Journal of the Mexican Chemical Society
ISSN: 1870-249X
editor.jmcs@gmail.com
Sociedad Química de México
México

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Journal of the Mexican Chemical Society, vol. 60, núm. 4, octubre-diciembre, 2016, pp. 194-199
Sociedad Química de México
Distrito Federal, México

Article

J. Mex. Chem. Soc. **2016**, *60(4)*, 194-199

ISSN 1870-249X

Synthesis and Characterization of Three New di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV)

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Received June 16, 2016; Accepted October 12, 2016.

Abstract: The synthesis of novel di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV) (alkyl = octyl, dodecyl, octadecyl) **2a-2c** is reported. The complexes were characterized by ^{1}H, ^{13}C, ^{119}Sn NMR, IR, MS and elemental analyses. For compound **2a** the ^{119}Sn NMR showed the presence of five signals suggesting the existence of a mixture of five hexa–coordinated species in equilibrium, two trans (I, II) and three cis (III, IV, V). In contrast, the compounds **2b** and **2c** present only unique hexa–coordinated specie.
Keywords: Diorganotin; Sn (IV) complex; tin; organotin; spectroscopy.

Resumen: Se reporta la síntesis de nuevos di-*n*-butil [bis (alquil-ácido aminopropiónico)] de estaño (IV) (alquil = octil, dodecil y octadecil) **2a-2c**. Los complejos fueron caracterizados por ^{1}H, ^{13}C, ^{119}Sn RMN, IR, MS y análisis elemental. Para el compuesto **2a** el espectro de ^{119}Sn RMN muestra la presencia de cinco señales sugiriendo la existencia de cinco especies hexa-coordinadas en equilibrio, dos trans (I, II) y tres cis (III, IV, V). En contraste, los compuestos **2b** y **2c** presentan solo una especie hexa-coordinada.
Palabra clave: Diorganoestaño; complejos de Sn (IV); estaño, organoestaño; espectroscopía.

Introduction

Organotin compounds have been extensively studied in the structural chemistry. For organotin (IV) compounds present frequently the formation of polyhedra with tetrahedral, trigonal-bipyramidal, octahedral and pentagonal-bipyramidal coordination geometries, the final coordination number depends strongly on the number of organic substituents attached to tin atom and the nature of the ligands [1-6].

The organotin (IV) compounds and them derives exhibit antitumor activity [7, 8], biocides [9], material with non-linear optical properties [10], fluorescence probes for DNA traces [11], model systems like nucleotides [12], amino acids [13], peptides [14] and corrosion inhibitors [15].

In particular, diorganotin (IV) compounds derived from ligands containing nitrogen and oxygen donor atoms present increasing number of reports, mainly derivates of aminoacids and analogues have been investigated [16, 17]. Our current interest in the synthesis of organotin compounds and structural analysis [18-22], prompted us to extend our investigation in the synthesis of new organotin (IV) compounds, which could have corrosion inhibitors properties [23].

This paper describes the synthesis of three new di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV) (alkyl = octyl, dodecyl and octadecyl) **2a-2c**, by the reaction of di-*n*-butyl (IV) oxide and alkyl aminopropionic acid (alkyl = octyl, dodecyl and octadecyl) **1a-1c** in a 1:2 molar ratio. All compounds were characterized by ^{1}H, ^{13}C, ^{119}Sn NMR, infrared spectroscopy, mass spectrometry and elemental analyses.

Results and Discussion

Herein we report the reaction of alkyl aminopropionic acids **1a-1c**, with di-*n*-butyltin (IV) oxide in a 2:1 ratio to obtain di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV) **2a-2c** as white solids (Fig. 1). The compounds **1a-1c** were obtained through the reaction of alkyl amine (alkyl = octyl, dodecyl and octadecyl) and acrylic acid [24].

