

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

ENTEGRIS, INC.
Petitioner

v.

INPRIA CORP.
Patent Owner

Case No. IPR2025-00267
Patent 11,673,903

PETITION FOR *INTER PARTES* REVIEW OF
U.S. PATENT NO. 11,673,903

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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”).

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
1010	U.S. Patent No. 11,697,660.
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.

MANDATORY NOTICES

Real Party in Interest: Entegris, Inc. is the petitioner and a real party-in-interest. No other party was involved in any aspect of this Petition.

Related Matters: The '903 Patent is currently being asserted by Inpria Corp. ("Patent Owner" or "PO") against Lam Research Corp., Case No. 1:22-cv-01359 in the United States District Court for the District of Delaware (the "Delaware Litigation"), but has not otherwise been asserted against any other entity, including Petitioner.

Lead Counsel and Back-Up Counsel: Petitioner designates lead and back-up counsel as noted below. A Power of Attorney is filed concurrently herewith under 37 C.F.R. § 42.10(b).

LEAD COUNSEL	BACK-UP COUNSEL
Eliot D. Williams (Reg. No. 50,822) eliot.williams@bakerbotts.com BAKER BOTTS L.L.P. 700 K Street, N.W. Washington, D.C. 20001-5692 Phone: 202.639.7700 Fax: 202.639.7890	Katharine Burke (<i>pro hac vice</i> application forthcoming) katharine.burke@bakerbotts.com BAKER BOTTS L.L.P. 700 K Street, N.W. Washington, D.C. 20001-5692 Phone: 202.639.7700 Fax: 202.639.7890 Frank Zhu (Reg. No. 71,214) frank.zhu@bakerbotts.com BAKER BOTTS L.L.P. 30 Rockefeller Plaza New York, NY 10112-4498 212.408.2500 Amy E. Bergeron (Reg. No. 76,974) amy.bergeron@bakerbotts.com BAKER BOTTS L.L.P. 910 Louisiana Street Houston, Texas 77002 713.229.1234

Service Information: A copy of the entire Petition, including all Exhibits and Power of Attorney is being served by FedEx, costs prepaid, to the address of the attorney or agent of record for PO at the USPTO: Douglas Christensen, Christensen, Fonder, Dardi & Herbert PLLC, 11322 86th Ave. N., Maple Grove, MN 55369.

Correspondence to lead and back-up counsel can be provided at the postal and electronic addresses above. Petitioner also consents to service by electronic mail at DLEntegris-InpriaIPR@Bakerbotts.com.

A Power of Attorney is filed concurrently herewith under 37 C.F.R. § 42.10(b).

CLAIMS LISTING

Claim 1

[1pre]	A high purity liquid composition comprising:
[1a]	a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ and
[1b]	no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,
[1c]	wherein R is a hydrocarbyl group with 1-31 carbon atoms, and
[1d]	wherein R' is a hydrocarbyl group with 1-10 carbon atoms.

Claim 2

[2pre]	The composition of claim 1
[2a]	wherein R is a branched alkyl ligand represented by $\text{R}^1\text{R}^2\text{R}^3\text{C—}$, 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.

Claim 3

[3pre]	The composition of claim 1
[3a]	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 ($\text{CH}_3(\text{CH}_3\text{CH}_2)\text{CH—}$), neopentyl ($(\text{CH}_3)_3\text{CCH}_2\text{—}$), cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl.

Claim 4

[4pre]	The composition of claim 1
[4a]	wherein R' comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.

Claim 6

[6pre]	The composition of claim 1
[6a]	comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.

Claim 7

[7]	A solution comprising the composition of claim 1 and an organic solvent.
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Claim 8

[8]	The solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.
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Claim 9

[9]	The solution of claim 7 wherein R comprises methyl (CH ₃ —), ethyl (CH ₃ CH ₂ —), isopropyl (CH ₃ CH ₂ CH—), tert-butyl ((CH ₃) ₃ C—), tert-amyl (CH ₃ CH ₂ (CH ₃) ₂ C—), sec-butyl (CH ₃ (CH ₃ CH ₂)CH—), neopentyl (CH ₃) ₃ CCH ₂ —, cyclohexyl, cyclopentyl, cyclobutyl, or cyclopropyl, and wherein R' comprises a methyl group, ethyl group, isopropyl group, t-butyl group, or t-amyl group.
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Claim 10

[10pre]	A method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination, the method comprising,
[10a]	reacting the composition of claim 1 with an alcohol represented by the formula HOR'' in an organic solvent to form RSn(OR'') ₃ ,
[10b]	wherein R'' is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,
[10c]	wherein the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.

