

## TRIORGANOTIN DERIVATIVES OF 1,2-BIS(2'-CARBOXYPHENYLAMINO)-ETHANE AND -PROPANE, AND ETHYLENEDIAMINETETRAACETIC ACID

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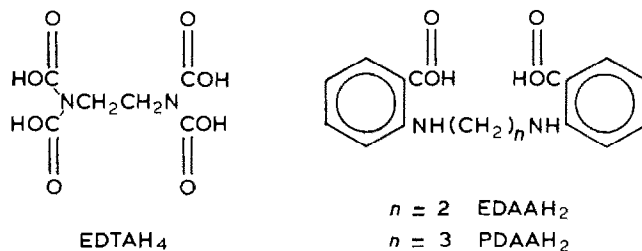
(Received May 13th, 1986)

### Summary

Fifteen new complexes have been prepared of the type  $(R_3Sn)_2L$ , where L is the dianion of 1,2-bis(2'-carboxyphenylamino)-ethane and -propane, and ethylenediaminetetraacetic acid, and R is Me, n-Pr, n-Bu, Ph, or cyclohexyl (Cy). Characterisation by IR,  $^1H$  NMR and  $^{119}Sn$  Mössbauer spectroscopy indicates that the ligands are bound only through oxygen. In most cases the carboxylates are bidentate and the tin five-coordinate. The  $Ph_3Sn$  and  $Cy_3Sn$  derivatives contain tetrahedral tin, with monodentate carboxylates.

### Introduction

The tributyltin derivative of ethylenediaminetetraacetic acid has been shown to be a useful biocide [1], but has not been otherwise characterised. The diorganotin derivatives of this ligand are monomeric chelates,  $R_2Sn(EDTAH_2) \cdot 2H_2O$ , and have been characterised by their infrared spectra [2]. We now describe the preparation and characterisation of bis-triorganotin derivatives of  $EDTAH_4$  and the new ligands  $EDA AH_2$  and  $PDA AH_2$ .



## Experimental

Trimethyl- and tributyl-tin chlorides were obtained from Alfa Products and Fluka Chemicals respectively, and used as such. Tripropyl-, triphenyl- and tri-cyclohexyl-tin chlorides were prepared by published methods [3].

### *1,2-Bis(2'-carboxyphenylamine)ethane (EDA<sub>2</sub>H<sub>2</sub>)*

To a suspension of *o*-aminobenzoic acid (13.7 g, 0.10 mol) was added sodium hydroxide (4.0 g, 0.10 mol), and the mixture was warmed to give a clear solution. 1,2-Dibromoethane (9.4 g, 4.4 cm<sup>3</sup>, 0.05 mol) was added and the mixture refluxed until a light-yellow solid began to separate (8–10 h). More solid separated on cooling, which was washed twice or thrice with boiling water. The solid was dried, ground to a powder, stirred with the minimum amount of glacial acetic acid, filtered, washed with water, and dried. Yield 60%; M.p. 228°C. Found: C, 63.64; H, 5.58; N, 9.13. C<sub>16</sub>H<sub>16</sub>N<sub>2</sub>O<sub>4</sub> calcd.: C, 64.00; H, 5.33; N, 9.23%.

### *1,3-Bis(2'-carboxyphenylamine)propane (PDA<sub>2</sub>H<sub>2</sub>)*

This compound was prepared analogously from anthranilic acid and 1,3-dibromopropane. Yield 40%; M.p. 220–221°C (dec.). Found: C, 66.25; H, 5.88; N, 8.40. C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub> calcd.: C, 65.85; H, 6.09; N, 8.53%.

### *Sodium salts of EDA<sub>2</sub>H<sub>2</sub> and PDA<sub>2</sub>H<sub>2</sub>*

The acid (0.023 mol) and sodium hydroxide (0.046 mol) were dissolved in ethyl alcohol (50 cm<sup>3</sup>) and refluxed until a clear solution was obtained. The alcohol was removed by distillation, benzene (20 cm<sup>3</sup>) was added, and the distillation continued to remove water azeotropically, using a Dean and Stark apparatus. The solid salt was filtered off, washed with dry ether, and dried in vacuum. The silver salt of EDTAH<sub>4</sub> was obtained by the addition of silver nitrate to a solution of the disodium salt.