The ^{1}H NMR spectra in $CDCl_3$ of all the compounds **2a-2c** (^{1}H NMR data are summarized in Table 1) show only one set of signal for each magnetically equivalent H nucleus, thus both $(n\text{-}Bu)_2Sn$ proton and those of the ligands are in 1:2 ratio, respectively. For **2b** and **2c** compounds, this indicate the formation of unique species or a fast-exchanging equilibrium on the NMR time scale (Support information). For the compound **2b** for example, the H3 and H4 (3.008 and 2.823 ppm) proton appeared as broad signals at higher field ($\Delta\delta$ = 0.05 and 0.083) with respect to the free ligand **1b** (Support information). This suggests a shielding of these protons upon coordination giving evidence for the formation of a Sn-O bonded and NH→Sn coordination bond.

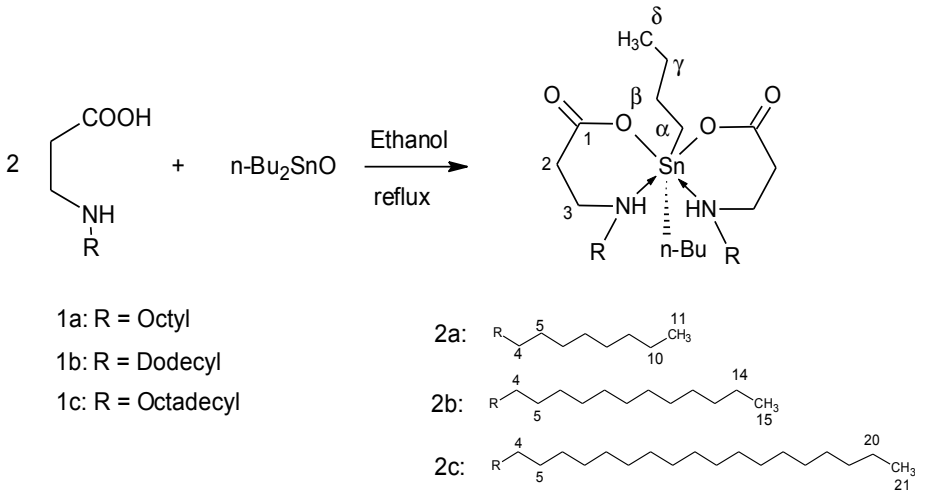
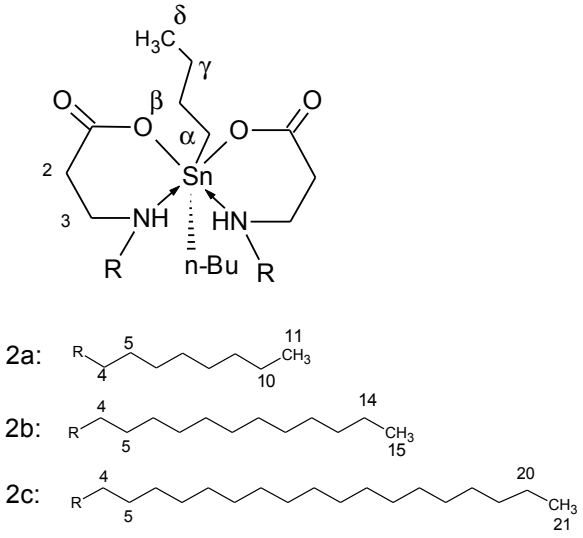


Fig. 1. Synthesis of new di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV).

Table 1. Chemical shift of ^{1}H NMR for compounds 2a-2c.



Compound			
	2a	2b	2c
H2	2.504 (b)	2.504 (b)	2.367 (b)
H3	3.045(b)	3.008 (b)	2.914 (m)
H4	2.869 (b)	2.823 (b)	2.725 (m)
H5	1.662 (b)	1.635 (b)	1.532 (b)
H6-H10	1.262 (m)		
H11	0.877 (t, J=6.6)		
H6-H14		1.242 (m)	
H15		0.869 (t, J=6.6)	
H6-H19			1.135 (m)
H20			0.744 (m)
Hα-Hβ	1.215 (m)	1.242 (m)	1.135 (m)
Hγ	1.180 (m)	1.242 (m)	1.135 (m)
Hδ	0.877 (t, J=6)	0.869 (t, J=6.6)	0.744 (m)

d (^{1}H) relative to $Si(CH_3)_4$; solvent $CDCl_3$. b: broad; m: unresolved pattern; t: triplet and J Hz.

