

# Synthesis, characterisation and X-ray structures of diorganotin(IV) and iron(III) complexes of dianionic terdentate Schiff base ligands

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## Abstract

Diorganotin(IV) complexes,  $[\text{SnR}_2\text{L}]$  (1)–(4), ( $\text{R} = \text{Me}, \text{Ph}$ ), of the terdentate Schiff bases *N*-[(2-pyrrolyl)methylidene]-*N'*-tosylbenzene-1,2-diamine ( $\text{H}_2\text{L}^1$ ) and *N*-[(2-hydroxyphenyl)methylidene]-*N'*-tosylbenzene-1,2-diamine ( $\text{H}_2\text{L}^2$ ) have been synthesised. The complexes were obtained by addition of the appropriate ligand to a methanol suspension of the corresponding diorganotin(IV) dichloride in the presence of triethylamine. However, the reaction between the precursor  $[\eta^5\text{-C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2\text{SnCl}_2$  and the Schiff bases in the presence of triethylamine gave  $[\text{Et}_3\text{NH}][\text{FeL}_2^1]$  (5) and  $[\text{Et}_3\text{NH}][\text{FeL}_2^2]$  (6), respectively. The crystal structures of the ligands and complexes have been studied by X-ray diffraction. The structure of  $[\text{SnR}_2\text{L}]$  complexes shows the tin to be five-coordinate in a distorted square pyramidal environment with the dianionic ligand acting in a terdentate manner. In 5 and 6, the iron atom is in a slightly distorted octahedral environment and is meridionally coordinated by two ligands. Spectroscopic data for the ligands and complexes (IR,  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{119}\text{Sn}$  NMR and mass spectra) are discussed and related to the structural information.

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**Keywords:** Organotin(IV) complexes; Iron(III) complexes; Schiff base ligands; Amide ligand; Crystal structure;  $^{119}\text{Sn}$  NMR

## 1. Introduction

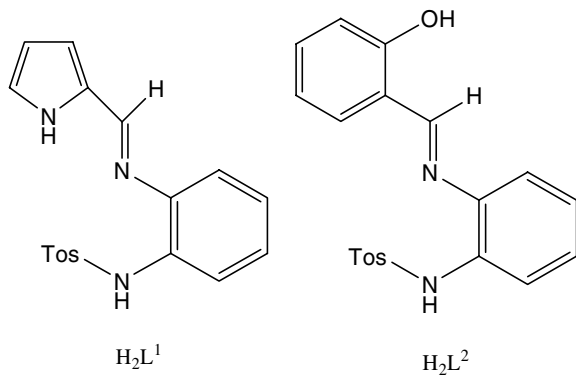
Metal complexes of Schiff base ligands have played an important role since the early days of Coordination Chemistry, not only from an inorganic point of view but also because of the possible biological interest in such compounds [1]. A great deal of work has been performed on the synthesis and characterisation of transition metal compounds with these ligands. More recently, organotin complexes with Schiff base ligands have been studied, not only due to their novel structural features caused by the multidenticity of these ligands but also in view of their pharmacological and antitumour activity [2]. Several

reviews dealing with the synthesis, structural behaviour and biological applications of these materials have been published [3]. In particular, complexes with Schiff bases derived from amino acids [4–6] and ONO and NNO dianionic terdentate Schiff bases have been widely reported [6–12]. Recently, the synthesis and toxicological activity of organotin(IV) compounds with a sulfonamide imine ligand have been also described, although crystal structures for these compounds were not reported [13].

As a continuation of our previous work dealing with the study of the interaction of tin and diorganotin(IV) with Schiff bases, we report here the synthesis and characterisation of new dimethyl-, diphenyl- and di[(cyclopentadienyldicarbonyl)iron]tin(IV) complexes with *N*-[(2-pyrrolyl)methylidene]-*N'*-tosylbenzene-1,2-diamine ( $\text{H}_2\text{L}^1$ ) and *N*-[(2-hydroxyphenyl)methylidene]-*N'*-tosylbenzene-1,2-diamine ( $\text{H}_2\text{L}^2$ ). These ligands contain, in addition to a pyrrole N–H or phenolic O–H group, respectively, a

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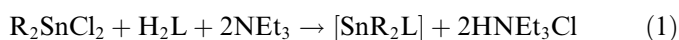
Fig. 1.  $H_2L^1$  and  $H_2L^2$ .

sulfonamide N–H group (Fig. 1). The nature of the groups bonded to tin was chosen in an effort to assess whether the steric hindrance produced by them influences the structure of the complexes formed.

