

# Synthesis and characterization of tribenzyltin(IV) and dibenzyltin(IV) complexes of 2- $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetic acid: Crystal structure of tribenzyl $\{2$ - $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetato $\}$ tin(IV)

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Published online: 16 February 2007  
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**Abstract** Reactions of equimolar quantities of potassium 2- $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetate, with  $R_n\text{SnX}_{4-n}$  ( $R$ : benzyl- and  $n = 2$  or 3) in methanol yielded products of compositions  $\text{LHSn}(\text{PhCH}_2)_3$  and  $\text{LSn}(\text{PhCH}_2)_2$ , respectively. The complexes were characterized by microanalysis, IR, NMR ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{119}\text{Sn}$ ) and  $^{119\text{m}}\text{Sn}$  Mössbauer spectroscopy. A full characterization of the structure of the complex, tribenzyl $\{2$ - $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetato $\}$ tin(IV), was carried out by single crystal X-ray crystallography. The compound exists as centrosymmetric dimers in which two ligand molecules bridge the two tin centres. Each of the tin atoms in the dimeric unit is five coordinate in an approximately trigonal bipyramidal configuration, with carbon atoms

in the equatorial positions and oxygen atoms arranged axially.

## Keywords

2- $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetic acid · Tribenzyltin · Dibenzyltin · NMR ·  $^{119}\text{Sn}$  Mössbauer · Crystal structure

## Introduction

The synthesis of Schiff base–amino acid ligands, and their interactions with organotin(IV), have attracted much interest owing to their novel structural possibilities [1] and broad therapeutic activity [2]. Recent reports on the structures of such complexes [3–7] show that Schiff base–amino acid systems are very versatile ligands, with considerable configurational flexibility creating the possibility of a variety of coordination modes, which are well characterised. In search of new and better therapeutic agents, we have for some time studied the interactions of these compounds in vivo with Ehrlich ascites carcinoma cells [5].

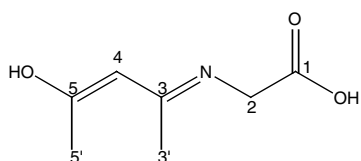
In this paper, we report the continuation of our systematic investigations with the results of an analogous study with the aim of clarifying the organotin–ligand bonding patterns in  $\text{LHSnBz}_3$  and  $\text{LSnBz}_2$  complexes. One of the complexes, tribenzyl $\{2$ - $\{[(2Z)$ -3-hydroxy-1-methyl-2-butenylidene]amino $\}$ acetato $\}$ tin(IV) has been characterized by single crystal X-ray crystallography in order to give deeper insight into the structure and bonding. The solution behaviour of the complexes is investigated using  $^{119}\text{Sn}$  NMR in a non-coordinating solvent.

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2-[(Z)-3-hydroxy-1-methyl-2-butenylidene]aminoacetic acid: (LHH')

**Fig. 1** Structure of the ligand framework and abbreviation

## Experimental

### Starting materials

The solvents used in the reactions were of AR grade and dried using standard procedures.  $(\text{PhCH}_2)_3\text{SnCl}$  and  $(\text{PhCH}_2)_2\text{SnCl}_2$  were prepared and purified as described by Sisido et al. [8]. The potassium salt (LKH) of the ligand (LHH', Fig. 1) was generated in situ by reacting the appropriate amino acids with KOH and their subsequent condensation with acetylacetone under cold conditions and followed by proper workup as described in our earlier report [7].

