

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

ENTEGRIS, INC.
Petitioner

v.

INPRIA CORP.
Patent Owner

IPR2025-00267
Patent 11,673,903

DECLARATION OF DR. GERARD PARKIN

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PETITIONER’S EXHIBIT LIST

Ex[No.]¹	Description of Documents
1001	U.S. Patent No. 11,673,903 (“’903 Patent”).
1002	Declaration of Dr. Gerard Parkin dated December 12, 2024 (“Parkin Decl.”).
1003	Declaration of Dr. Drew Hood dated December 10, 2024 (“Hood Decl.”).
1004	Prosecution File History for the ’903 Patent (“’903 Patent File History”).
1005	D. Hänssgen et al., “Synthesis of The First Mono-t-Butyltin Element Compounds,” 293 Journal of Organometallic Chemistry, pp. 191-195 (1985) (“Hänssgen”).
1006	U.S. Patent Application Publication No. 2016/0116839, entitled “Organometallic Solution Based High Resolution Patterning Compositions and Corresponding Methods,” to Meyers et al. (“Meyers”).
1007	U.S. Patent Application Publication No. 2011/0166268, entitled “High Purity Monoalkyltin Compounds and Uses Thereof,” to B. J. Deelman et al. (“Deelman”).
1008	Declaration of Sylvia D. Hall-Ellis, Ph.D. dated November 29, 2024.
1009	K. Jones and M.F. Lappert, “Amino-derivatives of Metals and Metalloids. Part I. Preparation of Aminostannanes, Stannylamines, and Stannazanes,” Journal of Chemical Society, pp. 1944-1951 (1965) (“Jones”).
1010	U.S. Patent No. 11,697,660.

¹ Citations to issued patents are made by column and line number. Citations to patent application publications are made by paragraph number. Citations to the remaining exhibits are to the page number added by the exhibit label.

Ex[No.] ¹	Description of Documents
1011	Excerpt of Prosecution File History for U.S. Patent No. 11,697,660.
1012	<i>Inpria Corp. v. Lam Research Corp.</i> , Case No. 1:22-cv-01359-CJB (D. Del.), Joint Claim Construction Brief, ECF No. 199.
1013	U.S. Patent Application Publication No. 2022/0242888, entitled “Process for Preparing Organotin Compounds,” to D. Kuiper et al.
1014	U.S. Patent No. 6,517,901.
1015	Walter Caseri, “Initial Organotin Chemistry.” 751 Journal of Organometallic Chemistry, pp. 20-24 (2014).
1016	K. Jones and M.F. Lappert, “Amino Derivatives of Metals and Metalloids. III. Metal Amines as Reagents for Synthesis of Organometallics; Especially the Reactions of Aminostannanes with Protic Species.” Journal of Organometallic Chemistry, Vol. 3, pp. 295-307 (1965).
1017	Clark Shove Robinson, “The Elements of Fractional Distillation,” McGraw-Hill Book Co., Inc. (1st ed. 1922).
1018	U.S. Patent No. 10,642,153.
1019	Procedures for Synthesis of $i\text{PrSn}(\text{NMe}_2)_3$ developed by Dr. Parkin.
1020	Procedures for Synthesis of $t\text{BuSn}(\text{NEt}_2)_3$ developed by Dr. Parkin.
1021	M. Froneman, T. A. Modro and S.M. Vather, “Dialkylamidoderivatives of Sn(II) and Sn(IV) in Phosphorus Chemistry,” 164 Inorganica Chimica Acta, pp. 17-21 (1989).
1022	U.S. Patent Application Publication No. 2022/0064192 A1.

I, Dr. Gerard Parkin, declare as follows:

I. INTRODUCTION

1. I make this declaration based upon my own personal knowledge and, if called upon to testify, would testify competently to the matters contained herein.

2. I have been asked to provide technical assistance in the *inter partes* review of U.S. Patent No. 11,673,903 (the “’903 Patent”). This declaration is a statement of my opinions on issues related to the unpatentability of Claims 1-4 and 6-10 of the ’903 Patent (the “Challenged Claims”). I am being compensated at my normal rate, plus reimbursement for expenses, for my analysis. My compensation does not depend on the content of my opinions or the outcome of this proceeding.

II. EXPERIENCE AND QUALIFICATIONS

3. In formulating my opinions, I have relied upon my knowledge, training, and experience in the relevant art. My qualifications are stated more fully in my curriculum vitae, which has been provided as Appendix A. Here, I provide a brief summary of my qualifications.

4. I am a Professor in the Department of Chemistry at Columbia University, New York, New York.

5. I received a B. A. (First Class), M. A., and D. Phil. in Chemistry from the Queen’s College, Oxford University. I joined the faculty of Columbia University as Assistant Professor in 1988 and was promoted to Associate Professor in 1991 and

to Professor in 1994. I served as Chairman of the Department from 1999 to 2002. I have also served as Chair of the New York Section of the American Chemical Society, Chair of the Inorganic Chemistry and Catalytic Science Section of the New York Academy of Sciences, Chair of the Organometallic Subdivision of the American Chemical Society Division of Inorganic Chemistry, and Chair of the Gordon Research Conference in Organometallic Chemistry. I am an elected Fellow of both the Royal Society of Chemistry and the American Chemical Society, and also the American Association for the Advancement of Science.

6. Among other awards, I am a recipient of the Presidential Faculty Fellowship Award, an Alfred P. Sloan Research Fellowship, the Camille and Henry Dreyfus Teacher-Scholar Award, the American Chemical Society Award in Pure Chemistry, the American Chemical Society Award in Organometallic Chemistry, the Royal Society of Chemistry Corday Morgan Medal, the Royal Society of Chemistry Award in Organometallic Chemistry, the Royal Society of Chemistry Chem Soc Rev Lecture Award, the James Flack Norris Award for Outstanding Achievement in the Teaching of Chemistry, the Columbia University Presidential Award for Outstanding Teaching, the Lenfest Distinguished Columbia Faculty Award, the New York Section of the American Chemical Society Outstanding Four-Year University with Graduate School Chemistry Faculty Teaching Award, the American Chemical Society Project SEED Outstanding Mentor Award and the

Columbia University Presidential Award for Outstanding Teaching. I also received the Presidential Award for Excellence in Science, Mathematics and Engineering Mentoring, an award that was presented at a White House ceremony.

7. Other recognitions that I have received include the Distinguished Monsanto Lecture (Indiana University), the Denkwalter Lecture (Loyola University), the Inaugural Distinguished Catalysis Lecturer (University of Florida), Scholar's Day Lecturer (Western Washington University), the Dow Lecturer (University of California, Berkeley), the Distinguished Raymond Siedle Lecture (Indiana University), the Coates Lecture (University of Wyoming), the Malcolm L. H. Green Lecture (Oxford University), the Anton B. Burg Memorial Lecture (University of Southern California), the Distinguished Karcher-Barton Awardee Lecturer (University of Oklahoma), and the Senator Penny Williams Distinguished Lecturer (Oklahoma School of Science and Mathematics Foundation).

8. I have taught various courses on inorganic chemistry, which include "Introduction to Inorganic Chemistry," "Advanced Inorganic Chemistry," "Structural Methods in Inorganic Chemistry" and "Applications of NMR Spectroscopy to Inorganic Chemistry." In addition, I also teach "General Chemistry I."

9. For approximately the last 40 years, the focus of my work has been concerned with synthetic, structural, and mechanistic inorganic chemistry. By virtue

of my education and experience, I am familiar with a wide variety of metal coordination compounds, including those that contain nitrogen donor ligands, and I have also published on tin chemistry.

10. I have published more than 350 research papers and have given more than 275 research presentations at various universities, national and international chemistry conferences, and companies. My research has been supported by many sources, which include the National Science Foundation, the National Institutes of Health, the Department of Energy, the American Chemical Society Petroleum Research Fund, United Technologies Research Center, the Alfred P. Sloan Foundation, the Camille and Henry Dreyfus Foundation, and the Kanagawa Academy of Science and Technology.

11. My work on inorganic chemistry has led me to serve as a consultant to a number of companies. These companies include Exxon, Purdue Pharma, Redox Pharmaceutical Corporation, Symyx, and United Technologies Research Center.

12. My work on inorganic chemistry has led me to serve on the Editorial Advisory Boards of a number of chemistry journals, which include *Accounts of Chemical Research*, *Inorganic Chemistry*, *Main Group Chemistry*, *J. Chem. Soc.*, *Dalton Trans.*, *Polyhedron*, *J. Organomet. Chem.*, *Organometallics*, and *Structure and Bonding*.

13. I have been an editor of *Polyhedron* since 2000 and was also an editor for *SciTopics*. In addition, I have served as an editor for *Comprehensive Coordination Chemistry 2 & 3*, *Comprehensive Organometallic Chemistry 3 & 4*, and *Structure and Bonding*.

14. I have organized a variety of chemical conferences and symposia. For example, I was the Chair of the 2006 Gordon Research Conference in Organometallic Chemistry. I also organized the following symposia: “Main Group Organometallic Compounds: Novel Structures and Reactivity,” 206th ACS National Meeting, Chicago, Illinois, August 1993; “New Vistas in Inorganic Chemistry,” 211th ACS National Meeting, New Orleans, Louisiana, March, 1996; “Scorpionate Ligands, Thirty-Five Years Later,” 225th ACS National Meeting, New Orleans, Louisiana, March 2003; “Organometallic Chemistry: The New Frontiers,” 248th American Chemical Society National Meeting, San Francisco, California, 2014; and the Pure Chemistry Award, 251st American Chemical Society National Meeting, San Diego, California, March 2016.

15. I appeared on National Geographic Channel’s *Going Deep with David Rees* episode on “How to light a match” (Season 1, Episode 8, 2014) and was Science Advisor for the *Tonight Show* with Jimmy Fallon (2014 – 2019).

III. LEGAL STANDARDS

16. I have been informed by counsel for Entegris regarding several principles relevant to this matter that I have used in arriving at my stated conclusions in this declaration.

17. I have been informed and understand that the patentability analysis is conducted on a claim-by-claim and element-by-element basis, and that there are several possible reasons that a patent claim may be found to be unpatentable.

18. I have been informed and understand that earlier publications and patents may act to render a patent unpatentable for one of two reasons: (1) anticipation and (2) obviousness.

19. I have been informed and understand that the claims of a patent are anticipated by a prior art reference if each and every element of the claim is found either explicitly or inherently in a single prior art reference. I have been informed and understand that for a claimed limitation to be inherently present, the prior art need not expressly disclose the limitation, so long as the claimed limitation necessarily flows from a disclosure in the prior art. I further have been informed and understand that inherency requires a showing that the missing descriptive matter in the claim is necessarily or implicitly present in the allegedly anticipating reference, and that it would have been so recognized by a person of ordinary skill in the art (“POSITA”). In addition, I have been informed and understand that an enabling

disclosure is a disclosure that allows a POSITA to make the invention without undue experimentation. Although anticipation typically involves the analysis of a single prior art reference, I have been informed and understand that additional references may be used to show that the primary reference has enabling disclosure, to explain the meaning of a term used in the primary reference, and/or to show that a characteristic is inherent in the primary reference.

20. I have been informed and understand that the prior art may render a patent claim “obvious.” I have been informed and understand that two or more prior art references (*e.g.*, prior art articles, patents, or publications) that each disclose fewer than all elements of a patent claim may nevertheless be combined to render a patent claim obvious if the combination of the prior art collectively discloses all elements of the claim and one of ordinary skill in the art at the time would have been motivated to combine the prior art in such a way. I have been informed and understand that this motivation to combine need not be explicit in any of the prior art, but may be inferred from the knowledge of one of ordinary skill in the art at the time the patent was filed. I also have been informed and understand that one of ordinary skill in the art is not an automaton, but is a person having ordinary creativity. I further have been informed and understand that one or more prior art references, articles, patents or publications that disclose fewer than all of the elements of a patent claim may render a patent claim obvious if including the missing

element would have been obvious to one of skill in the art (*e.g.*, the missing element represents only an insubstantial difference over the prior art or a reconfiguration of a known system).

21. I have been informed and understand that under the doctrine of obviousness, a claim may be invalid if the differences between the invention and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a POSITA to which the subject matter pertains.

22. I have been informed and understand that, to assess obviousness, I am to consider the scope and content of the prior art, the differences between the prior art and the claim, the level of ordinary skill in the art, and any secondary considerations to the extent they exist.

23. I have been informed and understand that any evidence of secondary considerations of non-obviousness should be considered when evaluating whether a claimed invention would have been obvious to one of ordinary skill at the time of invention. I have been informed and understand these secondary considerations of non-obviousness may include, for example:

- a long felt but unmet need in the prior art that was satisfied by the claimed invention;
- commercial success of processes claimed by the patent;
- unexpected results achieved by the invention;
- praise of the invention by others skilled in the art;

- the taking of licenses under the patent by others; and
- deliberate copying of the invention.

24. I have been informed and understand that there must be a nexus between any such secondary considerations and the claimed invention.

25. I have also been informed and understand that there are additional considerations that may be used as further guidance as to when the above factors will result in a finding that a claim is obvious, including the following:

- the claimed subject matter is simply a combination of prior art elements according to known methods to yield predictable results;
- the claimed subject matter is a simple substitution of one known element for another to obtain predictable results;
- the claimed subject matter uses known techniques to improve similar devices or methods in the same way;
- the claimed subject matter applies a known technique to a known device or method that is ready for improvement to yield predictable results;
- the claimed subject matter would have been “obvious to try” choosing from a finite number of identified, predictable solutions, with a reasonable expectation of success;
- there is known work in one field of endeavor that may prompt variations of it for use in either the same field or a different one based on design incentives or other market forces if the variations would have been predictable to a POSITA;
- there existed at the time of conception and reduction to practice a known problem for which there was an obvious solution encompassed by the patent’s claims; and

- there is some teaching, suggestion, or motivation in the prior art that would have led a POSITA to modify the prior art reference or to combine prior art reference teachings to arrive at the claimed subject matter.

26. Finally, I have been informed and understand that a claim may be deemed invalid due to obviousness in light of a single prior art reference, without the need to combine references, if the elements of the claim that are not found in the reference can be supplied by the knowledge or common sense of one of ordinary skill in the relevant art.

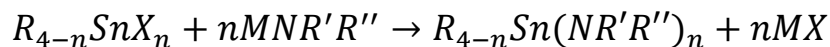
IV. STATE OF THE ART AT THE TIME OF THE '903 PATENT

27. I provide the below technology overview for context, the subject matter of which was well-known to persons of ordinary skill in the art before the time of the alleged invention (*i.e.*, April 11, 2018).

A. Organotin compounds

28. Organotin compounds, or stannanes, are a sub-class of organometallic compounds, which contain at least one tin-to-carbon bond in which the carbon is part of an organic group. Organotin compounds were discovered more than a century ago—the first organotin compound, diethyltin diiodide ((CH₃CH₂)₂SnI₂), was discovered in 1849, and was followed by a rapid expansion of new organotin compounds in early 1900s. EX1015 (Walter Caseri, “Initial Organotin Chemistry,” 751 Journal of Organometallic Chemistry, pp. 20–24 (2014)). By the 1960s, reactions and methodologies to form a broad range of aminostannanes, including

alkyltin amides, became well established, *e.g.*, through reactions between tinhalides and lithium amide compounds, as illustrated below:



(EX1009, 4), where R, R', and R'' = alkyl or aryl, X = Cl or Br, M = Li, and n = 1, 2, 3, or 4. See EX1009, 4.² In other words, given the above lithium amide chemistry, a POSITA was able to derive mono-, di-, or trialkyl tin amides from corresponding alkyltin halides as early as the 1960s. EX1009, 4.

29. During the same time period, certain properties, and reactions of aminostannanes had already become evident. For example, it was “noticed when preparing aminostannanes, that the exclusion of atmospheric moisture was necessary to avoid their decomposition.” EX1016, 4 (K. Jones and M.F. Lappert, “Amino Derivatives of Metals and Metalloids. III. Metal Amines as Reagents for Synthesis of Organometallics; Especially the Reactions of Aminostannanes with Protic Species,” *Journal of Organometallic Chemistry*, Vol. 3, pp. 295-307 (1965)). Besides water, it was further understood that aminostannanes are reactive towards other protic species including alcohols “due to the very weak metal-nitrogen bond.” EX1016, 4. The ready alcoholysis (*i.e.*, reaction with alcohol) of aminostannanes

² I note that there are typographical errors in reaction (4) of Jones, which I corrected herein, and are clear to a POSITA given the textual descriptions and balancing of the chemical reaction. See footnote 4.

had been utilized for the preparation of tin alkoxides, which had been difficult to obtain in a pure composition otherwise. EX1016, 4 and Table 2.

30. Organotin compounds have found significant applications in many areas. For example, organotin compounds are commercially applied as stabilizers “for chlorine-containing polymers, especially PVC polymers, post-chlorinated PVC and copolymers thereof, as well as for uses as a catalyst, or in glass coating compositions.” EX1007, [0009]. Moreover, it is also known that aminostannanes can be used to prepare precursor solutions that can be deposited “to form organometallic coatings that can be patterned with very high resolution with radiation” (EX1006, [0002], [0152]) or used as an intermediate for other useful organotin compounds (EX1016, 4). As another example, tin alkoxides can be useful for formation of coatings as “electrodes for the glass substrates of emissive screens of the flat screen type, electrodes for photovoltaic cells or arrays of conductive elements for heated windows, windows provided with an antenna or glazing with a conductive coating having the function of a shield towards electromagnetic waves.” EX1014, 1:23-30 (U.S. Patent No. 6,517,901).

31. Like other chemicals, high purity organotin compounds are of general interest to chemists because high purity compounds can minimize side reactions and undesirable impurities. While reactions can be designed to generate specifically targeted compounds and optimized to minimize secondary reactions and impurities,

purification steps are routinely conducted to remove solvents and reaction by-products and to improve the purity of the target compounds.

B. Fractional Distillation

32. Fractional distillation is a technique, established many years ago, to separate a mixture of two or more volatile substances into its components, “by causing the mixture to vaporize by suitable application of heat, [and] condensing the vapors in such a way that fractions of varying boiling points are obtained;” that process can be repeated “until the degree of separation desired is finally obtained.” EX1017,16 (Clark Shove Robinson, “The Elements of Fractional Distillation,” McGraw-Hill Book Co., Inc. (1st ed. 1922)).

33. While fractional distillation may be conducted using different equipment, setup, and conditions, the underlying principle remains that when a mixture of volatile liquids is distilled, the distillate contains a greater proportion of the more volatile material than the residue. Given sufficient quantities of the starting mixture and sufficient numbers of repeated distillation cycles, components of the mixture tend to be eventually separated and purified. While fractional distillation is often a laborious and expensive process, it has been employed widely in industry to separate and purify volatile substances including organotin compounds. EX1007, [0028].

C. Nuclear Magnetic Resonance (“NMR”) Spectroscopy

34. NMR spectroscopy is a well-known method used for characterizing organic compounds and their structures. NMR is further able to provide quantitative analysis of trace amount of materials, and is ideally suited for determining impurity levels in an organometallic compound.

35. The principle of NMR spectroscopy is based on characteristics of nuclei that have a nonzero nuclear spin. When such nuclei are subject to a strong magnetic field and are perturbed by a radio-frequency pulse, the nuclei produce an electromagnetic signal with a characteristic frequency, which depends on the strength of the applied magnetic field, the chemical environment, and the magnetic properties of the isotope. NMR spectroscopy provides a wealth of information regarding the structure of a molecule and has been used as a standard method for identifying and quantifying organic compounds.

V. OVERVIEW OF THE '903 PATENT

A. Subject Matter of the '903 Patent

36. The '903 Patent is entitled “Monoalkyl Tin Compounds With Low Polyalkyl Contamination, Their Compositions and Methods.” EX1001.

37. The “Field of the Invention” section of the '903 Patent states the “invention relates to high-purity compositions of monoalkyl tin triamides,

monoalkyl tin trialkoxides, or monoalkyl triamido tin and the methods to make them.” EX1001, 1:7-9.

38. In the Background, the Patent Owner describes the use of “organometallic compounds” to “provid[e] metal ions in a solution processable form.” EX1001, 1:13-14. Specifically, “[a]llyl tin compounds provide a radiation sensitive Sn-C bond that can be used to pattern structures lithographically[,]” which makes them “promising advanced materials” for semiconductor processing, where the “ever shrinking dimensions result[] in demands for more versatile materials to achieve desired patterning resolution[.]” EX1001, 1:14-21.

39. The '903 Patent states that the “invention pertains to a composition comprising a monoalkyltin trialkoxide compound represented by the chemical formula $\text{RSn}(\text{OR}')_3$ or a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and no more than 4 mole% dialkyltin compounds relative to the total tin amount, where R is a hydrocarbyl group with 1-31 carbon atoms, and where R' is a hydrocarbyl group with 1-10 carbon atoms.” EX1001, 1:25-32.

40. The '903 Patent acknowledges that the compounds of the alleged invention can either “be used as synthesized or further purified[,]” including purification by “fractional distillation to effectively reduce polyalkyl contaminants

below levels that may already be relatively low from the direct synthesis.” EX1001, 3:2-5, 3:13-17.

41. The '903 Patent acknowledges that “[m]ethods for the preparation of monoalkyl tin triamides” were known prior to the invention, stating that “t-butyl tris(diethyl-amido)tin, (t-BuSn(NEt₂)₃), can be synthesized with a lithium reagent according to the method of Hänssgen, D.; Puff, H.; Beckerman, N. J. Organomet. Chem. 1985, 293, 191.” EX1001, 3:29-36. The '903 Patent states that this method, “however, can produce a mixture of monoalkyl and dialkyl tin products” and “it can be desirable to reduce any polyalkyl byproducts, e.g., dialkyl tin contaminants.” EX1001, 3:29-44. Separation of the monoalkyl and dialkyl species “can be [done] for some compounds,” but Patent Owner asserts “the separation or purification process generally raises the cost of manufacturing.” EX1001, 3:44-52. However, none of the Challenged Claims recite limitations precluding a purification process. Moreover, “[p]olyalkyl tin impurity compositions may affect condensation and contribute to photoresist outgassing during lithographic processing, which increases the potential for tin contamination of equipment used for film deposition and patterning.” EX1001, 4:40-46. Thus, according to the '903 Patent, “it can be desirable to synthesize the monoalkyl tin compounds with higher purity such that any subsequent purification, such as with fractional distillation, if desired, results in even lower dialkyl or polyalkyl contamination.” EX1001, 3:49-52.

B. Prosecution History of the '903 Patent

42. I have been informed and understand that the application that ultimately issued as the '903 Patent was filed on April 11, 2018 as Application No. 15/950,286 ("286 Application"). EX1004, 4. I have been informed and understand that the application was filed with 16 total claims; claims 1-10, and 12-16 were directed to a composition, while claim 11 was directed to a method of formation. Claims 1, 11, and 12 were independent claims, with claims 2-10 depending from claim 1, and claims 13-16 depending from claim 12. EX1004, 1242-1243.

i. September 20, 2019 Restriction/Election and Response

43. I have been informed and understand that on September 20, 2019, the Examiner issued a "Restriction/Election" requirement, requiring the applicant to elect one invention to be examined between claims 1-10, which were "drawn to a composition, classified in C07F7/2224," claim 11, "drawn to a method to selectively form a monoalkyltin trialkoxide compound, classified in C07F7/2224," and claims 12-16, which were "drawn to a composition, classified in C07F7/2285." EX1004, 1199-1205.

44. I have been informed and understand that Applicant filed a Response to the Restriction/Election Requirement on November 20, 2019, electing claims 1-10. EX1004, 1193.

ii. March 4, 2020 Rejection and Response

45. I have been informed and understand that on March 4, 2020, the Examiner issued a Non-Final Rejection rejecting claims 1-10. EX1004, 1135-1142. Claims 1-9 were provisionally rejected on the ground of nonstatutory double patenting as being obvious over claims 14-21 of copending Application No. 16/446,732. EX1004, 1137-38.

46. I have been informed and understand that claims 1, 3, 6-7, and 9-10 were further rejected as anticipated under 35 U.S.C. § 102(a)(1) by Bernhardt (DE 3109309). EX1004, 1139-40. I have been informed and understand that the Examiner found that Bernhardt “teaches a composition comprising an organic tin compound in an organic solvent wherein the organic tin compound is a monoalkyl tin triethoxide (which satisfies a composition comprising no dialkyltin compounds)” and “monoalkyl triethoxide (RSn(OR)_3) or monoalkyl tin triamide ($\text{RSN(NR}'_2)_3$) wherein R can be an alkyl or cyclohexyl (which satisfies a composition comprising no dialkyltin compounds).” EX1004, 1139-40.