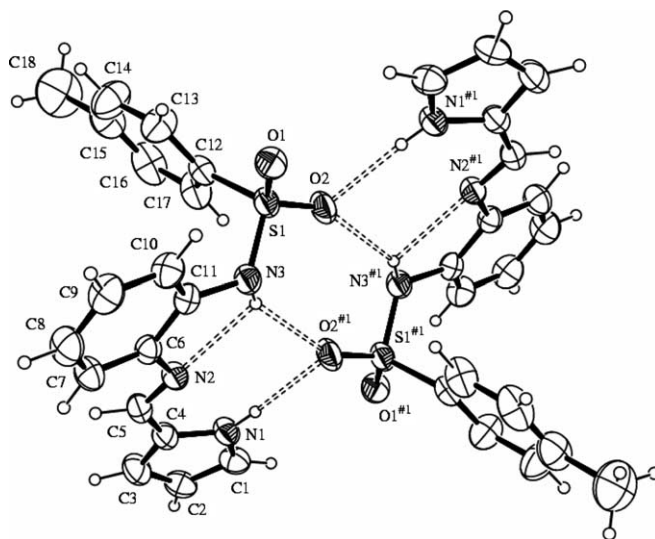
## 2. Results and discussion

### 2.1. Synthesis

Room temperature reaction between  $SnR_2Cl_2$  ( $R = Me, Ph$ ) and an equimolar amount of the Schiff base ligand,  $[H_2L]$ , in methanol in the presence of triethylamine, Eq. (1), led to yellow crystalline compounds 1–4. The analytical data for these complexes are consistent with the formula  $[SnR_2L]$ .



These compounds are stable to air and moderately soluble in methanol and chlorinated solvents. Crystallisation from the mother liquor afforded crystals of 1–4 that were suitable for X-ray studies.

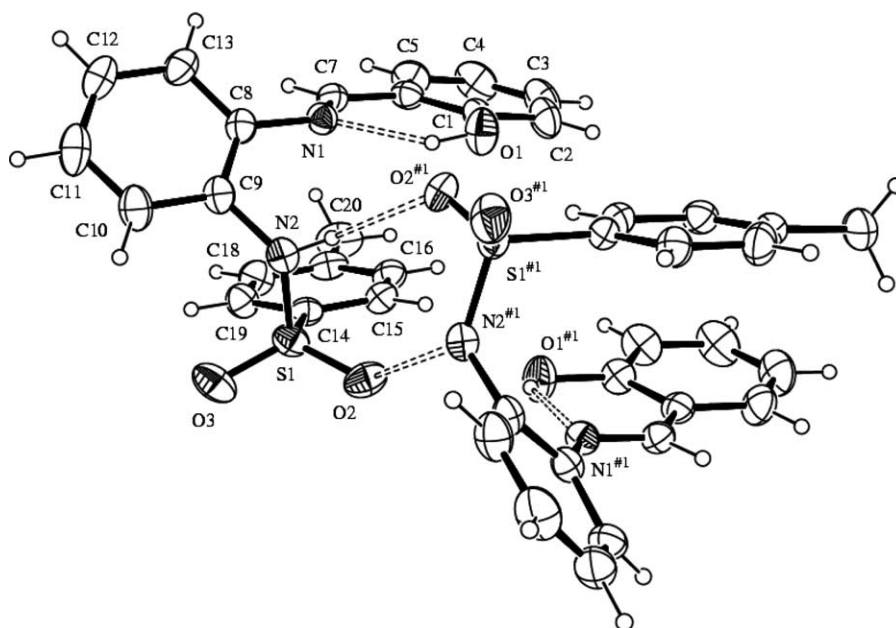
Fig. 2. The molecular structure of  $H_2L^1$ , showing the intra- and intermolecular hydrogen bonding.

In the case of  $[\eta^5-C_5H_5Fe(CO)_2]_2SnCl_2$ , the reaction produced crystalline solids of composition  $[Et_3NH][FeL^1_2]$  (5) and  $[Et_3NH][FeL^2_2]$  (6). The structures of both of these compounds show an iron atom octahedrally coordinated by two dianionic terdentate ligands in a meridional way (vide infra).

### 2.2. Molecular structures of ( $H_2L^1$ ) and ( $H_2L^2$ )

ORTEP views of  $[H_2L^1]$  and  $[H_2L^2]$  are shown in Figs. 2 and 3 along with the atom-labelling scheme. Selected bond lengths and angles, with the estimated deviations, are given in Tables 1 and 2.

The structural parameters for both ligands are as expected, with a relatively short bond distance for the

Fig. 3. The molecular structure of  $H_2L^2$ , showing the intra- and intermolecular hydrogen bonding.

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