I. INTRODUCTION

Entegris, Inc. (“Petitioner”) respectfully submits this Petition for *inter partes* review (“IPR”) of Claims 1-4 and 6-10 (the “Challenged Claims”) of U.S. Patent No. 11,673,903 (the “’903 Patent”).

As PO recognized during prosecution, the claimed monoalkyltin triamide compositions were well-known in the art long before the filing of the ’903 Patent. The **only** purported novelty of the Challenged Claims is the level of purity of these well-known compositions. The prior art disclosures made clear that the claimed purity level was known, desirable, and achievable. However, the Office allowed the claims based on declaration statements made by the inventor during prosecution asserting that following the prior art’s teachings could not produce the claimed level of purity. As Petitioner demonstrates herein, PO’s assertions during prosecution were wrong, and following the prior art’s teachings (and obvious variants thereof) does, as the Examiner initially believed, result in high purity monoalkyl tin triamide compositions within the scope of the claims.

II. MANDATORY NOTICES UNDER 37 C.F.R. 42.8(A)(1)

A. Grounds for Standing

Petitioner certifies that the ’903 Patent is available for IPR and that Petitioner is not barred or otherwise estopped.

B. Identification of Challenge and Statement of Precise Relief Requested

This IPR, supported by the declaration of Dr. Gerard Parkin (EX1002), requests cancellation of Claims 1-4 and 6-10 of the '903 Patent under the following grounds.

Ground	Claims	Statutory Basis (35 U.S.C. §)	Prior Art
1A	1–4, 6-10	102	<i>Hänssgen</i>
1B	1–4, 6–10	103	<i>Hänssgen in view of the knowledge of a person of ordinary skill in the art (“POSITA”)</i>
2A	1–4, 6	103	<i>Meyers in view of Deelman</i>
2B	1-4, 6-10	103	<i>Meyers in view of Deelman and Hänssgen</i>
3A	1–4, 6	103	<i>Deelman in view of Jones</i>
3B	1–4, 6-10	103	<i>Deelman in view of Jones and Hänssgen</i>

III. OVERVIEW OF THE '903 PATENT AND PROSECUTION HISTORY

A. The '903 Patent

The alleged invention of the '903 Patent involves either of two compositions: “a monoalkyltin trialkoxide compound represented by the chemical formula RSn(OR')_3 or a monoalkyl tin triamide compound represented by the chemical

formula $\text{RSn}(\text{NR}'_2)_3$...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. The '903 Patent acknowledges that such compounds, including “monoalkyl tin triamides,” previously existed, citing the description in Hänssgen of synthesizing “t-butyl tris(diethyl-amido)tin, $(\text{t-BuSn}(\text{NEt}_2)_3)$ ” by reactions with a lithium reagent. EX1001, 3:29-44.

The alleged invention instead relates to a desired purity characteristic of such known compounds. EX1001, 3:29-44. For example, the '903 Patent asserts that the method of Hänssgen “can produce a mixture of monoalkyl and dialkyl tin products,”² but that “it can be desirable to reduce any polyalkyl byproducts, e.g., dialkyl tin contaminants.” EX1001, 3:29-44. In particular, the patent states that “[p]olyalkyl tin impurity compositions [such as dialkyl tin] 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.

Notably, the patent acknowledges that methods of purifying monoalkyl compounds also previously existed, stating that separation of monoalkyl and dialkyl species “can be [done] for some compounds[.]” EX1001, 3:44-52. The '903 Patent also admits that the compounds of the alleged invention can either “be used as

² All emphases added unless otherwise noted.

synthesized or further purified,” including subsequent purification by “fractional distillation” (a method known in the art) “to effectively reduce polyalkyl contaminants below levels that may already be relatively low from the direct synthesis.” EX1001, 3:2-5, 3:13-17. The patent asserts that such a “separation or purification process generally raises the cost of manufacturing,” EX1001, 3:44-52, but, notably, the Challenged Claims do not recite limitations requiring particular cost or efficiency or precluding a subsequent purification process.³ Indeed, according to the ’903 Patent, “subsequent purification, such as with fractional distillation” can be desirable because it “results in even lower dialkyl or polyalkyl contamination.” EX1001, 3:44-52.

