

# A NEW HETEROMETALLIC $[Al^{III}-Bu_2Sn^{IV}-B^{III}]$ -M- OXOISOPROPOXIDE AND ITS DERIVATIVES WITH $\beta$ - DIKETONES: SYNTHETIC, SPECTRAL AND ELEMENTAL STUDIES

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## ABSTRACT

New heterometallic derivative  $[Al^{III} - Bu_2Sn^{IV} - B^{III}] - \mu - \text{oxoisopropoxide}$  has been prepared by the thermal condensation of compound  $Bu_2Sn^{IV} - B^{III} - \mu - \text{oxoisopropoxyacetate}$  (OAc)  $Bu_2SnOB(OPr^i)_2$  with  $Al(OPr^i)_3$  in 1:1 molar ratio in xylene with continuous liberation of isopropyl acetate formed during the course of reaction. The  $Bu_2Sn^{IV} - B^{III} - \mu - \text{oxoisopropoxyacetate}$  was synthesized by reaction of tin diacetate with triisopropyl borate in 1:1 molar ratio in toluene. The reaction of  $\mu$ -oxoisopropoxide with  $\beta$ -diketones (HL) in 1:1 and 1:2 molar ratios yielded  $Bu_2SnO_2AlB(OPr^i)_3L$ , and  $Bu_2SnO_2AlB(OPr^i)_2L_2$  respectively. (where L = acetylacetonate/benzoylacetonate/p-bromo benzoylacetonate anion) respectively. The  $\mu$ -oxoisopropoxide compound has been characterized by elemental and spectral (IR,  $^1H$ ,  $^{13}C$ ,  $^{27}Al$ ,  $^{119}Sn$ ,  $^{11}B$  NMR). The  $\beta$  diketones of  $[Bu_2SnO_2AlB(OPr^i)_4]$  have been characterized by elemental, liberated isopropanol and spectral analysis (IR,  $^1H$ ,  $^{13}C$  &  $^{27}Al$  NMR).

Keywords: Tin, Aluminium, Boron, alkoxides,  $\beta$ -diketones

## INTRODUCTION

The elucidation of structural features and reactivity patterns of polymetallic alkoxides has aroused deep scientific interest, because the complexes have been used as precursors for super or semi-conducting, ferroelectric, dielectric and even biocompatible oxide materials [1,2]. Further, the mixed metal oxides prepared from heterometallic- $\mu$ -oxoalkoxides [3-6] have been used for absorbing harmful chemicals [7] and gases such as  $SO_2$ ,  $CCl_4$ , and decontaminating chemical warfare agents [8]. Recently,  $MgAl_2O_4$  prepared from  $[MgO_2Al_2(OPr^i)_4]_2$  have been used to destructively adsorb paraxon [diethyl-4-nitrophenol phosphate (DNPP)] [9]. In view of the solubility of metal alkoxides [10,11] and oxoalkoxides [12,13] in organic solvents, these materials are strongly preferred as precursors in sol-gel processes. In heterometallic- $\mu$ -oxoalkoxides M-O-M' linkage is present, which makes the M-O-M' bond strong and stable as compared to other precursors. Non-cleavage of the M-O-M' bond even upon hydrolysis followed by dehydration, makes homogenous oxides of high surface area mixed

metal oxides nanoparticles. Therefore, these compounds are considered as especially suitable precursors over other precursors such as metal nitrate, acetate, monodispersed metal hydrous oxides, mainly due to the ease of their purification, solubility in organic solvents, volatility and their extremely facile hydrolyzability. The rate of hydrolysis depends upon the nature of metal in terms of electrophilicity and ability to expand its coordination number<sup>[14]</sup>. The hydrolysis rates transition metal alkoxides (especially in the case of heterobimetallic alkoxides) are very high due to their highly electrophilic nature and their ability to expand its coordination number which complicates the problem by causing phase segregation.