### Preparation of $\text{LHSn}(\text{PhCH}_2)_3$ (1)

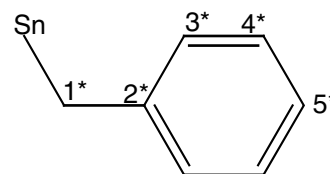
$\text{Bz}_3\text{SnCl}$  (0.61 g, 1.42 mmol) in anhydrous methanol (5.0 mL) was added dropwise with continuous stirring to a warm methanol solution (20 mL) containing LHK (0.28 g, 1.43 mmol). The reaction mixture was then refluxed for 8 h and the solvent was removed using a rotary evaporator. The residue was washed thoroughly with hexane, dried, extracted into warm benzene (20 mL) and filtered. The filtrate was concentrated up to one-fourth of its initial solvent volume, precipitated with hexane and filtered. The yellow coloured crude product was collected, dissolved in ethanol and filtered to remove any suspended particles. Slow evaporation of an ethanol solution of the product deposited shining block-shaped crystals of the product. Yield: 0.44 g (57%). Melting point: 128–129°C. Anal. Calcd. for  $\text{C}_{28}\text{H}_{31}\text{NO}_3\text{Sn}$  (MW = 548.23) (%): C, 61.34; H, 5.69; N, 2.55. Found (%): C, 61.30; H, 5.75; N, 2.59. IR ( $\text{cm}^{-1}$ ): 1650  $\nu(\text{OCO})_{\text{asym}}$ , 1600  $\nu(\text{C}=\text{N})$ , 1386  $\nu(\text{OCO})_{\text{sym}}$ , 1245  $\nu(\text{Ph}(\text{C}-\text{O}))$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ): Ligand skeleton: 10.9 (brs, 1H, H-5,  $\text{D}_2\text{O}$  exchangeable), 5.10 (s, 1H, H-4), 3.87 (d, 2H, H-2), 1.84 and 2.05 (s, 6H, H-3'/H-5'); Sn-Bz skeleton (ppm): 7.17 (m, 6H, H-3\*), 7.06 (t, 3H, H-5\*), 6.81 (m, 6H, H-4\*), 2.67 (s, 6H, H-1\*).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ): Ligand skeleton: 195.7 (C-1), 174.4 (C-3), 161.9 (C-5), 96.3 (C-4), 45.0 (C-2), 18.8 and 29.0 (C-3'/C-5'); Sn-Bz skeleton (ppm): 138.3 (C-2\*), 128.8 (C-4\*), 128.0 (C-3\*), 124.7 (C-5\*), 24.3 (C-1\*).  $^{119}\text{Sn}$  NMR ( $\text{CDCl}_3$ ): 0.82 ppm.  $^{119}\text{Sn}$

Mössbauer:  $\delta = 1.50$ ,  $\Delta = 3.27$ ,  $\Gamma_1 \pm$  and  $\Gamma_2 \pm = 0.88$  and  $0.89 \text{ mm s}^{-1}$ .

### Preparation of $\text{LHSn}(\text{PhCH}_2)_2$ (2)

This compound was prepared by reacting LHK (0.26 g, 1.34 mmol) and  $\text{Bz}_2\text{SnCl}_2$  (0.50 g, 1.34 mmol) in methanol by following the method described for 1. The product was recrystallized from benzene. Yield: (0.35 g) 58%. Melting point: 174–175 °C. Anal. Calcd for  $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{Sn}$  (MW = 456.10) (%): C, 55.30; H, 5.08; N, 3.07. Found (%): C, 55.40; H, 5.18; N, 3.01. IR ( $\text{cm}^{-1}$ ): 1635  $\nu(\text{OCO})_{\text{asym}}$ , 1587  $\nu(\text{C}=\text{N})$ , 1394  $\nu(\text{OCO})_{\text{sym}}$ , 1272  $\nu(\text{Ph}(\text{C}-\text{O}))$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ): Ligand skeleton: 4.73 (s, 1H, H-4), 3.19 (s, 2H, H-2), 1.65 and 1.91 (s, 6H, H-3'/H-5'); Sn-Bz skeleton (ppm): 7.17 (m, 4H, H-3\*), 7.08–7.02 (m, 6H, H-4\* and H5\*), 2.86 and 2.89 (AB system of prochiral methylene group, 4H, H-1\*).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ): Ligand skeleton: 185.9 (C-1), 175.7 (C-3), 172.2 (C-5), 96.4 (C-4), 50.8 (C-2), 25.1 and 26.6 (C-3'/C-5'); Sn-Bz skeleton (ppm): 137.6 (C-2\*), 128.4 (C-4\*), 128.2 (C-3\*), 124.8 (C-5\*), 30.5 (C-1\*).  $^{119}\text{Sn}$  NMR ( $\text{CDCl}_3$ ):  $-249.1$  ppm.  $^{119}\text{Sn}$  Mössbauer:  $\delta = 1.28$ ,  $\Delta = 2.89$ ,  $\Gamma_1 \pm$  and  $\Gamma_2 \pm = 0.80$  and  $1.09 \text{ mm s}^{-1}$ .