### *Triorganotin(IV) complexes*

Ethanol solutions of the triorganotin chlorides (2.0 mmol) were treated with EDAANa<sub>2</sub> or PDAANa<sub>2</sub> and refluxed until clear solutions were obtained (about 3 h). The bulk of the ethanol was removed by distillation and the separated sodium chloride was removed by filtration. Thiophene-free dry benzene (20 cm<sup>3</sup>) was added to the filtrate and distillation continued, using a Dean and Stark apparatus. The filtration/reflux cycle was repeated until removal of sodium chloride was complete. Solvent was then removed, and the residue dried under vacuum. The sodium chloride produced was dissolved in water and determined with silver nitrate. Compounds **1** and **2** were recrystallised from ethanol, and compounds **10**, **11**, **13**, and **14** were washed with petroleum ether (60–80°C) to remove traces of triorganotin chloride. Compounds **4–7** were purified by dissolution in petroleum ether, in which the ligands are insoluble. (Analytical data and the numbering scheme are shown in Table 1.)

EDTA derivatives were obtained using EDTAH<sub>2</sub>Ag<sub>2</sub> in place of the above sodium salt. A mixture of the complex and silver chloride was precipitated from the ethanol. The solvent was removed by distillation, and the complex extracted from the porous residue with ethanol and thiophene-free benzene in a Soxhlet apparatus

(2–3 d). The complex was then recovered by distillation of the solvent. Any remaining traces of triorganotin chloride were removed by washing with petroleum ether.

#### *Physical measurements*

Melting points were determined in open capillaries, and are uncorrected. Elemental analyses for C, H, and N were carried out by the Microanalytical Service of Calcutta University, tin was determined as  $\text{SnO}_2$ , and chlorine (in the product sodium chloride) volumetrically. Molecular weights were determined cryoscopically in benzene and by the Rast method in molten camphor. Infrared spectra were recorded on a Pye-Unicam SP3-300 spectrometer as neat liquids or as KBr discs.  $^1\text{H}$  NMR spectra were obtained with a Tesla B487 spectrometer at 80 MHz, using TMS as internal standard; the free ligands were examined in trifluoroacetic acid,  $\text{EDTAH}_2\text{Na}_2 \cdot 2\text{H}_2\text{O}$ , in  $\text{D}_2\text{O}$ , and the tin complexes in  $\text{CDCl}_3$ .  $^{119}\text{Sn}$  Mössbauer spectra were obtained with a Harwell 6000 series spectrometer, with samples at ca. 80 K and source (Pd/Sn) at room temperature; isomer shifts are relative to  $\text{SnO}_2$  at room temperature.

#### **Results and discussion**

A series of new dinuclear triorganotin complexes of the ligands  $\text{EDAA}^{2-}$ ,  $\text{PDAA}^{2-}$ , and  $\text{EDTAH}_2^{2-}$  have been obtained by the reaction of the triorganotin chloride with the sodium or silver salt of the ligand. Molecular-weight data (Table 1) indicate that most of the complexes are monomers, but two (**6** and **9**) may be polymeric at room temperature but are monomeric in molten camphor; three others (**3**, **12**, **15**) are insoluble in molten camphor, and are presumably polymeric.

The assignments of important infrared bands [4] are shown in Table 2 both for the ligands and the complexes. The  $\nu(\text{N-H})$  band of the ligands is either unshifted or moved to higher frequency in the complexes, suggesting that the amino-nitrogen atom is not coordinated to tin. The mode of binding of the carboxyl groups can be assessed by the separation,  $\Delta\nu$ , of the symmetric and asymmetric COO stretching modes [5–8]. For eight of the complexes (**10–15**) of Table 2, values of  $\Delta\nu$  in the range  $250\text{--}270\text{ cm}^{-1}$  indicate either asymmetric or unidentate binding to tin; the remainder have  $\Delta\nu$  values of  $235\text{--}245\text{ cm}^{-1}$ , comparable to those of sodium salts, indicating bidentate binding. All the EDTA derivatives show a band at  $1665 \pm 10\text{ cm}^{-1}$  due to the free carboxylic acid group. The Sn–C stretching frequencies ( $500\text{--}600\text{ cm}^{-1}$  for alkyl derivatives, and  $250\text{--}300\text{ cm}^{-1}$  for aromatic compounds) allow the disposition of the organic groups to be assessed. All the complexes show two such bands although that at lower frequency is often quite weak. For compounds **1**, **3**, **10**, **11**, and **13**, the bands are of comparable intensity, indicating considerable deviation from planarity [9–12].