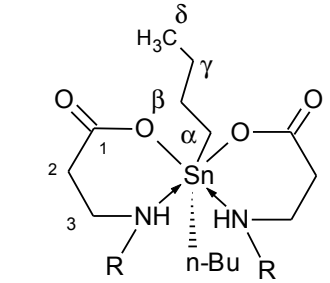
For the compound **2b** the ^{13}C NMR spectrum in $CDCl_3$, the carbon C3 and C4 appeared as broad signals (47.623 and 44.748 ppm) at low field ($\Delta\delta$ = 0.521 and 0.170) with respect to the free ligand **1b**, the chemical shift of these carbons gave evidence for the formation of NH→Sn coordination bond. Likewise, the chemical shift of the signals of the compound **2b** the carbons C1 and C2 appeared as broad signals (177.242 and 33.154 ppm) with respect to the carbons of free ligand **1b** ($\Delta\delta$ = 0.316 and 1.254) giving evidence for the formation of Sn-O bonded. The ^{13}C NMR data are summarized in Table 2.

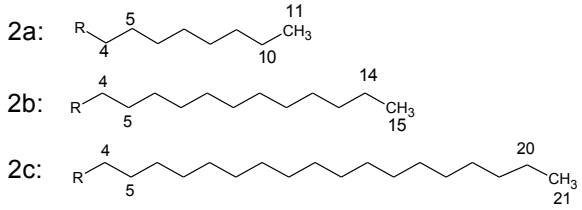
All compounds **(2a-2c)** exhibited $^{1}J(^{119}Sn\text{-}^{13}C)$ and $^{2}J(^{117}Sn\text{-}^{13}C)$ coupling 749/608, 720/690 and 705/633 Hz respectively within the range of trans six-coordinated tin compound [16-19] The ^{119}Sn NMR spectra of **2b** and **2c** at room temperature in chloroform show only one signal at -203.96 and -204.20 ppm respectively. This can indicate unique species or a fast exchanging equilibrium, on the NMR time scale. The chemical shifts are within the range of di-*n*-butyl (IV) hexacoordinated compounds [3-6, 18-22]. Neverthelees, for compound **2a,** the ^{119}Sn NMR spectrum show five signals in ca. 0.9:1.0:1.1:1.5:1.0 ratio, δ -176.21, -186.10, -194.50, -204.37 and -215.91 ppm associate to the hexa-coordinated species, this result suggested the existence of a mixture of an equilibrium between five different hexacoordinated isomers, two trans (I, II) and three cis (III, IV, V) in solution at room temperature, the isomerism is attributed a different arrangement of the ligands [28, 21] as show in Fig. 2, a possible explanation in formation of hexacoordinate isomers due to the presence of chain short, giving as a result the existence of equilibrium dynamic.

The IR spectra for the compounds **2a-2c** exhibit one $\upsilon_{(C=O)}$ carbonyl band in 1654 cm^{-1}. Also a broad band one of them assigned to the N-H in 1566 cm^{-1} and other due Sn-O band is observed in 430 cm^{-1}.

The mass spectra in all three cases for **2a-2c**, (15 eV IE) displayed the presence of molecular ion confirming of the

Table 2. Chemical shift of ^{13}C NMR for compounds 2a-2c.





Compound			
	2a	2b	2c
C1	177.242	177.242	177.037
C2	33.066	33.154	32.524
C3	47.579	47.623	47.343
C4	44.723	44.782	44.460
C5	31.646	31.807	31.821
C6	29.117		
C7	29.068		
C8-C9	26.813		
C10	22.507		
C11	13.995		
C6-C11		29.257-29.537	
C12-C13		26.710-26.888	
C14		22.581	
C15		14.014	
C6-C17			29.156-29.626
C18			26.696
C19			26.154
C20			22.581
C21			14.014
Cα-	27.399 [749/608]	27.487 [720/690]	27.370 [705/633]
Cβ- Cγ	27.252-27.399	27.106-27.252	27.062-27.208
Cδ	13.560	13.604	13.574

Chemical shifts in ppm respect to TMS $^{1}J(^{13}C\text{-}^{119}Sn)$, $^{2}J(^{13}C\text{-}^{117}Sn)$ coupling constant between square brackets.

formation of the products, the fragmentation process start with the loss of a ligand, followed by loss of *n*-butyl groups (yielding the base peak for **2a**), and later loss of Sn atom giving the base peak for compounds **2b** and **2c**. The spectra show others fragment ion and a possible fragmentation pattern is given in Fig. 3.