Numbering scheme for Sn-Bz skeleton is as follows:



### Physical measurements

Carbon, hydrogen and nitrogen analyses were performed with a Perkin-Elmer 2400 series II instrument. IR spectra in the range 4000–400  $\text{cm}^{-1}$  were obtained on a BOMEM DA-8 FT-IR spectrophotometer with samples investigated as KBr discs. The  $^1\text{H}$ -,  $^{13}\text{C}$ - and  $^{119}\text{Sn}$  NMR spectra were recorded on a Bruker ACF 300 spectrometer and measured at 300.13, 75.47 and 111.92 MHz, respectively. The  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{119}\text{Sn}$  chemical shifts were referred to  $\text{Me}_4\text{Si}$  set at 0.00 ppm,  $\text{CDCl}_3$  set at 77.0 ppm and tetramethyltin set at 0.00 ppm. Mössbauer spectra were recorded on solid samples at liquid nitrogen temperature using a conventional constant acceleration spectrometer, coupled with a multichannel analyser (a.e.n., Ponteranica (BG), Italy) equipped with a cryostat Cryo (RIAL, Parma, Italy). The solid samples placed in the cryostat were dried with a flow of helium followed by vacuum. A  $\text{Ca}^{119}\text{SnO}_3$  Mössbauer

**Table 1** Crystallographic data for LHSn(PhCH<sub>2</sub>)<sub>3</sub> (**1**)

Empirical formula	C <sub>28</sub> H <sub>31</sub> NO <sub>3</sub> Sn
F <sub>w</sub>	548.23
Crystal size (mm <sup>3</sup> )	0.30 × 0.68 × 0.70
Temperature (K)	93(2)
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	
<i>a</i> (Å)	26.805(3)
<i>b</i> (Å)	15.068(17)
<i>c</i> (Å)	12.948(14)
$\alpha$ (°)	90
$\beta$ (°)	99.181(2)
$\gamma$ (°)	90
<i>V</i> (Å <sup>3</sup> )	5163.0(10)
<i>Z</i>	8
<i>D</i> <sub>calcd</sub> (g cm <sup>-3</sup> )	1.411
<i>F</i> (000)	2240
$\mu$ (Mo K $\alpha$ ) (mm <sup>-1</sup> )	1.018
Transmission factors (min, max)	0.770, 0.894
$\theta$ range (°)	2.14 to 28.31
Reflections measured	19267
Independent reflection ( <i>R</i> <sub>int</sub> )	6268 (0.0229)
Reflections with <i>I</i> > 2 $\sigma$ ( <i>I</i> )	6268
reflections	
Number of parameters	303
Number of restraints	0
<i>R</i> indices [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0210 <i>wR</i> <sub>2</sub> = 0.0530
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0257 <i>wR</i> <sub>2</sub> = 0.0561
<i>GOF</i> ( <i>F</i> <sup>2</sup> )	1.06
Max, min $\Delta\rho$ (e Å <sup>-3</sup> )	0.50, -0.27

source, 10 mCi (from Ritverc, St. Petersburg, Russia), moving at room temperature with constant acceleration in a triangular waveform was used. The velocity calibration was made using a <sup>57</sup>Co Mössbauer source, 10 mCi, and an iron foil as absorber (from Ritverc, St. Petersburg, Russia).