B. Prosecution History and PO’s Declarations

The ’903 Patent application was filed on April 11, 2018. After a Final Rejection on various grounds, PO filed an RCE amending Claim 1 to specify the claimed composition is a “high purity liquid composition” and to modify the purity limitation as follows: “no more than [[4]]0.5 mole% dialkyltin compounds as an impurity relative to the total tin amount[.]” EX1004, 893. PO also submitted an inventor declaration “regarding the novelty of the claimed purity with respect to dialkyl impurities,” and presenting “results of in-house experiments” purportedly

³ In the Delaware Litigation, PO asserts the claims do not exclude a purification step. EX1012, 43-53. Petitioner applies PO’s proposed construction in this proceeding.

following the admitted “state of the art” method of Hänssgen. EX1004, 896. The declaration claimed that the “post-distillation product had an average of approximately 2% dialkyltin triamide, $\text{tBu}_2\text{Sn}(\text{NEt}_2)_2$,” and, based on that, concluded “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, 897.

Subsequently, the Examiner again rejected the claims on several grounds, including (1) the combination of Bernhardt (DE3109309) in view of Merz III (U.S. Patent Application No. 2013/028125); and (2) Frenkel (U.S. Patent Application No. 2017/0002180). For claim 1, the Examiner found Bernhardt disclosed everything except for “no more than 0.5 of 0.1 mole% of dialkyltin impurity compounds and a branched alkyl group” but Merz III taught production of “monoalkyltin trihalide with an overall yield of nearly 100%,” satisfying the purity limitation. EX1004, 631-632. The Examiner also found Frenkel disclosed “a composition comprising at least one monooctyl tin compound $(\text{n-OC})\text{Sn}(\text{T})_3$ wherein tin is an alcoholate[] having a purity greater than 85%, preferably up to 99wt%[,],” and found the purity limitation obvious in view of Frenkel’s disclosure of “as low as 0.1 wt% of dioctyl tin compounds and this overlaps with the instantly claimed range.” EX1004, 634-635.

The Examiner considered PO's declaration but stated "the experimental methods disclosed...are not comparable" to the art cited in the rejections. EX1004, 637.

In response, PO distinguished Frenkel by amending Claim 1 to delete "a monoalkyltin trialkoxide compound" (leaving only "a monoalkyl tin triamide compound"). EX1004, 618, 623-624. PO argued that Bernhardt's Grignard-reaction synthesized chloride products result in "technical problems" and "high dialkyl impurities," and the known "[p]urification techniques...do not reasonably enable the claimed level of mono-alkyl purity." EX1004, 621.

In response, the Office issued a Final Office Action on January 6, 2022, which, among other things, withdrew the rejections over Bernhardt and Frenkel in view of Applicant's amendments, but rejected Claims 1-9 as obvious over U.S. Patent Application No. 2011/0166268 ("Deelman") in view of U.S. Patent Application No. 2016/0116839 ("Meyers"). EX1004, 599-600. For the purity limitation, the Examiner noted that "Deelman ... fails to specifically disclose no more than 0.5 or 0.1 mole% dialkyltin compounds," but found it would have been obvious 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.

PO submitted a response on February 28, 2022, arguing, based on the inventor's 2021 declaration, that the claimed purity was not achievable through an

alkyl lithium reaction, and that Deelman and Meyers fail to teach the claimed purity. EX1004, 592. The Examiner was unconvinced and issued an Advisory Action on April 1, 2022, rejecting PO's arguments because the claims were directed to the composition, not the method of manufacture. EX1004, 585.

On September 16, 2022, PO filed an RCE, repeating the same arguments from the February 28, 2022 response and arguing that the method of making the composition affects both the product produced and its purity. EX1004, 546-547. PO also filed another inventor declaration which purportedly "presents results of an in-house experiment that **adapted** the method of Deelman to try to make a monoalkyltin triamide product." EX1004, 547 (emphasis in original). Specifically, "Applicant reacted an $\text{RSn}(\text{Hal})_3$ compound (n-butyltin trichloride) with a 3-fold molar excess of an H-T compound (diethylamine), **and water**, under stirring, to try to form an $\text{RSn}(\text{T})_3$ product (nbutyltin trisdiethylamine)." EX1004, 547. The inventor observed that "[i]mmmediately upon combining the reagents, a bulk, white precipitate was formed[]" "consistent with the formation of nBuSnOOH ," and concluded "the claimed high purity liquid compositions according to claim 1 are not achievable through the method disclosed by Deelman et al." EX1004, 552-553. Notably, nothing in Deelman directs synthesis of monoalkyltin triamide in an **aqueous** solution because it is well-known (as admitted by the inventor) that

“monoalkyltin triamides would be expected to hydrolyze in aqueous solution[.]” EX1002 ¶69 (citing EX1004, 553).

