Short Communication

Jonathan O. Bauer*

The crystal structure of the first ether solvate of hexaphenyldistannane $[(\text{Ph}_3\text{Sn})_2 \cdot 2 \text{THF}]$

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Abstract: Structural investigations of molecular crystal solvates can provide important information for the targeted crystallization of particular inclusion compounds. Here, the crystal structure of the first ether solvate of hexaphenyldistannane $[(\text{Ph}_3\text{Sn})_2 \cdot 2 \text{THF}]$ is reported. Structural features in terms of host-guest interactions and in the context of the previously reported polymorphs and solvates of (Ph$_3$Sn)$_2$ are discussed.

Keywords: crystalline solvates; inclusion compounds; stannanes; tin; weak interactions; X-ray crystallography

Molecules in the solid state can arrange in more than just one crystal packing (polymorphs) and can crystallize by incorporating stoichiometric or non-stoichiometric amounts of solvent molecules in the crystal lattice to form inclusion compounds (solvates) (Bernstein, 2002; Lee et al., 2011). Usually, even the inclusion of small solvent molecules into a molecular crystal network leads to a significant change of the crystal structure (Atwood et al., 2017). Polymorphism and solvatomorphism are structural phenomena in solid-state chemistry that can substantially affect the physicochemical properties of a crystalline molecular compound (Brittain, 2012a; Nassimbeni, 2003) and provide valuable information for crystal engineering (Desiraju, 1995). Therefore, nonsolvated polymorphs and solvates of molecular crystals have gained great importance in pharmaceutical science (Brittain, 2012b; Shan and Zaworotko, 2008). Different crystal structures and crystalline solvates of the same compound can for example influence the stability, solubility, and dissolution rate of an active pharmaceutical ingredient so that clinically relevant effects on bioavailability may result (Rexrode et al., 2019). In general, crystalline solvates can provide valuable information on the correlation between crystal structure, molecular structure, and molecule-solvent interaction. The noncovalent intermolecular interactions that determine the structure of molecular crystalline systems can vary widely in type and strength (Steed and Atwood, 2009) and range from strong ionic interactions (100-350 kJ mol$^{-1}$) via hydrogen bonds (20-40 kJ mol$^{-1}$), C–H⋯O and O–H⋯π interactions (2-20 kJ mol$^{-1}$) through to weak nondirectional interactions like C⋯H in aromatic compounds (240 kJ mol$^{-1}$) (Desiraju, 1995), which are often summarized as van der Waals forces (< 5 kJ mol$^{-1}$) (Nassimbeni, 2003). Although hydrogen bonding ranging from weak to strong interactions is considered to be the most important structure-forming principle in supramolecular crystal construction (Desiraju, 2002; Steiner, 2002), there are also intriguing examples of highly stable supramolecular systems that are held together only by weak dispersive interactions and used for the interstitial inclusion of highly volatile gases like methane (Atwood et al., 2002).

In the course of our studies on the structural behaviour of triphenyl-substituted group 14 elements (Bauer and Strohmann, 2010; Bauer and Strohmann, 2015; Bauer and Strohmann, 2017; Bauer et al., 2010), we already reported on the inclusion of pyrrolidine into triphenylsilanol-based hydrogen-bonded arrangements (Bauer and Strohmann, 2015). Recently, we reported the methanol solvate of enantiomerically pure (S)-2-(triphenylsilyl)pyrrolidinium chloride (Bauer and Strohmann, 2017) as a further crystal solvate in addition to the previously described chloroform-incorporating hydrochloride (Bauer et al., 2010). Hexaphenyldistannane, the lightest representative within the group 14, is a hypothetical compound (Lewars, 2008) that has been the subject of intense research since its presumed discovery in 1900 (Gomberg, 1900a; Gomberg, 1900b), leading to a new conceptual understanding of the interplay between steric repulsion and noncovalent dispersive attraction (Rösel et al., 2017). Although many structural data have been collected during the last decades, single-crystals of...
pure hexaphenylsilane suitable for single-crystal X-ray diffraction have not hitherto been obtained (Bernert et al., 2016; Brendler et al., 2012).