Experimental section

Materials

The reagents were purchased from Aldrich Co. Melting points were determined in open capillaries on Electrothermal melting apparatus (UK) and are uncorrected. IR spectra were recorded on Bruker Tensor-27 FT-IR using KBr pellets. ^{1}H, ^{13}C and ^{119}Sn NMR spectra were recorded on VARIAN Mercury 200-BB spectrometer. The ^{1}H and ^{13}C chemical shifts [ppm] are relative to internal $SiMe_4$ (TMS) and the ^{119}Sn chemical shifts d [ppm] are relative to internal $SnMe_4$. Results are presented as, chemical schift in ppm, multiplicity coupling constants in Hz, number of protons, proton's position. Multiplicities are shown as the abbreviations: s (singlet), d (doublet), t (triplet), and m (multiplet). Mass spectra were obtained in HP 5973 MSD eV = 15.2 (Direct insertion probe). Elemental analyses were performed on CHNS analysis in Perkin Elmer Series II 2400. Solvent were commercially available reagent grade.

Synthesis of alkyl-aminopropionic acid 1a-3a (alkyl = octyl, docecyl and octadecyl)

Octyl-aminopropionic acid 1a: 5.0 g (0.038 mol) of octylamine was slowly added 3.35 g (0.046 mol) of acrylic acid; the reaction was placed in flask equipped with a magnetic stirred, thermometer and reflux system. The reaction was carried to 90-95 ºC during 2 hours. After being cooled to room temperature the residue was treated with hexane, the solution is filtered and the residue was treated with chloroform, the solution was filtered and the solvent was evaporated under vacuum to yield 7.82 g (95 %) of compound **1a** as white solid. Yield 95%; mp 86-87 °C; IR (KBr, υ_{max}/cm^{-1}) 3375 (N-H), 1655 (C=O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.856 (t, J = 6.6 Hz, 3H, H-11), 1.241 (m, 10H, H-6 to H-10), 1,721 (m, 2H, H-5), 2.506 (t, J = 5.5 Hz, 2H, H-2), 2.895 (t, J = 7.8 Hz, 2H, H-4), 3.055 (t, J = 5.5 Hz, 2H, H-3); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 176.920 (C-1), 46.964 (C-3), 44.518 (C-4), 31.792 (C-2), 31.675 (C-5), 29.068 (C-6, C-7), 26.710 (C-8), 26.169 (C-9), 22.566 (C-10), 14.043 (C-11); elemental analysis: $C_{11}H_{23}NO_2$ Calc.: C 65.97, H 12.22, N 6.95; found: C 65.65, H 12.18, N 6.94.

Dodecyl-aminopropionic acid 1b: 5.0 g (0.027 mol) of dodecylamine and 2.3 g (0.032 mol) of acrylic acid gave 6.52 g (94%) of compound **1b** as white solid. Yield 94%; mp 89-90 °C; IR (KBr, υ_{max}/cm^{-1}) 3374 (N-H), 1655 (C=O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.879 (t, J = 6.6 Hz, 3H, H-15),