$^1\text{H}$  NMR data are shown in Table 3. Integrations agree well with the ratios expected except for the triphenyltin complexes, where severe overlapping of the two sets of phenyl signals occurs. The signals for the amino-group protons are either unshifted relative to those of the ligands, or are moved slightly upfield, indicating that the nitrogen atoms are not coordinated to tin. In the trimethyltin derivatives, multiplet signals for the methyl groups suggest either the non-equivalence of the eighteen protons or a non-planar arrangement of methyl groups.

(Continued on p. 318)

TABLE I

PHYSICAL AND ANALYTICAL DATA FOR TRIORGANOTIN(IV) DERIVATIVES (EDAAH<sub>3</sub> = 1,2-bis(2'-carboxyphenylamino)ethane; PDAAH<sub>3</sub> = 1,3-bis(2'-carboxyphenylamino)propane; EDTA · 2H<sub>2</sub>O = 1,2-diaminoethane-tetra-acetic acid disodium dihydrate)

Complex	Yield (%)	M.p. (°C)	Analysis (Found (calcd.)) (%)				Molecular weight			
			C	H	N	Sn	Cl <sup>b</sup>	C	R	Calcd.
{(CH <sub>3</sub> ) <sub>3</sub> Sn} <sub>2</sub> EDAA	85	152-154	41.90 (42.21)	4.85 (5.11)	4.05 (4.47)	37.85 (37.95)	17.65 (17.83)	598.4	580.4	625.4
{(CH <sub>3</sub> ) <sub>3</sub> Sn} <sub>2</sub> PDAA	85	189-190	43.68 (43.16)	4.98 (5.31)	4.70 (4.37)	36.90 (37.12)	17.60 (17.83)	601.3	598.6	639.4
{(CH <sub>3</sub> ) <sub>3</sub> Sn} <sub>2</sub> EDTA	80	203-205	30.85 (31.09)	5.07 (5.18)	4.18 (4.53)	38.31 (38.45)	-	-	-	617.4
{(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> Sn} <sub>2</sub> EDAA <sup>a</sup>	80	-	51.05 (51.42)	6.85 (7.05)	3.10 (3.52)	29.75 (29.92)	12.30 (12.53)	747.2	750.6	793.4
{(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> Sn} <sub>2</sub> PDAA <sup>a</sup>	84	-	51.90 (52.01)	6.88 (7.18)	3.95 (3.46)	29.30 (29.40)	12.20 (12.53)	778.8	770.4	807.4
{(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> Sn} <sub>2</sub> EDTA	75	103-105	42.55 (42.78)	6.88 (7.13)	4.09 (3.56)	29.84 (30.22)	-	-	721.8	785.4
{(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> Sn} <sub>2</sub> EDAA <sup>a</sup>	85	-	54.25 (54.70)	7.28 (7.75)	2.90 (3.19)	26.80 (27.05)	10.70 (10.91)	839.8	820.5	877.4