Hexaphenyldistannane, the tin homologue of hexaphenylethane, was first synthesized by Krause and Becker in 1920 by reduction of triphenyltinchloride with sodium and crystallized as the benzene solvate (Ph₃Sn)₂•2C₆H₆ (Krause and Becker, 1920). The physicochemical properties of this compound have been described very carefully by the same authors and it has been found that (Ph₃Sn)₂ can be crystallized from m-xylene, diethyl ether, and chloroform solvent-free. The crystal structures of a monoclinic (Preut et al., 1973) and a triclinic polymorph (Dakternieks et al., 2001) of solvent-free hexaphenyldistannane have been reported. In addition to the parent phenyl-substituted compound, crystal structures of the symmetric hexaaryldistannanes (p-Tol₃Sn)₂, (Schneider-Koglin et al., 1991), (o-Tol₃Sn)₂, (Schneider-Koglin et al., 1993), and [(C₆F₃)₂Sn]₂ (Bojan et al., 2010), and two co-crystalline compounds with included hexakis(2,6-diethylphenyl)distannane (Sita and Bickerstaff, 1989) and included [(o-(CH₃NMe)₂C₆H₅)]Ph₃Sn₂ (Turek et al., 2009), respectively, have been reported. The dianion Sn₂Ph₆⁻ has been the subject of investigation in the context of phenyl-substituted Zintl anions of the heavier main-group elements (Scotti et al., 1997; Wiesler et al., 2006). To my knowledge, only for two solvates of (Ph₃Sn)₂ singe-crystal X-ray structure determinations have so far been performed, i.e. the trigonal compounds (Ph₃Sn)₂•2C₆H₆ (Piana et al., 1991) and (Ph₃Sn)₂•2C₆H₅CH₃ (Eckardt et al., 1995; Karlov et al., 2013). Remarkably, thermogravimetry and X-ray powder diffraction of the benzene, toluene, fluorobenzene, chlorobenzene, and aniline solvates proved that all known crystal solvates are isomorphous and have the space group R̅3̅, regardless of whether they are formed by symmetric or asymmetric aromatic solvents (Eckardt et al., 1995). The reason is obvious, since all asymmetric solvents in the crystal lattice are rotationally disordered leading to a highly symmetric crystallographic pseudo threefold axis (Eckardt et al., 1995). This was also observed for the toluene solvate of hexaphenylsiloxane (Hönle et al., 1990). In addition, (Ph₃Sn)₂ was structurally characterized as part of a composite crystal (Lu and Cui, 2007). Hexaphenyldistannane can be considered as an interesting and simplified molecular model for investigating polymorphism and solvatomorphism in symmetrical and sterically shielded all-aryl-substituted group 14 compounds that cause crystal lattices with cavities of suitable size for the incorporation of solvent molecules.

This contribution presents the single-crystal X-ray diffraction analysis of the first crystalline ether solvate of hexaphenyldistannane [(Ph₃Sn)₂•2THF] that was obtained by reductive Sn–Sn coupling in the presence of tert-butyllithium, presumably in an analogous manner as previously described using Grignard reagents (Dakternieks et al., 2001). (Ph₃Sn)₂•2 THF crystallized from a solvent mixture of tetrahydrofuran/diethyl ether/pentane (2:1:1) at −30°C in the triclinic crystal system, space group P̅1, as colourless needles suitable for single-crystal X-ray diffraction analysis (see Table 1 and Figure 1). The crystallographic asymmetric unit contains a half molecule of hexaphenyldistannane and one tetrahydrofuran molecule arranged around an inversion center. The two THF molecules are located at both ends of the extended Sn–Sn bond axis. The phenyl groups along the Sn–Sn axis are arranged in a gauche conformation (C–Sn–Sn–C angles: 180.0°; 58.20°; 59.96°; 61.84°), which, after comparison with the other known structures of hexaphenyldistannane, is unaffected by the packing. However, unlike the previously known solvates (space group R̅3̅) consisting of symmetric (benzene) and