47. I have been informed and understand that claims 2, 4-5, and 8 were rejected as obvious under 35 U.S.C. § 103 over Bernhardt. EX1004, 1140-42. While the Examiner acknowledged that “Bernhardt fails to specifically disclose a branched alkyl group and the amount of the compound in the solvent,” the Examiner stated these limitations “would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention.” EX1004, 1140-42.

48. I have been informed and understand that Applicant filed a response to the Non-Final Rejection on May 29, 2020. EX1004, 1125-29. I have been informed and understand that with respect to the provisional rejection for nonstatutory double patenting, the Applicant argued that “the scope of the ’732 claims are not accurately stated in the Office Action,” as they “are directed to a solution with a tin concentration from about 0.004 to about 1.0 M,” while the claims of the ’286 Application limit the “amount of dialkyltin compounds” to “no more than 4 mole% of the total tin amount.” EX1004, 1125. I have been informed and understand that applicant further argued that Bernhardt did not anticipate or render obvious the claims of the ’286 Application because “Bernhardt does not teach or suggest” the “highly pure monoalkyltin compounds as instantly claimed.” EX1004, 1125-1129.

iii. September 18, 2020 Rejection and Response

49. I have been informed and understand that a Final Rejection was issued on September 18, 2020. EX1004, 908-916. I have been informed and understand that the Examiner maintained the nonstatutory double patenting rejection of Claims 1-9, the rejection of Claims 1, 3, 6-7, and 9-10 as anticipated by Bernhardt, and the rejection of claims 2, 4-5, and 8 as obvious over Bernhardt. EX1004, 908-916. I have been informed and understand that the Examiner rejected Applicant’s arguments regarding the teaching of Bernhardt, stating “the instant claims are drawn to the

composition and not the method of making monoalkyl tin trialkoxide.” EX1004, 913.

50. I have been informed and understand that Applicant conducted an interview with the Examiner on October 6, 2020, where the Applicant indicated “their invention has a higher purity than the Bernhardt reference” and “Applicant is considering filing a declaration to provide evidence of the difference in purity compared to Bernhardt.” EX1004, 904-905.

51. I have been informed and understand that Applicant filed a Request for Continued Examination on January 19, 2021. EX1004, 886-898. I have been informed and understand that Applicant amended claim 1 to specify in the preamble that the claimed composition was a “high purity liquid composition” and amended the claim limitation regarding the purity of the composition to require “no more than ~~[[4]]~~0.5 mole% dialkyltin compounds as an impurity relative to the total tin amount.” EX1004, 893. I have been informed and understand that claim 6 was amended to the “composition of claim 1 comprising essentially ~~no more than about 1 mole%~~ dialkyltin compounds as measured with an approximate 0.1% detection limit.” EX1004, 893.

52. I have been informed and understand that Applicant argued that the nonstatutory double patenting rejection should be withdrawn because the claims of the '732 Application do not recite “a high purity liquid composition having a percent

range of dialkyltin compounds as an impurity.” EX1004, 889-90. With respect to the rejections over Bernhardt, I have been informed and understand that Applicant argued that Bernhardt does not “teach or suggest monoalkyltin compounds having the claimed purity with respect to dialkyl compounds.” EX1004, 890-892.

53. I have been informed and understand that the Applicant further stated “[t]o more fully address the Examiner’s concerns regarding the novelty of the claimed purity with respect to dialkyl impurities, the Applicant files a Declaration under 37 C.F.R. § 1.132 signed by inventor Joseph Edson” (the “2021 Declaration”). EX1004, 890-891. I have been informed and understand that the 2021 Declaration “presents results of in-house experiments that [purportedly] followed the method of Hänssgen (HÄNSSGEN ET AL., “Synthesis of the First Mono-t-butyltin Element Connections”, *Organometallic Chemistry*, 293:191-195 (1985)) since this reaction was considered to be the state of the art at the time of filing of the instant application.” EX1004, 890. I have been informed and understand that Applicant’s January 19, 2021 Response further stated the “experiments considered the reaction of tBuLi with Sn(NEt₂)₄ in diethyl ether. The reaction product was purified using standard fractional distillation, as described by Hänssgen, to selectively isolate the major product tBuSn(NEt₂)₃. The post-distillation product was subjected to ¹¹⁹Sn NMR analysis. The post-distillation product had an average of approximately 2% dialkyltin triamide, tBu₂Sn(NEt₂)₂.” EX1004, 890-891. Thus, according to

Applicant, “the claimed purity of the monoalkyltin compounds was not achievable through the use of an alkyl lithium reaction, through the use of an alkyl lithium reaction with a subsequent distillation step, or through other synthesis and/or purification processes that were then known in the art.” EX1004, 891.

iv. June 10, 2021 Rejection and Response

54. I have been informed and understand that a Non-Final Rejection was mailed on June 10, 2021. EX1004, 628-638. I have been informed and understand that the Examiner withdrew the anticipation rejection of Claims 1, 3, 6-7, and 9-10 over Bernhardt “together with Applicants amendment,” but maintained the rejection of claims 2, 4-5 as obvious over Bernhardt. EX1004, 630. I have been informed and understand that claim 6 was rejected under 35 U.S.C. § 112 as being indefinite; the Examiner argued that the claim term “0.1% detection limit” was “unclear if the percentage is mole% or weight%.” EX1004, 630-31. I have been informed and understand that claims 1-7, 9-10 were rejected as obvious over Bernhardt together with Merz III (U.S. Patent Application No. 2013/028125). EX1004, 631-34. I have been informed and understand that the Examiner argued that Bernhardt disclosed all the claim limitations except for “no more than 0.5 of 0.1 mole% of dialkyltin impurity compounds and a branched alkyl group.” EX1004, 632. “Merz III et al teaches producing monoalkyltin trihalide with an overall yield of nearly 100% (which satisfies no more than 0.5 of 0.1 mole% of dialkyl tin compounds.” EX1004,

632. I have been informed and understand that the Examiner argued that “it would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to have provided a nearly 100% yield of monoalkyl tin trichloride with less than 0.5 of 0.1 mole% dialkyl tin compounds as Bernhardt teaches controlling the reaction to produce the intended amounts of product and because Merz III et al teaches having a monoalkyl tin compound at nearly 100% yields helps to avoid or minimize toxicity issues.” EX1004, 632.

55. I have been informed and understand that claim 8 was rejected as obvious over Bernhardt together with Merz III together with Minami et al (EX1014 - U.S. Patent No. 6,517,901). EX1004, 633. I have been informed and understand that the Examiner argued that while Bernhardt together with Merz III “fail to specifically disclose an alcohol solvent and the amount of the tin compound in the solvent,” Minami, which is “in the same field of endeavor,” “teaches a process for depositing a layer on a glass substrates comprising a dopant such as alkyltin alkoxides...and a solvent such as ethanol or isopropanol.” EX1004, 633.

56. I have been informed and understand that the Examiner further rejected Claims 1-6 as obvious over Frenkel et al (U.S. Patent Application No. 2017/0002180), arguing Frenkel disclosed “a composition comprising at least one mono-octyl tin compound (n-OC)Sn(T)₃ wherein tin is an alcoholate[] having a purity greater than 85%, preferably up to 99wt%. Frenkel et al further teaches dioctyltin

compounds in an amount less than 8wt% and in a range of 0.1 to 5 wt% (which overlaps the instantly claimed range).” EX1004, 634-35. I have been informed and understand that the Examiner found that Frenkel failed to specifically disclose “R is methyl, ethyl, isopropyl, t-butyl, or t-amyl, R’ is methyl, ethyl, isopropyl, t-butyl, or t-amyl, and less than 0.5/0.1mole% of dialkyl tin compounds,” but found such limitations would be obvious. EX1004, 634-35.

57. I have been informed and understand that claims 7-9 were rejected as obvious over Frenkel together with Tartarin et al (European Patent 2123659) together with Minami. EX1004, 635-36. I have been informed and understand that the Examiner found “Tartarin et al teaches monoalkyl tin compounds used in glass coating compositions and solutions.” EX1004, 635-36. I have been informed and understand that the Examiner found “[i]t would have been obvious...to [provide] a solution comprising the tin compound of Frenkel et al and an alcohol solvent together with Tartarin et al and Minami et al in order to form a coating/film composition for coating glass substrates.” EX1004, 635-36.

58. I have been informed and understand that the Examiner considered Applicant’s arguments regarding the 2021 Declaration and rejected them, stating “the experimental methods disclosed in the declaration in paragraph 8 are not comparable to the Bernhardt reference. Furthermore, new rejections have been

applied...in regards to the impurity of the dialkyl tin compounds, as recited in the newly amended claims.” EX1004, 636.

59. I have been informed and understand that the Applicant filed a response on October 1, 2021. EX1004, 617-624. I have been informed and understand that claim 1 was amended to delete “a monoalkyltin trialkoxide compound represented by the chemical formula $\text{RSn}(\text{OR}')$,” so the claim only requires “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$.” EX1004, 618. I have been informed and understand that claim 6 was amended to “the composition of claim 1 comprising essentially no dialkyltin compounds as measured with an approximate 0.1mole% detection limit.” EX1004, 618.

60. I have been informed and understand that Applicant argued the amendment to claim 6 overcame the indefiniteness rejection. EX1004, 620. I have been informed and understand that Applicant further argued that the rejections over Bernhardt were improper because “Bernhardt’s vague description does not inherently teach the claimed compositions and does not enable their formation. Bernhardt does not provide a reasonable expectation of success.” EX1004, 620-21. I have been informed and understand that Applicant specifically argued that Bernhardt’s disclosure of Grignard-reaction synthesized chloride products result in “technical problems” and “high dialkyl impurities.” EX1004, 621. I have been informed and understand that Applicant argued that the “[p]urification techniques

taught in these references do not reasonably enable the claimed level of mono-alkyl purity.” EX1004, 621.

61. I have been informed and understand that Applicant further argued that the rejections involving Frenkel should be withdrawn because claim 1 was amended to require a “monoalkyl tin triamide compound,” which Applicant argued Frenkel does not teach. EX1004, 623-624.

v. January 6, 2022 Rejection and Responses

62. I have been informed and understand that a Final Office Action was mailed January 6, 2022. EX1004, 597-603. I have been informed and understand that the Examiner withdrew the rejection of claim 6 as indefinite and the rejections over Bernhardt and Frenkel in response to Applicant’s amendments. EX1004, 599.

63. I have been informed and understand that the Examiner issued new rejections, rejecting Claims 1-9 over U.S. Patent Application No. 2011/0166268 to Deelman et al. (“Deelman”) together with U.S. Patent Application No. 2016/0116839 to Meyers et al. (“Meyers”). EX1004, 600-603. I have been informed and understand that the Examiner stated “Deelman et al teaches a high purity monoalkyltin compound” with “up to 99.999wt% of monoalkyl tin compound of formula $R\text{Sn}(T)_3$, wherein R is a linear or branched C1-C20 alkyl and T is a ligand.” EX1004, 600. I have been informed and understand that the Examiner noted “Deelman fails to specifically disclose no more than 0.5 or 0.1 mole% dialkyltin

compounds.” EX1004, 600. I have been informed and understand that Meyers teaches “a coating solution comprising an organic solvent and organometallic compound,” “alkyl tin compounds with organic ligands such as dialkylamides,” and “solvents such as alcohols.” EX1004, 600. With respect to the “no more than 0.5 or 0.1 mole% dialkyltin compounds” limitation, I have been informed and understand that Examiner found it would have been obvious based on Deelman to have such amounts of dialkyltin compounds because Deelman “teaches as high as 99.999wt% of monoalkyl tin compounds in the composition and as low as 0.001wt% of dialkyl tin compounds,” which “overlaps the instantly claimed range.” EX1004, 600.

64. I have been informed and understand that Applicant submitted a response after Final Rejection on February 28, 2022, arguing, based on the 2021 Declaration, that the claimed purity of the monoalkyltin compounds was not achievable through the alkyl lithium reaction, and that the combination of Deelman and Meyers failed to teach the claimed purity of the compound. EX1004, 588-593.

65. I have been informed and understand that the Examiner issued an Advisory Action on April 1, 2022, indicating that the February 28, 2022 response had not placed the application in condition for allowance and that the arguments regarding Deelman and Meyers were not convincing because the claims were directed to the composition, not the method of manufacture. EX1004, 583-585.

66. I have been informed and understand that Applicant filed a Notice of Appeal on April 5, 2022. EX1004, 578.

67. I have been informed and understand that on September 16, 2022, Applicant filed a Request for Continued Examination, amending the specification and claims to correct typographical errors. EX1004, 540-553. I have been informed and understand that in the September 16, 2022 response, Applicant argued, again, based on the 2021 Declaration, that the claimed purity of the monoalkyltin compounds was not achievable through the alkyl lithium reaction, and the claims were not obvious over Deelman together with Meyers. EX1004, 546-548. I have been informed and understand that Applicant refuted Examiner's position in the Advisory Action, arguing that the method of making the composition affects both the product produced and its purity, so the method of manufacture is relevant to the claims. EX1004, 547.

68. I have been informed and understand that Applicant also filed another Declaration under 37 C.F.R. § 1.132 ("2022 Declaration") with the September 16, 2022 response, which "presents results of an in-house experiment that adapted the method of Deelman to try to make a monoalkyltin triamide product." EX1004, 547-548, 551-553 (2022 Declaration). Specifically, I have been informed and understand that the response stated "Applicant reacted an $\text{RSn}(\text{Hal})_3$ compound (nbutyltin trichloride) with a 3-fold molar excess of an H-T compound (diethylamine), **and**

water, under stirring, to try to form an RSn(T)_3 product (nbutyltin trisdiethylamine).” EX1004, 547.³ I have been informed and understand that the inventor observed that “[i]mmediately upon combining the reagents, a bulk, white precipitate was formed” “consistent with the formation of nBuSnOOH[.] ” EX1004, 547. Thus, I have been informed and understand that Applicant concluded “the claimed high purity liquid compositions according to claim 1 are not achievable through the method disclosed by Deelman et al.” EX1004, 548.

69. In my opinion, Applicant’s statements regarding the in-house experiments it supposedly adapted from Deelman are misleading. A POSITA would have understood that the reactions for production of monoalkyltin triamide compounds according to Deelman would not efficiently proceed in an aqueous mixture, and to the extent the reaction was even able to proceed in an aqueous mixture at all, the purity of any resulting monoalkyltin triamide compound would be very low. Indeed, a POSITA would have understood, as the inventor’s 2022 Declaration admits, that “monoalkyltin triamides would be expected to hydrolyze in aqueous solution.” EX1004, 553; *see also* EX1004, 448-449 (Dr. Edson’s 2023 Declaration citing to prior work by Patent Owner, U.S. Patent No. 10,642,153 at Examples 1 and 2, which demonstrate, respectively, that both t-butyl tris(diethylamido)tin ($\text{t-BuSn(NEt}_2)_3$) and i-propyltin trichloride (i-PrSnCl_3) react

³ All emphasis added unless otherwise indicated.

with water to form a hydrolysate solution.). Thus, because Applicant attempted to conduct the experiment “in aqueous solution,” a POSITA would not expect the resulting compound to show the predicted signals for the monoalkyltin compounds during NMR analysis.

70. Finally, I have been informed and understand that Applicant argued that a “recently published patent application US 2022/0242888, filed by Entegris, Inc.,” supported the patentability of the rejected claims because it allegedly “asserts an unmet need for highly pure monoalkyl tin triamide compounds” and is “objective evidence” supporting Applicant’s arguments “regarding the limitations of the teachings of Deelman and Meyers as well as the need for the instantly claimed products and methods.” EX1004, 548-550.

vi. October 27, 2022 Non-Final Rejection and Response

71. I have been informed and understand that the Examiner issued a Non-Final Rejection on October 27, 2022, which rejected claims 1-9 for nonstatutory double patenting over copending Application No. 17/198,669 (the “’669 Application”), which the Examiner stated “claims a high purity liquid composition comprising a compound having the formula $(CH_2)_5CHSn(N(CH_3)_2)_3$ and no more than 4 mole% dialkyltin compounds as an impurity,” and is “much more specific” than the current claims, so “a patent to the genus would improperly extend the right

to exclude granted by a patent to the species should the genus issue as a patent after the species.” EX1004, 465-473.

72. I have been informed and understand that the Examiner maintained the obviousness rejection over Deelman together with Meyers, and stated the “declaration under 37 CFR 1.132 is insufficient to overcome the rejection...because...[it] does not give information regarding the test conditions of the process, such as reaction temperature and time and the neutralizing reaction taught in Deelman. It is unclear if the differences in the reaction conditions will result in differences in the results as the same test conditions from Deelman et al have not been described in the process.” EX1004, 471-472.

73. I have been informed and understand that the Applicant conducted a telephonic interview with the Examiner on February 17, 2023. EX1004, 461-462. I have been informed and understand that the summary states “Applicant provided a proposed declaration and arguments regarding the information in the declaration. The Examiner explained that time was needed to review the proposed declaration and would get back to Applicant. On 2/21/23 the Examiner called Applicant to inform the Applicant, that if they submit the declaration on the record, this would overcome the previous 103 rejection.” EX1004, 461-462.

74. I have been informed and understand that Applicant filed a Terminal Disclaimer to overcome the nonstatutory double patenting rejection over the ’669

Application on February 24, 2023, which was approved that day. EX1004, 454-455, 458.

75. I have been informed and understand that on February 24, 2023, Applicant filed a Response to Non-Final Office Action, submitting a revised Declaration Under 37 C.F.R. § 1.132 (the “2023 Declaration”). EX1004, 441-450. I have been informed and understand that the response states the “in-house experiment” “followed the general procedure of Example 2 of Deelman[]” and the 2023 Declaration “discloses details of the test conditions that are consistent with” Example 2 of Deelman. EX1004, 444. Specifically, the experiment purportedly “modifies the procedure disclosed by Deelman by reacting an ‘RSn(Hal)₃’ reagent (*i.e.*, n-BuSnCl₃) with an ‘H-T’ reagent that is not taught by Deelman” because, according to the Applicant, “Deelman does not teach reacting an RSn(Hal)₃ with an H-NR₂ compound, and, as explained in the Declaration, the target nBuSn(NEt₂)₃ product was not formed.” EX1004, 444-445.⁴ I have been informed and understand that Applicant further argued that Meyers does not “remedy the failure of Deelman

⁴ I note that Deelman does not limit its teaching to “reacting an ‘RSn(Hal)₃’ reagent [] with an ‘H-T’ reagent,” nor does Deelman preclude first reacting the H-T reagent with an alkyl lithium base before reacting with ‘RSn(Hal)₃’ reagent. EX1007, [0023] (“As regards the ligand T, it is any ligand known in the art[.]”); EX1009, 7 (teaching reacting dimethylamine with n-butyl-lithium before reacting with trimethylchlorostannane).

to teach a monoalkyl tin triamide compound and no more than 0.5 mole% dialkyltin compounds as an impurity.” EX1004, 445.

76. The 2023 Declaration again stated that the “in-house experiment” was conducted reacting “ nBuSnCl_3 with HNEt_2 *in aqueous solution* and a subsequent neutralizing reaction with sodium hydroxide.” EX1004, 448. Example 2 of Deelman, which Applicant purported to adapt to synthesize $\text{nBuSn}(\text{NEt}_2)_3$, was directed to the synthesis of “Mono-Octyltin Tris((2-Ethylhexyl)Mercaptoacetate),” not a monoalkyl tin triamide compound. EX1007, [0190]-[0192].

77. Again, in my opinion, the Applicant’s statements regarding its supposed attempts to conduct the reactions described in Deelman to obtain $\text{nBuSn}(\text{NEt}_2)_3$ are misleading. A POSITA would have understood that the reactions for production of monoalkyltin triamide compounds according to Deelman would not efficiently proceed in an aqueous mixture, and to the extent the reaction was even able to proceed in an aqueous mixture at all, the purity of the resulting organic compound would be very low. Thus, because the Applicant attempted to conduct the experiment “in aqueous solution,” a POSITA would not expect the resulting compound to be the target $\text{nBuSn}(\text{NEt}_2)_3$ product.

vii. March 23, 2023 Notice of Allowance

78. I have been informed and understand that on March 23, 2023, the “Examiner called” Applicant “to request a Terminal Disclaimer to put the case in condition for allowance.” EX1004, 363.

79. I have been informed and understand that Applicant filed a Terminal Disclaimer to U.S. Patent No. 10,787,466 on March 23, 2023, which was accepted the same day. EX1004, 398, 402.

80. I have been informed and understand that on March 23, 2023, a Notice of Allowance issued with an Examiner’s Amendment rejoining claim 11. EX1004, 355-362.

81. I have been informed and understand that the Examiner provided the following Reasons for Allowance:

The obviousness rejection over Deelman et al in view of Meyers et al is withdrawn in view of Applicants arguments. The declaration under 37 CFR 1.132 filed February 24, 2023 is sufficient to overcome the rejection of claims 1-9 based upon the obviousness rejection over Deelman et al in view of Meyers. As Applicants argues and have shown in the declaration, the method of Deelman et al would not produce the claimed invention.

EX1004, 361.

VI. LEVEL OF ORDINARY SKILL IN THE ART

82. I have been informed and understand that that a POSITA is a hypothetical person who is presumed to know the relevant prior art. I have been

informed and understand that that the actual inventor's skill is not determinative of the level of ordinary skill. I further I have been informed and understand that that factors that may be considered in determining level of skill include: (i) type of problems encountered in art; (ii) prior art solutions to those problems; (iii) rapidity with which innovations are made; (iv) sophistication of the technology; and (v) educational level of active workers in the field. I have been informed and understand that that not all such factors may be present in every case, and one or more of them may predominate.

83. In my opinion, a POSITA at the time of invention of the '903 Patent would typically have had a Bachelor of Science in Chemistry, or an equivalent field, as well as four years of experience in the synthesis of organometallic compounds. Persons with a higher level of education but less practical experience can also meet this requirement. Around the time of invention (*i.e.*, April 11, 2018), I was familiar with this level of skill for organometallic chemistry because I already had a Ph.D. in chemistry and have worked and taught in the field for over thirty years.

VII. CLAIM CONSTRUCTION

84. I have been informed and understand that "claim construction" is the process of determining a patent claim's meaning. I have been informed and understand that, during an *inter partes* review proceeding, a claim construction analysis begins with the plain meaning of the claim term, which is the ordinary and

customary meaning given to the term by those of ordinary skill in the art at the time of the invention. I have been informed and understand that that the claims of a patent must be interpreted in light of the specification—that is, the claim language itself, the written description, and the figures in the patent—as well as the patent’s prosecution history and/or any reexamination history.

85. I am informed and understand that Patent Owner in a pending district court proceeding took the position that the ordinary, customary meaning should apply for all relevant terms of the ’903 Patent. For purposes of this declaration, I adopt Patent Owner’s claim construction positions and interpret all claim terms in accordance with their ordinary and customary meaning.

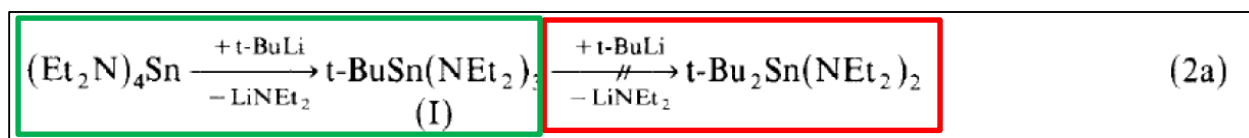
VIII. SUMMARY OF PRIOR ART

86. As I explained above (§ IV.A), the claimed monoalkyl tin triamide compounds were known in the art and efficient reaction pathways to arrive selectively at the claimed monoalkyl tin triamide compounds were also well-established long before the alleged invention of the ’903 Patent. § IV.A. I summarize below a few references that illustrate such reaction pathways known to a POSITA prior to the alleged invention. The purity of such monoalkyl tin triamide compounds can be further improved with known separation techniques including fractional distillation.

A. Hänssgen (EX1005)

87. “Hänssgen,” a paper entitled “Synthesis of The First Mono-t-Butyltin Element Compounds,” by D. Hänssgen, H. Puff, and N. Beckerman was published in the Journal of Organometallic Chemistry in 1985. EX1005; EX1008 (Hall-Ellis Decl.), ¶¶ 48, 50, 54, 64. As such, Hänssgen predates the '903 Patent by over three decades and I understand that Hänssgen is prior art.