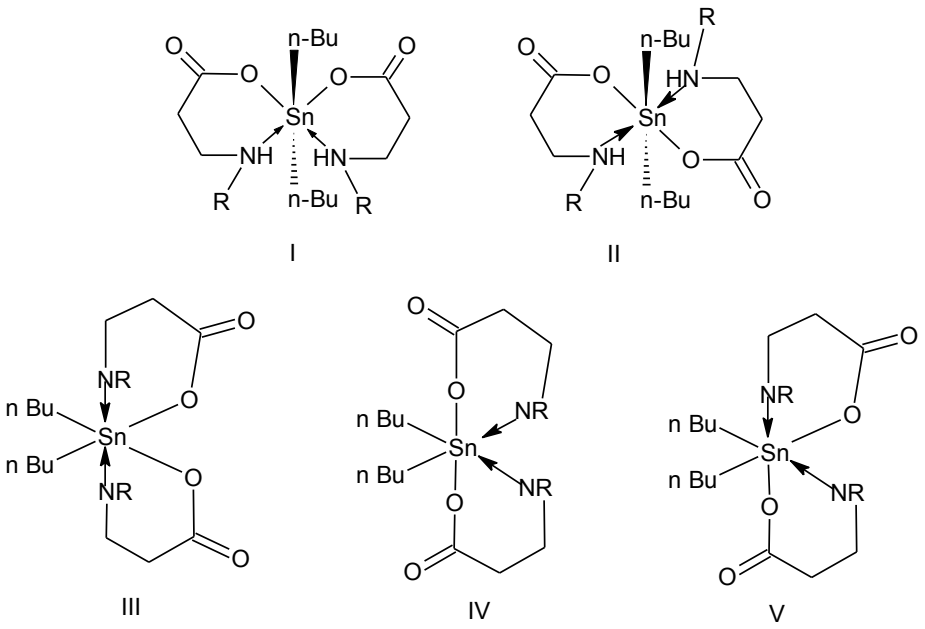


Fig. 2. Hexacoordinated isomers for the compound **2a.**

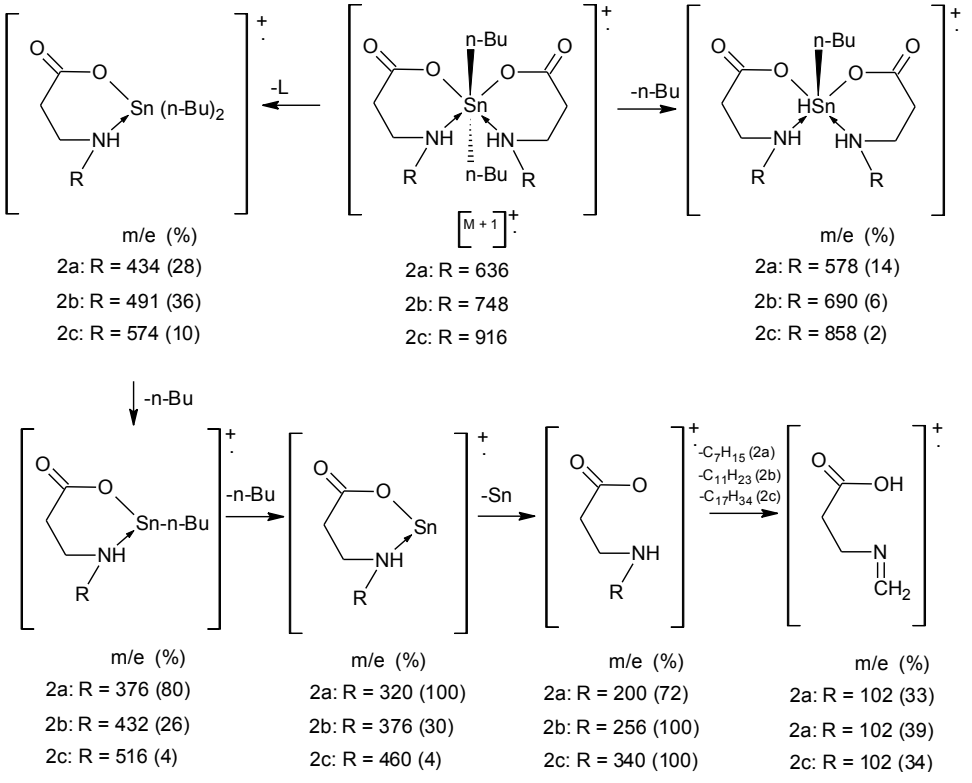


Fig. 3. Mass spectra for compounds **2a-2c**

1.250 (m, 18H, H-6 to H-14), 1,746 (m, 2H, H-5), 2.522 (t, J = 6.0 Hz, 2H, H-2), 2.906 (t, J = 7.8 Hz, 2H, H-4), 3.057 (t, J = 6.0 Hz, 2H, H-3); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 176.926 (C-1), 47.102 (C-3), 44.612 (C-4), 31.900 (C-2), 31.564 (C-5), 29.103-29.613 (C-6 to C-11), 26.731 (C-12), 26.394 (C-13), 22.689 (C-14), 14.137 (C-15); elemental analysis: $C_{15}H_{31}NO_2$ Calc.: C 67.42, H 12.45, N 5.26; found: C 67.46, H 12.50, N 5.26.