### X-ray crystallography

The intensity data for a pale yellow block-shaped crystal of complex **1** were measured at 93 K on a Bruker SMART 1K CCD diffractometer using Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å) using the omega scan technique. Absorption corrections were made using SADABS [9]. The structures were solved by direct methods. All hydrogen atoms were given calculated positions. Neutral atom scattering factors were taken from the literature [10]. All computations were performed using the TEXSAN system of crystal structure solving programmes [11]. All refinements were performed by full-matrix least-squares on *F*<sup>2</sup>. The data were corrected for Lorentz and polarization effects.

**Table 2** Selected bond lengths (Å) and angles (°) for LHSn(PhCH<sub>2</sub>)<sub>3</sub> (**1**)

Bond length (Å)		
Sn–C(21)		2.1583(15)
Sn–C(31)		2.1610(15)
Sn–C(11)		2.1805(17)
Sn–O(2)#1		2.2166(11)
Sn–O(1)		2.3712(11)
O(1)–C(2)		1.2746(18)
O(2)–C(7)		1.2897(18)
O(2)–Sn#1		2.2166(11)
O(3)–C(7)		1.2345(19)
N–C(4)		1.3275(19)
N–C(6)		1.457(2)
N–H(1N)		0.83(2)
C(1)–C(2)		1.505(2)
C(2)–C(3)		1.413(2)
C(3)–C(4)		1.399(2)
C(4)–C(5)		1.504(2)
C(6)–C(7)		1.531(2)
Bond angles (°)		
C(21)–Sn–C(31)		131.66(6)
C(21)–Sn–C(11)		113.74(6)
C(31)–Sn–C(11)		113.71(6)
C(21)–Sn–O(2)#1		92.55(5)
C(31)–Sn–O(2)#1		92.70(5)
C(11)–Sn–O(2)#1		94.27(5)
C(21)–Sn–O(1)		82.49(5)
C(31)–Sn–O(1)		85.74(5)
C(11)–Sn–O(1)		93.81(5)
O(2)#1–Sn–O(1)		171.71(4)
C(2)–O(1)–Sn		147.76(10)
C(7)–O(2)–Sn#1		108.07(9)
C(4)–N–C(6)		127.57(14)
C(4)–N–H(1N)		116.4(14)
C(6)–N–H(1N)		115.7(14)
O(1)–C(2)–C(3)		122.33(14)
O(1)–C(2)–C(1)		118.85(14)

Note. Symmetry transformations used to generate equivalent atoms: #1,  $-x + 1/2$ ;  $-y + 1/2$ ;  $-z$ .

## Results and discussion

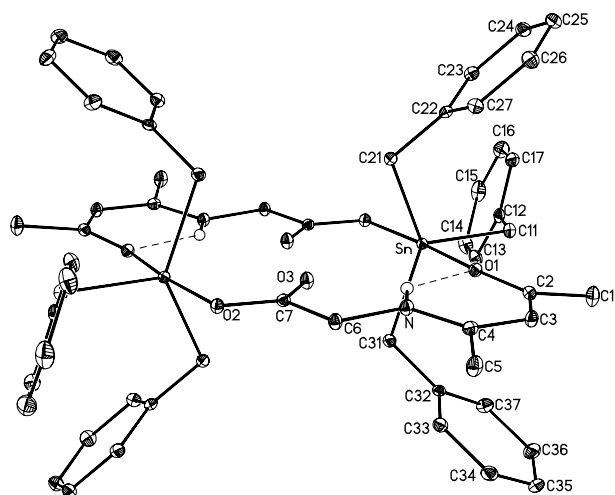
### Synthetic and spectroscopic properties

Potassium salts of the ligand, LHK with one equivalent of (PhCH<sub>2</sub>)<sub>3</sub>SnCl or (PhCH<sub>2</sub>)<sub>2</sub>SnCl<sub>2</sub> in methanol afforded the

**Table 3** Hydrogen bond parameters (Å and °) for LHSn(PhCH<sub>2</sub>)<sub>3</sub> (**1**)

D–H...A	<i>d</i> (D–H)	<i>d</i> (H...A)	<i>d</i> (D...A)	<(DHA)
N–H(1N)···O(1)	0.83(2)	2.01(2)	2.6703(18)	136.7(18)
N–H(1N)···O(3)	0.83(2)	2.16(2)	2.5890(17)	112.4(17)

Note. Symmetry transformations used to generate equivalent atoms: #1,  $-x + 1/2$ ;  $-y + 1/2$ ;  $-z$ .