88. Hänssgen describes the synthesis of “t-Butyl-tris(diethylamino)stannane (I)” by “Sn-alkylation of tetrakis(diethylamino)stannane with lithium-t-butyl,” as highlighted in green in the following reaction:

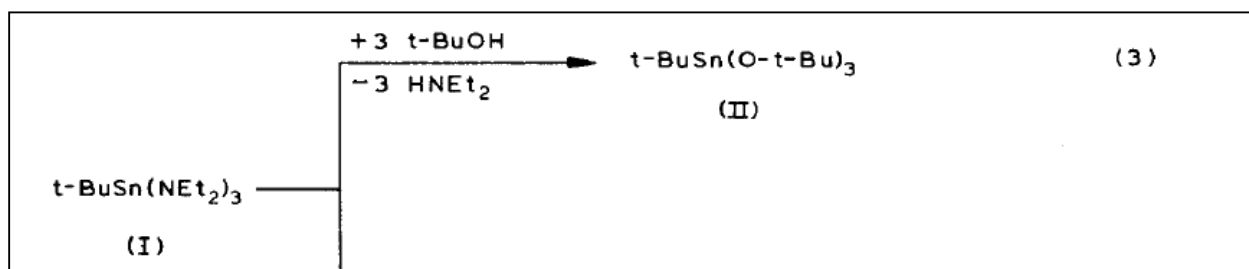


EX1005, 2.

89. Hänssgen states that the “reaction proceeds practically quantitatively; there are no indications of secondary reactions.” EX1005, 2. In the above chemical reaction (2a), highlighted in red, Hänssgen indicates that the reaction does not proceed further to generate the dialkyltin compound.

90. Hänssgen further provides that “lithium diethyl amide can be separated from the reaction mixture by precipitation with benzene and analytically pure [compound] I can be obtained by fractiona[l] distillation of the benzene phase.” EX1005, 2.

91. In addition, Hänssgen discloses further derivatization of monoalkyl tin triamides by reacting monoalkyl tin triamides with an alcohol in an organic solvent to form monoalkyl tin trialkoxides. Specifically, Hänssgen teaches the “alcoholysis of [compound] (I),” which is $t\text{-BuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3, results in the ‘displacement reaction’ [6] typical for aminostannanes to trialkoxystannane (II).” EX1005, 3. Equation 3 is shown below:



EX1005, 3.

B. Meyers (EX1006)

92. “Meyers” is United States Patent Application No. 2016/0116839. Meyers is entitled “Organometallic Solution Based High Resolution Patterning Compositions and Corresponding Methods.” Meyers was filed October 22, 2015 and published April 28, 2016, which is more than 1 year prior to the earliest filing date of the application that led to the ’903 Patent (April 11, 2018). As such, I understand Meyers is prior art to the ’903 Patent. I have been informed and understand that Patent Owner did not dispute Meyers’s status as prior art during prosecution of the ’903 Patent.

93. Meyers teaches “precursor solutions that can be deposited to form organometallic coatings that can be patterned with very high resolution with radiation.” EX1006, [0002].

94. Meyers provides several examples of potential precursor solutions that can be used in the disclosed coatings and methods. Specifically, Example 7 of Meyers teaches preparation of “a water-reactive precursor, isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃)” by mixing LiNMe₂ and anhydrous hexanes, and then adding i-PrSnCl₃ by addition funnel to the reaction flask dropwise over a period of two hours. EX1006, [0150]-[0151]. After the reaction was brought to room temperature, the reaction mixture was filtered, the solvent was removed and the residue distilled. EX1006, [0151]. ¹H and ¹¹⁹Sn NMR of the distillate in a C₆D₆ solvent “match[ed] the predicted spectra for i-PrSn(NMe₂)₃” and the “primary ¹¹⁹Sn resonance” was “consistent with a major product with a single tin environment, with a chemical shift comparable to reported monoalkyltin amido compounds.” EX1006, [0151].

95. Meyers further recognized a need for high purity monoalkyltin compounds: “As described herein, low metal contamination synthesis approaches can be accomplished with appropriate alternative hydrolysis approaches and high-purity alkyltin precursors.” EX1006, [0063].

C. Deelman (EX1007)

96. “Deelman” is U.S. Patent Application No. 2011/0166268. Deelman is entitled “High Purity Monoalkyltin Compounds and Uses Thereof.” Deelman was filed as a PCT application on May 14, 2009, has a 371 date of January 28, 2011, and was published July 7, 2011, which is prior to the earliest filing date of the application that led to the '903 Patent. Given the publication date, I have been informed and understand that Deelman is prior art to the '903 Patent. I have been informed and understand that Patent Owner did not challenge Deelman's status as prior art during prosecution of the '903 Patent.

97. Deelman teaches “high purity monoalkyltin compounds” and specifically “alkyltin compound compositions containing monoalkyltin as a major compound, and minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract. Deelman further teaches that “high purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.” EX1007, [0010].

98. Deelman explains “[s]electively produced mono-alkyltin trihalides have been found to be easily converted to mono-alkyltin compounds of formula RSn(T)_3 by reacting mono-alkyltin halides RSn(Hal)_3 with a compound of formula H-T in the presence of a suitable base.” EX1007, [0031]. “[T]he monoalkyltin

trihalides can be separated from the reaction medium by...distillation, solvent extraction, crystallization, and the like.” EX1007, [0057].

99. The “Detailed Description of the Invention” states that the monoalkyltin compound compositions may have “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula RSn(T)_3 , in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]. “As regards the ligand T, it is any ligand known in the art[.]” EX1007, [0023]. In another aspect, Deelman states the composition can comprise a “weight ratio of (mono-alkyltin compound)/(di-alkyltin compound) of not less than 90/10, preferably of not less than 95/5, most preferably of not less than 97/3.” EX1007, [0017].

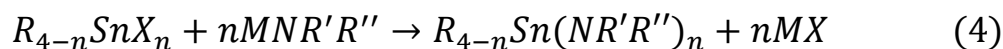
100. Deelman teaches that the compositions can include “organic, aprotic or even protic solvents,” including ethers and alcohols, and specifically lists “tetrahydrofuran (THF), ethanol, and 1,2-dimethoxyethane (DME)” as suitable solvents. EX1007, [0052].

D. Jones (EX1009)

101. “Jones,” a paper entitled “Amino-derivatives of Metals and Metalloids. Part I. Preparation of Aminostannanes, Stannylamines, and Stannazanes,” by K. Jones and M.F. Lappert was published in the *Journal of the Chemical Society* in 1965. EX1009; EX1008 (Hall-Ellis Decl.), ¶¶ 57, 59, 63, 64. As such, I understand

that Jones predates the '903 Patent by nearly five decades and is prior art to the '903 Patent.

102. Jones discloses that a “representative series of amino-derivatives of tin(IV) have been prepared,” including “aminostannanes $R_n\text{Sn}(\text{NR}'_2)_{4-n}$.” EX1009, 3-4. Specifically, Jones teaches preparation of an “aminostannane [see reaction (3)], by way of the lithium salt of the amine. We have extended reaction (3) [see reaction (4), R, R', and R'' = alkyl or aryl, X = Cl or Br, M = Li, and n = 1, 2, 3, or 4] to the preparation by transmetallation of numerous derivatives.” EX1009, 4. The relevant chemical reactions taught by Jones are as follows:⁵



EX1009, 4.

IX. SUMMARY OF PRIOR ART GROUNDS⁶

Ground	Claims	Statutory Basis (35 U.S.C. §)	Prior Art
1A	1-4, 6-10	102	<i>Hänssgen</i>

⁵ I note that there are two typographical errors in reaction (4) that I have corrected herein: (a) molecular formula $R_4\text{SnX}_{4-n}$ should be instead be $R_{4-n}\text{SnX}_n$, because it would have been clear to a POSITA that the subscripts are incorrect given the mass balance requirement and description of the text; and (b) Li should instead be M, as consistent with the immediately following text description of reaction (4)—“in some cases with M = Na, K, or R'''Mg.” EX1009, 4.

⁶ I note that Grounds 1B, 2A, 2B, 3A, and 3B present different combinations than the prior art described and purportedly replicated in three inventor declarations submitted during prosecution of the '903 Patent.

1B	1–4, 6–10	103	<i>Hänssgen together with the knowledge of a POSITA</i>
2A	1–4, 6	103	<i>Meyers together with Deelman</i>
2B	1-4, 6-10	103	<i>Meyers together with Deelman and Hänssgen</i>
3A	1–4, 6	103	<i>Deelman together with Jones</i>
3B	1–4, 6-10	103	<i>Deelman together with Jones and Hänssgen</i>

X. EXPERIMENTS CONDUCTED UNDER MY SUPERVISION

103. As I explained above (§ IV.A), monoalkyl tin triamide compounds were well-known long before the alleged invention. Indeed, I have been informed and understand that Patent Owner did not dispute that the claimed monoalkyl tin triamide compounds were known during prosecution of the '903 Patent, but instead argued that “at the time of filing the present application, the claimed purity of the monoalkyltin compounds was not achievable through the use of an alkyl lithium reaction, through the use of an alkyl lithium reaction with a subsequent conventional distillation step, or through other synthesis and/or purification processes that were then known in the art.” EX1004, 891. To that end, a named inventor of the '903 Patent, Dr. Joseph B. Edson, submitted three declarations citing in-house experiments to rebut the prior art references cited by the Examiner, including

Hänssgen, Deelman, and Meyers. EX1004, 896-898 (1/19/2021 Edson Declaration), 551-553 (9/16/2022 Edson Declaration), 447-450 (2/24/2023 Edson Declaration).

104. But, as I explained above (§§ IV.A. and VIII.A through VIII.D), the reaction pathways that can selectively lead to high purity monoalkyl tin triamide were well-known, including those identified in Hänssgen, Deelman, and Meyers. Based on my review of the inventor declarations (EX1004, 896-898 (1/19/2021 Edson Declaration), 551-553 (9/16/2022 Edson Declaration), 447-450 (2/24/2023 Edson Declaration)) and the prior art references, it is clear to me that the inventor failed to follow proper protocols and, because of that, he was unable to reproduce the compound with desired purity.

105. As I explained above (§§ V.B.ii. through V.B.vii.), the conditions used in the inventor's first declaration lack sufficient details to allow a POSITA to assess the appropriateness of the conditions. Specifically, the 2021 Declaration describes in a single paragraph (¶ 8) that the inventor allegedly recreated the procedure of Hänssgen without disclosing any specific reaction conditions that were used to achieve the purity level that is below that reported by Hänssgen. EX1004, 896-898.

106. Next, the conditions used in the in-house experiments described in the inventor's second and third declarations, the 2022 and 2023 Declarations, contradict what a POSITA would have applied and were apparently designed to lead to failures of the experiments. In both the 2022 and 2023 Declarations the inventor attempted

to adapt the synthesis procedures described in Deelman to synthesize a monoalkyltin triamide compound. However, the inventor followed a protocol to synthesize a different compound, and conducted the experiment in aqueous medium. Specifically, Example 2 of Deelman, which the inventor purported to adapt, does not relate to synthesis of moisture sensitive monoalkyltin triamide compounds, but an entirely different category of compound, namely mono-octyltin tris((2-ethylhexyl) mercaptoacetate), which a POSITA would recognize is stable in aqueous medium. A POSITA would recognize that the reaction to synthesize a monoalkyltin triamide compound, on the other hand, would not proceed efficiently in an aqueous mixture because the monoalkyltin triamide compound could also react with water. In fact, the Meyers reference cited by the Examiner, of which the Dr. Edson (the declarant) is also a named inventor, expressly teaches synthesis of a monoalkyltin triamide in “anhydrous” (*i.e.*, water free) medium using a monoalkyltin trihalide as the starting point, consistent with synthesis approach disclosed in Deelman. EX1006, [0151]. Given this known reactivity of monoalkyltin triamide with water, no POSITA would have chosen an aqueous solution for its synthesis, and PO’s in-house experiments were based on a flawed methodology.

107. A POSITA would also have understood that the monoalkyl trihalide (the starting compound disclosed by Deelman, as explained below (*see* § XIII.A)) could react with the water to form a hydrolysate, thereby preventing formation of a

monoalkyltin triamide compound. In fact, prior work of the Patent Owner confirms that the inventor was aware of such a possibility. Specifically, Dr. Edson's 2023 Declaration cites to prior work by the Patent Owner, U.S. Patent No. 10,642,153 (including Dr. Edson as a named inventor), at Examples 1 and 2, which confirm, respectively, that both t-butyl tris(diethylamido)tin ($\text{t-BuSn}(\text{NEt}_2)_3$) and i-propyltin trichloride (i-PrSnCl_3) react with water to form a hydrolysed compound. EX1004, 448; EX1018, 27:16-28:13 (U.S. Patent No. 10,642,153). Thus, the Patent Owner should have been aware that the conditions used in the experiments submitted to the Patent Office were likely to generate a different compound, and were not likely to generate the target monoalkyltin triamide compound.

108. Thus, because the inventor attempted to conduct the experiment "in aqueous solution"—in spite of a clear understanding that the presence of water could cause a different reaction—a POSITA would not expect the resulting compound to show the predicted peaks for a monoalkyltin triamide compound during NMR analysis.

109. Accordingly, in my opinion, Patent Owner's in-house experiments are irrelevant to a POSITA's understanding of the teaching of the Deelman patent regarding the synthesis of monoalkyltin triamide, and also not relevant to whether the approach disclosed in Deelman is able to achieve the claimed the purity levels.

110. I reviewed the synthesis steps disclosed in Hänssgen, Deelman, and Meyers, and directed experiments to replicate the synthesis methods disclosed in the prior art. Together with the instruments required and skilled operators needed to replicate these organometallic compound synthesis methods, I used Petitioner's facilities and its personnel to conduct experiments under my direction and supervision.

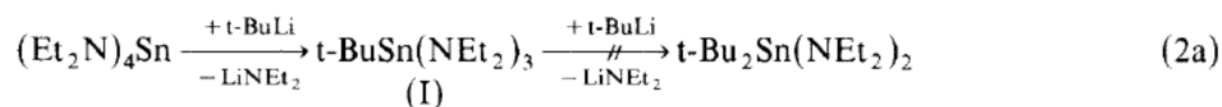
111. I provided instructions to Dr. Drew Hood of Entegris, a qualified chemist, to conduct the experimental steps disclosed in Hänssgen to reproduce the synthesis of t-butyl-tris(diethylamido)tin (*i.e.*, $\text{tBuSn}(\text{NEt}_2)_3$), and separately to conduct the synthesis methods disclosed in Example 7 of Meyers to produce isopropyl-tris(dimethylamido)tin (*i.e.*, $\text{iPrSn}(\text{NMe}_2)_3$). More specifically, I discussed the availability of chemical reagents and instruments with Dr. Hood, prepared general synthesis procedures according to the prior art disclosures, and provided them to Dr. Hood. EX1019 (Procedures for Synthesis of $\text{iPrSn}(\text{NMe}_2)_3$) and EX1020 (Procedures for Synthesis of $\text{tBuSn}(\text{NEt}_2)_3$). I explained these procedures to Dr. Hood both remotely and in-person, inspected the synthesis system that Dr. Hood had set up according to my procedures, and further oversaw his work on conducting the synthesis methods and characterizing the reaction products with NMR. Dr. Hood prepared a declaration that summarizes the synthesis methods and the characterization results in detail. EX1003. I have thoroughly reviewed Dr. Hood's

declaration and confirm that Dr. Hood's declaration accurately reflects the experiments conducted under my supervision to the best of my knowledge.

112. I further explain below the experimental procedures and how they conform to the teachings of the prior art.

A. Synthesis of tBuSn(NEt₂)₃

113. As I explained in § VIII.A. above, Hänssgen discloses a reaction pathway to synthesize tBuSn(NEt₂)₃ selectively. EX1005, 2. Hänssgen teaches: "The reaction of (Et₂N)₄Sn with equimolar quantities of lithium-t-butyl, by splitting a tin-nitrogen bond according to equation 2a, yields triamino-t-butylstannane (I) and lithium diethylamide." EX1005, 2.



EX1005, 2. Hänssgen further explains that the reaction between (Et₂N)₄Sn and lithium-t-butyl "proceeds practically quantitatively" and "there are no indications of secondary reactions." EX1005, 2. Accordingly, equation (2a) shows that this reaction stops at monoalkyl tin triamide (I) and does not proceed with a further alkylation reaction. In other words, this reaction does not lead to a dialkyl tin diamide compound under the reaction conditions provided. Hänssgen explains that this inhibition of the secondary reaction is due to the fact that "[t]hree Et₂-N groups and the (sterically even more exacting) t-butyl group shield the coordination sphere of I

so strongly that no two-stage substitutions occur.” EX1005, 3. Hänssgen further explains that even “reactions of $(\text{Et}_2\text{N})_4\text{Sn}$ with lithium-*t*-butyl in excess under moderate conditions always yield only the mono-substitution product I.” EX1005, 3. A POSITA would have understood that this reaction pathway naturally leads to the monoalkyltin triamide compound with no or negligible amount of dialkyltin impurity.

114. Accordingly, I provided instructions for Dr. Hood to replicate reaction (2a) of Hänssgen to confirm Hänssgen’s finding. Because Hänssgen discloses both using equimolar quantities of lithium-*t*-butyl and using an excess amount of lithium-*t*-butyl, experiments were replicated under both conditions, and Dr. Hood reported the results under (1) “Synthesis of $\text{tBuSn}(\text{NEt}_2)_3$ using 1eq of tBuLi ” and (2) “Synthesis of $\text{tBuSn}(\text{NEt}_2)_3$ using 1.2eq of tBuLi .” EX1003, §§II.b and II.c.

115. The experimental procedures that I provided closely follow the detailed experimental conditions disclosed in Hänssgen, including using the same amounts of reagents. *Compare* EX1020 *with* EX1005. The relevant experimental portion of Hänssgen is reproduced below:

t-butyl-tris(diethylamino)stannane (I)

To a solution of 4.08 g (10 mmol) $(\text{Et}_2\text{N})_4\text{Sn}$ in 50 ml diethyl ether, at -78°C under agitation, 6.2 ml of a 1.6 M solution of t-BuLi was added by drops into *n*-pentane. It was allowed to come to room temperature and agitated for an additional 2 h. The ether was distilled off and the residue was dissolved in 30 ml benzene. The precipitated

LiNEt₂ was filtered out and the benzene phase was fractionated in a vacuum.

Yield 3.68 g (93% theoretical density); viscous, pale yellow oil, boiling point 122°C (2 mbar).

EX1005, 4. Because benzene is no longer used in the industry as an organic solvent due to its carcinogenic effect, it was replaced with diethyl ether, a solvent commonly used in organometallic compound synthesis that can serve a similar purpose. I note that, in Hänssgen, benzene was merely used to extract the reaction product, *i.e.*, *t*-butyl-tris(diethylamino)stannane, and thus changing it to diethyl ether for extraction purposes would not have impacted the reaction in anyway.

116. Following my general experimental procedures, Dr. Hood performed the experiments using standard equipment and exercising precautions as a POSITA would. He recorded detailed experimental steps that were reported in his declaration. EX1003, §§II.b and II.c. Because a larger quantity of products would be necessary for further fractional distillation, Dr. Hood performed a simple vacuum distillation to collect the target compound but no attempt was made to further purify the reaction products with fractional distillation. As I explained above, additional fractional distillation of the final product would have further increased the purity of the monoalkyltin triamide product and lowered the amount of dialkyltin impurity. I further oversaw NMR measurement of the reaction products and reviewed the NMR analysis results, which I agreed with. These results are again presented in Dr. Hood's declaration.

117. The NMR analysis shows relatively simple ^{119}Sn spectra, as expected, since each organotin compound of the reaction mixture only contains a single tin atom. EX1003, ¶¶ 9-10, Figs. 5-6, 15-16.⁷ The relative molar percentage values are directly calculated from the ^{119}Sn NMR peak areas of an individual chemical species over the total peak area because the signal strength is directly proportional to the number of ^{119}Sn atoms detected. The peak assignments are also straightforward since spectra contain only a few NMR peaks, separated apart from each other.⁸ The chemical shift values are consistent with values reported in the literature, the expected trend between different organotin compounds, and chemical shifts determined from reference samples. *See, e.g.*, EX1003, ¶¶ 9-10, Figs. 5-6, 15-16; EX1021, 2-3 (M. Froneman, T. Modro and S. M. Vather, “Dialkylamidoderivatives of Sn(II) and Sn(IV) in Phosphorus Chemistry,” 164 *Inorganica Chimica Acta*, pp. 17-21 (1989)) (disclosing ^{119}Sn chemical shift of $\text{Sn}(\text{NEt}_2)_4$ at -120 ppm); EX1022, [0102] (U.S. Patent Application No. 2022/0064192A1) (disclosing ^{119}Sn chemical shift of $\text{tBuSn}(\text{NEt}_2)_3$ at -81.69 ppm).

⁷ ^{119}Sn spectra were collected at 149.1 MHz using proton decoupling with a neat sample and averaged over 10240 scans to maximize the signal to noise ratio for reliable quantitation. EX1003, ¶10. The detection limit obtained in Dr. Hood’s measurements are better than the detection limit of 0.1 mole % as claimed in claim 6 of the ’903 Patent, judging from the background noise level of the spectra in comparison to the impurity signal.

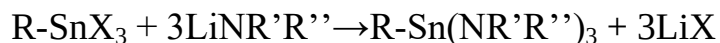
⁸ I note that the intense peak possesses a number of defined satellite peaks, which are included in the integration of the peak area.

118. Experimental results further show that the synthesis of $\text{tBuSn}(\text{NEt}_2)_3$ using 1eq of tBuLi yielded a monoalkyltin product with only 0.17 mole % dialkyltin impurity $\text{tBu}_2\text{Sn}(\text{NEt}_2)_2$ and the synthesis of $\text{tBuSn}(\text{NEt}_2)_3$ using 1.2 eq of tBuLi (*i.e.*, 20% excess of tBuLi) led to only 0.34 mole % $\text{tBu}_2\text{Sn}(\text{NEt}_2)_2$. EX1003, ¶¶ 17-18, 24-25. This is consistent with the disclosure of Hänssgen that the reaction between $(\text{Et}_2\text{N})_4\text{Sn}$ and tBuLi leads almost exclusively to the monoalkyltin triamide compound without appreciable amount of dialkyltin impurity, in this case, less than 0.5 mole % relative to the total tin amount. Thus, an excess amount of tBuLi does not appreciably lead to secondary reactions, while it improves the conversion rate of the starting $\text{Sn}(\text{NEt}_2)_4$ compound. EX1003 ¶¶ 17-18, 24-25.

B. Synthesis of $\text{iPrSn}(\text{NMe}_2)_3$

119. As I explained in § VIII.B above, Meyers discloses the synthesis of monoalkyltin triamides by reacting monoalkyltin trihalide with lithium amides, a chemical reaction well-known in the art. For example, Example 7 of Meyers provides a detailed description of the method for synthesizing isopropyl tris(dimethylamido)tin (*i.e.*, $\text{i-PrSn}(\text{NMe}_2)_3$) by reacting LiNMe_2 with i-PrSnCl_3 . EX1006, [0150]-[0151]. As further explained in § VIII.C. above, ultra-high purity monoalkyltin trihalides (*e.g.*, 99.999 wt%) can be prepared by using the methods disclosed in Deelman. EX1007, [0013]. It is my opinion that, for a POSITA interested in a high purity monoalkyl tin triamide, it would have been obvious to

start with high purity monoalkyltin trihalides and replace the halides with amide groups to arrive at the high purity monoalkyltin triamides as shown in the following chemical reaction:



where R, R', and R'' are alkyl or aryl groups. *See, e.g.*, EX1009. The reaction is a well-defined exchange pathway in which the highly nucleophilic amide ion displaces the halide ion, and thus, when properly controlled, would lead to high purity monoalkyltin triamides.

120. To demonstrate the viability of using this known reaction pathway to achieve high purity monoalkyltin triamide, I provided instructions for Dr. Hood to first replicate the synthesis procedures described in Example 7 of Meyers. EX1003, ¶¶ 8, 27-37. The resulting products contain over 93% of monoalkyltin triamide but also include a small quantity of dialkyltin diamide impurity due to secondary reactions. EX1003, ¶¶ 36-37. It is apparent that Example 7 of Meyers was not optimized for achieving high purity monoalkyltin triamide, but instead focused on the hydrolysis reactions of the compound. EX1006, [0152] (“An i-propyltin oxide hydroxide hydrolysate was prepared from the i-propyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃), via H₂O hydrolysis using two different methods.”); *see also* EX1006, [0150-0154].

121. Accordingly, I instructed Dr. Hood to modify the synthesis process. Namely, instead of directly adding the pure monoalkyl tin trihalide (i-PrSnCl₃) into the LiNMe₂ hexane suspension as described in Example 7 of Meyers, I asked Dr. Hood to first dilute i-PrSnCl₃ with tetrahydrofuran (“THF”), a known good solvent for monoalkyl tin trihalide, and then add the i-PrSnCl₃ THF solution to the reaction mixture. EX1003, ¶¶39-47; *see, e.g.*, EX1007, [0052] (disclosing THF as a suitable solvent for monoalkyltin trihalide). As expected, the modified process minimized secondary reactions that could convert monoalkyltin to multialkyltin compounds. EX1003, ¶¶46-47.