Octadecyl-aminopropionic acid 1c: 5.0 g (0.018 mol) of octadecylamine and 1.6 g (0.022 mol) of acrylic acid gave 5.52 g

(90%) of compound **1c** as white solid. Yield 90%; mp 91-92 °C; IR (KBr, υ_{max}/cm^{-1}) 3374 (N-H), 1655 (C-O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.871 (t, *J* = 6.6 Hz, 3H, H-21), 1.244 (m, 26H, H-6 to H-20), 1,740 (m, 2H, H-5), 2.522 (t, *J* = 6.0 Hz, 2H, H-2), 2.900 (t, *J* = 7.8 Hz, 2H, H-4), 3.059 (t, *J* = 6.0 Hz, 2H, H-3); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 176.920 (C-1), 47.052 (C-3), 44.548 (C-4), 31.909 (C-2), 31.734 (C-5), 29.127-29.698 (C-6 to C-17), 26.740 (C-18), 26.227 (C-19), 22.683 (C-20), 14.116 (C-21); elemental analysis: $C_{21}H_{43}NO_2$ Calc.: C 73.06, H 13.26, N 4.15; found: C 72.99, H 13.45, N 4.11.

Synthesis of di-*n*-butyl [bis(alkyl-aminopropionic acid)]tin (IV) 2a-2c (alkyl = octyl, docecyl and octadecyl)

Complex di-*n*-butyl [bis(octyl-aminopropionic acid)]tin (IV) 2a: A solution of 0.5 g (0.00248 mol) of **1a** in 100.0 ml of ethanol was placed in flask equipped with a magnetic stirred and dean-Stark trap and 0.3 g (0.0012 mol) of di-*n*-butyltin (IV) oxide were added. The suspension was refluxed during 10 hours. After being cooled to room temperature, the solvent was evaporated under vacuum. The residue was treated with chloroform, the solution was filtered and the solvent was evaporated under vacuum to yield 1.25 g (80 %) of compound **2a** as yellow solid. Yield 80%; mp 90-91 °C; IR (KBr, υ_{max}/cm^{-1}) 1654 (C=O), 1566 (N-H), 430 (Sn-O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.877 (t, *J* = 6.6 Hz, 6H, H-11), 1.262 (m, 10H, H-6 to H-10), 1,662 (broad, 2H, H-5), 2.504 (broad, 2H, H-2), 2.869 (b, 2H, H-4), 3.045 (b, 2H, H-3), 1.215 (m, 4H, Hα-Hβ), 1.180 (m, 2H, Hγ), 0.877 (t, J=6 Hz, 3H, Hδ); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 177.242 (C-1), 47.579 (C-3), 44.723 (C-4), 33.066 (C-2), 31.646 (C-5), 29.117 (C-6), 29.068 (C-7), 26.813 (C-8, C-9), 22.507 (C-10), 13.995 (C-11), 27.399 [Ca, $^{1}J(^{119}Sn-^{13}C)$=749, $^{1}J(^{117}Sn-^{13}C)$=608 Hz], 27.252-27.399 (Cβ-Cγ), 13.560 (Cδ); ^{119}Sn NMR (200MHz, $CDCl_3$, δ ppm) -176.21, -186.10, -194.50, -204.37, -215.91 ppm; elemental analysis: $C_{30}H_{62}N_2O_4Sn$ Calc.: C 53.90, H 10.44, N 4.32; found: C 53.79, H 10.52, N 4.27.