**Fig. 2** Molecular structure and crystallographic numbering scheme for  $\text{LHSn}(\text{PhCH}_2)_3$  (**1**)

desired products in moderate yield by appropriate workup of the reaction mixture. The complexes **1–2** are pale yellow in colour, quite stable in air and can successfully be recrystallized from suitable organic solvents.

Compounds **1** and **2** were characterized by elemental analysis that supported the formulation of the products. The  $^1\text{H}$  and  $^{13}\text{C}$  chemical shift assignments of the benzyltin moiety and the ligand are straightforward from the multiplicity patterns and/or resonance intensities [3–7] and also by standard distortionless enhancement by polarization transfer (DEPT) experiments. The  $^1\text{H}$ - and  $^{13}\text{C}$  NMR spectra show the expected resonances and integration. The compound **1** in a non-coordinating solvent (deuteriochloroform) exhibited the  $\delta(^{119}\text{Sn})$  value as a sharp singlet at 0.82 ppm. The value found for **1** falls within the range +55.0 to –25.0 ppm specified for four-coordinate tribenzyltin species in solution [12]. This indicates that the five-coordinate dimeric structure of the solid (see X-ray discussion, *vide infra*) breaks up in solution. However, compound **2** exhibits one  $^{119}\text{Sn}$  resonance at –249.1 ppm, which is assigned to the five-coordinate tin centre, in line with the data on cognate systems [3].

The isomer shift ( $\delta$ ) values are typical for quadrivalent organotin derivatives and the full width at half maximum ( $\Gamma_{\pm}$ ) of these resonance absorptions are approximately  $0.90 \text{ mm s}^{-1}$ , further suggesting the presence of a single tin centre in the complexes [13]. The quadrupole splitting ( $\Delta$ ) values, 3.27 and  $2.89 \text{ mm s}^{-1}$  for **1** and **2** respectively, lie in the range  $2.47\text{--}4.11 \text{ mm s}^{-1}$  specified for equatorial- $\text{R}_3\text{Sn}$  trigonal bipyramidal geometry and in the range  $2.21\text{--}3.88 \text{ mm s}^{-1}$  specified for equatorial- $\text{R}_2\text{Sn}$  trigonal bipyramidal complexes [5–7, 13]. In **1**, the two oxygen atoms of the ligand occupy the apical positions, while the benzyl groups take up the equatorial plane, in excellent agreement with the X-ray structure of the complex (see later). The  $\Delta$  parameter

value of **2** indicates a trigonal bipyramidal geometry with two apical oxygen atoms, and the positions and the equatorial plane occupied by two benzyl groups and an imino nitrogen atom of the ligand [5, 13]. The C–Sn–C angle of the two benzyl groups bonded to tin was calculated applying the Parish relation and its value is  $117^\circ$  [14].

### Crystal structure

The results of the X-ray crystallographic study on compound **1** are in excellent agreement with the spectroscopic evidence presented earlier. Crystallographic data for **1** are summarized in Table 1, and selected bond lengths and bond angles are presented in Table 2. The H-bond parameters ( $\text{D}\cdots\text{H}$ ,  $\text{H}\cdots\text{A}$ ,  $\angle\text{D}\cdots\text{H}\cdots\text{A}$  and  $\text{D}\cdots\text{H}\cdots\text{A}$ ) for the fairly strong bifurcated  $\text{N}\cdots\text{H}\cdots\text{O}$  H-bonds that are important in determining the solid state structure of **1** are presented in Table 3, while Fig. 2 shows the molecular structure and the crystallographic numbering scheme.