122. NMR analysis of the resulting compound shows that this synthesis approach yielded a monoalkyltin product (i-PrSn(NMe₂)₃) (-65.19 ppm) with only 0.19 mole % dialkyltin impurity i-Pr₂Sn(NMe₂)₂ (-19.38 ppm). EX1003, ¶¶46-47. For the same reasons explained above, peak assignments of ¹¹⁹Sn NMR spectra of the reaction products are also straightforward since the spectra contain only a few NMR peaks that are separated apart from each other.⁹ EX1003, ¶26, Figs. 26-27, Figs. 37-38. The chemical shift values are consistent with values reported in the literature, the expected trend between different organotin compounds, and chemical

⁹ I note that the intense peak possesses a number of defined satellite peaks, which are include in the integration of the peak area.

shifts determined from reference samples. *See, e.g.*, EX1003, ¶¶36-37, 46-47; EX1018, 33:9-13 (disclosing ^{119}Sn chemical shift of $i\text{-PrSn}(\text{NMe}_2)_3$ at -65.4 ppm).

XI. GROUND 1A: ANTICIPATION BY HÄNSSGEN

A. Claim 1

i. [1Pre] “A high purity liquid composition comprising:”

123. To the extent that the preamble (“a high purity liquid composition”) is limiting, Hänssgen discloses “a high purity liquid composition.” For example, Hänssgen discloses “t-Butyl-tris(diethylamino)stannane,” *i.e.*, $\text{tBuSn}(\text{NEt}_2)_3$ and referred to by Hänssgen as (I), that was obtained “by Sn-alkylation of tetrakis(diethylamino)stannane with lithium-t-butyl.” EX1005, 1, Summary. Hänssgen provides a detailed description of the reactions employed for synthesizing (I) and further explains that “analytically pure I can be obtained by fractiona[l] distillation of the benzene phase” of the reaction mixture. Moreover, Hänssgen describes this “analytically pure” compound as a “viscous, pale yellow oil” (*i.e.*, a liquid) with a “boiling point [of] 122°C (2 mbar).” EX1005, 4.

124. Accordingly, Hänssgen discloses a high purity liquid composition, namely the analytically pure $\text{tBuSn}(\text{NEt}_2)_3$.

ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and”

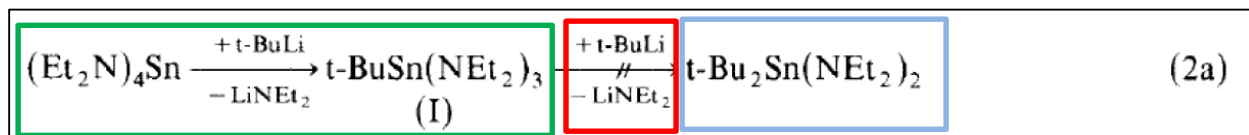
125. Hänssgen discloses “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$.” As I explained above, Hänssgen discloses the

synthesis of compound (I), which has the chemical formula of $t\text{BuSn}(\text{NEt}_2)_3$. EX1005, 2. This chemical formula corresponds to a monoalkyl tin triamide compound of composition $\text{RSn}(\text{NR}'_2)_3$, where R corresponds to the t-butyl hydrocarbyl group and R' corresponds to the ethyl hydrocarbyl group.

126. Accordingly, Hänssgen discloses “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$.”

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

127. Hänssgen discloses “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.” Hänssgen teaches that the “reaction of $(\text{Et}_2\text{N})_4\text{Sn}$ with equimolar quantities of lithium-t-butyl, by splitting a tin-nitrogen bond according to equation 2a, yields triamino-t-butylstannane (I) and lithium diethyl amide,” as shown in the below chemical equation and highlighted in green:



EX1005, 2.

128. As I explained above, a POSITA would have understood that equation 2a above also shows that the chemical reaction does not proceed to generate the dialkyl tin compound, *i.e.*, $\text{t-Bu}_2\text{Sn}(\text{NEt}_2)_2$ (highlighted in blue), as indicated by the double hash marks through the line above, highlighted in red. Hänssgen confirms this understanding, stating that equation 2a “proceeds practically quantitatively;

there are no indications of secondary reactions.” EX1005, 2. Thus, a POSITA would have understood that equation 2a shows there is no or negligible dialkyltin impurity in the reaction product, and therefore discloses claim limitation [1b].

129. Hänssgen further confirms that “analytically pure I [tBuSn(NEt₂)₃] can be obtained by fractiona[l] distillation of the benzene phase.” EX1005, 2. A POSITA would have understood “analytically pure I [tBuSn(NEt₂)₃]” to mean no or negligible dialkyltin impurity in the reaction product.

130. Moreover, as I explained above in § X.A., I directed and supervised experiments to replicate experimental processes disclosed in Hänssgen, including the reaction of (Et₂N)₄Sn with equimolar quantities of lithium-t-butyl and, alternatively, with 20% excess of lithium-t-butyl. The results show that the synthesis of tBuSn(NEt₂)₃ using 1eq of tBuLi yielded a monoalkyltin product with only 0.17 mole % dialkyltin impurity tBu₂Sn(NEt₂)₂ relative to the total tin amount, which is within the scope of claim limitation [1b]. EX1003, ¶¶17-18. Even when an excess amount of tBuLi (*i.e.*, 20% excess of tBuLi) is used, the reaction product contains only 0.34 mole % tBu₂Sn(NEt₂)₂ relative to the total tin amount. EX1003, ¶¶24-25.

131. Accordingly, based on the above teachings of Hänssgen and the experiments conducted by Dr. Hood at my direction, a POSITA would have understood that the no or negligible dialkyltin impurity taught by Hänssgen is within

the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

132. Hänssgen discloses “R is a hydrocarbyl group with 1-31 carbon atoms.”

Hänssgen discloses the synthesis of compound (I), which has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2. Tert-butyl (tBu) is a hydrocarbyl group with four (4) carbon atoms, which is within the claimed range of 1-31 carbon atoms.

133. Accordingly, Hänssgen discloses “wherein R is a hydrocarbyl group with 1-31 carbon atoms.”

v. [1d] “wherein R’ is a hydrocarbyl group with 1-10 carbon atoms.”

134. Hänssgen discloses “R’ is a hydrocarbyl group with 1-10 carbon atoms.” Hänssgen discloses the synthesis of compound (I), which has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2. Ethyl (Et) is a hydrocarbyl group with two (2) carbon atoms, which is in the claimed range of 1-10 carbon atoms.

135. Accordingly, Hänssgen discloses wherein R’ is a hydrocarbyl group with 1-10 carbon atoms.

B. Claim 2

i. [2pre] “The composition of claim 1”

136. Hänssgen discloses the composition of claim 1 for the reasons set forth in XI.A.

- ii. [2a] “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

137. Hänssgen discloses “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.” Hänssgen discloses the synthesis of compound (I), which has the chemical formula of $tBuSn(NEt_2)_3$. EX1005, 2. Tert-butyl (tBu) is a branched alkyl ligand R represented by $R^1R^2R^3C—$ where R^1 and R^2 are independent alkyl groups with one (1) carbon atom each, and R^3 is an alkyl group with one (1) carbon atom, each of which is within the claimed range of carbon atoms.

138. Accordingly, Hänssgen discloses wherein R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.

C. Claim 3

- i. [3pre] “The composition of claim 1”

139. Hänssgen discloses the composition of claim 1 for the reasons set forth in XI.A.

- ii. [3a] “wherein R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH_2—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

140. Hänssgen discloses “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$), sec-butyl ($\text{CH}_3(\text{CH}_3\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.” Hänssgen discloses the synthesis of compound (I), which has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2. Thus, Hänssgen discloses that R comprises tert-butyl, which is within the scope of claim limitation [3a].

D. Claim 4

i. [4pre] “The composition of claim 1”

141. Hänssgen discloses the composition of claim 1 for the reasons set forth in XI.A.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

142. Hänssgen discloses “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.” Hänssgen discloses the synthesis of compound (I), which has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2. Thus, Hänssgen discloses that R’ comprises ethyl, which is within the scope of claim limitation [4a].

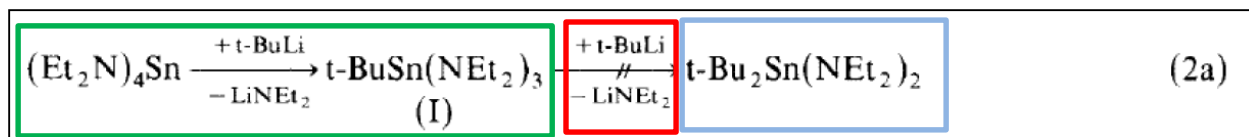
E. Claim 6

i. [6pre] “The composition of claim 1”

143. Hänssgen discloses the composition of claim 1 for the reasons set forth in XI.A.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

144. Hänssgen discloses the composition “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.” Hänssgen teaches that the “reaction of $(\text{Et}_2\text{N})_4\text{Sn}$ with equimolar quantities of lithium-t-butyl, by splitting a tin-nitrogen bond according to equation 2a, yields triamino-t-butylstannane (I) and lithium diethyl amide,” as shown in the below chemical equation and highlighted in green:



EX1005, 2.

145. A POSITA would have understood that equation 2a above also shows that the chemical reaction does not proceed to generate any dialkyl tin compound, *e.g.*, $\text{t-Bu}_2\text{Sn}(\text{NEt}_2)_2$ (highlighted in blue), as indicated by the double hash marks through the line above, highlighted in red. Hänssgen confirms this understanding, stating that equation 2a “proceeds practically quantitatively; there are no indications of secondary reactions.” EX1005, 2. Thus, a POSITA would have understood that equation 2a shows that there would have been no or negligible amount of dialkyltin impurity in the reaction product, *i.e.*, essentially no dialkyltin compounds.

146. Hänssgen further confirms that “analytically pure I [$\text{tBuSn}(\text{NEt}_2)_3$] can be obtained by fractiona[l] distillation of the benzene phase.” EX1005, 2. A POSITA

would have understood “analytically pure I [tBuSn(NEt₂)₃]” to mean no or negligible dialkyltin impurity in the reaction product.

147. Moreover, as I explained above in § X.A., I directed and supervised experiments to replicate experimental processes disclosed in Hänssgen, including the reaction of (Et₂N)₄Sn with equimolar quantities of lithium-t-butyl and, alternatively, with 20% excess of lithium-t-butyl. The results show that the synthesis of tBuSn(NEt₂)₃ using 1eq of tBuLi yielded a monoalkyltin product with only 0.17 mole % dialkyltin impurity tBu₂Sn(NEt₂)₂ relative to the total tin amount. EX1003, ¶¶17-18. Even when an excess amount of tBuLi (*i.e.*, 20% excess of tBuLi) is used, the reaction product contains only 0.34 mole % tBu₂Sn(NEt₂)₂ relative to the total tin amount. EX1003, ¶¶24-25. Given the claim language requiring “essentially no dialkyltin impurity when measured with an approximate 0.1 mole % detection limit,” in my opinion, because the impurity amounts of 0.17 mole % and 0.34 mole % determined by Dr. Hood are just slightly above the specified detection limit of approximately 0.1 mole %, such impurity levels would be barely detectable due to the background noise level if it were to be measured with an approximate 0.1 mole % detection limit.¹⁰ In other words, it can be regarded that there is essentially no

¹⁰ As I explained above, ¹¹⁹Sn spectra measurements that I oversaw used proton decoupling with a neat sample and were averaged over 10240 scans to maximize the signal to noise ratio for reliable quantitation. EX1003, ¶10. The detection limit obtained in Dr. Hood’s measurements are better than the detection limit of 0.1 mole

dialkyltin compound impurity in the reaction product if it were to be measured with an approximate 0.1 mole % detection limit.

148. Accordingly, based on the teachings of Hänssgen and the experiments conducted by Dr. Hood, a POSITA would have understood that the no or negligible dialkyltin impurity taught by Hänssgen is within the claim limitation requiring the composition with essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.

F. Claim 7

i. [7] “A solution comprising the composition of claim 1 and an organic solvent.”

149. Hänssgen discloses the composition of claim 1 for the reasons set forth in XI.A.

150. Hänssgen further discloses the composition of claim 1 in an organic solvent. For example, Hänssgen teaches a “solution of 3.93 g (10 mmol) [Compound] I in dichloromethane.” EX1005, 4. Hänssgen’s Reaction product (I) has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$ and meets every limitation of claim 1. EX1005, 2. Dichloromethane is an organic solvent. Hänssgen further teaches that t-butanol, which is an alcohol and an organic solvent, is added to the solution. EX1005, 4.

% as claimed in claim 6 of the '903 Patent, judging from the background noise level of the spectra in comparison to the impurity signal.

151. Accordingly, Hänssgen discloses a solution comprising the composition of claim 1 and an organic solvent, and anticipates claim 7.

G. Claim 8

- i. [8] “The solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”**

152. Hänssgen discloses the solution of claim 7 for the reasons set forth in § XI.F.

153. Hänssgen further discloses that the solvent comprises an alcohol solvent. For example, Hänssgen teaches t-butanol, an alcohol solvent, added to the solution including compound (I). EX1005, 4. A POSITA would have understood that the solvent includes, or “comprises” an alcohol, as t-butanol is added into the solution. Moreover, a POSITA would have understood that while the alcohol, t-butanol, can further react with the monoalkyl tin triamide in the solution over time, it exists as part of the solvent before t-butanol is fully consumed by the reactions.¹¹

154. Hänssgen further teaches a solution concentration within the range from about 0.005M to about 0.5M. For example, Hänssgen teaches a “solution of 3.93 g (10 mmol) [Compound] I in 20 ml dichloromethane.” EX1005, 4. The molar concentration of Compound I in 20 ml dichloromethane is about 0.5 M (*i.e.*, 10

¹¹ This is consistent with the '903 Patent, which refers to various alcohol reactants as solvents. EX1001, 10:6-11.

mmol/20 ml= 0.5 M)¹². After 2.2 g (30 mmol) t-butanol (the additional solvent), is added to the solution, the molar concentration decreases slightly, *i.e.*, slightly lower than about 0.5M, which is within the claimed range of about 0.005M to about 0.5M.

155. Accordingly, Hänssgen discloses solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent includes (comprises) an alcohol, and anticipates claim 8.

H. Claim 9

- i. **[9] “The solution of claim 7 wherein R comprises methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₃HC—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₃(CH₃)₂C—), sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R’ comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”**

156. Hänssgen discloses the solution of claim 7 for the reasons set forth in § XI.F.

157. Hänssgen further discloses that R includes one of the claimed listed options (“methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₃HC—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₃(CH₃)₂C—)¹³, sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl”),

¹² I note this calculation does not account for the volume from Compound I itself, and thus slightly overestimates the molar concentration of the solution, which, in any case, does not impact my conclusion herein.

¹³ I note that claim 9 appears to have an error in the chemical formula for tert-amyl, which should be (CH₃CH₂(CH₃)₂C—).

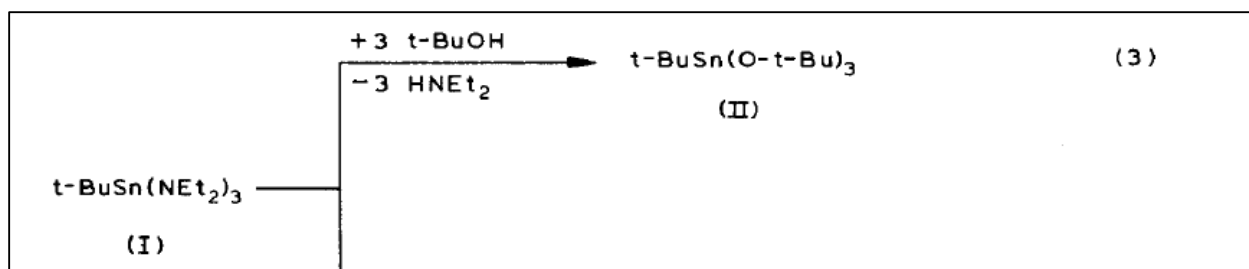
and wherein R' includes one of the claimed listed options (“a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group”). Specifically, as I explained above, Hänssgen teaches the synthesis of compound (I), which has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2. Thus, Hänssgen discloses R comprises tert-butyl and R' comprises ethyl, which are each within the scope of claim 9.

158. Accordingly, Hänssgen anticipates claim 9.

I. Claim 10

i. [10Pre] “A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination, the method comprising,”

159. To the extent the preamble (“A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination”) is limiting, Hänssgen discloses a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination. As I explained above regarding claim 1, Hänssgen teaches the synthesis of a monoalkyltin triamide compound (I) with very low dialkyl tin contamination. Hänssgen further teaches the “alcoholysis of [compound] (I),” *i.e.*, $\text{tBuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3, results in the ‘displacement reaction’ [6] typical for aminostannanes to trialkoxystannane (II).” EX1005, 3. Equation 3 is shown below:



EX1005, 3.

160. Hänssgen further provides a specific example to follow to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination: “to a solution of 3.93 g (10 mmol) in 20 ml dichloromethane, at 20°C, 2.2 g (30 mmol) t-butanol is added by drops. The solvent is evaporated and the residue heated for 4 h to 90°C. II is obtained as a colorless liquid by vacuum distillation of the residue.”

EX1005, 4. Compound II is tri-t-butoxy-t-butylstannane.

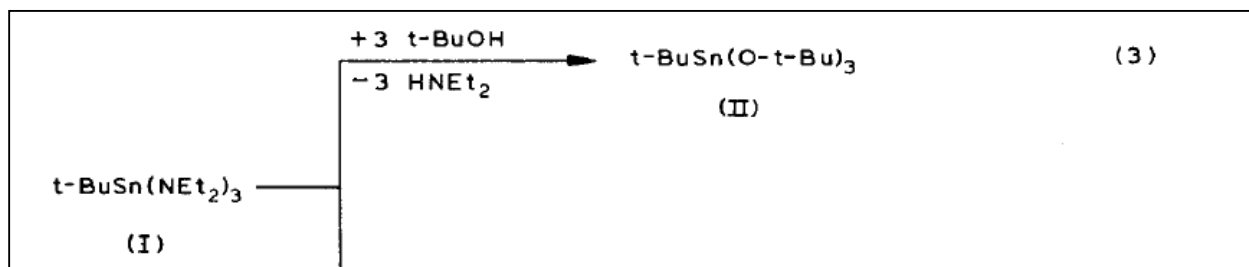
161. Because the above alcoholysis reaction impacts only the amido groups and not the alkyl group, the reaction does not change the dialkyl tin contamination level. In other words, a POSITA would have expected the resulting monoalkyltin trialkoxide compound from the alcoholysis reaction to have same low dialkyl tin contamination as the monoalkyltin triamide compound (I) does.

162. Accordingly, Hänssgen discloses a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.

- ii. **[10a] reacting the composition of claim 1 with an alcohol represented by the formula HOR'' in an organic solvent to form $\text{RSn}(\text{OR}'')_3$,**

163. Hänssgen discloses the composition of claim 1 for the reasons set forth in § XI.A.

164. As explained above, Hänssgen teaches reacting the composition of claim 1, $\text{t-BuSn}(\text{NEt}_2)_3$, with an alcohol represented by the formula HOR'' in an organic solvent to form $\text{RSn}(\text{OR}'')_3$. Specifically, Hänssgen teaches the “alcoholysis of [compound] (I),” which is $\text{t-BuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3, results in the ‘displacement reaction’ [6] typical for aminostannanes to trialkoxystannane (II).” EX1005, 3. Equation 3 is shown below:



EX1005, 3.

165. t-butanol is an alcohol represented by the formula HOR'' , wherein R'' is t-butyl, and $\text{t-BuSn}(\text{O-t-Bu})_3$ can be represented by the formula $\text{RSn}(\text{OR}'')_3$.

166. Moreover, Hänssgen teaches the reaction proceeds in an organic solvent that includes dichloromethane. EX1005, 3.

167. Accordingly, Hänssgen discloses reacting the composition of claim 1 with an alcohol represented by the formula HOR'' in an organic solvent to form $\text{RSn}(\text{OR}'')_3$.

iii. [10b] wherein R" is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,

168. Hänssgen discloses "R" is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition." As I explained above, Hänssgen teaches the "alcoholysis of [compound] (I)," which is $\text{tBuSn}(\text{NEt}_2)_3$, with "t-butanol" to synthesize $\text{t-BuSn}(\text{O-t-Bu})_3$, which is a product composition. In this chemical formula, R" corresponds to t-butyl, a hydrocarbyl group with 4 carbon atoms, which is within the claimed range of 1-10 carbon atoms.

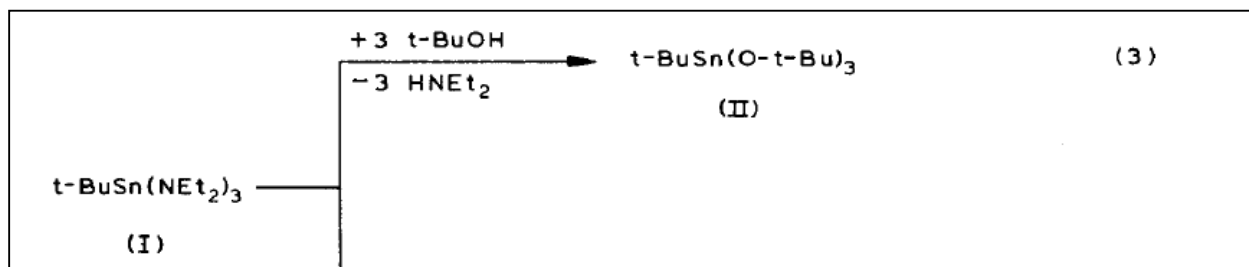
169. Accordingly, Hänssgen discloses wherein R" is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition.

iv. [10c] wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

170. Hänssgen discloses "the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin." As I explained above regarding claim 1, Hänssgen teaches a monoalkyl tin triamide composition (I) that contains "no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount." § XI.A.iii.

171. As I also explained above, Hänssgen further teaches that the "alcoholysis of [compound] (I)," which is the "analytically pure" $\text{tBuSn}(\text{NEt}_2)_3$, with "t-butanol, as described by equation 3, results in the 'displacement reaction' [6]

typical for aminostannanes to trialkoxystannane (II).” EX1005, 3. Equation 3 is shown below:



EX1005, 3.

172. Because the above alcoholysis reaction impacts only the amido groups and not the alkyl group, a POSITA would have understood the reaction does not change the dialkyl tin contamination level. In other words, the resulting monoalkyltin trialkoxide compound from the alcoholysis reaction has the same very low dialkyl tin contamination as the monoalkyltin triamide compound (I) does, and for the same reason, it meets the limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” as the monoalkyltin triamide compound (I) does.

173. A POSITA would have understood that performing the alcoholysis reaction on “analytically pure” $t\text{BuSn}(\text{NEt}_2)_3$ with no or negligible mole % dialkyltin in the reaction mixture would lead to the synthesis of $t\text{-BuSn}(\text{O-t-Bu})_3$, as shown in Equation 3, which is within the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.”

174. Accordingly, Hänssgen discloses the product composition has “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin” as required by this claim limitation. It is my opinion that Hänssgen anticipates claim 10.

XII. GROUND 1B: OBVIOUSNESS BASED ON HÄNSSGEN AND THE KNOWLEDGE OF A POSITA

175. It is my opinion that Claims 1-4 and 6-10 of the '903 Patent would have been obvious given the disclosures of Hänssgen together with the knowledge of one of ordinary skill in the art.

176. To the extent Patent Owner may argue Hänssgen does not expressly disclose any particular limitation of the claims of the '903 Patent, in my opinion, those claim limitations would have been obvious to a POSITA in light of the disclosure of Hänssgen and his or her own knowledge as I describe further below.

A. Motivation to Modify Hänssgen with the Knowledge of POSITA

177. As I explained above (§ XI), Hänssgen (EX1005) does explicitly disclose each of the claim limitations of claims 1-4 and 6-10. In particular, Hänssgen teaches that the “Sn-alkylation of tetrakis(diethylamino)stannane with lithium-t-butyl” “proceeds practically quantitatively” and “yields triamino-t-butylstannane (I)” with “no indications of secondary reactions.” EX1005, 2. Hänssgen further teaches that “analytically pure I can be obtained by fraction[al] distillation” of the reaction products. EX1005, 2. Hänssgen further observes that

“reactions of $(\text{Et}_2\text{N})_4\text{Sn}$ with lithium-*t*-butyl in excess under moderate conditions always yield only the mono-substitution product I.” EX1005, 3.

178. To the extent that Patent Owner argues that the specific purity limitations—“no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount” (as in claim limitation [1b]) and “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” (as in claim limitation [6b])—are not disclosed by Hänssgen, the purity limitations would have been obvious to a POSITA before the alleged invention of the '903 Patent (April 11, 2018).

