



A discrete unsymmetrically substituted dihydrodioxadistannetane with both η^1 and intramolecular $\eta^2\mu_2$ sulfonate bondings

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ABSTRACT

Hydrolysis of the aryltripropynyltin (**3**) in the presence of an arylsulfonic acid resulted in the formation of a new tetrasulfonato 1,3-dihydro-1,3-dioxa-2,4-diaryl-2,4-distannetane (**4**). Its crystal structure revealed that the molecule is the smallest discrete tin sulfonate cluster reported up to now with absence of intermolecular interactions. Molecule **4** also exhibits unusual η^1 and intramolecular $\eta^2\mu_2$ bonding modes for the sulfonate groups. Moreover, while only symmetrical 1,3-dihydro-1,3-dioxa-distannetanes have been described so far, the tin atoms in **4** are differently substituted.

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1. Introduction

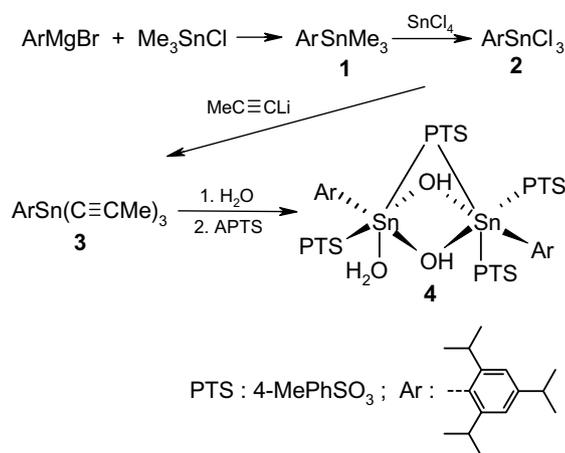
Four-membered cyclic 1,3-dihydro-1,3-dioxa-2,4-distannetanes that contain the $[\text{Sn}_2(\mu\text{-OH})_2]$ core were first described in a mixed chloro-hydroxide inorganic tin compound [1]. Then, a few examples where the tin atoms are substituted by one [2–8] or two [9–18] organic groups have been structurally characterized. They belong to a class of organotins that are of importance due to their structural properties as well as for their useful applications in the field of catalysis. They have been successfully used in acetylation [17], deacetylation reactions [19] and for the production of diphenyl carbonate [15]. They have also been involved in various carbon bond forming reactions such as Mukaiyama-aldol as well as Mukaiyama–Michael reactions and allylations of aldehydes [20]. In some cases, [10,12,17,18] where the tin atoms bear two organic groups, they have been isolated as dications that are dissociated in solution. Various anionic substituents of the tin atoms are found in the 1,3-dihydro-1,3-dioxa-2,4-distannetanes: chloro, nitro, η^1 and $\eta^2\mu_2$ carboxylato, η^1 and $\eta^2\mu_2$ phosphinato and η^1 and $\eta^2\mu_2$ sulfonato groups. The $\eta^2\mu_2$ bonded ligands can be bridging either intra- or intermolecularly. None of the 1,3-dihydro-1,3-dioxa-2,4-distannetanes described up to now are monomeric in the solid state. They form large supramolecular networks where the molecules are linked in two ways. Either bidentate ligands bridge two metal atoms of vicinal molecules,

or hydrogen bonds between the cyclic hydroxy groups (or a coordinated water) and the heteroatoms of the ionic substituents link the molecules. 1,3-Dihydro-1,3-dioxa-2,4-distannetanes are usually prepared in two different ways, either by hydrolysis of tin halides or by condensation between a tin oxide or hydroxide and an acid, often under Dean–Stark conditions. During the course of our studies concerning the hydrolysis of organotrialkynyltins [21–25] and bridged organohexaalkynyliditins [26–29] we showed that the use of a bulky substituent on the tin, such as the 2,4,6-triisopropylphenyl group, could allow the isolation of clusters of monoorganotin oxides–hydroxides of various sizes and shapes. When hydrolysis was conducted in the presence of an aromatic sulfonic acid, cationic polysulfonates with three or ten tin atoms and two types of sulfonate bondings, an electrostatic and a bridging one, were isolated [22]. We report here that, under different conditions, hydrolysis of a bulky organotrialkynyltin in the presence of an aromatic sulfonic acid leads to a very unusual 1,3-dihydro-1,3-dioxa-2,4-diaryl-2,4-distannetane (**4**) with an unsymmetrical substitution of the tin atoms and two bonding modes of the sulfonate groups, an η^1 and an intramolecular $\eta^2\mu_2$. Compound **4** appears to be the first instance of a 1,3-dihydro-1,3-dioxa-2,4-organo-2,4-distannetane with one aromatic substituent on each tin atom (see Scheme 1).