$[(n-C_4H_9)_3Sn]_2PDAA^a$ (8)	85	—	55.30 (55.19)	7.60 (7.85)	3.30 (3.14)	26.50 (26.63)	10.60 (10.91)	838.7	825.2	891.4
$(n-C_4H_9)_3Sn_2EDTA$ (9)	70	128–130	46.60 (46.92)	7.40 (7.82)	2.80 (3.22)	27.00 (27.30)	—	—	794.0	869.4
$[(C_6H_5)_3Sn]_2EDAA$ (10)	70	145–147	62.10 (62.56)	4.15 (4.41)	3.10 (2.80)	23.65 (23.80)	8.90 (9.21)	910.2	925.4	977.4
$[(C_6H_5)_3Sn]_2PDAA$ (11)	75	169–170	63.18 (62.88)	4.23 (4.54)	3.10 (2.76)	23.30 (23.47)	9.00 (9.21)	945.2	955.5	1011.4
$[(C_6H_5)_3Sn]_2EDTA$ (12)	80	brown at 250°C and black at 310°C (dec.)	55.60 (55.79)	4.20 (4.44)	3.10 (2.82)	23.71 (23.99)	—	—	—	989.4
$[(c-C_6H_{11})_3Sn]_2EDAA$ (13)	85	160–162	60.65 (60.38)	7.95 (7.74)	3.10 (2.71)	22.80 (22.97)	8.60 (8.80)	975.2	980.6	1033.4
$[(c-C_6H_{11})_3Sn]_2PDAA$ (14)	85	176–177	60.40 (60.72)	7.45 (7.82)	2.33 (2.67)	22.45 (22.66)	8.70 (8.80)	1014.2	1022.9	1047.4
$[(c-C_6H_{11})_3Sn]_2EDTA$ (15)	75	fuses at 122°C and melts at 138–140°C	53.45 (53.83)	8.07 (7.80)	3.10 (2.73)	22.86 (23.15)	—	—	—	1025.4

<sup>a</sup> Compound liquid in nature. <sup>b</sup> Corresponds to the Cl obtained as NaCl/AgCl from the reaction. <sup>c</sup> Insoluble in benzene and camphor.

TABLE 2

INFRARED SPECTRAL DATA ( $\text{cm}^{-1}$ ) (KBr and neat in polyethylene strips) (s = strong; b = broad; sp = sharp; m = medium; w = weak;  $\Delta\nu = \nu(\text{COO})_{\text{asym}} - \nu(\text{COO})_{\text{sym}}$ )

Compound	$\nu(\text{N-H})$ ( $\text{H}_2\text{O}$ )	$\nu(\text{COOH})$	$\nu(\text{COO})_{\text{asym}}$	$\nu(\text{COO})_{\text{sym}}$	$\Delta\nu$	$\nu(\text{Sn-C})$	$\nu(\text{Sn-O})$
EDAAH <sub>2</sub>	3380w,sp	2600m,b 2880m,b	1660vs,sp	1245vs,sp	415	—	—
EDAA-Na <sub>2</sub>	3340m,b	—	1610vs,sp	1390s,sp	220	—	—
PDAAH <sub>2</sub>	3380s,sp	2870m,b 2600mb	1655vs,sp	1240s,b	415	—	—
PDAANa <sub>2</sub>	3340-3280s,b	—	1615s,sp	1395vs,sp	220	—	—
EDTA-Na <sub>2</sub> ·2H <sub>2</sub> O	3380m,b 3510m,b	2580w,b 2770w,b	1610s,b	1310s 1390s	— 220	—	—
EDJAAg <sub>2</sub>	3340m,b 3350m,sp	—	1620vs,sp 1600vs,sp	1220w,sp 1390s	230 235	—	—
<b>1</b>	3240s,sp	—	1590s,sp	1350vs,sp	240	550s,sp 525m,sp 550s,sp	460m,sp 405m,sp 470s
<b>2</b>	3280m,b 3360-3440	—	1665vs 1650vs 1630vs	1385vs	245	520w,sp 550s,sp 505m,sp	470m,sp 450sb
<b>3</b>	3330s,sp	—	1620vs,sp	1385s,b	240	580m,b 515b 580m,b	480m,b 480m,b 480m,b
<b>4<sup>a</sup></b>	3330s,sp	—	1620vs,sp	1385s	235	515b 580m,b	480m,b 480m,b
<b>5<sup>a</sup></b>	3330s,sp	—	1620vs,sp	1385s	235	515b	480m,b

<b>6</b>	3400m,b	-	1660s 1630vs,b 1615s	1380s	235	550w,b 520w,b	475w,b
<b>7<sup>a</sup></b>	3320m	-	1615vs,sp	1380s	235	590w,sp 510w,b	480w,b 440sh
<b>8<sup>a</sup></b>	3300s,sp	-	1605vs,sp	1360s	245	570w 515w	470w
<b>9</b>	3430b	-	1660s 1640s 1625s	1380s	245	600w,b 515w,b	400w,b
<b>10</b>	3330m	-	1615vs,sp	1350s,sp	265	265m	365w,b
<b>11</b>	3360m	-	1620vs,sp	1360s	260	240m 280m,sp	335w,b 365w,b
<b>12</b>	3400b	-	1690vs,sp 1645vs	1375	270	250m 245m	330w,b 385m
<b>13</b>	3320m,sp	-	1610vs,sp	1355s	255	230 495s 475w	315m 385w 360w
<b>14</b>	3340m,sp	-	1620s,sp	1350s,sp	270	420m 485m,sp 415w	380w
<b>15</b>	3430m,b	-	1650s,sp 1620w,wp	1350s,sp	270	495wb 425w,sp	405w,sh

<sup>a</sup> Neat in polyethylene strips.

