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## NOTE

# POLYMERIZATION OF 1-BUTYL-2-TRIMETHYLSILYLACETYLENE BY TRANSITION METAL CATALYSTS

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Key Words: Disubstituted Polyacetylene, 1-Butyl-2-trimethylsilylacetylene, Transition Metal Catalysts, Desilylation

## ABSTRACT

New silicon-containing disubstituted polyacetylene was prepared by the polymerization of BTMSA having two bulky substituents (n-butyl and trimethylsilyl) using transition metal catalysts. The polymerization proceeded in a mild manner and the final polymer yield was generally lower than those of silicon-containing monosubstituted acetylenes by the similar catalyst system. The characterization on the polymer structure revealed that the resulting poly(BTMSA) have a conjugated backbone system, but the polymers have peculiar copolymer composition of poly(BTMSA) and poly(1-hexyne) due to the spontaneous desilylation during the polymerization. The fluoride-ion induced desilylation of poly(BTMSA) using  $n\text{-Bu}_4\text{N}^+\text{F}^-$  in THF yielded a completely desilylated product, pure poly(1-hexyne).

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## INTRODUCTION

The polymerization of acetylene and its derivatives has been carried out extensively using organometallic catalysts of Ziegler-Natta type, and anionic, cationic, and radical initiators [1-3]. Tungsten and molybdenum-based catalyst systems have been employed for the olefin metathesis and the ring-opening polymerization of cycloolefins [4-7]. It was also found that these catalyst systems were very effective for the polymerization of some acetylene derivatives such as phenylacetylene [8-10], *o*-CF<sub>3</sub>-phenylacetylene [11], β-naphthyl-acetylene [12], propiolic acid [13], nitrophenyl propargyl ethers [14], etc.

Tungsten and molybdenum chlorides were found to be effective for the polymerization of some acetylene derivatives. However, the cocatalysts such as organotin compounds (Ph<sub>4</sub>Sn, n-Bu<sub>4</sub>Sn, Me<sub>4</sub>Sn) [9] and polar protic compounds (H<sub>2</sub>O, CH<sub>3</sub>CH<sub>2</sub>OH, HC≡CCH<sub>2</sub>OH) [9, 12, 15] were necessary for the acetylene polymerization in order to result in high yield and high molecular weight.

The organoaluminium compounds such as Et<sub>3</sub>Al, Et<sub>2</sub>AlCl, and EtAlCl<sub>2</sub> have been found to be effective cocatalysts of tungsten- and molybdenum chloride for the metathesis polymerizations of cycloolefins and acetylene derivatives [4, 7, 16-18].

Conjugated polymer systems obtained from acetylene derivatives have been studied as organic semiconductors [19-22], as membranes for gas separation and for liquid-mixture separation [23-27], as materials for enantioseparation of racemates by high performance liquid chromatography [28], as a side-chain liquid crystal [29-33], as materials for chemical sensors [34-37], and as materials for nonlinear optical property [38-42] and for photoluminescence and electroluminescence properties [43, 44].

Especially, the studies on the potential applications of silicon-containing substituted polyacetylenes have been extensively performed over past two decades [45]. Oxygen enrichment is applicable to combustion furnaces, car engines, and respiration-aiding apparatus. The transport of oxygen dissolved in water can be applied to contact lenses and artificial lungs. Liquid-mixture separation can be applied to the concentration of ethanol from fermented bio-mass. Polymer degradation has relevance to resist materials for microlithography.

We reported the polymerization of silicon-containing disubstituted acetylenes, 1-aryl-2-trimethylsilylacetylenes (aryl: phenyl, 2-thienyl, 2-furyl, 2-pyridyl) using various transition metal catalysts [46, 47]. This paper deals with the synthesis of silicon-containing disubstituted polyacetylene having n-butyl

and trimethylsilyl groups as substituents and the characterization of the resulting polymer.

## EXPERIMENTAL

### Materials

1-Hexyne (Aldrich Chemicals, 97%) and chlorotrimethylsilane (Aldrich Chemicals, redistilled, 99+%) were dried with calcium hydride and distilled before use. n-Butyllithium (Aldrich Chemicals, 2.5 M solution in hexanes) was used as received.  $WCl_6$  and  $MoCl_5$  (Aldrich Chemicals, resublimed, 99+%), and organoaluminum compounds ( $Et_2AlCl$  and  $EtAlCl_2$ , Aldrich Chemicals, 1.8 M solution in toluene) were used as received. Tetraphenyltin (Aldrich Chemicals, 97%) was purified by recrystallizing twice from carbon tetrachloride. Tetramethyltin (Aldrich Chemicals, 99+%) and tetra-n-butyltin (Aldrich Chemicals, tech., 93%) were used after simple distillation. The polymerization solvent, chlorobenzene, was of analytical grade material. It was dried with calcium hydride and fractionally distilled.