2. Results and discussion

2,4,6-Triisopropylphenyl tripropynyltin (**3**) was first prepared from the corresponding trichloride **2**, itself obtained from **1** by a

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Scheme 1. Synthesis of **4**.

method [28] already used for lipophilic organotin trichlorides. It involves the reaction of an aryltrimethyltin with an excess of tin tetrachloride followed by the distillation of the volatile methyltin chlorides formed. Subsequent alkylation with propynyllithium at low temperature introduced the propynyl groups on the tin atoms and provided crystalline **3** in a good yield. Then treatment of **3** with 12 equiv. of water in THF at reflux for 8 days was followed by the addition of 1 equivalent of *p*-toluenesulfonic acid. A further reflux for 6 h followed by a slow cooling gave crystalline **4** in a 32% yield.

¹H NMR of a chloroform solution of **4** was not very informative, showing only the presence of unequivalent *p*-toluenesulfonyl and 2,4,6-triisopropylphenyl groups and of THF. In ¹¹⁹Sn NMR, the presence of two peaks of about equal intensity at –644.7 and –676.2 indicated the presence of two unequivalent six-coordinated monoorganotins. However, molecule **4** is unstable in solution. It undergoes slow decomposition resulting in three sets of resonances at –643, –652 and –676 ppm in ¹¹⁹Sn NMR after 60 h. The tin signals were not observed after 25 days. This is probably due to the formation of large and ill-defined stannic oxopolymeric species which are known to be NMR silent [30].

The molecular structure of **4** was unambiguously established by X-ray diffraction analysis. Colorless crystals of **4** were obtained from a toluene mixture at 10 °C. Crystallographic data for the structural analyses of compound **4** are shown in Table 1 and main bond parameters are listed in Fig. 1. The molecule is organized around a central cyclic four-membered dihydrodioxadistannetane core [Sn₂(μ-OH)₂] linked with four sulfonate fragments. Three sulfonates (S1, S2 and S4) are linked with tin centers in monodentate fashion and the fourth (S3) exhibits a bridging mode of coordination. The central cycle is not planar, as in the other examples described up to now, and shows neither an inversion center nor a symmetry plane as in similar rings. It is puckered, with one of the tin atoms about 0.4 Å apart from the plane passing through the other tin atom and the oxygen atoms. Bond lengths and angles of the cycle are in the range of the previously reported values. The tin atoms that are six-coordinated and show a distorted octahedral geometry are situated in different coordination environments. Sn1 is linked to one aryl and to two sulfonate groups in a monodentate coordination mode. The aryl group and one of the sulfonate groups (S4) are situated in the plane of the cycle while the other sulfonate group (S2) is perpendicular to it. Sn2 is surrounded by an aryl group and a sulfonate group (S1) in a monodentate coordination mode and by a molecule of water, that occupies an axial position. The tin–oxygen (water) distance, 2.120 Å, is rather short when compared to dialkyltetrachlorodihydrodioxadistannetane [1,2,4]

Table 1
Crystal data and structure refinements for **4**

Compound	4
Empirical formula	C ₆₄ H ₉₀ O _{16.50} S ₄ Sn ₂
Formula weight	1484.95
Crystal system	Triclinic
Space group	P1̄ (2)
<i>a</i> (Å)	12.8613(2)
<i>b</i> (Å)	15.3578(3)
<i>c</i> (Å)	21.0761(4)
α (°)	69.369(1)
β (°)	73.908(1)
γ (°)	68.178(1)
<i>V</i> (Å ³)	3565.25(11)
<i>Z</i>	2
<i>D</i> _{calc} (Mg/m ³)	1.383
Reflections collected	32 429
Independent reflections	16 266
Data/restraints/parameters	12 641/10/779
<i>R</i> _{int}	0.0378
<i>R</i> ₁ (<i>I</i> > 2σ(<i>I</i>))	0.060
<i>wR</i> ₂	0.1700