179. A POSITA would have understood that a high purity compound is known to be desirable for many applications. *See, e.g.*, EX1007, [0010] (“[H]igh purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.”). If a higher purity level than what is achieved through the selective reactions disclosed in Hänssgen is desired, a POSITA would have been motivated to further optimize the process conditions, including by use of fractional distillation as expressly taught in Hänssgen. As I explained above, fractional distillation is an effective method to separate a mixture of similar compounds according to their different boiling points, thereby improving the purity of the main product. Indeed, the '903 Patent itself admits: “The monoalkyl tin triamides produced using the methods described above or other methods not

explicitly described herein can be further purified using fractional distillation.” EX1001, 9:39-41; *see also* EX1001, 10:39-42 (“If desired, a fractional distillation can be performed to further purify the monoalkyl tin trialkoxides from polyalkyl contaminants.”). The ’903 Patent further includes an example (Example 8) to “demonstrate[] the effectiveness of fractional distillation to purify t-BuSn(NMe₂)₃,” a compound with similar properties to t-BuSn(NEt₂)₃, which is disclosed in Hänssgen. In my opinion, the fractional distillation conditions used in Example 8 were not materially different from known fractional distillation methods at the time of the alleged invention (*i.e.*, April 11, 2018). *See, e.g.*, EX1001, 9:46-48 (“A suitable fractional distillation column can be used with a volume suitable for the process, and these are commercially available.”); § IV.B. Moreover, in my opinion, any difference between the fractional distillation used in the ’903 Patent and conventional fractional distillation methods would impact only the efficiency of the distillation process; it would not alter whether the compound mixture is separable through fractional distillation.

180. In addition, the ’903 patent neither shows nor even alleges that these recited purity limitations are critical or unexpected. As I explained above (XI.A), they are not. A POSITA would have found it obvious to further improve the purity levels of the reaction products disclosed in Hänssgen using known methods and conditions, as disclosed in Hänssgen and admitted by the ’903 Patent to arrive at the

claimed limitation with a reasonable expectation of success, as such modification involves no more than applying a known technique (fractional distillation) to a known product (monoalkyltin triamide) ready for improvement.

B. Claim 1

i. [1Pre] “A high purity liquid composition comprising:”

181. Hänssgen discloses the claim limitation requiring “a high purity liquid composition” for the reasons set forth in § XI.A.i.

ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and”

182. Hänssgen discloses the claim limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ ” for the reasons set forth in § XI.A.ii.

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

183. Hänssgen discloses “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount” for the reasons set forth in § XI.A.iii.

184. To the extent Patent Owner may argue Hänssgen does not expressly disclose this limitation, as I explained above in § XII.A, it would have been obvious to a POSITA before the time of filing of the '903 Patent (April 11, 2018) based upon the disclosure of Hänssgen to optimize the reaction conditions and/or apply known purification methods to known monoalkyl triamide products to yield predictable success, *i.e.*, to optimize the purity of the monoalkyl tin triamide composition to

remove the dialkyltin compounds, including up to “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” as required by this claim limitation.

185. Indeed, a POSITA would have understood that purification of any compound is often necessary to obtain the desired product, and the level of purification needed for any given compound or any given use of a compound is simply a matter of economy, as purification steps take additional manufacturing time and may cost some yield. *See, e.g.*, EX1007, [0010] (“[H]igh purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.”).

186. As explained above, a POSITA at the time of filing of the '903 Patent (April 11, 2018) would have understood that many known methods of purification existed, including for monoalkyl tin triamide compounds. For example, the '903 Patent specifically admits that monoalkyl tin triamide compounds can “be further purified,” including purification by “fractional distillation to effectively reduce polyalkyl contaminants below levels that may already be relatively low from the direct synthesis.” EX1001, 3:2-5, 3:13-17. And, as noted in § IV.B, other references published long before the '903 Patent was filed (April 11, 2018) explained that fractional distillation could be used to purify compounds, and such purification could

be done in multiple times, if needed, to continue increasing the purity to a sufficiently high purity level for the desired application. *See* § IV.B.

187. I have been informed and understand that during prosecution, the Patent Owner admitted that Hänssgen is prior art that taught a method of the synthesis of monoalkyl tin triamide compounds. EX1004, 890. Hänssgen also teaches that the known reaction “proceeded quantitatively” with no evidence of “secondary reactions,” and that “analytically pure I [tBuSn(NEt₂)₃] can be obtained by fractionated distillation of the benzene phase.” EX1005, 2.

188. Thus, Hänssgen acknowledges the problem of secondary reactions and specifically provides a reason that would have led a POSITA to use the known process of fractional distillation to achieve the desired “analytically pure” compound.

189. The '903 Patent does not provide any description of unexpected results from the limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount” as compared to other analytically pure monoalkyl tin triamide compounds. Indeed, the '903 Patent states that “it can be desirable to synthesize the monoalkyl tin compounds with higher purity such that any subsequent purification, such as with fractional distillation, if desired, results in even lower dialkyl or polyalkyl contamination.” EX1001, 3:49-52.

190. Thus, in my opinion, to the extent Hänssgen does not explicitly disclose the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” such a limitation would have been obvious to a POSITA at the time of filing of the '903 Patent (April 11, 2018) because Hänssgen described a high purity monoalkyl tin compound, and a POSITA would have merely needed to apply known techniques to the known synthesis process disclosed in Hänssgen to achieve the claimed purity level.

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

191. Hänssgen discloses the claim limitation requiring “wherein R is a hydrocarbyl group with 1-31 carbon atoms” for the reasons set forth in § XI.A.iv.

v. [1d] “wherein R’ is a hydrocarbyl group with 1-10 carbon atoms.”

192. Hänssgen discloses the claim limitation requiring “R’ is a hydrocarbyl group with 1-10 carbon atoms” for the reasons set forth in § XI.A.v.

C. Claim 2

i. [2pre] “The composition of claim 1”

193. Hänssgen discloses the composition of claim 1 for the reasons set forth in § XI.A.

ii. [2a] “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C-$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

194. Hänssgen discloses the claim limitation requiring “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C-$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms” for the reasons set forth in § XI.B.

D. Claim 3

i. [3pre] “The composition of claim 1”

195. Hänssgen discloses the composition of claim 1 for the reasons set forth in § XI.A.

ii. [3a] “wherein R comprises methyl (CH_3-), ethyl (CH_3CH_2-), isopropyl ($CH_3CH_2CH_2-$), tert-butyl ($(CH_3)_3C-$), tert-amyl ($CH_3CH_2(CH_3)_2C-$), sec-butyl ($CH_3(CH_3CH_2)CH-$), neopentyl ($(CH_3)_3CCH_2-$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

196. Hänssgen discloses the claim limitation requiring “wherein R comprises methyl (CH_3-), ethyl (CH_3CH_2-), isopropyl ($CH_3CH_2CH_2-$), tert-butyl ($(CH_3)_3C-$), tert-amyl ($CH_3CH_2(CH_3)_2C-$), sec-butyl ($CH_3(CH_3CH_2)CH-$), neopentyl ($(CH_3)_3CCH_2-$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl” for the reasons set forth in § XI.C.

E. Claim 4

i. [4pre] “The composition of claim 1”

197. Hänssgen discloses the composition of claim 1 for the reasons set forth in § XI.A.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

198. Hänssgen discloses the claim limitation requiring “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group” for the reasons set forth in § XI.D.

F. Claim 6

i. [6pre] “The composition of claim 1”

199. Hänssgen discloses the composition of claim 1 for the reasons set forth in § XI.A.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

200. Hänssgen discloses the claim limitation requiring the composition to “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” for the reasons set forth in § XI.E.

201. To the extent Patent Owner may argue Hänssgen does not expressly disclose this limitation requiring that the composition includes “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit,” for the same reasons discussed above in §§ XII.A and XII.B.iii, it would have been obvious to a POSITA before the time of filing of the ’903 Patent based upon the disclosure of Hänssgen to apply known purification methods to known methods of the synthesis of monoalkyl triamide compositions to yield predictable success, *i.e.*, to optimize the purity of the monoalkyl tin triamide composition to remove the

dialkyltin compounds, including up to “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” (as required by the claim).

202. The '903 Patent does not provide any description of unexpected results from the limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” as compared to other analytically pure monoalkyl tin triamide compounds. Indeed, the '903 Patent merely states that “it can be desirable to synthesize the monoalkyl tin compounds with higher purity such that any subsequent purification, such as with fractional distillation, if desired, results in even lower dialkyl or polyalkyl contamination.” EX1001, 3:49-52.

203. Thus, in my opinion, to the extent Hänssgen does not explicitly disclose the limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit,” such a limitation would have been obvious to a POSITA at the time of filing of the '903 Patent (April 11, 2018) because Hänssgen provided an explicit reason to achieve a high purity monoalkyl tin compound, and a POSITA would have merely needed to apply a known technique to a known product disclosed by Hänssgen, which is ready for improvement with a reasonable expectation of success.

G. Claim 7

- i. [7] “A solution comprising the composition of claim 1 and an organic solvent.”**

204. Hänssgen discloses the claim limitation requiring the “composition of claim 1 and an organic solvent” for the reasons set forth in § XI.F.

H. Claim 8

- i. [8] “The solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”**

205. Hänssgen discloses the claim limitation requiring the “solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol” for the reasons set forth in § XI.G.

I. Claim 9

- i. [9] “The solution of claim 7 wherein R comprises methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₂CH—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₂(CH₃)₂C—), sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—, cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R’ comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”**

206. Hänssgen discloses the claim limitation requiring the “solution of claim 7 wherein R comprises methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₂CH—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₂(CH₃)₂C—)¹⁴, sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—, cyclohexyl, cyclopentyl,

¹⁴ Again, I note that claim 9 appears to have an error in the chemical formula for tert-amyl, which should be (CH₃CH₂(CH₃)₂C—).

cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group” for the reasons set forth in § XI.H.

J. Claim 10

- i. [10Pre] “A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination, the method comprising,”**

207. To the extent the preamble (“A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination”) is limiting, Hänssgen discloses the claim limitation requiring a “method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination” for the reasons set forth in § XI.I.i.

- ii. [10a] reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form $\text{RSn}(\text{OR}'')_3$,**

208. Hänssgen discloses the claim limitation requiring “reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form $\text{RSn}(\text{OR}'')_3$ ” for the reasons set forth in § XI.I.ii.

- iii. [10b] wherein R” is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,**

209. Hänssgen discloses the claim limitation requiring “R” is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition” for the reasons set forth in § XI.I.iii.

iv. [10c] wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

210. Hänssgen discloses “the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin” for the reasons set forth in § XI.I.iv.

211. To the extent Patent Owner may argue Hänssgen does not expressly disclose “the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin,” in my opinion, it would have been obvious to a POSITA before the time of filing of the '903 Patent (April 11, 2018) based upon the disclosure of Hänssgen to apply known known purification to known methods of the synthesis of monoalkyl triamide compositions and known methods of performing the alcoholysis reactions on those compositions to yield predictable success, *i.e.*, to optimize the purity of the composition to remove the dialkyltin compounds, including up to no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin. See XII.A.

XIII. GROUND 2A: OBVIOUSNESS BASED ON MEYERS AND DEELMAN

212. It is my opinion that Claims 1-4, 6 of the '903 Patent would have been obvious given the disclosures of Meyers (EX1006) together with Deelman (EX1007).

A. Reasons to Combine Meyers and Deelman

213. Meyers discloses “precursor solutions that can be deposited to form organometallic coatings that can be patterned with very high resolution with radiation,” including preparation of “a water-reactive precursor, isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃)” by reacting a monoalkyltin trihalide compound i-PrSnCl₃ with a lithium amide compound LiNMe₂. EX1006, [0002], [0150]-[0151]. ¹H and ¹¹⁹Sn NMR of the distillate obtained by Meyers in a C₆D₆ solvent “match[ed] the predicted spectra for i-PrSn(NMe₂)₃” and the “primary ¹¹⁹Sn resonance” was “consistent with a major product with a single tin environment, with a chemical shift comparable to reported monoalkyltin amido compounds.” EX1006, [0151]. While Meyers notes that high purity alkyltin compounds can be accomplished (EX1006, [0063] (“As described herein, low metal contamination synthesis approaches can be accomplished with appropriate alternative hydrolysis approaches and high-purity alkyltin precursors.”)), Meyers does not explicitly disclose the specific purity levels of the isopropyl tris(dimethylamido)tin compound.

214. Deelman, however, explicitly discloses the importance of obtaining “high purity monoalkyltin compounds” with only “minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract. Specifically, Deelman teaches that “monoalkyltin compounds of formula RSn(T)₃,” which covers the monoalkyltin triamide compounds disclosed by Meyers, in which the monoalkyltin compound

compositions may have “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula $\text{RSn}(\text{T})_3$, in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]; *see also* EX1007, [0023] (“As regards the ligand T, it is any ligand known in the art[.]”).

215. In my opinion, a POSITA, looking at the disclosure of the monoalkyltin triamide compound of Meyers, and given Meyers’s explicit teaching that pure monoalkyltin compounds can be accomplished (*see* ¶ 213), would take into account Deelman’s disclosure (*see* ¶ 214) to determine a method to increase the purity of the monoalkyltin triamide compound.

216. In my opinion, a POSITA would have understood that Meyer’s monoalkyltin triamide compound could benefit from achieving the high purity levels disclosed by Deelman, with a minimal amount of dialkyltin impurity because both Meyers and Deelman disclose the same synthesis pathway for preparing monoalkyltin triamide compound, and both recognize the need for highly pure monoalkyltin compounds.

217. More specifically, Meyers teaches the synthesis of a monoalkyltin triamide compound $\text{i-PrSn}(\text{NMe}_2)_3$ by reacting a monoalkyltin trihalide compound i-PrSnCl_3 with a lithium amide compound LiNMe_2 ; Meyers reported that the NMR results were “consistent with a major product with a single tin environment, with a chemical shift comparable to reported monoalkyltin amido compounds.” EX1006,

[0151]. As I explained above, selective reaction pathways replacing halide ligands of an organometallic compound with amide ligands were well-known in the art. § IV.A.

218. Deelman also teaches that “high purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.” EX1007, [0010]. Deelman explains the monoalkyltin compound compositions may have up to “99.999 wt%” of formula $\text{RSn}(\text{T})_3$, “in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]. Deelman further teaches that “[H]igh purity monoalkyltin-based compounds of $\text{RSn}(\text{T})_3$ ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula $\text{RSn}(\text{Hal})_3$, in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. Accordingly, Deelman teaches the same synthesis strategy disclosed in Meyers for preparing monoalkyltin triamide compound. See ¶ 217 above.

219. In my opinion, a POSITA would have reasons, as described below, to combine Meyers and Deelman. Meyers and Deelman are in the same field of art and are directed to the same types of monoalkyltin compounds, with each focusing on converting monoalkyltin trihalides into other mono-alkyltin derivatives. Thus, a POSITA looking to improve the purity of the monoalkyltin triamide compositions disclosed by Meyers would naturally consider Deelman, as it relates to the same

desired compounds, *i.e.*, monoalkyltin compounds of the same generic chemical formula, and the same starting compounds.

220. In my opinion, the combination of Meyers and Deelman requires no more than applying known techniques to achieve a high purity compound using a known reaction pathway, with predictable success. For example, Deelman teaches that high purity monoalkyltin trihalides are desirable (EX1007, [0010]) and can be achieved not only through means already then known from the prior art, including “distillation of monoalkyltin halides from thermal redistribution mixtures containing mono-, di-, and tri-alkylhalides” (EX1007, [0028]) but also through the reactions disclosed in Deelman that selectively form monoalkyltin halides without multi-alkyl impurities (EX1007, [0031]-[0033]). Thus, Deelman provides a specific reason for a POSITA to obtain high purity monoalkyltin trihalides, and further derive other high purity monoalkyltin compounds, including triamides, by replacing halide groups with amido groups using a well-known reaction pathway including the reaction taught by Meyers. Indeed, as explained above (§ IV.A), replacing halide groups of a high purity monoalkyltin trihalide with amide groups was a well-known reaction pathway prior to the time of the alleged invention of the '903 Patent (*i.e.*, April 11, 2018). § IV.A.

221. Moreover, a POSITA would have a reasonable expectation of success in maintaining the high purity of the monoalkyltin trihalide during the conversion to

a high purity monoalkyltin triamide because such a reaction is a well-defined exchange pathway, and thus, when properly controlled, a POSITA would have expected production of high purity monoalkyltin triamides. § IV.A; § X.B.

222. Indeed, the experiments conducted by Dr. Hood at my direction confirmed that the steps described in Meyers, with adjustment to the solvent (as disclosed by Deelman), which proceeded through conversion of a monoalkyltin trihalide to a monoalkyltin triamide, successfully and predictably yielded a monoalkyltin triamide ($i\text{-PrSn}(\text{NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$. Thus, the combination of Meyers/Deelman is able to generate the claimed high purity monoalkyltin triamide compounds. While Petitioner's experiments were conducted recently, a POSITA would at the time of the alleged invention (April 11, 2018) have also expected the combination to generate the claimed high purity monoalkyltin triamide compounds as well because the reaction conditions used in Petitioner's experiments were all available at that time.

223. Even if the purity was not maintained, methods to further improve the purity of monoalkyltin triamide compounds, such as fractional distillation, were well known to a POSITA prior to the alleged invention of the '903 Patent (April 11, 2018), and a POSITA would have a reasonable expectation of success at increasing the purity of the resulting monoalkyltin triamide compounds to the extent further

purification was necessary to obtain the purity that Deelman teaches is desirable and achievable. § IV.B.

224. Accordingly, a POSITA, who would be interested in improving the purity level of the monoalkyltin triamide composition of Meyers given the disclosure of Meyers that high purity monoalkyltin compounds can be accomplished (EX1006, [0063]), would consider Deelman to incorporate its teaching of achieving high purity compositions, including by using a high purity monoalkyltin trihalide as a starting compound.

B. Claim 1

i. [1Pre] “A high purity liquid composition comprising:”

225. To the extent that the preamble (“a high purity liquid composition”) is limiting, Meyers together with Deelman renders obvious the claim limitation requiring “a high purity liquid composition.” For example, Meyers discloses preparation of “a water-reactive precursor, isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃)” by mixing LiNMe₂ and anhydrous hexanes, and then adding i-PrSnCl₃ to the reaction flask dropwise over a period of two hours. EX1006, [0150]-[0151]. After the reaction was brought to room temperature, the reaction solution was filtered, the solvent was removed and the residue distilled, resulting in a “pale yellow liquid.” EX1006, [0151]. Meyers further teaches that the synthesis of high purity monoalkyltin precursors can be accomplished. EX1006, [0063] (“As

described herein, low metal contamination synthesis approaches can be accomplished with appropriate alternative hydrolysis approaches and high-purity alkyltin precursors.”).

226. Deelman also discloses the importance of high purity “monoalkyltin compound compositions that may have “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula $\text{RSn}(\text{T})_3$, in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]. Deelman discloses examples of preparation of monoalkyltin compounds, including for example, “mono-octyltin trichloride” which is a “yellow-orange liquid.” EX1007, Example 5 [0197]-[0198].

227. For the reasons I discussed above (§ XIII.A), it would have been obvious to combine Meyers with Deelman to achieve a high purity liquid composition. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “a high purity liquid composition.”

ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and”

228. Meyers together with Deelman renders obvious the claim limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$.”

229. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃),” which is a monoalkyl tin triamide compound. EX1006, [0150]-[0151].

230. Accordingly, Meyers together with Deelman renders obvious “a monoalkyl tin triamide compound represented by the chemical formula RSn(NR'₂)₃.”

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

231. Meyers together with Deelman renders obvious the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

232. Meyers teaches the synthesis of an example of a monoalkyltin triamide compound i-PrSn(NMe₂)₃ by reacting a monoalkyltin trihalide compound i-PrSnCl₃ with a lithium amide compound LiNMe₂. EX1006, [0151]. Meyers further teaches that high purity monoalkyltin precursors can be accomplished. EX1006, [0063] (“As described herein, low metal contamination synthesis approaches can be accomplished with appropriate alternative hydrolysis approaches and high-purity alkyltin precursors.”). However, Meyers does not explicitly teach that the monoalkyltin triamide compound has “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

233. Deelman does however teach that “high purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.” EX1007, [0010]. Deelman explains that the monoalkyltin compound compositions may have up to “99.999 wt%” of formula RSn(T)_3 , “in which R is linear, branched or cyclic C1-C20 alkyl, preferably C1-C10 alkyl, and T is a ligand.” EX1007, [0013]. Deelman further teaches “[H]igh purity monoalkyltin-based compounds of RSn(T)_3 ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula RSn(Hal)_3 , in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. As a preferred embodiment, R can be the iso-propyl group, *i.e.*, i-PrSnCl_3 . EX1007, [0022].

234. Accordingly, a POSITA interested in improving the purity of monoalkyltin triamide, such as the ones disclosed in Meyers, which a POSITA would have been motivated to do (as I describe above in § XIII.A), would have used a high purity monoalkyltin trihalide, as taught by Deelman, in the synthesis process disclosed in Meyers, *i.e.*, reacting a monoalkyltin trihalide compound with a lithium amide compound to arrive at a high purity monoalkyltin triamide composition. It would have been obvious to use different conditions including trying a different type of solvent, such as THF, a preferred solvent as taught by Deelman, to dilute the reactants so as to minimize additional substitutions. Indeed, given the known

reaction pathway, replacing halide groups of a high purity monoalkyltin trihalide with amide groups, a POSITA would have a reasonable expectation of success to arrive at the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” consistent with the teachings of Deelman.

235. Indeed, as I explained above in § X.B, the experiment conducted at my direction following the steps described in Meyers, with a mere adjustment to the solvent, as disclosed by Deelman (EX1007, [0052] “In particular tetrahydrofuran (THF), ethanol, and 1,2-dimethoxyethane (DME) are suitable solvents.”), successfully yielded a monoalkyltin triamide (i-PrSn(NMe₂)₃) that contains only 0.19 mole % dialkyltin impurity i-Pr₂Sn(NMe₂)₂, which falls within the scope of “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

236. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

237. Meyers together with Deelman renders obvious the claim limitation requiring “wherein R is a hydrocarbyl group with 1-31 carbon atoms.”

238. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]. Isopropyl is a hydrocarbyl group with 3 carbon atoms, which is within the claimed range of 1-31 carbon atoms.

239. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “R is a hydrocarbyl group with 1-31 carbon atoms.”

v. [1d] “wherein R’ is a hydrocarbyl group with 1-10 carbon atoms.”

240. Meyers together with Deelman renders obvious the claim limitation requiring “R’ is a hydrocarbyl group with 1-10 carbon atoms.”

241. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]. Methyl is a hydrocarbyl group with one (1) carbon atom, which is within the claimed range of 1-10 carbon atoms.

242. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “R’ is a hydrocarbyl group with 1-10 carbon atoms.”

C. Claim 2

i. [2pre] “The composition of claim 1”

243. Meyers together with Deelman renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

ii. [2a] “wherein R is a branched alkyl ligand represented by R¹R²R³C—, where R¹ and R² are independently an alkyl group with 1-10 carbon atoms, and R³ is hydrogen or an alkyl group with 1-10 carbon atoms.”

244. Meyers together with Deelman renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

245. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]. Isopropyl is a branched alkyl ligand which includes three (3) carbon atoms and could be represented by $R^1R^2R^3C—$, where R^1 and R^2 are each one (1) carbon atom, and R^3 is hydrogen.

246. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

D. Claim 3

i. [3pre] “The composition of claim 1”

247. Meyers together with Deelman renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

- ii. [3a] “wherein R comprises methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₂CH—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₂(CH₃)₂C—), sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”**

248. Meyers together with Deelman renders obvious the claim limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$), sec-butyl ($\text{CH}_3(\text{CH}_3\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

249. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, ($\text{i-PrSn}(\text{NMe}_2)_3$).” EX1006, [0150]-[0151]. Thus, Meyers discloses that R comprises isopropyl, which is within the scope of claim limitation [3a].

250. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$), sec-butyl ($\text{CH}_3(\text{CH}_3\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

E. Claim 4

i. [4pre] “The composition of claim 1”

251. Meyers together with Deelman renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

252. Meyers together with Deelman renders obvious the claim limitation requiring “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

253. Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]. Accordingly, Meyers discloses that R’ comprises a methyl group, which is within the scope of limitation 4[a].

254. Accordingly, Meyers together with Deelman renders obvious the claim limitation requiring “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

F. Claim 6

i. [6pre] “The composition of claim 1”

255. Meyers together with Deelman renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

256. Meyers together with Deelman renders obvious the claim limitation requiring that the composition comprises “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

257. Meyers teaches the synthesis of an example monoalkyltin triamide compound i-PrSn(NMe₂)₃ by reacting a monoalkyltin trihalide compound i-PrSnCl₃ with a lithium amide compound LiNMe₂. EX1006, [0151]. While Meyers teaches

that high purity compounds are desirable, it does not explicitly disclose the purity level of the monoalkyltin triamide compound $i\text{-PrSn}(\text{NMe}_2)_3$.