TABLE 3  
<sup>1</sup>H NMR DATA (δ in ppm)

Compound	NH	Ring protons	N(CH <sub>2</sub> ) <sub>n</sub> /NCH <sub>2</sub> COO	Sn-R
EDAAH <sub>2</sub> <sup>a</sup>	8.43 (m,2H)	7.83 (m,8H)	4.30 (s,4H)	-
PDAAH <sub>2</sub> <sup>a</sup>	8.43 (m,2H)	7.80 (m,8H)	3.80 (m,6H)	-
EDTANa <sub>2</sub> ·2H <sub>2</sub> O <sup>b</sup>	7.67	-	3.91-3.69/ (m,4H)	-
			5.24-4.12 (m,8H)	
1 <sup>c</sup>	7.90 (m,2H)	7.23 (m,2H)	3.43 (s,4H)	1.0-0.875 (m,18H)
2 <sup>c</sup>	8.10 (m,2H)	7.75 (t,2H)	3.43 (m,6H)	2.2-0.25 (m,18H)
3 <sup>d</sup>	-	-	-	-
4 <sup>c</sup>	7.98 (bd,2H)	7.80-7.13 (q,4H)	3.50 (bs,4H)	1.70 (m,12H)
5 <sup>c</sup>	7.95 (bd,2H)	7.50 (m,4H)	3.33	1.35 (m,12H)
6 <sup>c</sup>	7.27 (2H)	-	2.48-2.27 (m,12H)	1.03 (t,18H)
				1.30 (q,12H)
				2.27-1.22 (m,24H)



7 <sup>c</sup>	8.05 (bd,2H)	7.33 (bt,4H)	6.68 (m,4H)	3.50 (bs,4H)	1.55 (m,36H)	0.93 (t,18H)
8 <sup>c</sup>	7.95 (bd,2H)	7.88–6.60 (m,4H)	6.60–6.38 (m,4H)	3.35 (m,6H)	2.20–1.23 (m,36H)	0.93 (t,18H)
9 <sup>c</sup>	7.27 (2H)	–	–	2.60 (bm,12H)	2.17–1.29 (m,36H)	0.91 (t,18H)
10 <sup>c</sup>	8.08 (bd,2H)	7.70 (m,12H)	7.35 (m,14H)	6.68 (m,12H)	–	–
11 <sup>c</sup>	8.05 (m,2H)	7.68 (m,12H)	7.33 (m,14H)	6.53 (bd,12H)	–	–
12 <sup>d</sup>	–	–	–	–	–	–
13 <sup>c</sup>	7.93 (m,2H)	7.23 (m,4H)	6.63 (m,4H)	3.48 (bs,4H)	2.25–1.00 (bm,66H)	–
14 <sup>c</sup>	7.80 (m,2H)	7.15 (m,4H)	6.53 (m,4H)	3.25 (m,6H)	2.10–0.88 (bm,66H)	–
15 <sup>c</sup>	7.13	–	–	3.54–2.90 (m,4H)	2.20–0.68 (bm,66H)	–
				6.80–5.48 (m,8H)		

<sup>a</sup> Spectrum recorded in trifluoroacetic acid (TFA). <sup>b</sup> Spectrum recorded in D<sub>2</sub>O. <sup>c</sup> Spectrum recorded in CDCl<sub>3</sub>. <sup>d</sup> Insoluble, so spectrum could not be recorded. Spectrum recorded in CDCl<sub>3</sub> + DMSO-*d*<sub>6</sub>.