### Preparation of 1-Butyl-2-trimethylsilylacetylene (BTMSA)

To a stirred, ice-cooled solution of 1-hexyne (15.0 g, 0.183 mol) in 200 mL ethyl ether was added 75 mL of n-butyllithium (2.5 M solution in hexanes, 0.188 mol) over a period of 20 minutes at 0°C under nitrogen atmosphere. After standing at 0°C for an additional 1 hours, 22.0 g (0.203 mol) of chlorotrimethylsilane were added over 20 minutes at 5-10°C and the reaction mixture was refluxed for 2 hours. Then, the reaction mixture was filtered through a sintered glass filter to remove the precipitated lithium chloride, and the solvents were removed by rotary evaporation. Fractional distillation of the residue gave 23.71 g (yield 84%) of BTMSA, b.p. 83°C/65 mmHg.

$^1H$ -NMR ( $CDCl_3$ ,  $\delta$  ppm): -0.02 (9H), 0.61-1.00 (3H), 1.18-1.60 (4H), 2.01-2.25 (2H). IR (KBr pellet, wavenumbers,  $cm^{-1}$ ): 2980 (s), 2184 (m), 1317 (m), 842 (s).

### Polymerization of BTMSA

All procedures for catalyst system preparation and polymerization were carried out under dry nitrogen atmosphere. A typical example of the polymerization procedure is as follows. A catalyst solution is prepared by mixing  $WCl_6$

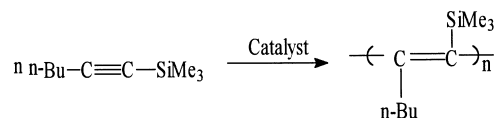
(1.30 mL 0.1 M chlorobenzene solution, 12.96 mmol),  $\text{EtAlCl}_2$  (1.30 mL 0.2 M chlorobenzene solution, 25.92 mmol), and polymerization solvent (0.4 mL chlorobenzene,  $[\text{M}]_0 = 1.5\text{M}$ ) and aged by keeping it at  $30^\circ\text{C}$  for 5 minutes. To this catalyst solution is added BTMSA (1.0 g, 6.48 mmol). Polymerization was carried out at  $80^\circ\text{C}$  for 24 hours and terminated with a small amount of methanol. This polymer solution was dissolved in chloroform followed by precipitation into a large excess of methanol. The precipitated polymers were filtered from the solution and dried to a constant weight under vacuum at  $40^\circ\text{C}$  for 24 hours. The polymer yield was calculated by gravimetry.

### Instruments

$^1\text{H-NMR}$  spectra were recorded on a Bruker AM-200 spectrometer in  $\text{CDCl}_3$ . IR spectra were obtained with a Perkin-Elmer 283B spectrometer using a KBr pellet. Elemental analyses were performed with Perkin-Elmer 240C Elemental Analyzer. UV-visible spectra were taken on a Cary 17 spectrophotometer in 1,2-dichloroethane solvent. Number-average molecular weight ( $\overline{M}_n$ ) was determined by gel permeation chromatography (Waters 150C) equipped with  $\mu$ -styragel columns using tetrahydrofuran as an eluent. Monodisperse polystyrene (PS) standard polystyrene (PS) standard samples were employed for molecular weight calibration.

## RESULTS AND DISCUSSION

The polymerization of BTMSA, a disubstituted acetylene having bulky *n*-butyl and trimethylsilyl substituents was carried out by tungsten and molybdenum-based transition metal catalysts as follows (Structure 1).