258. Deelman, however, teaches that “high purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives that are of interest for several existing applications.” EX1007, [0010]. Deelman explains that the monoalkyltin compound compositions may have up to “99.999 wt%” of formula $\text{RSn}(\text{T})_3$, “in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]. Deelman further teaches that “[H]igh purity monoalkyltin-based compounds of $\text{RSn}(\text{T})_3$ ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula $\text{RSn}(\text{Hal})_3$, in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. As a preferred embodiment, R can be iso-propyl group, *i.e.*, $i\text{-PrSnCl}_3$. EX1007, [0022].

259. Accordingly, a POSITA interested in improving the purity of monoalkyltin triamide, such as the ones disclosed in Meyers, which a POSITA would have been motivated to do (as I describe above in § XIII.A), would have used a high purity monoalkyltin trihalide, as taught by Deelman, in the synthesis process disclosed in Meyers, *i.e.*, reacting a monoalkyltin trihalide compound with a lithium amide compound to arrive at a high purity monoalkyltin triamide composition. It would have been obvious to use different conditions including trying a different type

of solvent, such as THF, a preferred solvent as taught by Deelman, to dilute the reactants so as to minimize additional substitutions. Given the known reaction pathway, replacing halide groups of a high purity monoalkyltin trihalide with amide groups, a POSITA would have a reasonable expectation of success to arrive at the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit,” consistent with the teachings of Deelman.

260. Indeed, as I explained above in § X.B, the experiment conducted at my direction following the steps described in Meyers, with a mere adjustment to the solvent, successfully yielded a monoalkyltin triamide ($i\text{-PrSn}(\text{NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$. A POSITA would have understood that, if needed, known purification techniques could be used to further purify the composition to arrive at the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

261. Accordingly, Meyers together with Deelman renders obvious claim 6 requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

XIV. GROUND 2B: OBVIOUSNESS BASED ON MEYERS TOGETHER WITH DEELMAN AND HÄNSSGEN

262. It is my opinion that Claims 1-4, 6-10 of the '903 Patent would have been obvious given the disclosures of Meyers (EX1006) together with Deelman (EX1007) and Hänssgen (EX1005).

A. Reasons to Combine Meyers and Deelman with Hänssgen

263. As I explained above in § XIII.A, Meyers discloses “precursor solutions that can be deposited to form organometallic coatings that can be patterned with very high resolution with radiation and to the coated substrates and coatings formed with the precursor solutions before and after patterning,” including preparation of a monoalkyltin triamide compound—“a water-reactive precursor, isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃)” by reacting a monoalkyltin trihalide compound i-PrSnCl₃ with a lithium amide compound LiNMe₂. EX1006, [0002], [0150]-[0151]. Meyers does not explicitly disclose the purity of the monoalkyltin triamide compound. § XIII.A.

264. Deelman discloses compounds similar to those in Meyers, including “mono-alkyltin compounds of formula RSn(T)₃” which can also be synthesized starting with a monoalkyltin trihalide. EX1007, [0031]. Deelman further teaches the importance of obtaining “high purity monoalkyltin compounds” with only “minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract. Specifically, Deelman teaches the monoalkyltin compound compositions may have “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula RSn(T)₃, in which R is linear, branched or cyclic C₁-C₂₀ alkyl, preferably C₁-C₁₀ alkyl, and T is a ligand.” EX1007, [0013].

265. As explained above, a POSITA would have understood that Meyers's teaching of the synthesis of a monoalkyltin triamide compound, $i\text{-PrSn}(\text{NMe}_2)_3$, and Deelman's teaching of the importance of achieving high purity levels for those same type compounds could be combined, including by beginning with the same starting compound (*i.e.*, a monoalkyltin trihalide compound), and used to achieve a high purity composition, with a minimal amount of dialkyltin compound as an impurity. See § XIII.A.

266. Similarly, as I explained above in § XI.I and § XII.A, Hänssgen teaches the synthesis of a monoalkyltin triamide compound, "triamino-*t*-butylstannane (I)" by "Sn-alkylation of tetrakis(diethylamino)stannane with lithium-*t*-butyl," which "proceeds practically quantitatively" with "no indications of secondary reactions." EX1005, 2. Hänssgen further teaches the "alcoholysis of [compound] (I)," *i.e.*, $t\text{BuSn}(\text{NEt}_2)_3$, with "*t*-butanol, as described by equation 3, results in the 'displacement reaction' ... typical for aminostannanes to trialkoxystannane (II)." EX1005, 3.

267. As explained above in § XIII.A, a POSITA would have been motivated to combine the teaching of Meyers and Deelman to arrive at a high purity monoalkyltin triamide composition, as claimed in the '903 Patent. A POSITA would likewise be motivated to combine the high purity monoalkyltin triamide composition of Meyers/Deelman with the disclosure of Hänssgen to arrive at the claimed

solutions of claims 7-9 of the '903 Patent by introducing the monoalkyltin triamide composition into a solution including an organic solvent, *i.e.*, and alcohol, and the resulting reaction of the monoalkyltin triamide compound with the alcohol to synthesize a high purity monoalkyltin trialkoxide, another useful and well-known compound, as recited by claim 10 of the '903 Patent. See § IV.A (describing use of monoalkyltin trialkoxide compounds in formation of coatings as electrodes for the glass substrates of emissive screens of the flat screen type, electrodes for photovoltaic cells or arrays of conductive elements for heated windows, windows provided with an antenna or glazing with a conductive coating having the function of a shield towards electromagnetic waves).

268. In my opinion, a POSITA would have had reasons to further combine Meyers/Deelman with the teaching of Hänssgen, to derive a high purity monoalkyltin trialkoxide compound by alcoholysis of the high purity monoalkyltin triamide compound taught by the combination of Meyers and Deelman. For example, Meyers, Deelman, and Hänssgen are in the same field of art and directed to the same types of monoalkyltin compounds with the same generic chemical formula. Moreover, as I explained in § IV.A, alcoholysis of organotin amides was well-known in the art prior to the alleged invention of the '903 Patent (April 11, 2018) with known benefits. Because of these known benefits, a POSITA would have

been motivated, for example, to convert the monoalkyltin triamide of Meyers/Deelman into a monoalkyltin trialkoxide as taught by Hänssgen.

269. In my opinion, a POSITA would be motivated to subject the high purity monoalkyltin compound derived from the combination of Meyers and Deelman with the alcoholysis reaction taught by Hänssgen. Indeed, Hänssgen discloses a typical alcoholysis reaction of monoalkyltin triamide compound to derive monoalkyltin trialkoxides, another useful class of compounds (see ¶ 266, § IV.A), and a POSITA would have expected success in placing the monoalkyltin triamide compound taught by Meyers/Deelman in solution with an organic solvent, like an alcohol, to undergo an alcoholysis reaction to form a monoalkyltin trialkoxide (as disclosed by Hänssgen). §§ IV.A, XI.G, and XI.I.

270. In my opinion, the combination of Meyers and Deelman with Hänssgen requires no more than applying a known chemical reaction (alcoholysis) to a known class of compounds (monoalkyltin triamide compounds) to arrive at another known compound (monoalkyltin trialkoxide), with predictable success.

271. Indeed, in my opinion, a POSITA would have a reasonable expectation of success applying the known alcoholysis reaction to the high purity monoalkyltin triamide composition taught by the combination of Meyers and Deelman because Hänssgen teaches the alcoholysis of a monoalkyltin triamide compound ($\text{tBuSn}(\text{NEt}_2)_3$) (see § XI.I) which is similar to the monoalkyltin triamide compound

formed by the combination of Meyers and Deelman (a high purity isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃). Moreover, as explained above (§ XI.I), the alcoholysis reaction of Hänssgen impacts only the amido groups and not the alkyl group (displacing the amido group with the alkoxyl group). As such, in my opinion, a POSITA would have understood that the reaction would not change the dialkyl tin contamination level of the monoalkyltin triamide compound. In other words, a POSITA would have expected the resulting monoalkyltin trialkoxide compound from the alcoholysis reaction to have the same low dialkyl tin contamination as the monoalkyltin triamide compound taught by Meyers/Deelman does.

272. Accordingly, a POSITA interested in deriving monoalkyltin trialkoxides would find it obvious to further subject the high purity monoalkyltin triamide composition of Meyers and Deelman to the typical alcoholysis reactions as taught by Hänssgen, with a predictable and reasonable expectation of success.

B. Claim 1

i. [1Pre] “A high purity liquid composition comprising:”

273. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “a high purity liquid composition” for the reasons set forth in § XIII.B.i.

ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula RSn(NR’₂)₃ and”

274. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ ” for the reasons set forth in § XIII.B.ii.

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

275. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount” for the reasons set forth in § XIII.B.iii.

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

276. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R is a hydrocarbyl group with 1-31 carbon atoms” for the reasons set forth in § XIII.B.iv.

v. [1d] “wherein R' is a hydrocarbyl group with 1-10 carbon atoms.”

277. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R' is a hydrocarbyl group with 1-10 carbon atoms” for the reasons set forth in § XIII.B.v.

C. Claim 2

i. [2pre] “The composition of claim 1”

278. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

- ii. **[2a] “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”**

279. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms” for the reasons set forth in § XIII.C.

D. Claim 3

- i. **[3pre] “The composition of claim 1”**

280. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

- ii. **[3a] “wherein R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH_2—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”**

281. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH_2—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl” for the reasons set forth in § XIII.D.

E. Claim 4

i. [4pre] “The composition of claim 1”

282. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

283. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group” for the reasons set forth in § XIII.E.

F. Claim 6

i. [6pre] “The composition of claim 1”

284. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

285. Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” for the reasons set forth in § XIII.F.

G. Claim 7

i. [7] “A solution comprising the composition of claim 1 and an organic solvent.”

286. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

287. Meyers together with Deelman together with Hänssgen further renders obvious the claim limitation requiring “a solution including the composition and an organic solvent.” For example, Hänssgen teaches creating a solution of “tBuSn(NEt₂)₃” in “dichloromethane.” EX1005, 4. Dichloromethane is an organic solvent. Hänssgen further teaches that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4. A POSITA would have understood that the solvent mixture includes an alcohol because t-butanol is added into the solution.

288. A POSITA would have understood that the high purity monoalkyltin triamide composition resulting from the combination of Meyers and Deelman could easily be combined with an organic solvent, like the alcohol, t-butanol, disclosed in Hänssgen to create the claimed solution with predictable success.

289. Accordingly, Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “a solution including the composition and an organic solvent.”

H. Claim 8

- i. [8] “The solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”**

290. Meyers together with Deelman and Hänssgen renders obvious the solution of claim 7 for the reasons set forth in § XIV.G.

291. Meyers together with Deelman and Hänssgen further renders obvious the claim limitation requiring “the solution having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.” As noted above (§ XI.G.), Hänssgen teaches a solution of “tBuSn(NEt₂)₃” in “dichloromethane,” and that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4. As I explained above (§ XI.G), Hänssgen further teaches the solution concentration from about 0.005M to about 0.5M for the reasons set forth in § XI.G.

292. In my opinion, a POSITA would have understood that the high purity monoalkyltin triamide composition resulting from the combination of Meyers and Deelman would similarly be combined with a solvent including dichloromethane and an alcohol, t-butanol, with a concentration from about 0.005 M to about 0.5M, as disclosed in Hänssgen for alcoholysis of the monoalkyltin with predictable success.

293. Accordingly, Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”

I. Claim 9

- i. [9] “The solution of claim 7 wherein R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$), sec-butyl ($\text{CH}_3(\text{CH}_2\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”

294. Meyers together with Deelman and Hänssgen renders obvious the solution of claim 7 for the reasons set forth in § XIV.G.

295. Meyers together with Deelman and Hänssgen further renders obvious the claim limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$)¹⁵, sec-butyl ($\text{CH}_3(\text{CH}_2\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group” for the reasons set forth in § XIII.D and in § XIII.E.

296. Accordingly, Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($(\text{CH}_3)_3\text{C—}$), tert-amyl ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$), sec-butyl ($\text{CH}_3(\text{CH}_2\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$),

¹⁵ Again, I note that claim 9 appears to have an error in the chemical formula for tert-amyl, which should be $(\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—})$.

cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”

J. Claim 10

i. [10Pre] “A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination, the method comprising,”

297. To the extent the preamble (“A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination”) is limiting, Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.”

298. As I explained above (§ XI.I.i), Hänssgen teaches a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination for the reasons set forth in § XI.I.i.

299. In my opinion, a POSITA would have been motivated to combine the high purity monoalkyltin triamide compositions formed from the combination of Meyers and Deelman with the method to form a monoalkyltin trialkoxide compound taught by Hänssgen for the reasons set forth in § XIV.A.

300. Accordingly, Meyers together with Deelman and Hänssgen renders obvious the claim limitation requiring “a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.”

- ii. [10a] reacting the composition of claim 1 with an alcohol represented by the formula HOR" in an organic solvent to form RSn(OR")₃,**

301. Meyers together with Deelman and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XIII.B.

302. Meyers together with Deelman and Hänssgen further render obvious the claim limitation requiring "reacting the composition of claim 1 with an alcohol represented by the formula HOR" in an organic solvent to form RSn(OR")₃."

303. As I explained above (§ XI.I.ii.), Hänssgen teaches reacting a composition like the high purity monoalkyltin triamide composition resulting from the combination of Meyers and Deelman, with an alcohol represented by the formula HOR" in an organic solvent to form RSn(OR")₃. Specifically, as I explained above in § XI.I.ii., Hänssgen describes the alcoholysis of tBuSn(NEt₂)₃ with t-butanol which "results in the 'displacement reaction' ... typical for aminostannanes to trialkoxystannane (II)," resulting in t-BuSn(O-t-Bu)₃, which can be represented by the formula RSn(OR")₃. EX1005, 3.

304. A POSITA would have been motivated to react the high purity monoalkyltin triamide composition resulting from the combination of Meyers and Deelman with the solvent disclosed by Hänssgen, t-butanol, or another alcohol, in an organic solvent to obtain a monoalkyltin trialkoxide of the formula RSn(OR")₃ for the reasons set forth in § XIV.A.

305. Accordingly, Meyers together with Deelman and Hänssgen render obvious the claim limitation requiring “reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form RSn(OR'')_3 .”.

iii. [10b] wherein R” is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,

306. Meyers together with Deelman and Hänssgen render obvious the claim limitation requiring “reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form RSn(OR'')_3 ” for the reasons set forth in § XI.I.iii.

iv. [10c] wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

307. Meyers together with Deelman and Hänssgen render obvious the claim limitation requiring “the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.”

308. As I explained above in § XIII.B.iii, the combination of Meyers and Deelman discloses the claim limitation requiring a monoalkyltin triamide composition that has no more than “0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.”

309. I explained in § IV.A that alcoholysis of organotin amides was well-known in the art, and in § XI.I. that Hänssgen expressly teaches alcoholysis of a

monoalkyltin triamide compound with t-butanol in an organic solvent. I also explained in § XIV.A that a POSITA would have been motivated to combine the high purity monoalkyltin triamide composition of Meyers and Deelman with the teachings of Hänssgen to arrive at a monoalkyltin trialkoxide compound with no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

310. In my opinion, it would have been further obvious to a POSITA before the time of filing of the '903 Patent (Apr. 11, 2018) based upon the disclosure of Meyers, Deelman, and Hänssgen to optimize the reaction conditions to minimize secondary reactions and/or apply known purification methods to known methods of the synthesis of monoalkyltin triamide compositions and known methods of performing the alcoholysis reactions on those compositions to yield predictable success, *i.e.*, to optimize the purity of the composition to remove the dialkyltin compounds, including up to no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

XV. GROUND 3A: OBVIOUSNESS BASED ON DEELMAN AND JONES

311. It is my opinion that Claims 1-4, 6 of the '903 Patent would have been obvious given the disclosures of Deelman (EX1007) together with Jones (EX1009).

A. Reasons to Combine Deelman and Jones

312. Deelman “relates to high purity monoalkyltin compounds, more specifically to alkyltin compound compositions containing monoalkyltin as major compound, and minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract. Deelman teaches such high purity monoalkyltin compound compositions can include “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula RSn(T)_3 , in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013].

313. Deelman further teaches “[H]igh purity monoalkyltin-based compounds of RSn(T)_3 ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula RSn(Hal)_3 , in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. Deelman explains that “monoalkyltin trihalide preparations are already known [from] the prior art” including “distillation of monoalkyltin halides from thermal redistribution mixtures containing mono-, di-, and tri-alkylhalides.” EX1007, [0027]. Deelman further teaches hydrostannylation of an alkene to selectively produce mono-alkyltin trihalides, which can “be easily converted to mono-alkyltin compound of formula RSn(T)_3 .” EX1007, [0031]-[0033].

314. Deelman, however, does not expressly teach an amino group as a preferred embodiment of ligand T. Jones expressly teaches ligand T can be an amino

group including those having the formula of $\text{NR}'\text{R}''$, where R' and R'' are alkyl or aryl groups. EX1009, 4. In the same field of organotin synthesis, Jones teaches a means to convert tin halides and alkyltin halides into alkyltin amides. EX1009, 4. More specifically, Jones explains that “[a] major advance in SnN chemistry was the preparation of an aminostannane” “by way of the lithium salt of the amine.” EX1009, 4. Jones refers to this type of reaction as “transmetallation,” illustrated in the following chemical reaction:



where “ R , R' , and R'' = alkyl or aryl, $\text{X}=\text{Cl}$ or Br , $\text{M} = \text{Li}$, and $n=1, 2, 3$, or 4 .” EX1009, 4. Jones further explains that “[r]eaction (4) invariably proceeded smoothly” (EX1009, 4). This is consistent with the common knowledge of a POSITA that the amide group from lithium amide replaces the halide ion on the metal center, leaving behind lithium halide as a byproduct. Jones further provides numerous examples of “Transmetallation with Lithium Salts of Secondary Amines” including monoalkyltin triamide compounds, such as $\text{PhSn}(\text{NMe}_2)_3$, $\text{Bu}^n\text{Sn}(\text{NMe}_2)_3$. EX1009, Tables 1 and 2.

315. Accordingly, a POSITA would have been motivated to combine the teachings of Deelman regarding the need for high purity monoalkyltin compounds

¹⁶ Again, I have corrected errors in equation 4 of Jones. See footnote 4.

of the formula $\text{RSn}(\text{T})_3$ with the disclosure of Jones that the T in the above formula could be an amino group with a reasonable expectation of success.

316. A POSITA would have had numerous reasons to make this combination. For example, Deelman and Jones are in the same field of art and directed to the same types of monoalkyltin compounds. A POSITA would have understood that incorporation of an amino group from Jones as “T” in the general chemical formula $\text{RSn}(\text{T})_3$ disclosed by Deelman would be nothing more than a simple substitution of a known ligand for another, involving known chemistry, with predictable success.

317. Indeed, the combination of Deelman and Jones requires no more than applying a known technique to achieve a high purity compound according to a known reaction pathway, with predictable success. For example, Deelman teaches: “[H]igh purity monoalkyltin-based compounds of $\text{RSn}(\text{T})_3$ ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula $\text{RSn}(\text{Hal})_3$, in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. In the same vein, Jones discloses the well-known chemical reaction to convert alkyltin halides into alkyltin amides by reacting the organotin halides with lithium amide salts. Accordingly, a POSITA interested in synthesizing high purity monoalkyltin triamide would have followed the approach taught by Deelman, *i.e.*, starting with a corresponding monoalkyltin trihalide and further adopted the well-

known reaction pathway taught by Jones, *i.e.*, to react the alkyltin halide with lithium amide salts, to convert high purity monoalkyltin trihalides into high purity monoalkyltin triamides.

318. Moreover, a POSITA would have a reasonable expectation of success in maintaining the level of purity of the monoalkyltin trihalide as disclosed by Deelman through the reaction with the lithium amide salt to derive a high purity monoalkyltin triamide as taught by Jones because such a reaction is a well-defined ion exchange pathway in which the highly nucleophilic amide ion displaces the halide ion, and thus, when properly controlled, a POSITA would have expected production of high purity monoalkyltin triamides.

319. Indeed, the experiments conducted by Dr. Hood at my direction confirm that conversion of a monoalkyltin trihalide to a monoalkyltin triamide, successfully and predictably yielded a monoalkyltin triamide ($i\text{-PrSn}(\text{NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$. See § X.B. A POSITA would have understood that the same type of reaction could be used to generate the specific monoalkyltin triamide compounds taught by the combination of Deelman and Jones. Moreover, while Petitioner's experiments were conducted recently, a POSITA at the time of the alleged invention (April 11, 2018) would have expected the combination to generate the claimed high purity monoalkyltin triamide

compounds as well because the reaction conditions used in Petitioner's experiments were all available at that time.

320. Even if the purity was not maintained (for example, due to improper controls on the well-known reaction pathway), methods to further improve the purity of monoalkyltin triamide compounds, such as fractional distillation, were well known to persons of ordinary skill in the art prior to the alleged invention of the '903 Patent (April 11, 2018), known to be useful for these compounds, and a POSITA would have a reasonable expectation of success at increasing the purity of the resulting monoalkyltin triamide compounds to the extent further purification was necessary to obtain the purity that Deelman teaches is desirable and achievable. See EX1007, [0027]; § IV.B.

B. Claim 1

i. [1Pre] "A high purity liquid composition comprising:"

321. To the extent that the preamble ("a high purity liquid composition") is limiting, Deelman together with Jones renders obvious the claim limitation requiring "a high purity liquid composition."

322. Deelman discloses high purity "monoalkyltin compound compositions that may have "from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula $R\text{Sn}(T)_3$, in which R is linear, branched or cyclic C1-C20 alkyl, preferably C1-C10 alkyl, and T is a ligand." EX1007, [0013]. Deelman discloses

examples of preparation of monoalkyltin compounds, including for example, “mono-octyltin trichloride” which is a “yellow-orange liquid.” EX1007, Example 5 [0197]-[0198].

323. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “a high purity liquid composition.”

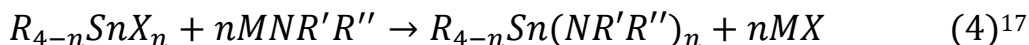
ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and”

324. Deelman together with Jones renders obvious the claim limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$.”

325. Deelman teaches “mono-alkyltin compounds of formula $\text{RSn}(\text{T})_3$.” EX1007, [0031]. Deelman further teaches “hydrostannylation of 1-alkene” to selectively produce mono-alkyltin trihalides, and acknowledges that these mono-alkyltin trihalides can “be easily converted to mono-alkyltin compound of formula $\text{RSn}(\text{T})_3$.” EX1007, [0031]-[0033]. Deelman does not expressly teach an amino group as a preferred embodiment of ligand T. See EX1007, [0023], [0024] (paragraphs discussing ligand “T” which do not list an amino group as a preferred embodiment).

326. Jones nevertheless does expressly teach that ligand T can be an amino group including those having the formula of $\text{NR}'\text{R}''$, where R' and R'' are alkyl or

aryl groups. EX1009, 4. Jones teaches the preparation of an aminostannane” “by way of the lithium salt of the amine,” as shown in the following equation from Jones:



where “R, R’, and R’’= alkyl or aryl, X=Cl or Br, M = Li, and n=1, 2, 3, or 4.”

EX1009, 4. When n=3, reaction 4 of Jones, teaches reacting monoalkyltin trihalide (RSnX₃) with a lithium amide compound to form monoalkyltin triamide compounds (RSn(NR'R'')₃), consistent with the teachings of Deelman. When R’ and R’’ are the same alkyl group and n=3, the disclosed R_{4-n}Sn(NR'R'')_n product corresponds to a monoalkyl tin triamide compound represented by the chemical formula RSn(NR'₂)₃. This is further supported by compound examples disclosed in Jones. *See, e.g.*, EX1009, Tables 1 and 2 (disclosing the synthesis of monoalkyltin triamide compounds represented by the chemical formula RSn(NR'₂)₃, including PhSn(NMe₂)₃ and BuⁿSn(NMe₂)₃).

327. It would have been obvious to combine Deelman and Jones, for the reasons I explained above. § XV.A. Accordingly, Deelman together with Jones renders obvious the limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula RSn(NR'₂)₃.”

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

¹⁷ Again, I have corrected errors in equation 4 of Jones. See footnote 4.