A rejection issued on October 27, 2022, maintaining the obviousness rejections over Deelman in view of Meyers, and stating that the “declaration...is insufficient to overcome the rejection...because...[it] does not give information regarding the test conditions of the process” so 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. PO conducted a telephonic Examiner interview on February 17, 2023, where “Applicant provided a proposed declaration and arguments regarding the information in the declaration,” and, “[o]n [February 21, 2023] 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, 462.

On February 24, 2023, PO submitted a revised Declaration, EX1004, 447-450, which stated that the “in-house experiment” “followed the general procedure of Example 2 of Deelman” “as appropriately modified for the target product composition.” EX1004, 444, 448. The substance of the “in-house experiment” did not change from the 2022 declaration and still described reacting “nBuSnCl₃ with HNEt₂ in **aqueous** solution and a subsequent neutralizing reaction with sodium hydroxide.” EX1004, 448. Notably, Example 2 of Deelman, which Applicant

purported to adapt to synthesize $\text{nBuSn}(\text{NEt}_2)_3$, is directed to synthesis of “Mono-Octyltin Tris((2-Ethylhexyl)Mercaptoacetate),” which is not a monoalkyl tin triamide compound. EX1002, ¶76. Nonetheless, PO concluded that the experiment replicated Example 2 of Deelman and did not successfully synthesize the “target $\text{nBuSn}(\text{NEt}_2)_3$ product.” EX1004, 445.

A Notice of Allowance issued with an Examiner’s Amendment. EX1004, 360-361. The Examiner’s “Reasons for Allowance” stated:

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.

IV. PO’S DECLARATIONS WERE MISLEADING AND INACCURATE

Despite initial skepticism, the Examiner allowed the Challenged Claims based on several inventor declarations purporting to replicate experiments to show that the prior art could not achieve the claimed purity. *E.g.*, EX1004, 360-361, 462. However, those declarations, and PO’s arguments stemming from them, are inaccurate, ambiguous, and misleading. A POSITA would have recognized that PO’s declaration statements lacked sufficient detail to allow a POSITA to assess the appropriateness of the reaction conditions, or plainly adopted reaction conditions

that are contrary to the knowledge of a POSITA. EX1004, 447-450, 551-553, 896-898; EX1002, ¶¶105-109.

Specifically, the 2021 declaration describes in a single paragraph that the inventor allegedly recreated the procedure of Hänssgen without disclosing any specific reaction conditions used to achieve the sub-threshold purity level. EX1004, 896-898; EX1002, ¶105. Without these details, a POSITA would not have had sufficient information to assess the appropriateness of the experiment. *Id.*

The 2022 declaration similarly lacked details and, more than that, described a synthesis method that was apparently designed to fail, as the inventor claimed to “follow[] the method of Deelman” but, in reality, conducted “the reaction of nBuSnCl_3 with HNEt_2 in **aqueous** solution” to synthesize monoalkyl tin triamide. EX1004, 552; EX1002, ¶¶106-108. Nothing in Deelman would have led a POSITA to take that path. Indeed, the inventor admitted that: “monoalkyltin triamides would be expected to hydrolyze in aqueous solution[.]” EX1004, 553. Given this well-known reactivity of monoalkyltin triamide with water, no POSITA would have chosen an aqueous solution for its synthesis. EX1002 ¶106. Accordingly, PO’s in-house experiment is irrelevant to a POSITA’s understanding of the teaching of Deelman regarding synthesis of monoalkyltin triamide, let alone the purity achievable by the disclosed approach. EX1002, ¶109.