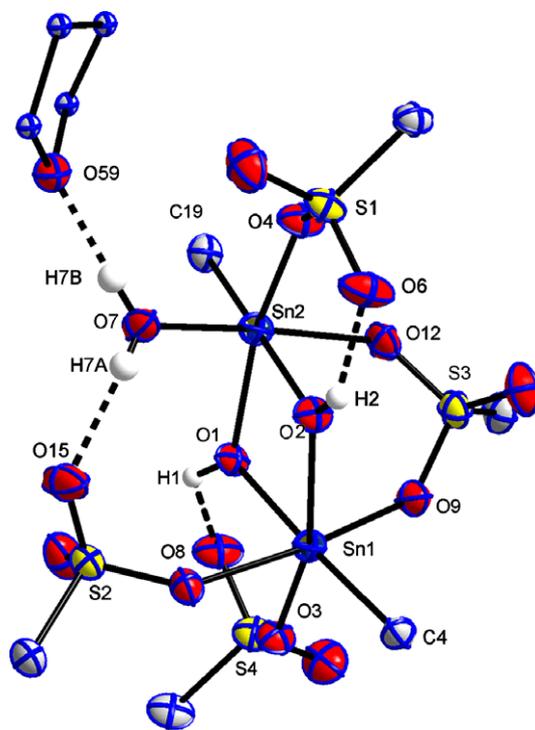


Fig. 1. Molecular structure of **4** with thermal ellipsoids at the 50% probability level. Aryl groups are omitted for clarity. Selected distances [Å]: Sn1–O1 2.057(4), Sn1–O2 2.112(4), Sn1–O8 2.103(4), Sn1–O3 2.113(4), Sn1–O9 2.159(4), Sn1–C4 2.158(6), Sn2–O1 2.078(4), Sn2–O2 2.065(4), Sn2–O4 2.098(4), Sn2–O7 2.120(5), Sn2–O12 2.169(4), Sn2–C19 2.129(6). Selected angles [°]: O1–Sn1–O8 86.25(16), O1–Sn1–O2 69.99(15), O8–Sn1–O2 95.51(16), O1–Sn1–O3 84.46(16), O8–Sn1–O3 85.39(17), O2–Sn1–O3 154.29(16), O1–Sn1–C4 176.85(19), O8–Sn1–C4 95.5(2), O2–Sn1–C4 107.18(19), O3–Sn1–C4 98.3(2), O1–Sn1–O9 83.10(16), O8–Sn1–O9 168.04(17), O2–Sn1–O9 85.95(16), O3–Sn1–O9 88.18(17), C4–Sn1–O9 95.4(2), O2–Sn2–O1 70.52(15), O2–Sn2–O4 86.78(16), O1–Sn2–O4 157.18(16), O2–Sn2–O7 85.11(18), O1–Sn2–O7 92.36(18), O4–Sn2–O7 83.19(19), O2–Sn2–C19 174.3(2), O1–Sn2–C19 105.22(19), O4–Sn2–C19 97.6(2), O7–Sn2–C19 99.1(2), O2–Sn2–O12 84.46(17), O1–Sn2–O12 87.32(16), O4–Sn2–O12 92.87(18), O7–Sn2–O12 169.04(19).

(2.243–2.346 Å depending on the alkyl group) or to neutral or cationic tetraalkylditrihydrodioxadistannetane [10,15] (2.364–2.409 Å). It is in the range of tin–water distances in inorganic tin chloride hydrates [31,32] (SnCl₄(H₂O)₂: 2.10 Å; SnCl₃(OH)(H₂O)₂: 2.06 Å). It is characteristic of the high electrophilic character of the tin conferred by the two electroattracting arylsulfonato groups.