The compound exists as centrosymmetric dimers, in which two ligand molecules bridge the two tin centres. Each of the tin atoms in the dimeric unit is five-coordinate with an approximately trigonal bipyramidal configuration. The three benzyl groups take up the equatorial positions, while the axial positions are occupied by a carboxylate oxygen from one ligand molecule O(2) and the alkoxy oxygen, O(1), of the second ligand molecule. The two O–Sn–O axes in each dimer are approximately parallel to each other. The two bridging ligand frameworks are essentially planar and lie in roughly the same plane as the two O–Sn–O axes, making the overall molecular shape flat, with protruding benzyl groups. There are no close contacts between the dimeric units. The carboxylate groups are monodentate. Each of the two ligands coordinates in the form of a zwitterion, where the alkoxy proton has migrated to the imine nitrogen, but is still hydrogen bonded to the alkoxy oxygen. The N–H bond distance is  $0.83(2) \text{ \AA}$ , while the (alkoxy) O–H distance is  $2.01(2) \text{ \AA}$ . In addition, there is also some hydrogen-bonding interaction with the free carboxylate oxygen atom of the same ligand, the hydrogen–oxygen separation being  $2.16(2) \text{ \AA}$ . The magnitudes of these hydrogen-bonding interactions are similar those reported for an analogous Schiff base complex [15]. Such proton transfer from oxygen to nitrogen atoms via hydrogen bond, and the formation of zwitterions in similar molecules to that discussed here, is quite commonly encountered [6, 16, 17] and facilitates coordination of the alkoxy or phenolic oxygens with the tin atoms.

As mentioned earlier, the environments of the two tin atoms in the dimeric units of **1** are the same. A description of the bonding pattern around one such tin atom is as follows. The sum of the carbon–tin–carbon angles in the trigonal plane of the complex is  $359.13(5)^\circ$ , very similar to those found in  $\text{Ph}_3\text{Sn}(2\text{-OHC}_6\text{H}_4\text{C}(\text{H})=\text{NCH}_2\text{COO})$  ( $359.21(14)^\circ$ )

and  $\text{Me}_3\text{Sn}(2\text{-OHC}_6\text{H}_4\text{C(H)=NCH}_2\text{COO})$  ( $359.41(17)^\circ$ ) [6]. The sums of these carbon–tin–carbon angles are also comparable to those observed in triphenyltin *N*-salicylidene-6-aminohexanoate [18] and some other similar complexes [19]. The O–Sn–O angle in **1** is  $171.71(4)^\circ$ , while the tin–oxygen distances are 2.2166(11) and 2.3712(11) Å, these being the tin–carboxylate oxygen and tin–alkoxy oxygen distances, respectively. These values are close to those reported for the corresponding bond distances in the complexes  $\text{Ph}_3\text{Sn}(2\text{-OHC}_6\text{H}_4\text{C(H)=NCH}_2\text{COO})$  (2.164(2), 2.357(2) Å) and  $\text{Me}_3\text{Sn}(2\text{-OHC}_6\text{H}_4\text{C(H)=NCH}_2\text{COO})$  (2.185(2), 2.353(2) Å), each of which have tin centres coordinated to both carboxylate and phenolic oxygen atoms [6]. The short C(7)=O(3) distance of 1.2345(19) Å in **1** indicates that the free carbonyl group on the ligand is not involved in coordination with another tin atom. Similar C=O bond lengths have also been reported for the corresponding non-bridging carbonyl groups in a number of structurally related compounds [6, 20, 21].

### Supplementary materials

Full tables of bond lengths and angles, tables of non-hydrogen and hydrogen atomic coordinates, anisotropic thermal parameters for non-hydrogen atoms have been deposited with Cambridge Crystallographic data center, CCDC No. 265648. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

**Acknowledgement** The financial support of the Department of Science & Technology, New Delhi, India (Grant No. SP/S1/F26/99, TSBB) is gratefully acknowledged. ER is indebted to the Università di Palermo, Italy for the support.

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