Finally, the inventor's 2023 declaration responding to the Examiner's criticisms that the 2022 declaration failed to "give information regarding the test conditions of the process," EX1004, 471-472, compounded these errors. The 2023 declaration is substantively the same as the 2022 declaration and depicts the same failed experiment—"reaction of nBuSnCl_3 with HNet_2 in aqueous solution"—but added the assertion that the experiment "follow[s] the general method as disclosed in Example 2 of Deelman." EX1004, 448. However, a POSITA would not have followed Example 2 of Deelman to synthesize $\text{nBuSn(NEt}_2)_3$ because it does not relate to synthesis of moisture sensitive monoalkyltin triamide compounds, and instead describes an entirely different category of compound—mono-octyltin tris((2-ethylhexyl) mercaptoacetate), which is stable in aqueous medium. EX1002, ¶¶106-108. In fact, the Meyers reference cited by the Examiner, of which 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. EX1002, ¶106 (citing EX1006, [0151]). PO's in-house experiment results did not follow these teachings and are simply irrelevant to the question PO purported to answer. EX1002, ¶¶108-109.

To demonstrate that the disclosures of Hänssgen, Deelman, and Meyers enable a POSITA to achieve the claimed purity of monoalkyltin triamide

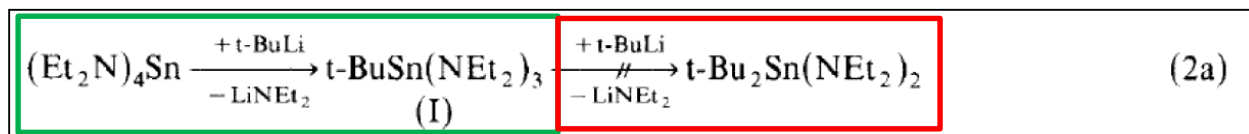
compounds, consistent with the Examiner's initial conclusions, Petitioner's expert, Dr. Gerard Parkin, supervised Dr. Drew Hood in conducting the synthesis methods under the **proper** conductions. EX1002, ¶¶110-111; EX1003, ¶8. Specifically, Petitioner's expert synthesized two monoalkyltin triamide products, $\text{tBuSn}(\text{NEt}_2)_3$ (following the Hänssgen procedure) and $\text{i-PrSn}(\text{NMe}_2)_3$ (following a procedure adapted from Meyers in view of Deelman). *Id.*; EX1003, §§II-III. The experiments confirmed synthesis of high purity monoalkyltin triamide compounds according to the prior art teachings results in levels of dialkyltin impurities (0.17 mole%, Hänssgen and 0.19 mole%, Meyers/Deelman) well below those recited in the Challenged Claims, and establish that PO's representations to the Office during prosecution that such high purity compounds were not achievable were incorrect and based on a flawed methodology. EX1002, ¶¶116-118, 121-122; EX1003, ¶¶17-18, 24-25, 46-47.

V. SUMMARY OF PRIOR ART

A. Hänssgen

"Hänssgen," a paper entitled "Synthesis of The First Mono-t-Butyltin Element Compounds," was first published in the Journal of Organometallic Chemistry in 1985. EX1005; EX1008, ¶¶48-54, 64. Thus, Hänssgen is prior art under at least 35 U.S.C. §102(a).

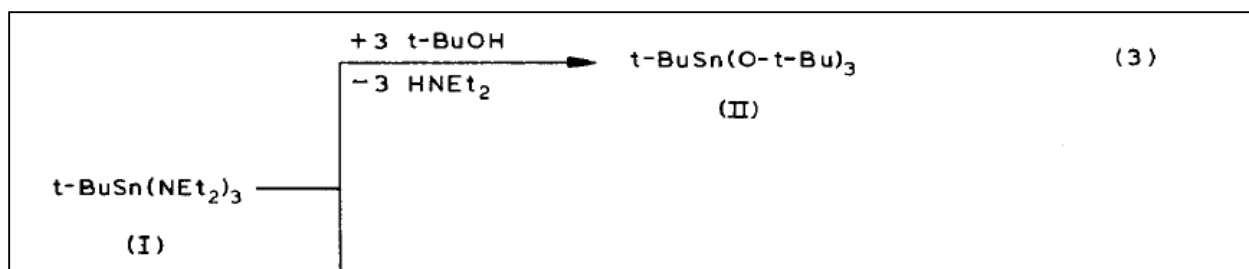
Hänssgen describes a monoalkyltin triamide. Specifically, Hänssgen describes synthesis of “t-Butyl-tris(diethylamino)stannane (I)” by “Sn-alkylation of tetrakis(diethylamino)stannane with lithium-t-butyl,” as highlighted in green below:



EX1005, 2.