To overcome this problem to provide modified precursor by replacing the alkoxide groups with other ligands for example acetylacetone, benzoylacetone which may undergo hydrolysis at slower rate,<sup>[15]</sup> structural features, solubilities and effect of chelating group on the stability of  $\mu$ -oxo compounds, their  $\beta$ -diketones have also been synthesized. Systematic studies have been made on the catalytic application of bimetallic-  $\mu$ -oxoalkoxides of transition metals reveals that these compounds rank among the best catalysts in ring opening polymerization of heterocyclic compounds like thiranes, oxiranes, epoxides and lactones<sup>[16-19]</sup>. It was observed that neither any alkoxide nor oxoalkoxides of Al-Bu<sub>2</sub>Sn-B as a soluble heterometallic single source precursor have been reported so far.

The above features underline the importance and utility of  $\mu$ -oxo compounds and its derivatives with  $\beta$ -diketones, so it was considered of interest to synthesis new heterometallic [Bu<sub>2</sub>SnO<sub>2</sub>AIB(OPr<sup>i</sup>)<sub>4</sub>] and its  $\beta$ -diketonates.

## EXPERIMENTAL

All manipulations have been carried out under anhydrous conditions<sup>[20]</sup> and the solvents and reagents used were purified and dried by standard methods<sup>[20]</sup>. The general technique and physical measurement were carried out as described elsewhere<sup>[6,21,22]</sup>. Tin diacetate [Bu<sub>2</sub>Sn(OAc)<sub>2</sub>] (Aldrich) and aluminium isopropoxide [Al(OPr<sup>i</sup>)<sub>3</sub>] (Aldrich) and triisopropyl borate B(OPr<sup>i</sup>)<sub>3</sub> (Aldrich) were used as received. Acetyl acetone was dried prior to use. Benzoyl acetone (Hi-media) and p- bromobenzoyl acetone was used as received. The estimation of isopropoxy groups in the  $\mu$ - oxoisopropoxide and isopropyl alcohol liberated in synthesis of  $\beta$ -diketonates of [Bu<sub>2</sub>SnO<sub>2</sub>AIB(OPr<sup>i</sup>)<sub>4</sub>] were carried out oxidimetrically<sup>[23]</sup>. Tin and aluminium were analyzed gravimetrically and boron was estimated volumetrically<sup>[22]</sup>. Further, spectral study of tin, aluminium and boron has also been carried out to find out their respective coordination number in order to elucidate the structure of  $\mu$ -oxo compounds. The complex [Bu<sub>2</sub>SnO<sub>2</sub>AIB(OPr<sup>i</sup>)<sub>4</sub>] and its  $\beta$ - diketonates were decomposed in conc. HCl and extracted in dilute HCl, tin was precipitated as sulphide (pH 5-6), filtered and estimated as Bu<sub>2</sub>SnO<sub>2</sub><sup>[22]</sup>. The H<sub>2</sub>S was boiled off completely from the filtrate and aluminium was estimated as aluminium oxinate<sup>[22]</sup>.

The Infrared spectra were recorded on a Perkin-Elmer 1710 FTIR spectrometer over the range of 4000-400 cm<sup>-1</sup>. The <sup>1</sup>H, <sup>13</sup>C, <sup>27</sup>Al, <sup>119</sup>Sn and <sup>11</sup>B NMR spectra were recorded in CDCl<sub>3</sub> on Bruker

Avance II 400 NMR spectrometer. TG study has been made on Diamond TG/DTA Perkin Elmer instrument and molecular weight data was determined by the cryoscopic method using a Beckman thermometer.