328. Deelman together with Jones renders obvious the limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

329. As I explained above in § XV.A, Deelman “relates to high purity monoalkyltin compounds, more specifically to alkyltin compound compositions containing monoalkyltin as major compound, and minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract. Deelman teaches such high purity monoalkyltin compound compositions can include “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula RSn(T)_3 , in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013]. Deelman further teaches that “high purity monoalkyltin-based compounds of RSn(T)_3 ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula RSn(Hal)_3 , in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. Deelman’s disclosure of 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula RSn(T)_3 is within the claim scope of no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.

330. Jones teaches the synthesis of a dialkyltin triamide of the formula $\text{R}_4\text{-}_n\text{Sn(NR'R'')}_n$ by converting tin halides and alkyltin halides into alkyltin amides. EX1009, 4.

331. As I explained above (§ XV.A), a POSITA would have been motivated to follow the approach taught by Deelman to obtain high purity monoalkyltin trihalides, and further to adopt the known reaction pathway taught by Jones to convert the high purity monoalkyltin trihalides into high purity monoalkyltin triamides. Indeed, in my opinion, given the known reaction pathway, replacing halide groups of a high purity monoalkyltin trihalide with amide groups, a POSITA would have a reasonable expectation of success to arrive at the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” consistent with the teachings of Deelman.

332. As I explained above in § X.B, the experiment conducted at my direction following this approach—starting with a high purity monoalkyltin trihalide compound ($i\text{-PrSnCl}_3$) and reacting with a lithium amine salt (LiNMe_2) under typical reaction conditions, successfully and predictably yielded a monoalkyltin triamide ($i\text{-PrSn(NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn(NMe}_2)_2$, which falls within the scope of the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.” *See also* EX1007, [0022] (identifying “i-propyl” as a preferred embodiment of R in RSn(T)_3); EX1009, 4 (identifying dimethylamino group as a suitable ligand for monoalkyltin triamides).

333. In my opinion, it would have been obvious to combine Deelman and Jones, for the reasons I explained above. § XV.A. Accordingly, Deelman together with Jones render obvious this limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

334. Deelman together with Jones renders obvious the claim limitation requiring “R is a hydrocarbyl group with 1-31 carbon atoms.”

335. Deelman discloses the synthesis of a monoalkyl tin triamide compound: “mono-alkyltin compounds of formula $R\text{Sn}(T)_3$.” EX1007, [0031]. Deelman teaches “R is a linear, branched or cyclic alkyl radical having from 1 to 20 (C_1 - C_{20} alkyl), preferably from 1 to 10 carbon atoms (C_1 - C_{10} alkyl).” EX1007, [0021]. And “[a]ccording to a preferred embodiment,” R can be methyl, iso-propyl, n-butyl, and phenyl groups. EX1007, [0021]-[0022]. Thus, Deelman discloses R is a hydrocarbyl group with 1-31 carbon atoms.

336. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “R is a hydrocarbyl group with 1-31 carbon atoms.”

v. [1d] “wherein R’ is a hydrocarbyl group with 1-10 carbon atoms.”

337. Deelman together with Jones renders obvious the claim limitation requiring “R’ is a hydrocarbyl group with 1-10 carbon atoms.”

338. Deelman discloses the synthesis of “monoalkyltin compound[s] of formula RSn(T)_3 , in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, [0013], [0031]. Deelman further teaches “[H]igh purity monoalkyltin-based compounds of RSn(T)_3 ” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula RSn(Hal)_3 , in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. Deelman does not expressly teach an amino group as a preferred embodiment of ligand T. See EX1007, [0023], [0024] (paragraphs discussing ligand “T” which do not list an amino group as a preferred embodiment).

339. Jones nevertheless expressly teaches ligand T can be an amino group including those having the formula of NR'R'' , where R' and R'' are alkyl or aryl groups. EX1009, 4. Starting with an alkyltin trihalide, as Deelman does, Jones further teaches the synthesis of alkyltin triamides of the formula $\text{R}_{4-n}\text{Sn(NR'R'')}_n$, where “R, R', and R''= alkyl or aryl, X=Cl or Br, M = Li, and n=1, 2, 3, or 4.” EX1009, 4. When n=3 and, R' and R'' are the same alkyl groups, Jones discloses the synthesis of a monoalkyltin triamide of the formula RSn(NR'_2)_3 . Jones further discloses several monoalkyltin triamide compounds, such as $\text{PhSn(NMe}_2)_3$, $\text{Bu}^n\text{Sn(NMe}_2)_3$. EX1009, Tables 1 and 2. The “methyl” groups in Jones’s listed compounds fall within the claim scope requiring “R' is a hydrocarbyl group with 1-10 carbon atoms” as they are a hydrocarbyl group with one (1) carbon atom.

340. It would have been obvious to combine Deelman and Jones, for the reasons I explained above. § XV.A. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “‘R’ is a hydrocarbonyl group with 1-10 carbon atoms.”

C. Claim 2

i. [2pre] “The composition of claim 1”

341. Deelman together with Jones renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [2a] “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C-$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

342. Deelman together with Jones renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C-$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

343. Deelman discloses the synthesis of “mono-alkyltin compounds of formula $R\text{Sn}(T)_3$,” where “R is a linear, branched or cyclic alkyl radical having from **1 to 20** (C1-C20 alkyl),” and preferably an iso-propyl group. EX1007, [0021]-[0022], [0031]. An iso-propyl group is a branched alkyl ligand that can be represented by $R^1R^2R^3C-$, where R^1 and R^2 are independently an alkyl group with

one (1) carbon atom each, and R^3 is an alkyl group with one (1) carbon atom, as required by the claim limitation.

344. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”

D. Claim 3

i. [3pre] “The composition of claim 1”

345. Deelman together with Jones renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [3a] “wherein R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH_2—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

346. Deelman together with Jones renders obvious the claim limitation requiring “wherein R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH_2—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

347. Deelman discloses the synthesis of “mono-alkyltin compounds of formula $RSn(T)_3$,” where “R is a linear, branched or cyclic alkyl radical having from

1 to 20 (C1-C20 alkyl),” and preferably an iso-propyl group. EX1007, [0021]-[0022], [0031]. An iso-propyl group is within the scope of claim [3a].

348. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “wherein R comprises methyl (CH₃—), ethyl (CH₃CH₂—), isopropyl (CH₃CH₂CH₂—), tert-butyl ((CH₃)₃C—), tert-amyl (CH₃CH₂(CH₃)₂C—), sec-butyl (CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—, cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”

E. Claim 4

i. [4pre] “The composition of claim 1”

349. Deelman together with Jones renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

350. Deelman together with Jones renders obvious the claim limitation requiring “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

351. Deelman discloses the synthesis of “mono-alkyltin compounds of formula RSn(T)₃,” where “R is a linear, branched or cyclic alkyl radical having from 1 to 20 (C1-C20 alkyl), and T is a ligand.” EX1007, [0013]. Deelman further teaches “[H]igh purity monoalkyltin-based compounds of RSn(T)₃” “are advantageously prepared from the corresponding monoalkyltin trihalides of formula RSn(Hal)₃, in

which R is as defined above and Hal is chosen from chlorine, bromine and iodine.”
EX1007, [0027].

352. While Deelman does not expressly disclose ligand T can be amino groups with the formula of NR'_2 , Jones expressly discloses that ligand T can be an amino group with the formula of NR'_2 . See § XV.A. Starting with an alkyltin trihalide, as Deelman does, Jones further teaches the synthesis of alkyltin triamides of the formula $\text{R}_{4-n}\text{Sn}(\text{NR}'\text{R}'')_n$, where “R, R', and R''= alkyl or aryl, X=Cl or Br, M = Li, and n=1, 2, 3, or 4.” EX1009, 4. When n=3 and, R' and R'' are the same alkyl groups, Jones discloses the synthesis of a monoalkyltin triamide of the formula $\text{RSn}(\text{NR}'_2)_3$. Jones further discloses several monoalkyltin triamide compounds, such as $\text{PhSn}(\text{NMe}_2)_3$, $\text{Bu}^n\text{Sn}(\text{NMe}_2)_3$. EX1009, Tables 1 and 2. The “methyl” groups in Jones’s listed compounds fall within the claim scope.

353. It would have been obvious to combine Deelman and Jones, for the reasons I explained above. See § XV.A. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “R' comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

F. Claim 6

i. [6pre] “The composition of claim 1”

354. Deelman together with Jones renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

355. Deelman together with Jones renders obvious the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

356. As I explained above in § XV.A, Deelman teaches “high purity monoalkyltin compounds” containing “minor quantities of di- and/or trialkyltin compounds,” which can include “from 85 wt% to 99.999 wt%...of at least one monoalkyltin compound of formula $\text{RSn}(\text{T})_3$, in which R is linear, branched or cyclic $\text{C}_1\text{-C}_{20}$ alkyl, preferably $\text{C}_1\text{-C}_{10}$ alkyl, and T is a ligand.” EX1007, Abstract, [0013]. Deelman further teaches these compounds “are advantageously prepared from the corresponding monoalkyltin trihalides of formula $\text{RSn}(\text{Hal})_3$, in which R is as defined above and Hal is chosen from chlorine, bromine and iodine.” EX1007, [0027]. Deelman’s disclosure of “85 wt% to 99.999 wt%¹⁸...of at least one monoalkyltin compound of formula $\text{RSn}(\text{T})_3$ ” overlaps with the claim scope of essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.

¹⁸ While weight percentage slightly differs from molar percentage, given the slight molecular weight difference between different organotin compound in the composition, 99.999 wt% purity undoubtedly meets the requirement of “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

357. As I explained above (§ XV.A.), while Deelman does not expressly disclose ligand T can be amino groups with the formula of NR'_2 , Jones does expressly disclose. EX1009, 4. Starting with an alkyltin trihalide, as Deelman does, Jones further teaches the synthesis of alkyltin triamides of the formula $\text{R}_4\text{-}_n\text{Sn}(\text{NR}'\text{R})_n$, where “R, R', and R''= alkyl or aryl, X=Cl or Br, M = Li, and n=1, 2, 3, or 4.” EX1009, 4. When n=3 and, R' and R'' are the same alkyl groups, Jones discloses the synthesis of a monoalkyltin triamide of the formula $\text{RSn}(\text{NR}'_2)_3$.

358. As I explained above (§ XV.A.), a POSITA would have been motivated to follow the approach taught by Deelman to obtain high purity monoalkyltin trihalides, and further to adopt the known reaction pathway taught by Jones to convert the high purity monoalkyltin trihalides into high purity monoalkyltin triamides. Indeed, given the known reaction pathway, replacing halide groups of a high purity monoalkyltin trihalide with amide groups, a POSITA would have a reasonable expectation of success to arrive at the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit,” consistent with the teachings of Deelman. To the extent necessary, a POSITA can further reduce dialkyltin impurity through known purification in the art including fractional distillation, which was known to be useful for these compounds.

359. Accordingly, Deelman together with Jones renders obvious the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

XVI. GROUND 3B: OBVIOUSNESS BASED ON DEELMAN AND JONES AND HÄNSSGEN

360. It is my opinion that Claims 1-4, and 6-10 of the '903 Patent would have been obvious given the disclosures of Deelman (EX1007) together with Jones (EX1009) and Hänssgen (EX1005).

A. Reasons to Combine Deelman and Jones and Hänssgen

361. As I explained above (§ XV.A.), Deelman discloses high purity “monoalkyltin compounds of formula $R\text{Sn}(T)_3$.” EX1007, [0031].

362. Jones teaches the synthesis of alkyltin triamides of the formula $R_4\text{Sn}(\text{NR}'\text{R})_n$ by converting tin halides and alkyltin halides into alkyltin amides by “transmetallation.” EX1009, 4. For the reasons discussed in § XV.A. above, a POSITA would have been motivated to combine Deelman and Jones to arrive at a high purity monoalkyltin triamide composition with very low dialkyltin contamination. § XV.A.

363. While the combination of Deelman and Jones does not expressly teach monoalkyltin trialkoxide, reactions for converting monoalkyltin triamide into monoalkyltin trialkoxide were known in the art, as expressly taught by Hänssgen. EX1005, 3; § XI.I. Moreover, as I explained above (§ IV.A), monoalkyltin alkoxides

are useful compounds. § IV.A. In my opinion, a POSITA would have been motivated to combine the high purity monoalkyltin triamide composition of Deelman/Jones into solutions with organic solvents in Hänssgen, as recited by claims 7-9, and the reaction of the monoalkyltin triamide compound with an alcohol to synthesize a high purity monoalkyltin trialkoxide, another useful and well-known compound, as recited by claim 10 of the '903 Patent. *See* § IV.A. (discussing uses of monoalkyltin trialkoxides).

364. As I explained above in §§ XI.A and XI.I, Hänssgen teaches the synthesis of a monoalkyltin triamide compound, “triamino-t-butylstannane (I)” by “Sn-alkylation of tetrakis(diethylamino)stannane with lithium-t-butyl,” which “proceeds practically quantitatively” with “no indications of secondary reactions.” EX1005, 2. Hänssgen further teaches the “alcoholysis of [compound] (I),” *i.e.*, $\text{tBuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3, results in the ‘displacement reaction’...typical for aminostannanes to trialkoxystannane (II).” EX1005, 3.

365. In my opinion, a POSITA would have had reasons, as described below, to further combine Deelman and Jones with the teaching of Hänssgen, to incorporate the high purity monoalkyltin triamide compound into certain solutions, including an organic solvent like an alcohol to derive a high purity monoalkyltin trialkoxide compound by alcoholysis of the high purity monoalkyltin triamide compound taught

by the combination of Deelman and Jones. A POSITA would have understood that Deelman, Jones and Hänssgen are in the same field of art and each directed to the same types of monoalkyltin compounds with the same generic chemical formula. Moreover, as I explained in § IV.A, alcoholysis of organotin amides was known in the art prior to the alleged invention of the '903 Patent (April 11, 2018), and tin alkoxide compounds were known to be useful compounds (as described in § IV.A). Thus, a POSITA would have been motivated, for example, to convert the monoalkyltin triamide of Deelman/Jones into a monoalkyltin trialkoxide as taught by Hänssgen.

366. In my opinion, the combination of Deelman and Jones with Hänssgen requires no more than applying a known chemical reaction (alcoholysis) to a known class of compounds (monoalkyltin triamide compounds) to arrive at another known beneficial compound (monoalkyltin trialkoxide), with predictable success. Indeed, a POSITA would have a reasonable expectation of success applying the known alcoholysis reaction to the high purity monoalkyltin triamide composition taught by the combination of Deelman and Jones because Hänssgen teaches the alcoholysis of a monoalkyltin triamide compound ($\text{tBuSn}(\text{NEt}_2)_3$) (*see* § XI.I), which is a very similar compound to the monoalkyltin triamide compounds taught by the combination of Deelman and Jones. Thus, because Hänssgen disclosed a successful alcoholysis reaction involving a monoalkyltin triamide compound, a POSITA would

have understood that such a reaction could be used with other monoalkyltin triamide compounds of the same class to synthesize a monoalkyltin trialkoxide.

367. Moreover, as explained above (*see* § XI.I), the alcoholysis reaction of Hänssgen impacts only the amido groups and not the alkyl group (displacing the amido group with the alkoxyl group, as shown by Equation 3 of Hänssgen). Thus, a POSITA would have understood that the alcoholysis reaction would not change the dialkyl tin contamination level of the monoalkyltin triamide compound. In other words, a POSITA would have expected the resulting monoalkyltin trialkoxide compound from the alcoholysis reaction to have the same low dialkyl tin contamination as the monoalkyltin triamide compound taught by Deelman/Jones does.

368. Accordingly, a POSITA interested in deriving monoalkyltin trialkoxides would find it obvious to further subject the high purity monoalkyltin triamide composition of Deelman and Jones to the typical alcoholysis reactions as taught by Hänssgen, with predictable success.

B. Claim 1

i. [1Pre] “A high purity liquid composition comprising:”

369. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “a high purity liquid composition” for the reasons set forth in § XV.B.i.

ii. [1a] “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and”

370. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ ” for the reasons set forth in § XV.B.ii.

iii. [1b] “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,”

371. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount” for the reasons set forth in § XV.B.iii.

iv. [1c] “wherein R is a hydrocarbyl group with 1-31 carbon atoms, and”

372. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “R is a hydrocarbyl group with 1-31 carbon atoms” for the reasons set forth in § XV.B.iv.

v. [1d] “wherein R' is a hydrocarbyl group with 1-10 carbon atoms.”

373. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “R' is a hydrocarbyl group with 1-10 carbon atoms” for the reasons set forth in § XV.B.v.

C. Claim 2

i. [2pre] “The composition of claim 1”

374. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

- ii. **[2a] “wherein R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms.”**

375. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “R is a branched alkyl ligand represented by $R^1R^2R^3C—$, where R^1 and R^2 are independently an alkyl group with 1-10 carbon atoms, and R^3 is hydrogen or an alkyl group with 1-10 carbon atoms” for the reasons set forth in § XV.C.

D. Claim 3

- i. **[3pre] “The composition of claim 1”**

376. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

- ii. **[3a] “wherein R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl ($CH_3(CH_3CH_2)CH—$), neopentyl ($(CH_3)_3CCH_2—$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.”**

377. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “R comprises methyl ($CH_3—$), ethyl ($CH_3CH_2—$), isopropyl ($CH_3CH_2CH—$), tert-butyl ($(CH_3)_3C—$), tert-amyl ($CH_3CH_2(CH_3)_2C—$), sec-butyl

(CH₃(CH₃CH₂)CH—), neopentyl (CH₃)₃CCH₂—), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl” for the reasons set forth in § XV.D.

E. Claim 4

i. [4pre] “The composition of claim 1”

378. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [4a] “wherein R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.”

379. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group” for the reasons set forth in § XV.E.

F. Claim 6

i. [6pre] “The composition of claim 1”

380. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

ii. [6a] “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.”

381. Deelman together with Jones and Hänssgen renders obvious the claim limitation requiring “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit” for the reasons set forth in § XV.F.

G. Claim 7

i. [7] “A solution comprising the composition of claim 1 and an organic solvent.”

382. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

383. Deelman together with Jones and Hänssgen further renders obvious the limitation requiring “solution comprising the composition of claim 1 and an organic solvent.” For example, Hänssgen teaches creating a solution of an alkyltin triamide compound, “tBuSn(NEt₂)₃,” in “dichloromethane.” EX1005, 4. Dichloromethane is an organic solvent. Hänssgen further teaches that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4. A POSITA would have understood that the solvent mixture includes an alcohol, as t-butanol is added into the solution.

384. In my opinion, a POSITA would have understood that the high purity monoalkyltin triamide composition resulting from the combination of Deelman and Jones could be easily combined with an organic solvent, like the dichloromethane and t-butanol, as disclosed in Hänssgen to create the claimed solution with predictable success. § XVI.A.

385. Accordingly, Deelman together with Jones and Hänssgen renders obvious the limitation requiring “solution comprising the composition of claim 1 and an organic solvent.”

H. Claim 8

- i. [8] **“The solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”**

386. Deelman together with Jones and Hänssgen renders obvious the solution of claim 7 for the reasons set forth in § XVI.G.

387. Deelman together with Jones and Hänssgen further renders obvious the limitation requiring “solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.” As noted above (§ XI.G), Hänssgen teaches a solution of “tBuSn(NEt₂)₃” in “dichloromethane,” and that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4. As I explained above, Hänssgen further teaches the solution concentration from about 0.005M to about 0.5M for the reasons set forth in § XI.G.

388. In my opinion, a POSITA would have understood that the high purity monoalkyltin triamide composition resulting from the combination of Deelman and Jones could be easily combined with an organic solvent comprising an alcohol, t-butanol, at a concentration from about 0.005 M to about 0.5M, as disclosed in Hänssgen to create the claimed solution with predictable success. § XVI.A.

389. Accordingly, Deelman together with Jones and Hänssgen renders obvious the limitation requiring “solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.”

I. Claim 9

- i. [9] “The solution of claim 7 wherein R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($((\text{CH}_3)_3\text{C—})$), tert-amyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH—}$), sec-butyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”

390. Deelman together with Jones and Hänssgen renders obvious the solution of claim 7 for the reasons set forth in § XVI.G.

391. Deelman together with Jones and Hänssgen renders obvious the limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($((\text{CH}_3)_3\text{C—})$), tert-amyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH—}$)¹⁹, sec-butyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group” for the reasons set forth in § XV.D. and in § XV.E.

392. Accordingly, Deelman together with Jones together with Hänssgen renders obvious the limitation requiring “R comprises methyl ($\text{CH}_3\text{—}$), ethyl ($\text{CH}_3\text{CH}_2\text{—}$), isopropyl ($\text{CH}_3\text{CH}_2\text{CH—}$), tert-butyl ($((\text{CH}_3)_3\text{C—})$), tert-amyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH—}$), sec-butyl ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$),

¹⁹ Again, I note that claim 9 appears to have an error in the chemical formula for tert-amyl, which should be $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH—}$.

cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.”

J. Claim 10

i. [10Pre] “A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination, the method comprising,”

393. Deelman together with Jones and Hänssgen renders obvious the limitation requiring “a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.”

394. As I explained above, Hänssgen teaches “a method to selectively form a monoalkyltin trialkoxide compound” for the reasons set forth in § XI.I.

395. In my opinion, a POSITA would have been motivated to combine the high purity monoalkyltin triamide compositions formed from the combination of Deelman and Jones with the method to form a monoalkyltin trialkoxide compound as taught by Hänssgen for the reasons set forth in § XVI.A.

396. Accordingly, Deelman together with Jones and Hänssgen renders obvious the limitation requiring “a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.”

ii. [10a] reacting the composition of claim 1 with an alcohol represented by the formula HOR’ in an organic solvent to form $R\text{Sn}(\text{OR}')_3$,

397. Deelman together with Jones and Hänssgen renders obvious the composition of claim 1 for the reasons set forth in § XV.B.

398. Deelman together with Jones and Hänssgen further renders obvious the limitation requiring “reacting the composition of claim 1 with an alcohol represented by the formula HOR'' in an organic solvent to form $\text{RSn}(\text{OR}'')_3$.”

399. As I explained above (§ XI.I.ii), Hänssgen teaches reacting a composition like the high purity monoalkyltin triamide composition resulting from the combination of Deelman and Jones, with an alcohol represented by the formula HOR'' in an organic solvent to form $\text{RSn}(\text{OR}'')_3$. Specifically, as I explained above in § XI.I.ii., Hänssgen teaches alcoholysis of $\text{tBuSn}(\text{NEt}_2)_3$, with t-butanol “results in the ‘displacement reaction’ ... typical for aminostannanes to trialkoxystannane (II),” resulting in $\text{t-BuSn}(\text{O-t-Bu})_3$, which can be represented by the formula $\text{RSn}(\text{OR}'')_3$. EX1005, 3.

400. In my opinion, a POSITA would have been motivated to react the high purity monoalkyltin triamide composition resulting from the combination of Deelman and Jones with the alcohol solvent disclosed by Hänssgen, t-butanol, or any other alcohol, to obtain a monoalkyltin trialkoxide of the formula $\text{RSn}(\text{OR}'')_3$ for the reasons set forth in § XVI.A.

401. Accordingly, Meyers together with Deelman and Hänssgen renders obvious the limitation requiring “reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form $\text{RSn}(\text{OR}')_3$.”

iii. [10b] wherein R' is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,

402. Deelman together with Jones and Hänssgen renders obvious the limitation requiring “R” is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition” for the reasons set forth in § XI.I.iii.

iv. [10c] wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

403. Deelman together with Jones and Hänssgen renders obvious the limitation requiring “wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin” for the reasons set forth in § XV.B.iii.

404. As I explained above in § XV.B.iii., the combination of Deelman and Jones renders obvious a monoalkyltin triamide composition that has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

405. As I further explained in § IV.A, alcoholysis of organotin amides were known in the art, and in § XI.I, that Hänssgen expressly teaches alcoholysis of a monoalkyltin triamide compound with t-butanol in an organic solvent. As I explained in § XVI.A, a POSITA would have been motivated to combine the high

purity monoalkyltin triamide composition of Deelman and Jones with the teachings of Hänssgen to arrive at a monoalkyltin trialkoxide compound with no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

406. In my opinion, it would have been further obvious to a POSITA before the time of filing of the '903 Patent (Apr. 11, 2018) based upon the disclosure of Deelman, Jones, and Hänssgen to optimize the reaction conditions to minimize secondary reactions and/or apply known purification methods to known methods of the synthesis of monoalkyltin triamide compositions and known methods of performing the alcoholysis reactions on those compositions to yield predictable success, *i.e.*, to optimize the purity of the composition to remove the dialkyltin compounds, including up to no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

407. Accordingly, Deelman together with Jones and Hänssgen renders obvious the limitation requiring “wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.”