This polymerization was found to proceed in a mild manner. Exothermic reaction was not observed. Table 1 shows the results for the polymerization of BTMSA by W- and Mo-based catalyst systems. In general, the polymer yields were low in comparison with those for the polymerization of monosubstituted and some disubstituted acetylenes [9, 15-17]. These low polymer yields are pri-

TABLE 1. Polymerization 1-Butyl-2-trimethylsilylacetylene by Transition Metal Catalysts<sup>a</sup>

Experiment no	Catalyst System <sup>b</sup>	M/C <sup>c</sup>	[M] <sub>0</sub>	Polymer Yield (%)	$\bar{M}_n^f$
1	WCl <sub>6</sub>	50	1.5	3	-
2	WCl <sub>6</sub> -Me <sub>4</sub> Sn(1:1)	50	1.5	7	2,080
3	WCl <sub>6</sub> -Ph <sub>4</sub> Sn(1:1)	50	1.5	10	4,600
4	WCl <sub>6</sub> -Et <sub>2</sub> AlCl(1:2)	50	1.5	13	5,700
5	WCl <sub>6</sub> -EtAlCl <sub>2</sub> (1:2)	50	1.5	17	7,620
6	WCl <sub>6</sub> -EtAlCl <sub>2</sub> (1:2)	50	1.0	9	4,210
7	WCl <sub>6</sub> -EtAlCl <sub>2</sub> (1:2)	50	0.5	Trace	-
8	WCl <sub>6</sub> -EtAlCl <sub>2</sub> (1:2)	30	1.5	15	7,200
9	MoCl <sub>5</sub>	50	1.5	Trace	-
10	MoCl <sub>5</sub> -n-Bu <sub>4</sub> Sn(1:1)	50	1.5	10	3,920
11	MoCl <sub>5</sub> -EtAlCl <sub>2</sub> (1:2)	50	1.5	5	-

<sup>a</sup>Polymerization was carried out at 80°C for 24 hours in chlorobenzene.

<sup>b</sup>Mixture of catalyst and cocatalyst solution was aged for 5 minutes at 30°C before use.

<sup>c</sup>Monomer to catalyst mole ratio.

<sup>d</sup>Initial monomer concentration of BTMSA.

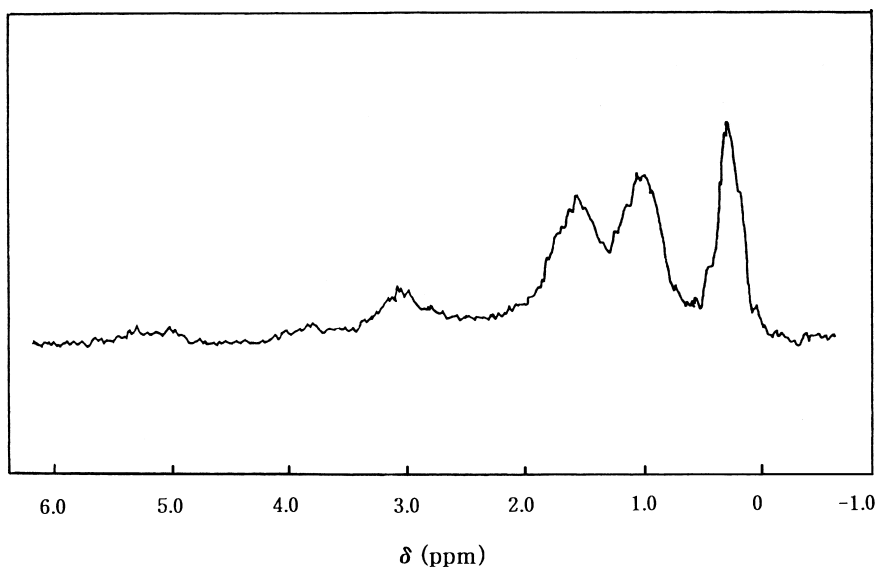
<sup>e</sup>Methanol-insoluble polymer yield.

<sup>f</sup>The molecular weights were obtained with GPC.

marily attributable to the high steric hindrance of n-butyl and trimethylsilyl substituents. WCl<sub>6</sub> or MoCl<sub>5</sub> itself gave only a trace amount of oligomeric materials. When the organotin compounds such as Me<sub>4</sub>Sn and Ph<sub>4</sub>Sn were used as cocatalyst for this polymerization of BTMSA by WCl<sub>6</sub> or MoCl<sub>5</sub>, the polymer yields slightly increased. In the WCl<sub>6</sub>-catalyzed polymerization of BTMSA, organoaluminum compound cocatalyst, especially EtAlCl<sub>2</sub>, showed a relatively good polymerization results in a viewpoint of polymer yield and molecular weight. As the initial monomer concentration ([M]<sub>0</sub>) increased, the polymer yield generally decreased (exp. no.; 5, 6, 7).