### Synthesis of $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$

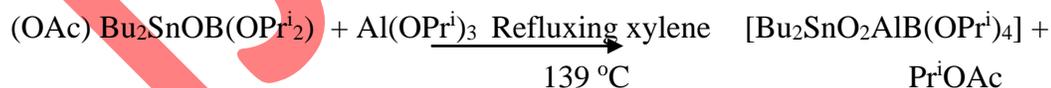
The  $[(\text{OAc})\text{Bu}_2\text{SnOB}(\text{OPr}^i)_2]$  compound was synthesized by refluxing the  $\text{Bu}_2\text{Sn}(\text{OAc})_2$  and  $\text{B}(\text{OPr}^i)_3$  in 1:1 molar ratio in toluene. The thermal condensation between  $[(\text{OAc})\text{Bu}_2\text{SnOB}(\text{OPr}^i)_2]$  (3.169 g, 9.811 mmol) and  $\text{Al}(\text{OPr}^i)_3$  (2.004 g, 9.811 mmol) in xylene gives the  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$ . The contents were refluxed for about 8 h on a fractionating column and the isopropyl acetate formed during the reaction was distilled off continuously from 78 °C to boiling point of xylene <sup>[6,13]</sup> (139 °C). The solvent xylene was completely removed at (~70 °C /1mm) yielding a pale yellow solid. The product was redissolved in benzene and slow evaporation of benzene resulted in a pale yellow glassy solid. The  $\mu$ -oxo compound was found to be soluble in common organic solvents such as  $\text{CHCl}_3$ , and  $\text{C}_6\text{H}_6$ , highly susceptible to hydrolysis and decomposed on heating (~210°C). [Yield: 96% ]. Found:  $\text{OPr}^i$ , 21.36; Sn, 21.88; Al, 4.5; B, 1.88 for  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$  Calcd:  $\text{OPr}^i$ , 22.26; Sn, 21.31; Al, 4.35; B, 1.59.

### Reaction of $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$ with acetylacetone (Hacac) in 1:1 molar ratio:

The compound  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$  (0.478 g, 0.915 mmol) and acetylacetone (0.0915 g, 0.915 mmol) were refluxed in ~ 50 ml benzene in a flask connected to short distillation column on an oil bath for about 4 h. The isopropanol liberated at 72-78 °C was fractionated as the binary azeotrope of isopropanol-benzene <sup>[13]</sup>. The azeotrope was collected and checked for completion of the reaction. The excess of the solvent was then removed under reduced pressure (45 °C /1mm) yielding a yellowish brown solid. The preparations of other  $\beta$ -diketonates of  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$  in different molar ratios were carried out by similar procedure and the analytical results have been summarized in Table-1.

## RESULTS AND DISCUSSION

The preparation of the heterometallic- $\mu$ -oxoisopropoxide  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$  follows the following reaction scheme:



### IR Spectra

The sharp band observed at ~ 1630  $\text{cm}^{-1}$  due to  $\text{C}=\text{O}$  in IR spectrum of tin diacetate is absent in that of  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$  indicating the complete removal of acetate groups in  $\mu$ -oxo compound <sup>[24]</sup>. Instead, a band exhibited in the region ~1370-1340  $\text{cm}^{-1}$  is assigned to *gem*-dimethyls of isopropoxy group. Similarly the bands observed at 1180-1160 and 1130- 1110  $\text{cm}^{-1}$  have been assigned to the combination bands  $\nu(\text{C}-\text{O}+\text{OPr}^i)$  of bridging and terminal isopropoxy group respectively. A band

appearing at approximately  $950\text{ cm}^{-1}$  is assigned to  $\nu(\text{C-O})$  stretching of bridging isopropoxy group [25,26]. A number of vibrations are observed in the region  $700\text{-}400\text{ cm}^{-1}$  due to M-O stretching vibrations [27] in  $\mu$ -oxo compound.

#### NMR Spectra

##### $^1\text{H}$ NMR

A sharp singlet observed at  $\delta$  2.1 ppm in the  $^1\text{H}$  NMR spectrum of tin diacetate is found to be absent in the spectrum of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  confirms the complete removal of acetate groups.  $^1\text{H}$  NMR spectrum of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  exhibits two doublet at  $\delta$  1.0 and 1.1 ppm assigning methyl protons of terminal and bridging isopropoxy groups [16,28]. A multiplet centered at  $\delta$  4.4 ppm is observed due to the methine proton of isopropoxy group in the  $\mu$ -oxo compound [16].