### Table 1: Crystal data and structure refinement of (Ph₃Sn)₂•2 THF.

| Parameter                  | Value                        |
|----------------------------|------------------------------|
| Empirical formula          | C₆H₅O₂Sn₂                    |
| Formula weight [g·mol⁻¹]   | 844.39                       |
| Crystal system             | Triclinic                    |
| Space group                | P̅1                          |
| a [Å]                      | 8.5992(3)                    |
| b [Å]                      | 11.2314(3)                   |
| c [Å]                      | 11.2870(3)                   |
| α [°]                      | 61.204(3)                    |
| β [°]                      | 84.396(2)                    |
| γ [°]                      | 84.581(2)                    |
| Volume [Å³]                | 949.34(5)                    |
| Z                          | 1                            |
| Density (calculated) [g·cm⁻³] | 1.477                      |
| Absorption coefficient μ [mm⁻¹] | 1.350                  |
| F(000)                     | 426                          |
| Crystal size [mm³]         | 0.40 × 0.30 × 0.30           |
| Theta range for data collection [°] | 2.063 – 25.999              |
| Index ranges               | -10 ≤ h ≤ 10                |
|                          | -13 ≤ k ≤ 13                |
|                          | -13 ≤ l ≤ 13                |
| Reflections collected      | 24331                        |
| Independent reflections   | 3733 (Rint = 0.0382)         |
| Completeness to θ = 27.00° | 100.0%                       |
| Max. and min. transmission | 0.6875 and 0.6142            |
| Data/restraints/parameters | 3733/0/277                  |
| Goodness-of-fit on F²      | 1.000                        |
| Final R indices [I > 2σ(I)] | R1 = 0.0219, wR2 = 0.0606    |
|                          | R1 = 0.0227, wR2 = 0.0610    |
| Largest diff. peak and hole [e·Å⁻³] | 1.012 and −0.359           |
asymmetric aromatic solvents (Eckardt et al., 1995; Piana et al., 1991), here the threefold axis of hexaphenyldistannane does not run perpendicularly through the THF molecules. The absence of this high symmetry in (Ph₃Sn)₂ • 2 THF, which in the previous cases was caused by rotational disorder, may indicate that the THF molecules are more tightly bound in the crystal lattice than the aromatic solvents. The Sn–Sn* distance with 2.7592(4) Å is quite similar to the Sn–Sn distance in the triclinic solvent-free polymorph of hexaphenyldistannane [2.7635(14) Å] (Dakternieks et al., 2001) and slightly shorter than the average Sn–Sn distance of the two independent molecules in the monoclinic form [2.759(4) Å and 2.780(4) Å] (Preut et al., 1973). The respective bond distances in the benzene and toluene solvates are 2.7666(4) Å and 2.7909(8) Å, respectively (Eckardt et al., 1995; Piana et al., 1991). The C–Sn distances are 2.138(3) Å, 2.141(3) Å, and 2.145(3) Å and are shorter than in the monoclinic solvent-free polymorph (average C–Sn distance: 2.18 Å) (Preut et al., 1973), but they are comparable to those of the other crystalline hexaphenyldistannanes and to related triphenylstannyl compounds (Bet tens et al., 2009; Dakternieks et al., 1998; Reeske et al., 2001). The ring angles around the aromatic carbon atoms bound to the tin atom (approx. 118°) deviate from the ideal angle (120°) by 2°, which is known for Ph₃Sn moieties (Eckardt et al., 1995). However, it has been found that this deviation is even more pronounced in triphenylsilyl-substituted derivatives (Bauer and Strohmann, 2010). The angels within the included tetrahydrofuran molecule range from almost ideal tetrahedral angles of 109.02(2) Å [C(22)–O–C(19)] to strongly distorted angles of 101.13(3) Å [C(22)–C(21)–C(20)]. Figure 2 shows some short intermolecular distances that are in the range of the sum of the van der Waals radii of the atoms involved (Rowland and Taylor, 1996). The shortest intermolecular distances can be found between the oxygen atom of the tetrahydrofuran molecule and hydrogen atoms of the phenyl rings of hexaphenyldistannane ranging from 2.628 Å [H(10)***···O] to 2.715 Å [H(16)*···O] (see Figure 2). Such weak C–H···O interactions are considered attractive and directional, and it is assumed that they can contribute significantly to the crystal packing, especially in the absence of strong hydrogen bonds (Desiraju, 1991; Desiraju, 1995). Deviations of this type of C–H···O hydrogen bonding from the directional preference of the hydrogen atoms towards the plane of the lone electron pairs have already been rationalized in the case of ethereal oxygen atoms (Desiraju, 1991; Legon and Millen, 1987). Even a short C···H distance of 2.861 Å between aryl rings can be located [H(2)–C(11)***] (see Figure 2).