The η^1 bonding of the simply bonded sulfonate groups (S1, S2 and S4) can be easily recognized by the different bond lengths of sulfur–oxygen bonds, 1.505 Å (mean) for the sulfur–oxygen(tin) bonds, and 1.439 Å (mean) for the sulfur–oxygen double bonds. Both tin atoms are also linked by two oxygen atoms of a sulfonate group (S3) in a bidentate coordination mode. The intramolecular bridging $\eta^2\mu_2$ character is characterized by two almost identical tin–oxygen bonds (2.169 and 2.158 Å) and sulfur–oxygen(tin) bonds (1.494 and 1.505) as well as by a shorter sulfur oxygen double bond (1.427 Å). The presence of monodentate or bridging (intermolecular) bidentate sulfonate groups in organotin sulfonates has been already reported, but never in the same molecule. So, to the best of our knowledge, the simultaneous existence of a monodentate mode and an intramolecular bridging bidentate binding mode in an organotin sulfonate is reported here for the first time. Another noteworthy feature is the presence of hydrogen bonds. The hydrogen atoms of the hydroxide groups are intramolecularly linked to oxygen atoms of equatorial sulfonate groups (S1 and S4) through normal hydrogen bonding (1.852 and 1.969 Å) forming two six-membered rings. One hydrogen atom of the coordinated water molecule is also bonded to the oxygen atom of an axial sulfonate group (S2) (1.950 Å). The other hydrogen atom of the coordinated water molecule ties the tetrahydrofuran solvate to the tin core through an hydrogen bond (1.771 Å).

The monomeric molecules of **4** associated to THF are well separated in space, unlike other chloro- or sulfonatodihydrodioxadistannetanes. The last compounds form coordination polymer networks that are built through coordination bonds between sulfonate groups and tin atoms [11,13,15] or hydrogen bonds between coordinated water and/or hydroxide and ligands [2–5,10,18]. In **4**, it is likely that the bulky organic ligands preclude close contacts from one molecule to another. When compared to other anions as carbonates, phosphates or phosphinates, this example shows that the weak aptitude of organosulfonates to build supramolecular networks by cooperative coordination or weaker intermolecular interactions [33,34] can even be suppressed.

In summary we have shown that a new tetrasulfonato-1,3-dihydro-1,3-dioxa-2,4-diaryl-2,4-distannetane **4** was obtained as the smallest discrete molecule from the hydrolysis of a bulky aryltripropynyltin in the presence of *p*-toluenesulfonic acid. Its crystal structure revealed several unusual features such as the unsymmetrical substitution of tin atoms and the η^1 and intramolecular $\eta^2\mu_2$ linkages for the sulfonate groups.

3. Experimental

3.1. General remarks

All reactions were carried out under a nitrogen atmosphere. THF and toluene were distilled from sodium-benzophenone ketyl prior to use. Tin tetrachloride was distilled before use. ^1H , ^{13}C , and ^{119}Sn NMR spectra were recorded on a Bruker AC 200, AC 250, or DPX300 spectrometer (solvent CDCl_3). Tin–hydrogen and tin–carbon coupling constants are given in square brackets. Elemental analyses were performed by the “Service d’Analyse du CNRS” at Vernaison, France.

3.2. Synthesis of **3**

An equimolar solution of arylmagnesium bromide (10 mmol) in THF (60 mL) was treated with Me_3SnCl (1.9 g, 10 mmol) in THF (75 mL) at room temperature, stirred for 12 h and the reaction mixture was quenched with a saturated solution of NH_4Cl , followed by extraction with hexane (200 mL). Evaporation of the solvents gave 3.4 g of **1** as a white solid (93%). Mp: 86 °C.