##### $^{13}\text{C}$ NMR

The  $^{13}\text{C}$  NMR spectrum of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  shows prominent peaks at  $\delta$  25.6, and 28.4 ppm assignable to the methyl carbon of non-bridging and bridging isopropoxy groups. The other peaks  $\delta$  62.6, and 67.9 ppm in the  $^{13}\text{C}$  NMR spectrum are due to the different types of methine carbon of the isopropoxy groups [29].

##### $^{27}\text{Al}$ NMR

The  $^{27}\text{Al}$  NMR spectrum of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  show a singlet at  $\delta$  62 ppm indicating tetrahedral environment about the Al atom and is surrounded by four oxygen atoms [30].

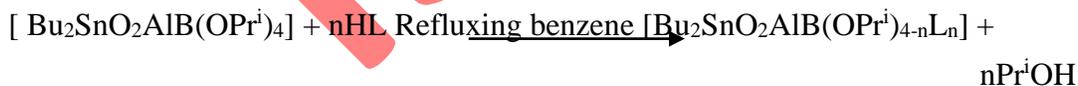
##### $^{119}\text{Sn}$ NMR

The  $^{119}\text{Sn}$  NMR spectrum of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  exhibits a sharp signal at  $\delta$  489 ppm assignable to the tetracoordination about Sn(IV) attributed to the polymeric nature of  $\mu$ -oxo compound [31].

##### $^{11}\text{B}$ NMR

The  $^{11}\text{B}$  NMR spectrum of the compound  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  display a signal at  $\delta$   $-3.23$  ppm indicating a tetra-coordinated environment about B atom which is surrounded by four oxygen atoms [32].

Further reactions of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  with  $\beta$ -diketones (HL) in various molar ratios have been performed in refluxing benzene yielded the compounds of the types  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_3\text{L}]$  and  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_2\text{L}_2]$  according to the following reaction scheme:



( $n = 1\text{-}2$ ,  $\text{L} = \text{acac}/\text{bzac}/\text{p-Brba}$ )

The isopropanol liberated during the reaction collected azeotropically (isopropanol-benzene) and estimated oxidimetrically to check the progress of the reaction. It was observed that only two out of the four of isopropoxy groups of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  could be replaced by  $\beta$ -diketones. Further replacement of isopropoxy groups could not be achieved even with an excess of ligand ( $\beta$ -diketones)

and prolonged refluxing time (approx. 12 h). This indicates the substitution of only terminal isopropoxy groups not bridging.

#### IR Spectra

The spectra of 1:1  $\beta$ -diketone derivatives of  $\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4$  show absorption bands in the region  $1360\text{-}1340\text{ cm}^{-1}$ ,  $1165\text{-}1150$  and  $1115\text{-}1110\text{ cm}^{-1}$  are the characteristics of *gem*-dimethyl portion and combination bands  $\nu(\text{C-O}+\text{OPr}^i)$  of the bridging and terminal isopropoxy group respectively. No peak is observed at  $1115\text{-}1110\text{ cm}^{-1}$  in the 1:2 and 1:3  $\beta$ -diketonates indicates the absence of terminal isopropoxy group. A band appeared at approximately  $950\text{ cm}^{-1}$  is due to  $\nu(\text{C-O})$  stretching of bridging isopropoxy group. Similar spectra were recorded for 1:4  $\beta$ -diketonates as found for 1:3  $\beta$ -diketone derivatives. The IR spectrum of  $\beta$ -diketonates<sup>[33]</sup> display strong bands at  $\sim 1600\text{-}1580\text{ cm}^{-1}$  and  $\sim 1520\text{-}1500\text{ cm}^{-1}$  due to  $\nu_{\text{sym}}(\text{C=O})$  and  $\nu_{\text{asym}}(\text{C=C})$  respectively along with a broad band at  $\sim 3100\text{-}2700\text{ cm}^{-1}$  due to enolic stretch. The absence of shift in  $\text{C=O}$  frequency and disappearance of broad band at  $3100\text{-}2700\text{ cm}^{-1}$  in the  $\beta$ -diketonates of  $\mu$ -oxoisopropoxide compound suggest that bonding takes place through both of the terminal oxygen of CO group. A number of vibrations are observed in the region  $700\text{-}400\text{ cm}^{-1}$  due to M-O stretching vibrations in all  $\beta$ -diketonates.