Hänssgen further discloses a pure monoalkyl compound. For example, Hänssgen describes that the “reaction proceeds quantitatively; there are **no** indications of secondary reactions.” EX1005, 2. As highlighted in red above, Hänssgen’s equation 2(a) expressly indicates that the reaction **does not** generate a dialkyltin compound. Hänssgen further states “***analytically pure*** [compound] I [i.e., the monoalkyltin triamide] can be obtained by fractiona[l] distillation of the benzene phase.” EX1005, 2.

As relevant to claims 7-10, Hänssgen discloses reacting monoalkyl tin triamides with an alcohol in an organic solvent to form monoalkyl tin trialkoxides. Specifically, Hänssgen teaches the “alcoholysis of [compound] (I)” (tBuSn(NEt₂)₃), with “t-butanol, as described by equation 3 [shown below], results in the ‘displacement reaction’ [6] typical for aminostannanes to trialkoxystannane (II):”



EX1005, 3.

B. Meyers

U.S. Patent Application No. 2016/0116839 (“Meyers”) published April 28, 2016—more than 1 year prior to the earliest filing date of the ’903 Patent application. EX1006. Meyers is thus prior art under at least 35 U.S.C. §102(a). PO did not dispute Meyers’ prior art status during prosecution of the ’903 Patent.

Meyers teaches “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.” EX1006, [0002].

Meyers provides several examples of potential precursor solutions that can be used in the disclosed coatings and methods, including a precursor solution of monoalkyltin triamide. 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 via syringe 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 removed, and the residue distilled. EX1006, [0151]. ^1H and ^{119}Sn NMR of the distillate in a C_6D_6 solvent “match[ed] the predicted spectra for $^i\text{PrSn}(\text{NMe}_2)_3$ ” and the “primary ^{119}Sn resonance” was “consistent with a major product with a single tin environment, with a chemical shift comparable to reported monoalkyltin amido compounds.” EX1006, [0151].

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

U.S. Patent Application No. 2011/0166268 (“Deelman”) published July 7, 2011—more than one year before the earliest filing date of the ’903 Patent application. EX1007. Thus, Deelman is prior art under at least 35 U.S.C. §102(a). PO did not challenge Deelman’s prior art status during prosecution of the ’903 Patent.

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].

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]; *see also* EX1007, [0023] (“As regards the ligand T, it is any ligand known in the art[.]”). Deelman 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]. “[T]he monoalkyltin trihalides can be separated from the reaction medium by any means such as...distillation, solvent extraction, crystallization, and the like.” EX1007, [0057].

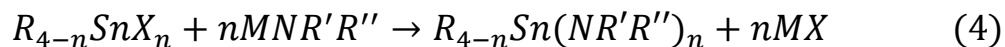
As relevant to claims 7-10, Deelman teaches the compositions can include “organic, aprotic or even protic solvents,” including ethers and alcohols, and specifically lists “tetrahydrofuran (THF), ethanol, and 1,2-dimethoxyethane (DME)” as suitable solvents. EX1007, [0052].

D. Jones

“Jones,” a paper entitled “Amino-derivatives of Metals and Metalloids. Part I. Preparation of Aminostannanes, Stannylamines, and Stannazanes,” was first

published in 1965 in Part II of *Journal of the Chemical Society* at pages 1944-1951. EX1009; EX1008, ¶¶57-64. Jones is prior art under at least 35 U.S.C. §102(a).

Jones discloses a monoalkyltin triamide and describes 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. 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 disclosed chemical reactions are:



EX1009, 4.⁴

VI. LEVEL OF ORDINARY SKILL

A POSITA at the time of the '903 Patent would 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. EX1002, ¶83.

⁴ There are two typographical errors in reaction (4) that have been corrected herein: (a) molecular formula $R_4\text{SnX}_{4-n}$ should instead be $R_{4-n}\text{SnX}_n$. A POSITA would have understood 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; EX1002, n.5.

VII. CLAIM CONSTRUCTION

Claim terms are construed according to their ordinary and customary meaning as understood by a POSITA. 37 C.F.R. § 42.100(b). In the Delaware Litigation, PO argued the ordinary and customary meaning should apply for all relevant terms of the '903 Patent. For purposes of this Petition, Petitioner interprets all claim terms in accordance with their ordinary and customary meaning. *See also supra* n.3.

VIII. INSTITUTION IS PROPER UNDER 35 U.S.C. §§ 314(a) AND 325(d)

A. § 314(a)

This is the first petition challenging the '903 Patent. The *General Plastic* factors therefore do not provide a basis for denying institution.