^1H NMR: δ 0.27 (s, 9H, [47], $\text{Sn}(\text{CH}_3)_3$), 1.16 (d, 6H, *p*- $\text{CH}(\text{CH}_3)_2$), 1.21 (d, 12H, *o*- $\text{CH}(\text{CH}_3)_2$), 2.82 (sept, 1H, *p*- $\text{CH}(\text{CH}_3)_2$), 2.91 (sept, 2H, *o*- $\text{CH}(\text{CH}_3)_2$), 6.98 (s, 2H, [28], ArH). ^{13}C NMR (CDCl_3 , 62.9): δ –4.0 ($\text{Sn}(\text{CH}_3)_3$), 24.5 (*p*- $\text{CH}(\text{CH}_3)_2$), 24.7 (*o*- $\text{CH}(\text{CH}_3)_2$), 34.7 (*p*- $\text{CH}(\text{CH}_3)_2$), 37.0 [48] (*o*- $\text{CH}(\text{CH}_3)_2$), 121.5 [42] (C(Ar)H), 138.34 (C(Ar)Sn), 149.8 (C(Ar)*p*- $\text{CH}(\text{CH}_3)_2$), 156.2 [35] (C(Ar)*o*- $\text{CH}(\text{CH}_3)_2$). ^{119}Sn NMR: δ –62.4. A 3:1 mixture of SnCl_4 (3.9 g, 15 mmol) and **1** (1.7 g, 5 mmol) was heated in a Schlenk tube at 120 °C for 5 h. Unreacted SnCl_4 and MeSnCl_3 by-product formed in the reaction mixture were removed by vacuum distillation (50–80 °C/0.01 mmHg). The resulted black solid was purified by vacuum distillation to give **2** as a colorless crystalline solid (130 °C/ 1×10^{-4} mmHg) (2 g, 92%). Mp: 113–115 °C; ^1H NMR: δ 1.17 (d, 6H, *p*- $\text{CH}(\text{CH}_3)_2$), 1.25 (d, 12H, *o*- $\text{CH}(\text{CH}_3)_2$), 2.83 (sept, 1H, *p*- $\text{CH}(\text{CH}_3)_2$), 3.11 (sept, 2H, *o*- $\text{CH}(\text{CH}_3)_2$), 7.15 (s, 2H, [65], ArH). ^{13}C NMR (CDCl_3 , 62.9): δ 24.2 (*p*- $\text{CH}(\text{CH}_3)_2$), 25.2 (*o*- $\text{CH}(\text{CH}_3)_2$), 34.8 (*p*- $\text{CH}(\text{CH}_3)_2$), 38.6 [55] (*o*- $\text{CH}(\text{CH}_3)_2$), 124.3 [127] (C(Ar)H), 135.7 (C(Ar)Sn), 154.9 (C(Ar)*p*- $\text{CH}(\text{CH}_3)_2$), 150.0 [23] (C(Ar)*o*- $\text{CH}(\text{CH}_3)_2$). ^{119}Sn NMR: δ –93.5 ppm. An excess of propyne (25 mmol) was condensed in toluene (50 mL) at –196 °C. At –90 °C, *n*-BuLi (4 mmol) in toluene (40 mL) was added dropwise to the above solution. Then the reaction mixture was slowly brought to room temperature and stirred for 15 min. Again, the reaction mixture was cooled to –90 °C and a solution of **2** (1.7 g, 4 mmol) in toluene (50 mL) was added dropwise. The reaction mixture was slowly brought to RT and stirred for 12 h. Insolubles were filtered through a MgSO_4 bedded G3 frit and removal of volatiles from the filtrate resulted in **3** in an excellent yield. (1.6 g, 90%). Mp: 129–131 °C. ^1H NMR: δ 1.17 (d, 6H, *p*- $\text{CH}(\text{CH}_3)_2$), 1.22 (d, 12H, *o*- $\text{CH}(\text{CH}_3)_2$), 1.83 (s, 9H [15], $\text{C}\equiv\text{CCH}_3$), 2.79 (sept, 1H, *p*- $\text{CH}(\text{CH}_3)_2$), 3.37 (sept, 2H, *o*- $\text{CH}(\text{CH}_3)_2$), 6.97 (s, 2H, [31], ArH). ^{13}C NMR (CDCl_3 , 62.9): δ 5.0 ($\text{C}\equiv\text{CCH}_3$), 23.8 (*p*- $\text{CH}(\text{CH}_3)_2$), 24.8 (*o*- $\text{CH}(\text{CH}_3)_2$), 34.2 (*p*- $\text{CH}(\text{CH}_3)_2$), 36.4 [48] (*o*- $\text{CH}(\text{CH}_3)_2$), 79.3 [850] ($\text{C}\equiv\text{CCH}_3$), 106.4 [176] ($\text{C}\equiv\text{CCH}_3$), 121.9 [72] (C(Ar)H), 131.4 (C(Ar)Sn), 151.0 (C(Ar)*p*- $\text{CH}(\text{CH}_3)_2$), 155.6 [52] (C(Ar)*o*- $\text{CH}(\text{CH}_3)_2$). ^{119}Sn NMR: δ –335.4 ppm. IR: 3043w, 2962s, 2930s, 2881s, 2169vs, 1589w, 1560w, 1463 m, 1423w, 1384w, 1362w, 1266w, 1101w, 996vs, 886w, 802w, 746w, 650w, 513w cm^{-1} . Anal. Calc. for $\text{C}_{24}\text{H}_{32}\text{Sn}$: C, 65.63; H, 7.34; Sn, 27.03. Found: C, 65.25; H, 7.56; Sn, 26.59%.