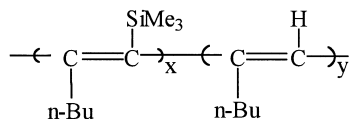
The polymerization of BTMSA using MoCl<sub>5</sub> based catalyst gave only a low yield of polymer. MoCl<sub>5</sub>-n-Bu<sub>4</sub>Sn (1:1) catalyst system exhibited 10% yield of polymer having low molecular weight ( $\bar{M}_n$ : 3,920).

The polymer structure of poly(BTMSA) was characterized by NMR, infrared, and UV-visible spectroscopies. Figure 1 shows the <sup>1</sup>H-NMR spectrum of poly(BTMSA) prepared by WCl<sub>6</sub>-EtAlCl<sub>2</sub> catalyst system. It shows the n-



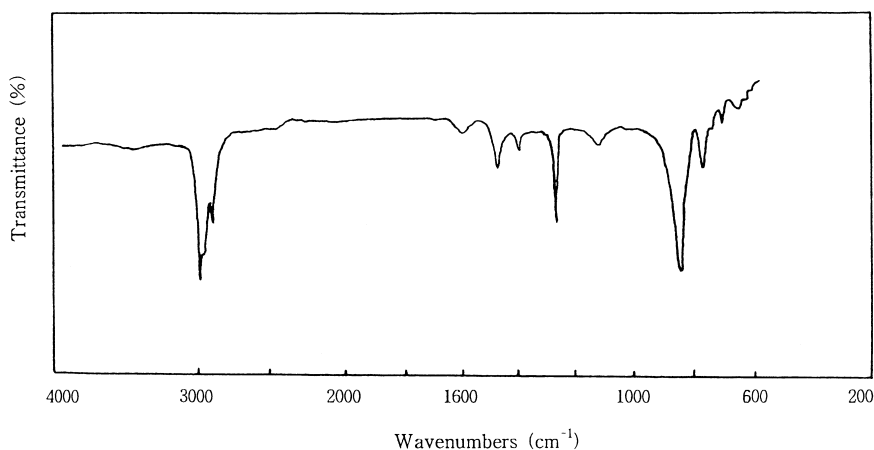
**Figure 1.**  $^1\text{H-NMR}$  spectrum of poly(BTMSA) in  $\text{CDCl}_3$ .

butyl protons and the trimethylsilyl protons at 0.73-2.97 ppm and -0.05-0.72 ppm, respectively. The integration ratio of the *n*-butyl protons and the trimethylsilyl protons is very different from that of ideal poly(BTMSA) structure. The integration ratio of trimethylsilyl groups was smaller than that of ideal polymer structure. From the  $^1\text{H-NMR}$  integration and elemental analysis, it was found that the resulting poly(BTMSA)s are partially desilylated one having the following structure (Structure 2).



The desilylation extent of the resulting poly(BTMSA) was calculated to be 70%. The broad peak at about 6.0 ppm was originated from the vinyl protons of partially desilylated poly(BTMSA).

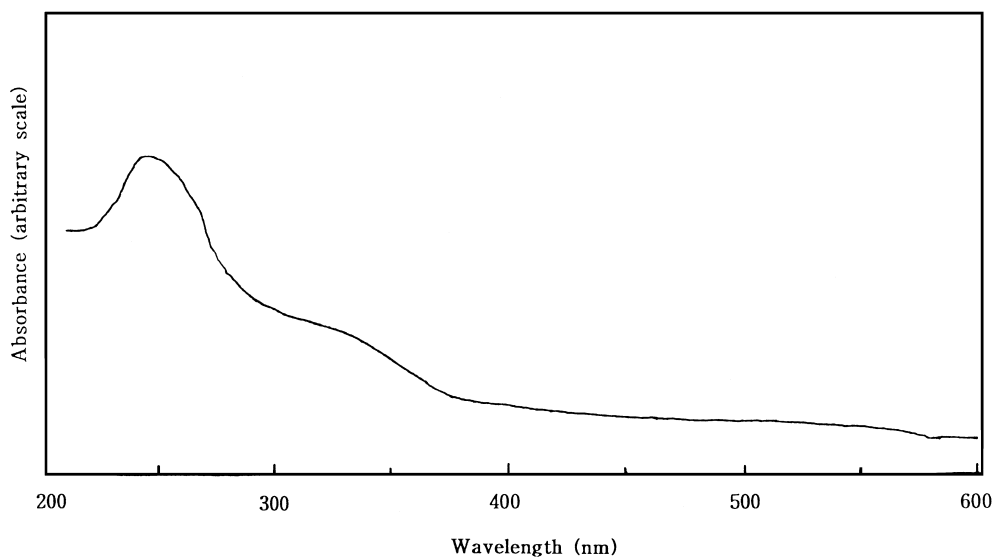
Figure 2 shows the IR spectra of poly(BTMSA) on KBr pellet. In the IR spectrum of poly(BTMSA), it does not show the acetylenic  $\text{C}\equiv\text{C}$  bond stretching frequency at  $2184\text{ cm}^{-1}$ . Instead, the  $\text{C}\equiv\text{C}$  stretching frequency of conjugated polymer backbone at  $1591\text{ cm}^{-1}$ , which did not be seen in the IR spectrum of



