

3.4. Derivatizations of lithiated tributylstannyl intermediates for GC

To this point, specific methods for the analysis of isolated (tributylstannyl)methanol had been developed. However, methods were still required to monitor (tributylstannyl)methanol processing through its two lithiated intermediates (Eq. (3)). Less expensive and less reactive derivatizing agents than BSTFA were sought for these more reactive species. Two methods of derivatization were evaluated to stabilize the lithiated intermediates: methylation with iodomethane (Eq. (6)) and trimethylsilylation with chlorotrimethylsilane (Eq. (7)):

$$R{-}OLi + CH_3I \rightarrow R{-}O\,CH_3 + LiI \quad (6)$$

$$R{-}OLi + TMSCl \rightarrow R{-}OTMS + LiCl \quad (7)$$

The results of these derivatizations for the end of the paraformaldehyde reaction in Eq. (3) (where Bu_3SnCH_2OLi is the main product in the reaction matrix) are shown in Fig. 4a and b. Significant amounts of $Bu_3SnCH_2OCH_3$ and $Bu_3SnCH_2OCH_2OCH_3$, the anticipated derivatization products of Bu_3SnCH_2OLi and $Bu_3SnCH_2OCH_2OLi$, were formed during the iodomethane reaction in Fig. 4a. However, a significant and unacceptable amount of another derivatization product (Bu_3SnCH_3) was unexpectedly generated, suggesting decomposition of one or more of the lithiated species during derivatization. The trimethylsilylation reaction represented in Fig. 4b was observed to form the anticipated derivatization products (Bu_3SnCH_2OTMS and $Bu_3SnCH_2OCH_2OTMS$) for the two major analytes present at the end of paraformaldehyde reaction. Other previously-discussed process impurities were unaffected by the derivatization procedure. Employing the chlorotrimethylsilane derivatization procedure for GC analysis, a typical pilot plant-scale reaction was successfully monitored to $<0.1\%$ Bu_3SnH and Bu_3SnLi starting materials, respectively, for the first two steps of the reaction in Eq. (3).

4. Conclusions

Reported direct and indirect chromatographic methods of trialkyltin analysis have been evaluated and found to be inadequately sensitive and specific to monitor the formation of (tributylstannyl)methanol from tributyltin hydride through unstable lithiated intermediates. As a result, alternative derivatization methods have been developed. Derivatization of the (tributylstannyl)methanol product with benzenesulfonylisocyanate produces the corresponding urethane analogue which may be assayed by reversed-phase liquid chromatography. However, process impurities such as Bu_4Sn and $Bu_3SnSnBu_3$ are not readily derivativized and therefore remain undetectable by UV. Trimethylsilyl derivatization with TMSCl has been found to quickly and reproducibly form single derivatization products for the lithiated intermediates (Bu_3SnLi, Bu_3SnCH_2OLi, and $Bu_3SnCH_2OCH_2OLi$); non-alcohol matrix components were unaffected by the derivatization and detectable with FID. Derivatization of the quenched product alcohols Bu_3SnCH_2OH and $Bu_3SnCH_2OCH_2OH$ to their respective TMS analogues with BSTFA, a more potent trimethylsilylating agent, was also successful. The silylation–GC method was applied to the laboratory optimization of the (tributylstannyl)methanol reaction and was also used to monitor the progress of pilot-scale reactions to $<0.1\%$ component levels.

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