3.3. Synthesis of **4**

To a stirred solution of **3** (1.8 g, 4 mmol) in THF (30 mL), H_2O (0.9 g, 49.2 mmol) was added dropwise at RT. The reaction mixture was heated under reflux for 8 days and *p*-toluenesulfonic acid monohydrate (0.8 g, 4 mmol) was added at once at RT. The resulting solution was then heated under reflux for 6 h. It was dried under reduced pressure to give a white solid, which was dissolved in THF (2 mL) and filtered to obtain colorless crystals of **4** at 10 °C. (0.98 g, 32%). Mp > 180 °C (dec). ^1H NMR (CDCl_3 , 250 MHz): δ 1.12 (m, 36 H, *p*- $\text{CH}(\text{CH}_3)_2$ and *o*- $\text{CH}(\text{CH}_3)_2$), 1.74 (m, 8H, THF), 2.27–2.28 (m, 12H, O_3SArCH_3), 2.65–2.84 (m, 6H, *p*- $\text{CH}(\text{CH}_3)_2$ and *o*- $\text{CH}(\text{CH}_3)_2$), 3.63 (m, 8H, THF), 6.81–7.84 (m, 20H, ArH). ^{119}Sn NMR (CDCl_3 , 74.6 MHz): δ –644.2, –676.2 ppm. IR(KBr): ν = 3403 s, 3207s, 3441s, 2961vs, 2864s, 1594w, 1563w, 1498w, 1464s, 1420m, 1385 m, 1364m, 1279s, 1153s, 1101vs, 1027s, 986vs, 877m, 838w, 814s, 788m, 742w, 708w, 680s, 568s cm^{-1} . Anal. Calc. for $\text{C}_{58}\text{H}_{78}\text{O}_{15}\text{S}_4\text{Sn}_2$, 1.5 THF (1489.1): C, 51.62; H, 6.09; S, 8.61; Sn, 15.94. Found: C, 51.21; H, 5.44; S, 8.23; Sn, 15.02%.

3.4. Structure determination for **4**

Crystal data collection and processing parameters are given in Table 1. A suitable crystal was covered with mineral oil (Aldrich), mounted onto glass fiber, and transferred directly to the 150-K

N₂ stream of a Nonius Kappa CCD diffractometer. The structure was solved by Direct Methods (SHELXS97) [35] and refined by full-matrix least squares against F^2 using SHELXS97 [36]. All non-H atoms were refined anisotropically with the exception of disordered atoms. H-atoms were fixed in calculated positions at parent C atoms. The locations of the largest peaks in the final difference Fourier map calculation as well as the magnitude of the residual electron densities in each case were of no chemical significance.

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Appendix A. Supplementary material

CCDC 691514 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.07.036.