**XVII. NO SECONDARY CONSIDERATIONS SUPPORT
NONOBVIOUSNESS**

408. I have been informed and understand that during prosecution of the '903 Patent the Patent Owner argued that a “recently published patent application US 2022/0242888, filed by Entegris, Inc.,” supported the patentability of the rejected

claims of the '903 Patent (*see* § XI-XVI discussing claims 1-4 and 6-10) because the Patent Owner argued that the Entegris Patent Application Publication (EX1013) shows there was an “unmet need for highly pure monoalkyl tin triamide compounds.” EX1004, 548-550 (File History showing Patent Owner argument). I disagree for the reasons described below that the Entegris Patent Application Publication, US 2022/0242888 (EX1013) shows an “unmet need for highly pure monoalkyl tin triamide compounds,” as the Patent Owner argued.

409. I have reviewed the Entegris Patent Publication (EX1013) and it does not state that there is an “unmet need for highly pure monoalkyl tin triamide compounds,” as the Patent Owner argued during prosecution. EX1004, 548-550. Rather, the Entegris Patent Publication states there was a need for “improved **methodology** for manufacturing” the organotin compounds “in highly pure forms for use in the deposition of highly pure tin oxide films.” EX1013, [0004]. Thus, the Entegris Patent Publication does not support Patent Owner’s argument that the Entegris Patent Publication discloses an unmet need for the high purity organotin compounds themselves.

410. By contrast, the claims of the '903 Patent discussed above (§§ XI-XVI) are directed to monoalkyl tin triamide compositions, and do not pertain to the need for a new synthesis process as identified in the Entegris Patent Publication. Thus, in my opinion, the Entegris Patent Publication does not support Patent Owner’s

assertion that there was an “unmet need for a highly pure monoalkyl tin triamide compounds.” The Entegris Patent Publication does not state there is an “unmet need for highly pure monoalkyl tin triamide compounds” and, as I explained above (§§ XI-XVI), the prior art already disclosed such compositions.

411. I am unaware of any other alleged evidence of secondary considerations with respect to the '903 Patent that would render any of the Challenged Claims (claims 1-4 and 6-10 of the '903 Patent) non-obvious but reserve the right to supplement my opinions in the future to respond to any additional secondary considerations raised by Patent Owner.

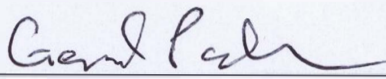
XVIII. RESERVATION OF RIGHTS

412. My opinions are based upon the information that I have considered to date. I reserve the right, however, to supplement my opinions in the future to respond to any arguments raised by the owner of the '903 Patent and to consider new information that becomes available to me.

XIX. DECLARATION

413. All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true. I further understand that willful false statements and the like are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code. I declare under penalty of perjury that the foregoing is true and correct.

Executed on December 12, 2024.

A handwritten signature in dark ink, appearing to read "Gerard Parkin", is written over a horizontal line.

Dr. Gerard Parkin

Attachment A

Gerard Parkin
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Personal

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Education

D. Phil.: The Queen's College, Oxford, England (1985)

M. A.: The Queen's College, Oxford, England (1984)

B. A.: The Queen's College, Oxford, England (1981)

Research Positions

Columbia University, New York, New York, Professor (1994 –); Chairman (1999 – 2002)

Columbia University, New York, New York, Associate Professor (1991)

Columbia University, New York, New York, Assistant Professor (1988)

California Institute of Technology, Pasadena, California, NATO Postdoct. Fell. (1985 – 88)

Honors and Awards

- American Chemical Society Project SEED Certificate of Recognition (2024)
- American Chemical Society Project SEED Outstanding Mentor Award (2022)
- Elected Fellow of the American Association for the Advancement of Science (2022)
- The Senator Penny Williams Distinguished Lecturer, Oklahoma School of Science and Mathematics Foundation (2022)
- Distinguished Karcher-Barton Awardee Lecturer, The University of Oklahoma (2022)
- New York Section of the American Chemical Society Outstanding Four-Year University with Graduate School Chemistry Faculty Teaching Award (2021)
- The Anton B. Burg Memorial Lecture, University of Southern California (2019)
- The Malcolm L. H. Green Lecture, Oxford University (2019)
- The Coates Lecture, The University of Wyoming (2018)
- James Flack Norris Award for Outstanding Achievement in the Teaching of Chemistry (2018)
- The Distinguished Raymond Siedle Lecture, Indiana University (2017)
- National Academies Education Fellow in the Sciences (2015 – 2016)
- Royal Society of Chemistry Ludwig Mond Award (2014)
- Lenfest Distinguished Columbia Faculty Award (2013)
- United States Presidential Award for Excellence in Science, Mathematics and Engineering Mentoring (2011)
- Dow Lecturer, University of California, Berkeley (2011)
- Scholar's Day Lecturer, Western Washington University (2011)
- Elected Fellow of the Royal Society of Chemistry (2010)

- Royal Society of Chemistry Chem Soc Rev Lecture Award (2010)
- Elected Inaugural Fellow of the American Chemical Society (2009)
- Columbia University Presidential Award for Outstanding Teaching (2008)
- American Chemical Society Award in Organometallic Chemistry (2008)
- Royal Society of Chemistry Award in Organometallic Chemistry (2004)
- Inaugural Distinguished Catalysis Lecturer, University of Florida, Gainesville (2004)
- Royal Society of Chemistry Corday Morgan Medal (1995)
- American Chemical Society Award in Pure Chemistry (1994)
- The Denkwalter Lecture, Loyola University, Chicago (1994)
- Elected Member of Sigma Xi, Columbia University (Kappa) Chapter
- The Distinguished Monsanto Lecture, Indiana University (1993)
- United States Presidential Faculty Fellowship (1992)
- Camille and Henry Dreyfus Teacher-Scholar Award (1991)
- Alfred P. Sloan Research Fellow (1991)
- *Polyhedron* Best Paper Award (1988)

Professional Activities

- New York Academy of Sciences, Inorganic Chemistry and Catalytic Science Section Chair (1993-1995)
- Editorial Advisory Board, *Accounts of Chemical Research* (1994-96)
- Editorial Advisory Board, *Inorganic Chemistry* (1995-97)
- Editorial Advisory Board, *Main Group Chemistry* (1996-)
- Symposium organizer: "Main Group Organometallic Compounds: Novel Structures and Reactivity," 206th American Chemical Society National Meeting, Chicago, Illinois, August 1993
- *Encyclopedia of Inorganic Chemistry*, Contributor
- *The Columbia Encyclopedia* (5th Edn.), Contributor
- Guest Editor, *Polyhedron* Symposium-in-Print: "Recent Developments in Organometallic Chemistry," January, 1995
- Symposium co-organizer on "New Vistas in Inorganic Chemistry," 211th American Chemical Society National Meeting, New Orleans, Louisiana, 24 – 28 March, 1996
- International Editorial Advisory Board, *J. Chem. Soc., Dalton Trans.* (1998 – 2003)
- Editorial Advisory Board, *Polyhedron* (1999 – 2000)
- Editorial Advisory Board, *J. Organomet. Chem.* (1999 – 2004)
- Editor, *Polyhedron* Symposia-in-Print (2000 –)
- ACS New York Section, Chair-Elect (2002); Chair (2003)
- Editorial Advisory Board, *Organometallics* (2002 – 2005)
- Symposium co-organizer on "Scorpionate Ligands Thirty-Five Years Later", 225th American Chemical Society National Meeting, New Orleans, Louisiana, 23 – 27 March, 2003

- *Comprehensive Coordination Chemistry 2*, Volume editor
- Editorial Advisory Board, *Inorganic Chemistry* (2003 – 2006)
- Gordon Research Conference in Organometallic Chemistry: vice-Chair (2005); Chair (2006)
- *Comprehensive Organometallic Chemistry III*, Volume editor
- Organometallic Subdivision of the American Chemical Society Division of Inorganic Chemistry: Chair-Elect (2005); Chair (2006)
- Editorial Board for *Structure and Bonding* (2007 – 2015)
- New York Academy of Sciences Blavatnik Awards for Young Scientists Selection Committee (2007-2011)
- ACS Awards Selection Committee (2007-2009)
- Editor, *SciTopics* (2008 – 2011)
- Editor, *Structure and Bonding*, Volume 136, “Metal–Metal Bonding”
- Department of Energy ARPA–E IMPACCT Review Panel (2010)
- Editorial Advisory Board, *Dalton Trans.* (2012 –)
- Reaxys PhD Prize Jury (2012 – 2021)
- Symposium co-organizer on “Organometallic Chemistry: The New frontiers”, 248th American Chemical Society National Meeting, San Francisco, California (2014)
- Chemist on National Geographic Channel’s *Going Deep with David Rees* episode on “How to light a match” (2014)
- Science Advisor, *Tonight Show* with Jimmy Fallon (2014 – 2019)
- Symposium organizer for Jonathan Owen Pure Chemistry Award, 251st American Chemical Society National Meeting, San Diego, California (2016)
- *Comprehensive Coordination Chemistry 3*, Co-editor-in-chief and section editor (2018 – 2021).
- *Comprehensive Organometallic Chemistry 4*, Co-editor-in-chief (2019 – 2022)
- Member of the NY ACS committee to recognize Marie Maynard Daly, the first African American woman to receive a PhD in chemistry in the United States, with an ACS National Historic Chemical Landmark at Columbia University (2022–).

Media Coverage of Research

- “Bond Stretch Isomerism may be an Artifact.” *Chemical and Engineering News* **1991**, March 4, 20 (R. Baum).
- “The Case of the Unlikely Molecular Twins.” *Science* **1991**, 254, 1452-1453 (I. Amato).
- “False Minima can Cause Crystal Structure Errors.” *Chemical and Engineering News* **1995**, October 2, 27-29 (S. Borman).
- “A Bridge to New Metallocene Chemistry” *Chemical and Engineering News* **1999**, June 28, 23.
- “Mo Reactivity Toward S and N Heterocycles.” *Chemical and Engineering News* **2002**, November 25, 18 (S. Ritter).
- “New Ligand Works in a Pinch.” *Chemical and Engineering News* **2002**, April 2, 24 (R. Baum).

- “Methylmercury detox.” *Chemical and Engineering News* **2007**, July 16, 10 (S. Ritter).
- “Model Enzyme Attacks Alkyl Mercury”. *Chemistry World* **2007**, July 12 (T. Westgate).
- “Working out Mercury.” *Environmentalresearchweb* **2007**, July 27 (L. Kalaugher).
- “Toward Methylmercury Bioremediation.” *Science* **2007**, 317, 205 (J. G. Omichinski).
- “Mercury–Alkyl Bond Cleavage Based on Organomercury Lyase” *Angew. Chem. Int. Ed.* **2007**, 46, 2 (H. Strasdeit).
- “Cleaving C-Hg Bonds: Two Thiolates are Better than One.” *Nature Chem. Biol.* **2007**, 3, 537 (S. M. Miller).
- “Selenium’s Role in Mercury’s Toxicity.” *Chemical and Engineering News* **2010**, January 11, 33 (S. Ritter).
- “Cracking Carbon–Carbon Bonds.” *Chemistry World* **2010**, March 29 (P. Broadwith).
- “Make or Break.” *Science* **2010**, 327, 923 (February 19), Editor’s Choice (J. S. Yeston).
- “Tungsten Breaks Tough Bond.” *Chemical and Engineering News* **2010**, January 26, 30 (S. Ritter).
- “Carbon–Carbon Bonds get a Break.” *Nature* **2010**, 463, 435, January 28 (A. S. Goldman).
- “Molybdenum Hydride Complexes Work Magic on Formic Acid.” *Chemical and Engineering News* **2015**, January 26, 30 (S. Ritter).
- “New Catalyst Puts CO₂ to Good Use”. *Chemical and Engineering News* **2018**, January 1, 8 (S. Ritter).
- “CO₂ converts into formaldehyde at room temperature via a shelf-stable intermediate.” *Chemical and Engineering News* **2019**, November 25, C. Zhang.
- “Formaldehyde from CO₂.” *Chemistry Views* **2019**, October 29
(https://www.chemistryviews.org/details/news/11193896/Formaldehyde_from_CO2.html) (S. Maier).
- “Three centres, two electrons, but how many bonds?” *Chemistry World*, **2019**, November, p. 33
(<https://www.chemistryworld.com/news/three-centres-two-electrons-but-how-many-bonds/4010498.article>) (F. Gomollón-Bel).
- “Lessons from an unlikely cadmium carbonyl compound” *Chemistry World*, 2020, December, p. 46
(<https://www.chemistryworld.com/news/lessons-from-an-unlikely-cadmium-carbonyl-compound/4012710.article>) (K. McRoberts).
- “A crystallographic conundrum” *Science* **2020**, 370, 928 (J. Yeston).

Gerard Parkin

Complete List of Publications

1. "Synthetic Pathways in Binuclear Molybdenum Chemistry: μ -Acetato, Chloro, and Tertiary Phosphine Derivatives; X-ray Structure Determination of *trans*-[MoCl(PBuⁿ)₃](μ -O₂CMe)₂." Malcolm L. H. Green, Gerard Parkin, Jim Bashkin, John Fail and Keith Prout *J. Chem. Soc., Dalton Trans.* **1982**, 2519-2525.
2. "Trimethylphosphine-tungsten Chemistry: Hydrido, Silyl, Fluoro, Hydroxy and Aquo Derivatives; Crystal structure of [W(PMe₃)₄H₂(OH₂)F]F." Malcolm L. H. Green, Gerard Parkin, Chen Mingqin and Keith Prout *J. Chem. Soc., Chem. Commun.* **1984**, 1400-1402.
3. "Formation of (η -Cyclopentadienyl)pentahydrido(trimethylphosphino)-tungsten from Cyclopentane, Trimethylphosphine and Tungsten atoms." Malcolm L. H. Green and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1984**, 1467-1468.
4. "Formation of an η^2 -Formaldehyde compound from Methanol and its Hydrogenation giving Methanol." Malcolm L. H. Green, Gerard Parkin, Kelly J. Moynihan and Keith Prout *J. Chem. Soc., Chem. Commun.* **1984**, 1540.
5. "Synthetic Use of Lithium Nitride, an Unusual Reducing agent. Formation of Ti₄ and Ti₆ Complexes." Melvyn Kilner, Gerard Parkin and Andrew G. Talbot *J. Chem. Soc., Chem. Commun.* **1985**, 34-35.
6. "Formation of (η -Benzene) or (η -Cyclohexa-1,3-diene) Compounds of Chromium, Tungsten and Rhenium from Cyclohexane and the Metal atoms." Malcolm L. H. Green, Dermot O'Hare and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1985**, 356-357.
7. " η -Arene and η -Cyclohexadiene Tungsten Chemistry: New Synthetic Routes to Tertiaryphosphine-Hydride Derivatives." Malcolm L. H. Green and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1985**, 771-773.
8. "The Synthesis of Electron-rich η -Cyclopentadienyl Trimethylphosphine Complexes of Tungsten." Malcolm L. H. Green and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1985**, 1383-1384.
9. "Tertiary Phosphine Complexes of Tungsten." Gerard Parkin *Reviews in Inorganic Chemistry* **1985**, 7, 251-297.
10. "Ethylene Insertion into the W-C bond of the η^2 -Formaldehyde Ligand System of W(PMe₃)₄(η^2 -CH₂O)H₂ giving the Oxometallacyclopentane Derivative W(OCH₂CH₂CH₂)(PMe₃)₂(C₂H₄)₂ and Related Studies." Malcolm L. H. Green and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1986**, 90-91.
11. "The reaction of [W(PMe₃)₄(η^2 -CH₂PMe₂H)] with Carbon Dioxide and Dihydrogen: Characterization of [(W(PMe₃)₃H₂(η^1 -PMe₂CH₂)₂(C₃H₂O₆))] using Two-dimensional Nuclear Magnetic Resonance Spectroscopy." Malcolm L. H. Green, Gerard Parkin, Dermot O'Hare, Luet-Lok Wong and Andrew E. Derome *J. Organomet. Chem.* **1986**, 317, 61-68.
12. "Lithium Nitride Reduction of Cp₂TiCl₂ and CpTiCl₃: The Synthesis of (Cp₂TiCl)₂, (CpTiCl₂)_n, Cp₂Ti(CO)₂ and Chloro-Ti₄ and Nitrido-Ti₆ Complexes." Melvyn Kilner and Gerard Parkin *J. Organomet. Chem.* **1986**, 302, 181-191.
13. "The Chemistry of [W(PMe₃)₄(η^2 -CH₂PMe₂H)]: Synthesis of Hydroxy-hydrido, Fluoro-hydrido and Silyl-hydrido complexes and the Dimerisation of Ethylene and Propylene giving η^4 -Diene Derivatives." Mingqin Chen, Malcolm L. H. Green, Gerard Parkin and Keith Prout *J. Chem. Soc., Dalton Trans.* **1986**, 2227-2236.
14. "Synthesis of (η -Cyclopentadienyl)(trimethylphosphine)tungsten Derivatives and Related Chemistry." Malcolm L. H. Green and Gerard Parkin *J. Chem. Soc., Dalton Trans.* **1987**, 1611-1618.
15. "Alpha-and Beta-Migratory Insertion and Elimination Processes for Alkyl Complexes of Permethylscandocene and Permethyltantlocene." Gerard Parkin, Emilio Bunel, Barbara J. Burger, Mark. S. Trimmer, Allan van Asselt and John E. Bercaw *J. Mol. Catalysis* **1987**, 41, 21-39.
16. "Structure of (Pentamethylcyclopentadienyl)(pentamethyl-cyclopentadienyloxy)dioxotungsten(VI), (η^5 -C₅Me₅)W(=O)₂(OC₅Me₅). Structural Evidence for the Strong *trans* influence of the Oxo Ligand." Gerard Parkin, Richard E. Marsh, William P. Schaefer and John E. Bercaw *Inorg. Chem.* **1988**, 27, 3262-3264.
17. "Halide, Hydride, Alkyl, Oxo and Related Derivatives of Bis(pentamethylcyclopentadienyl)tungsten(IV)." Gerard Parkin and John E. Bercaw *Polyhedron* **1988**, 7, 2053-2082.
18. "(η^5 -C₅Me₅)₂W=O: An Exceptionally Reactive Organometallic Oxo Derivative. Reduction with H₂ and Reaction with O₂ Resulting in Insertion of Oxygen into a Tungsten-Carbon Bond." Gerard Parkin and John E. Bercaw *J. Am. Chem. Soc.* **1989**, 111, 391-393.
19. "Direct Protonation of the W-H Bonds of Bis(pentamethylcyclopentadienyl)tungsten Dihydride." Gerard Parkin and John E. Bercaw *J. Chem. Soc., Chem. Commun.* **1989**, 255-257.
20. "Elimination Processes for Alkyl, Hydride, and Hydroxy Derivatives of Permethyltungstenocene." Gerard Parkin and John E. Bercaw *Organometallics* **1989**, 8, 1172-1179.
21. "Tris(pyrazolyl)hydroborato Magnesium and Aluminum Alkyl Derivatives: Alkyl Exchange with Methyl Iodide and Enolate Formation with Acetone." Runyu Han, Adrian Looney and Gerard Parkin *J. Amer. Chem. Soc.* **1989**, 111, 7276-7278.
22. "Poly(pyrazolyl)hydroborato and Poly(pyrazolyl)methane Aluminum Alkyl Derivatives." Adrian Looney and Gerard Parkin *Polyhedron* **1990**, 9, 265-276.
23. "Tris(pyrazolyl)hydroborato Zinc Alkyl Derivatives: Direct Comparison of the Reactivity of Zn-C and Mg-C Bonds." Ian B. Gorrell, Adrian Looney and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1990**, 220-222.
24. "Insertion of O₂ into the Mg-C Bonds of the Alkyl Derivatives { η^3 -HB(3-Bu^p)₃}MgR (R = CH₃, CH₂CH₃, CH(CH₃)₂, C(CH₃)₃): Formation of Alkylperoxo Derivatives and Oxygen Atom Transfer." Runyu Han and Gerard Parkin *J. Am. Chem. Soc.* **1990**, 112, 3662-3663.
25. "{Bis(3-tert-Butylpyrazolyl)hydroborato}zinc Alkyl Derivatives: Competitive Reactivity of Zn-C and B-H Bonds." Ian B. Gorrell, Adrian Looney, Gerard Parkin and Arnold L. Rheingold *J. Am. Chem. Soc.* **1990**, 112, 4068-4069.
26. "Tris(3-t-butylpyrazolyl)hydroborato Manganese, Iron, Cobalt and Nickel Halide Derivatives: Facile Abstraction of Fluoride from [BF₄]⁻." Ian B. Gorrell and Gerard Parkin *Inorg. Chem.* **1990**, 29, 2452-2456.
27. "Hexakis(trimethylphosphine)tungsten(0): Synthesis, Structure and Reactivity." Daniel Rabinovich and Gerard Parkin *J. Am. Chem. Soc.* **1990**, 112, 5381-5383.

28. "Competitive Alkyl and Halide Metathesis in the Reactions of Grignard Reagents with $\text{Ti}(\eta^3\text{-HB(3-Bu}^t\text{pz)}_3)_2$." Runyu Han, Max Bachrach and Gerard Parkin *Polyhedron* **1990**, 9, 1775-1778.
29. "Ligand Redistribution Reactions of $\text{Tris(3,5-dimethylpyrazolyl)hydroboratomagnesium Alkyl Derivatives: Crystal Structure of } \{\eta^3\text{-HB(3,5-Me}_2\text{pz)}_3\}_2\text{Mg}$." Runyu Han and Gerard Parkin *J. Organomet. Chem.* **1990**, 393, C43-C46.
30. "Reaction of O_2 with $\{\eta^3\text{-HB(3-Bu}^t\text{pz)}_3\}\text{MgCH}_2\text{Si(CH}_3)_3$: Facile Si-C Bond Cleavage and Formation of $\{\eta^3\text{-HB(3-Bu}^t\text{pz)}_3\}\text{MgOSi(CH}_3)_3$." Runyu Han and Gerard Parkin *Polyhedron* **1990**, 9, 2655-2657.
31. "Competitive sp^3 and sp^2 C-H Bond Activation of Phenols by $\text{W(PMe}_3)_6$ and $\text{W(PMe}_3)_4(\eta^2\text{-CH}_2\text{PMe}_2)\text{H}$: Formation of Four and Five-Membered Oxametallacycles." Daniel Rabinovich, Ross Zelman and Gerard Parkin *J. Am. Chem. Soc.* **1990**, 112, 9632-9633.
32. "Synthesis and Reactivity of Permethylyltantalocene Derivatives Possessing η^3 -Allyl, η^2 -Butadiene, η^2 -Methylallene and η^1 -Alkenylidene Ligands. Model Studies for the Role of Surface Vinylidenes in the Fischer-Tropsch Hydrocarbon-Chain-Lengthening Process." Vernon C. Gibson, Gerard Parkin and John E. Bercaw *Organometallics* **1991**, 10, 220-231.
33. "A Re-Investigation of the Molecular Structures of $\text{cis-mer-MoOCl}_2(\text{PR}_3)_3$: Do Bond-Stretch Isomers Really Exist?" Keum Yoon, Gerard Parkin and Arnold L. Rheingold *J. Am. Chem. Soc.* **1991**, 113, 1437-1438.
34. " $\text{Tris(pyrazolyl)hydroborato Magnesium Alkyl Derivatives: Synthetic and Structural Studies}$." Runyu Han and Gerard Parkin *Organometallics* **1991**, 10, 1010-1020.
35. " $\text{Tris(3-t-butylpyrazolyl)hydroborato Zinc Hydride: Synthesis, Structure and Reactivity of a Monomeric Zinc Hydride Derivative}$." Runyu Han, Ian B. Gorrell, Adrian G. Looney and Gerard Parkin *J. Chem. Soc., Chem. Commun.* **1991**, 717-719.
36. "Anion Coordination by Protonated $\text{Tris(pyrazolyl)hydroborato Derivatives: The Crystal Structure of the Host-Guest Complex } [\{\eta^3\text{-HB(3-Bu}^t\text{pzH)}_3\}\text{Cl}][\text{AlCl}_4]$." Adrian Looney, Gerard Parkin and Arnold L. Rheingold *Inorg. Chem.* **1991**, 30, 3099-3101.
37. "The Syntheses, Structures, and Reactivity of Monomeric Tungsten(IV) and Tungsten(VI) Bis(sulfido) Complexes: Facile Elimination of H_2 from H_2S ." Daniel Rabinovich and Gerard Parkin *J. Am. Chem. Soc.* **1991**, 113, 5904-5905.
38. "A Mononuclear Zinc Hydroxide Complex Stabilized by a Highly Substituted $\text{Tris(pyrazolyl)hydroborato}$ Ligand: Analogies with the Enzyme Carbonic Anhydrase." Ralf Alsasser, Swiatoslaw Trofimenko, Adrian Looney, Gerard Parkin and Heinrich Vahrenkamp *Inorg. Chem.* **1991**, 30, 4098-4100.
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PATENTS

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