#### NMR spectra

##### $^1\text{H}$ NMR

$^1\text{H}$  NMR spectra of all the  $\beta$ -diketone derivatives of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  show broad multiplet centered between  $\delta 0.8\text{-}1.2$  ppm due to the intermixing of methyl protons of isopropoxy groups. A broad multiplet centered at  $\delta 4.0\text{-}4.2$  is due to the methine proton of isopropoxy groups in the spectra of all derivatives. All the  $\beta$ -diketone derivatives of  $\mu$ -oxoisopropoxide compound show singlet at  $\delta 2.1$  ppm and  $\sim \delta 5.8$  ppm due to methyl and methine proton of the ligand moiety respectively. Further, the peaks observed in the region  $\delta 7.0\text{-}7.7$  ppm in benzoylacetone derivative of  $[\text{Bu}_2\text{SnO}_2\text{AIB}(\text{OPr}^i)_4]$  are due to the phenyl ring protons.

##### $^{13}\text{C}$ NMR

The  $^{13}\text{C}$  NMR spectra of 1:1  $\beta$ -diketone derivatives of  $\mu$ -oxoisopropoxide compound show two prominent peaks between  $\delta 26.4\text{-}27.7$  ppm and  $\delta 27.9\text{-}28.4$  ppm assignable to the methyl carbon of terminal and bridging isopropoxy groups. The two peaks observed at  $\delta 62.6\text{-}62.8$  ppm and  $\delta 63.1\text{-}63.4$  ppm assignable to the methine carbons of isopropoxy groups in the derivatives. Two single peak are observed at  $\sim \delta 26.1$  and about  $\sim \delta 62.4$  ppm in 1:2 and 1:3 derivatives due to the methyl and methine carbon of terminal isopropoxy group. Two peaks observed in the range  $\delta 190.2\text{-}181.7$  ppm and  $\delta 98.4\text{-}92.3$  ppm are due to carbonyl carbon and methine carbon of ligand moiety in all the  $\beta$ -diketone derivatives of  $\mu$ -oxoisopropoxide compound. The peaks observed at  $127.1$ ,  $126.4$ ,  $125.3$  and  $136.7$  ppm are due to ortho, meta, para and substituted carbon of the phenyl ring respectively in the spectra of benzoylacetone derivative of  $\mu$ -oxoisopropoxide compound. Further, the spectra of *p*-bromobenzoylacetone derivatives of  $\mu$ -oxoisopropoxide show the shifting of the para carbon peak from  $125.3$  to  $\sim 131$  ppm due to the bromo group substituted at para position.

The 1:1  $\beta$ -diketone derivatives spectra show the presence of tetrahedral and octahedral environment about aluminium atom. But the spectra of 1:2 and 1:3  $\beta$ -diketone derivatives of  $\mu$ -oxoisopropoxide indicate that the aluminium atom is surrounded octahedrally by oxygen atoms in these compounds [34].

The aforesaid spectral, and elemental analysis suggest the following tentative structures of  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_4]$ , and its  $\beta$ -diketone derivatives of the type  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_3(\text{L})]$ ,  $[\text{Bu}_2\text{SnO}_2\text{AlB}(\text{OPr}^i)_2(\text{L})_2]$  are given as below (Fig. 1).