**Figure 2.** Infrared spectrum of poly(BTMSA) in KBr pellet.

monomer, is newly observed. An absorption due to SiC-H deformation is seen at  $1255\text{ cm}^{-1}$ , and a group of absorptions due to Si-C stretching is seen at  $842\text{ cm}^{-1}$ .

Figure 3 shows the UV-visible spectrum of the resulting poly(BTMSA) in 1, 2-dichloroethane. A characteristic peak of the conjugated polymer, broad and weak  $\pi\rightarrow\pi^*$  transitions which are absent in monomer, appeared at longer



**Figure 3.** UV-visible spectra of poly(BTMSA) in 1,2-dichloroethane.



wavelength upto 580 nm. In the UV-visible spectrum of highly twisted poly[1-(trimethylsilyl)-1-propyne], no absorption was seen above 340 nm [48]. However, a broad absorption from the UV to visible region was observed in the UV-visible spectrum of the present polymer. These facts were mainly attributable to the partial desilylation freeing remarkably twisted main chain conformations.

The resulting poly(BTMSA)s were generally obtained as light-brown powders. The number-average molecular weights ( $M_n$ ) of the resulting polymers were generally and were in the range of 2,080-7,620.

Table 2 shows the solubility behavior of poly(BTMSA). The solubility test was performed for powdery samples in excess solvent. The poly(BTMSA) was completely soluble in benzene, chlorobenzene, THF, and  $CCl_4$  but insoluble in DMF, ethyl ether, methanol, formic acid, and n-hexane.

The chemical modification of poly(trimethylsilylacetylene), a monosubstituted polyacetylene containing trimethylsilyl group, had been performed with  $n-Bu_4N^+F^-$  or HCl gas to obtain polyacetylene [49, 50]. However, the resulting product was the copolymer of poly(trimethylsilylacetylene) and polyacetylene. Despite the choice of appropriate reaction conditions, the reaction did not proceed in completion. The reason of partial desilylation was attributed to the insolubility of the product even at moderate conversion.

The desilylation of the present poly(BTMSA) using  $n-Bu_4N^+F^-$  in THF at room temperature for 5 hours yielded a completely desilylated one, pure poly(1-hexyne). It had been also found the similar reaction of the disubstituted polyacetylenes having aromatic substituents (phenyl, 2-thienyl, and 2-furyl) instead

TABLE 2. Solubility Behaviors of Poly(BTMSA)

Solvent	Solubility <sup>a</sup>
Benzene	+
Chlorobenzene	+
Tetrahydrofuran	+
Chloroform	+
Carbon tetrachloride	±
Hexanes	-
Methanol	-
Formic acid	-
N,N-Dimethylformamide	-

<sup>a</sup>Solubility (+: soluble, ±: partially soluble, -: insoluble).

of n-butyl substituent proceeded completely [47]. The complete desilylation may be due to the excellent solubility of the resulting monosubstituted polyacetylene.

## CONCLUSION

Novel Si-containing disubstituted polyacetylene was prepared by the polymerization of BTMSA having bulky two substituents (n-butyl and trimethylsilyl) using transition metal catalysts. The polymerization proceeded in a mild manner and the final polymer yield was generally lower than those of silicon-containing monosubstituted acetylenes by the similar catalyst system. The characterization on the polymer structure revealed that the resulting poly(BTMSA)s have a conjugated backbone system, but the polymers have peculiar copolymer composition of poly(BTMSA) and poly(1-hexyne) due to the spontaneous desilylation during the polymerization. The fluoride-ion induced desilylation of poly(BTMSA) using n-Bu<sub>4</sub>N<sup>+</sup>F<sup>-</sup> in THF yielded a completely desilylated product, pure poly(1-hexyne).

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