References

- [1] N.G. Bokii, Y.T. Struchkov, Zh. Strukt. Khim. 12 (1971) 253.
- [2] C. Lecomte, J. Protas, M. Devaud, Acta Cryst. B32 (1976) 923.
- [3] R.R. Holmes, S. Shafieezad, V. Chandrasekhar, J.M. Holmes, R.O. Day, J. Am. Chem. Soc. 110 (1988) 1174.
- [4] H. Puff, H. Reuter, J. Organomet. Chem. 364 (1989) 57.
- [5] B. Zobel, A. Duthie, D. Dakternieks, E.R.T. Tiekink, Organometallics 20 (2001) 2820.
- [6] R.R. Holmes, K.C. Kumara Swamy, C.G. Schmid, R.O. Day, J. Am. Chem. Soc. 110 (1988) 7060.
- [7] K.C. Kumara Swamy, R.O. Day, R.R. Holmes, Inorg. Chem. 31 (1992) 4184.
- [8] V. Chandrasekhar, P. Thilagar, J.F. Bickley, A. Steiner, J. Am. Chem. Soc. 127 (2005) 11556.
- [9] A.M. Domingos, G.M. Sheldrick, J. Chem. Soc. Dalton Trans. (1974) 475.
- [10] K. Sakamoto, Y. Hamada, H. Akashi, A. Orita, J. Otera, Organometallics 18 (1999) 3555.
- [11] S.P. Narula, S. Kaur, R. Shankar, S. Verma, P. Venugopalan, S.K. Sharma, R.K. Chadha, Inorg. Chem. 38 (1999) 4777.
- [12] K. Sakamoto, H. Ikeda, H. Akashi, T. Fukuyama, A. Orita, J. Otera, Organometallics 19 (2000) 3242.
- [13] R. Kapoor, A. Gupta, P. Kapoor, P. Venugopalan, J. Organomet. Chem. 619 (2001) 157.
- [14] V. Chandrasekhar, S. Nagendran, K. Gopal, A. Steiner, S. Zacchini, Chem. Commun. (2003) 862.
- [15] H. Lee, L.Y. Bae, O.S. Kwon, S.J. Kim, S.D. Lee, H.S. Kim, J. Organomet. Chem. 689 (2004) 1816.
- [16] V. Chandrasekhar, P. Thilagar, A. Steiner, J.F. Bickley, Chem. Eur. J. 12 (2006) 8847.
- [17] B. Kasna, L. Dostal, I. Cisarova, R. Jambor, Organometallics 26 (2007) 4080.
- [18] V. Chandrasekhar, P. Singh, K. Gopal, Organometallics 26 (2007) 2833.
- [19] A. Orita, Y. Hamada, T. Nakano, S. Toyoshima, J. Otera, Chem. Eur. J. 7 (2001) 3321.
- [20] D.L. An, Z. Peng, A. Orita, A. Kurita, S. Man-e, K. Ohkubo, X. Li, S. Fukuzumi, J. Otera, Chem. Eur. J. 12 (2006) 1642.
- [21] G. Prabusankar, B. Jousseau, T. Toupance, H. Allouchi, Angew. Chem. Int. Ed. 45 (2006) 1255.
- [22] G. Prabusankar, B. Jousseau, T. Toupance, H. Allouchi, Dalton Trans. (2007) 3121.
- [23] G. Vilaça, B. Jousseau, C. Mahieux, T. Toupance, C. Belin, H. Cachet, M.-C. Bernard, V. Vivier, Adv. Mater. 18 (2006) 1073.
- [24] S. Boutet, B. Jousseau, M. Biesemans, R. Willem, C. Labrugère, L. Delattre, Chem. Mater. 17 (2005) 1803.
- [25] M.-L. Dumartin, H. Elhamzaoui, B. Jousseau, M.-C. Rasclé, T. Toupance, H. Allouchi, Organometallics 26 (2007) 5576.
- [26] H. Elhamzaoui, B. Jousseau, H. Riague, T. Toupance, P. Dieudonné, C. Zakri, M. Maugey, H. Allouchi, J. Am. Chem. Soc. 126 (2004) 8130.
- [27] H. Elhamzaoui, B. Jousseau, T. Toupance, C. Zakri, M. Biesemans, R. Willem, H. Allouchi, Chem. Commun. (2006) 1304.
- [28] H. Elhamzaoui, B. Jousseau, T. Toupance, H. Allouchi, Organometallics 26 (2007) 3908.
- [29] H. Elhamzaoui, T. Toupance, M. Maugey, C. Zakri, B. Jousseau, Langmuir 23 (2007) 785.
- [30] C. Eychenne-Baron, F. Ribot, C. Sanchez, J. Organomet. Chem. 567 (1998) 137.
- [31] J.C. Barnes, H.A. Sampson, T.J.R. Weakley, Dalton Trans. (1980) 943.
- [32] N.W. Isaacs, C.H.L. Kennard, J. Chem. Soc. (1970) 1257.
- [33] A.P. Cote, G.K.H. Shimizu, Coord. Chem. Rev. 245 (2003) 49.
- [34] J. Cai, Coord. Chem. Rev. 248 (2004) 1061.
- [35] G.M. Sheldrick, Acta Crystallogr. A 46 (1990) 467.
- [36] G.M. Sheldrick, SHELXL-97, Program for Crystal Structure Refinement, Universität von Göttingen, Göttingen, Germany, 1997.