In summary, the ether solvate [(Ph₃Sn)₂ • 2 THF] shows that also non-aromatic guest molecules...
can be incorporated in the crystal lattice of hexaphenyldistannane. Remarkable are the only small deviations of the intramolecular bonding parameters in the hexaphenyldistannane molecule of the here described tetrahydrofuran solvate when compared to the other known solvatomorphs and the crystal structures of the solvent-free host compound. Evidently, the guest molecules can be exchanged over a wide structural range without requiring large changes in the packing of the host compound. Weak intermolecular C–H···O interactions may cause an additional anchoring of the entrapped ether molecules within the host structure. This may give rise to further structural model studies regarding the interstitial crystallization behaviour of all-C-substituted main group element compounds.

**Experimental**

**General**

The title compound has been obtained as a by-product during the preparation of substituted triphenylstannanyl compounds from tert-butyllithium and triphenyltinchloride in a solvent mixture of tetrahydrofuran/diethyl ether/pentane (2:1:1). Colourless needles suitable for single-crystal X-ray diffraction analysis were obtained at –30°C after one day.

**X-ray crystallography**

Single-crystal X-ray diffraction analysis of (Ph3Sn)2 • 2 THF was performed on an Oxford Diffraction CCD Xcalibur S Diffractometer (Oxford Diffraction Ltd., 2008) at 173(2) K using graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). The crystal structure was solved with direct methods [SHELXS-97 (Sheldrick, 1997; Sheldrick, 2008)] and refined against F2 with the full-matrix least-squares method [SHELXL-2018/3 (Sheldrick, 2015; Sheldrick, 2018)] using the SHELX program package as implemented in WinGX (Farrugia, 2012). A multi-scan absorption correction using the implemented CrysAlis RED program was employed (Oxford Diffraction Ltd., 2008). The non-hydrogen atoms were refined using anisotropic displacement parameters. The aromatic hydrogen atoms were located on the difference Fourier map and refined independently. All other hydrogen atoms were placed in idealized geometric positions and each was assigned a fixed isotropic displacement parameter based on a riding-model with Uiso(H) = 1.2Ueq(C) for the methylene hydrogen atoms. The C–H distances constrained to 0.99 Å for the methylene groups. Crystal and refinement data are collected in Table 1. Figures 1 and 2 were created using Mercury 4.1.0 (Macrae et al., 2006). Crystallographic data of (Ph3Sn)2 • 2 THF have been deposited with The Cambridge Crystallographic Data Centre. CCDC 1955228 contains the supplementary crystallographic data for (Ph3Sn)2 • 2 THF. Copies of these data may be obtained free of charge from The Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk), or via www.ccdc.cam.ac.uk/data_request/cif.

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