Complex di-*n*-butyl [bis(dodecyl-aminopropionic acid)]tin (IV) 2b: 0.5 g (0.00194 mol) of **1b** and 0.242 g (0.00097 mol) of di-*n*-butyltin (IV) oxide gave 1.15 g (81 %) of compound **2b** as yellow solid. Yield 81%; mp 91-92 °C; IR (KBr, υ_{max}/cm^{-1}) 1654 (C=O), 1566 (N-H), 430 (Sn-O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.869 (t, *J* = 6.6 Hz, 3H, H-15), 1.242 (m, 18H, H-6 to H-14), 1,635 (b, 2H, H-5), 2.504 (b, 2H, H-2), 2.823 (b, 2H, H-4), 3.008 (b, 2H, H-3), 1.242 (m, 4H, Hα-Hβ), 1.242 (m, 2H, Hγ), 0.869 (t, J=6.6 Hz, 3H, Hδ); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 177.242 (C-1), 47.623 (C-3), 44.782 (C-4), 33.154 (C-2), 31.807 (C-5), 29.257-29.537 (C-6 to C-11), 26.888 (C-12), 26.710 (C-13), 22.581 (C-14), 14.014 (C-15), 27.487 [Cα, $^{1}J(^{119}Sn-^{13}C)$ = 720, $^{2}J(^{117}Sn-^{13}C)$=690 Hz], 27.252-27.106 (Cβ-Cγ), 13.604 (Cδ); ^{119}Sn NMR (200MHz, $CDCl_3$, δ ppm) -203.96 ppm; elemental analysis: $C_{38}H_{78}NO_4Sn$ Calc.: C 58.31, H 10.87, N 3.50; found: C 58.33, H 10.85, N 3.48.

Complex di-*n*-butyl [bis(octadecyl-aminopropionic acid)] tin (IV) 2c: 1.5 g (0.0044 mol) of **1c** and 0.547 g (0.0022 mol) of di-n-butyltin (IV) oxide gave 2.5 g (78 %) of compound **2c** as yellow solid. Yield 78%; mp 93 °C; IR (KBr, υ_{max}/cm^{-1}) 1654 (C=O), 1566 (N-H), 430 (Sn-O); ^{1}H NMR (200MHz, $CDCl_3$, δ ppm): 0.744 (m, 3H, H-21), 1.135 (m, 26H, H-6 to H-20), 1.532 (b, 2H, H-5), 2.367 (b, 2H, H-2), 2.725 (b, 2H, H-4), 2.914 (b, 2H, H-3), 1.135 (m, 6H, Hα,Hβ,Hγ), 0.744 (m, 3H, Hδ); ^{13}C NMR (50MHz, $CDCl_3$, δ ppm); 177.037 (C-1), 47.343 (C-3), 44.460 (C-4), 32.524 (C-2), 31.821 (C-5), 29.156-29.629 (C-6 to C-17), 26.696 (C-18), 26.154 (C-19), 22.581 (C-20), 14.014 (C-21), 27.370 [Cα, $^{1}J(^{119}Sn-^{13}C)$=705, $^{1}J(^{117}Sn-^{13}C)$=633 Hz], 27.062-27.208 (Cβ-Cγ), 13.574 (Cδ); ^{119}Sn NMR (200MHz, $CDCl_3$, δ ppm) -204.20 ppm; elemental analysis: $C_{50}H_{102}N_2O_4Sn$ Calc.: C 73.51, H 11.06, N 4.37; found: C 73.19, H 11.11, N 4.42.

Conclusions

Novel di-*n*-butyl [bis (alkyl-aminopropionic acid)]tin (IV) (alkyl= octyl, dodecyl and octadecyl) **2a-2c** were obtained. The hexacoordinated complexes were characterized by ^{1}H, ^{13}C, ^{119}Sn NMR, infrared spectroscopy, mass spectrometry and elemental analyses. The compound **2a** showed five hexa–coordinate species indicating the existence of a mixture in equilibrium between two trans (I, II) and three cis (III, IV, V) hexacoordinated isomers. Whereas for the compounds **2b** and **2c** only unique species were observed due to due to steric hindrance of alkyl substituents.

Acknowledgments

The authors want to acknowledge the Mexican Institute of Petroleum (IMP), Project D.00402 “Escalamiento y evaluación a nivel industrial de un nuevo inhibidor de corrosion para medios básicos característicos de plantas FCC IMP-ICIM-8000”.

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