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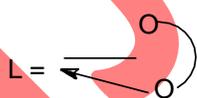
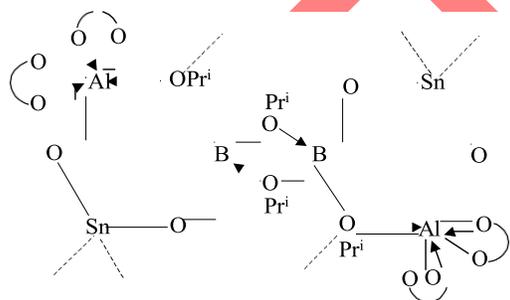
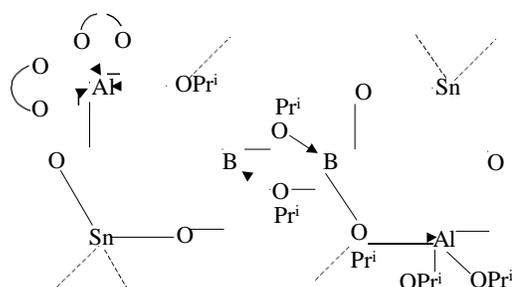
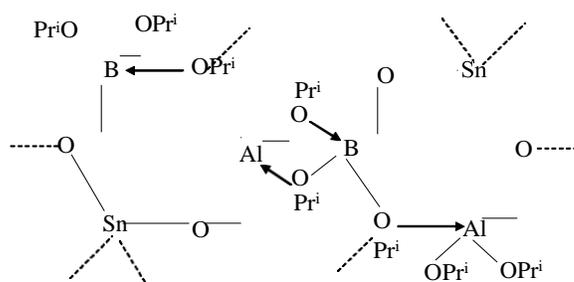


Fig. 1

Table 1 : Analytical data

S.No.	Compound	Ligand				Molar	Refluxing	Product
	Anal. Calcd. (found)							
	g (mmol)	g(mmol)				ratio	time (hrs.)	
	HOPr <sup>i</sup> Sn Al B							
								g (%)
	g (%) (%) (%)							
1.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hacac				1:1	4	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>3</sub> (acac)]
	0.055 21.59 4.80 1.74							
	0.478 (0.915)	0.0915 (0.915)						0.448(72.9)
	(0.53) (21.62) (5.72) (2.1)							
2.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hacac				1:2	5	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>2</sub> (acac) <sub>2</sub> ]
	0.120 19.56 4.34 1.68							
	0.524 (1.002)	0.200 (2.00)						0.543 (78.5)
	(0.12) (19.58) (4.37) (1.58)							
3.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hbzac				1:1	3	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>3</sub> (bzac)]
	0.061 18.57 4.12 1.60							
	0.534 (1.023)	0.166 (1.023)						0.578 (79.6)
	(0.059) (18.61) (4.08) (1.63)							
4.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hbzac				1:2	5	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>2</sub> (bzac) <sub>2</sub> ]
0.130	14.90 3.28 1.58							
	0.539 (1.08)	0.350 (2.16)						0.735 (84.8)
	(0.128) (14.86) (3.30) (1.52)							
5.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hp-Brba				1:1	4	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>3</sub> (p-Brba)]
	0.06 16.63 3.45 1.61							
	0.524 (1.007)	0.243 (1.007)						0.654 (83.9)
	(0.059) (16.67) (3.48) (1.66)							
6.	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>4</sub> ]	Hp-Brba				1:2	6	[Bu <sub>2</sub> SnO <sub>2</sub> AlB(OPr <sup>i</sup> ) <sub>2</sub> (p-Brba) <sub>2</sub> ]
	0.12 13.11 2.42 1.28							
	0.534 (1.02)	0.492 (2.04)						0.842(85.8)
	(0.12) (13.16) (2.44) (1.28)							