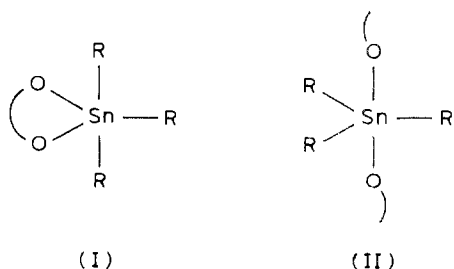
TABLE 4

 $^{119m}\text{Sn}$  MÖSSBAUER DATA (at 77 K; in  $\text{mm s}^{-1}$ )

Complex	$IS$ ( $\text{SnO}_2$ ) ( $\pm 0.03$ )	$QS$ ( $\pm 0.03$ )	Line widths ( $\pm 0.03$ )	
<b>1</b>	1.36	3.64	1.02	1.00
<b>2</b>	1.35	3.32	0.99	0.93
<b>3<sup>a</sup></b>	1.35	3.62	0.92	0.87
<b>4</b>	1.45	3.02	1.00	1.07
<b>5</b>	1.45	2.95	1.07	0.97
<b>6</b>	1.47	3.46	1.03	1.35
<b>7</b>	1.43	2.86	0.95	0.96
<b>8</b>	1.43	2.94	0.96	1.00
<b>9<sup>b</sup></b>	1.45	3.64	0.80	0.94
	1.19	2.31	0.80	0.80
<b>10</b>	1.20	2.31	1.14	0.79
<b>11</b>	1.23	2.40	1.03	0.90
<b>12</b>	1.49	2.72	0.99	0.97
<b>13</b>	1.47	2.51	0.88	0.99
<b>14</b>	1.53	3.21	0.80	1.09

<sup>a</sup> An additional component (ca. 30%) had  $IS$  0.95,  $QS$  2.15  $\text{mm s}^{-1}$ . <sup>b</sup> An additional component (ca. 30%) had  $IS$  1.2,  $QS$  2.3  $\text{mm s}^{-1}$ .

The  $^{119}\text{Sn}$  Mössbauer spectra (Table 4) are best interpreted on the basis of the quadrupole splitting ( $QS$ ) values [13]. The majority of the compounds have relatively large  $QS$  ( $> 3 \text{ mm s}^{-1}$ ), consistent with five-coordination for tin. Since these compounds are also monomeric and contain bidentate carboxylate groups, the most likely structure is I, for which the estimated  $QS$  is ca. 3.6  $\text{mm s}^{-1}$  for  $R = \text{alkyl}$  and



ca. 3.3  $\text{mm s}^{-1}$  for  $R = \text{Ph}$  [14]. Some of the observed values are rather lower, in the range normally associated with polymeric carboxylates with structure II [13,15], for which  $QS_{\text{est}}$  3.1 (alkyl), 2.9 (Ph)  $\text{mm s}^{-1}$ ; such a structure is not inconsistent with the IR data. These are also the compounds involving the larger alkyl groups. A high  $QS$  value is found for  $(\text{C}_3\text{Sn})_2\text{EDTAH}_2$  which appears to involve monodentate carboxylate coordination; there may, therefore be some interaction with the second carboxyl group.

The  $\text{Ph}_3\text{Sn}$  and  $\text{C}_3\text{Sn}$  derivatives of  $\text{EDAA}^{2-}$  and  $\text{PDAA}^{2-}$  have much lower  $QS$  values, and involve monodentate carboxyl groups. Both properties are consistent with four-coordination for tin, as found for other triorganotin carboxylates with bulky  $\alpha$ -substituents [16].

## Conclusion

The three ligands considered here bind to triorganotin residues solely through the carboxylate groups; there is no evidence of chelative involvement of the nitrogen atoms of the amine groups. The  $\text{Ph}_3\text{Sn}$  and  $\text{Cy}_3\text{Sn}$  derivatives are monomers, with monodentate binding of the carboxylate groups to tetrahedral tin. The remaining compounds involve five-coordination for tin, probably with structure I (bidentate carboxylate), although the more conventional carboxylate-bridged structure II cannot be ruled out.

## Acknowledgements

We are grateful to U.G.C., New Delhi for a Teacher Research Fellowship, to the President D.A.V. College Managing Committee, Chitergupta Road, New Delhi for study leave (S.P.V.), and to the Government of Lebanon for a Research Scholarship (L.S.M.).

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