There is also no basis for discretionary denial under *Apple Inc. v. Fintiv, Inc.*, because Petitioner is not a party to the Delaware Litigation. IPR2020-00019, Paper 11 (PTAB Mar. 20, 2020) (designated precedential May 5, 2020); *see also* Director Vidal's Memorandum dated June 21, 2022 at 1 (noting *Fintiv* addresses parallel "proceeding involving the same parties"). Regardless, Petitioner also presents compelling merits, as the claims were allowed based on the Office's reliance on PO's factual assertions during prosecution that were misleading and/or inaccurate. *See* §IV.

B. § 325(d)

Discretionary denial under 35 U.S.C. § 325(d) is not warranted. Although the prior art relied upon by Petitioner was reviewed and, in certain cases, relied upon by

the Office, reconsideration of these references is necessary because (1) Petitioner relies upon significantly different arguments regarding these references than were advanced in prosecution; and (2) the Office erred in a manner material to the patentability of the Challenged Claims based on the unclear and inaccurate declarations submitted by PO. *See Advanced Bionics, LLC v. MED-EL Elektromedizinische Geräte GmbH*, IPR2019-01469, Paper 6 at 8 (PTAB Feb. 13, 2020) (precedential).

1. The Petition Presents Different Arguments

The Petition's grounds rely on Hänssgen, Jones, Meyers, and Deelman.

Hänssgen and Jones are listed on the face of the '903 Patent, and Hänssgen was considered the "state of the art" before the purported invention, EX1004, 890, but the Examiner did not rely on or substantively address either reference for any rejections. *See generally* EX1004. As shown in §§IX-X, the Office also did not have the benefit of Petitioner's experiments modeled on Hänssgen, which, contrary to PO's statements during prosecution, confirm that Hänssgen invalidates the Challenged Claims. Moreover, Petitioner also identifies several combinations of references that were not considered by the Examiner, including the combinations of Deelman and Jones and further in combination with Hänssgen as rendering the Challenged Claims obvious. As the disclosures of Hänssgen and Jones were not properly considered, institution should be granted.

Regarding Meyers and Deelman, the Examiner considered these references and rejected the claims over “Deelman in view of Meyers.” EX1004, 469-471. But Petitioner’s arguments differ from the Examiner’s. Specifically, Petitioner relies on: Meyers in view of Deelman, Meyers/Deelman in view of Hänssgen, Deelman in view of Jones, and Deelman/Jones in view of Hänssgen. None of these combinations were considered during prosecution. Nor did the Examiner have the benefit of Petitioner’s properly conducted experiments, which show that a high purity monoalkyltin triamide compound of the Challenged Claims is achievable through the combination of Meyers and Deelman.

Even for Meyers and Deelman, Petitioner presents a different combination, starting with Meyers as the primary reference, and identifying different portions of Meyers than were considered by the Examiner. For example, during prosecution, the Examiner cited only paragraphs 6, 61, 63, and 69 of Meyers relating to a “coating solution” with an “organic solvent and organometallic compound,” “solvents such as alcohols” and Meyers’ teaching of “further teaches alkyl tin compounds with organic ligands such as dialkylamides.” EX1004, 600. The Examiner did not consider that paragraphs 150 and 151 of Meyers disclose an exemplary synthesis of a monoalkyltin triamide composition—limitations for which the Examiner relied upon Deelman. EX1004, 600-601. The Examiner did not consider that Meyers expressly discloses all challenged limitations except for the claimed purity, and did

not have the benefit of Petitioner’s experiments showing the claimed level of purity was achievable by combining Meyers and Deelman. *See* §IV; EX1002, ¶¶119-122; EX1003, ¶¶46-47.

Because Petitioner advances numerous different arguments to show the obviousness of the Challenged Claims, reconsideration is necessary.

2. The Office Erred in Its Prior Analysis as a Result of PO’s Incorrect and Misleading Statements

Part 2 of the *Advanced Bionics* framework also favors Institution because the Petition shows how PO’s statements during prosecution caused the Examiner to incorrectly allow the claims. *See Quasar Sci. LLC v. Colt Int’l Clothing, Inc.*, IPR2023-00611, Paper 10 at 14 (PTAB Oct. 10, 2023) (“A petitioner’s showing that the challenged claims are unpatentable over the asserted prior art may itself be evidence of material error by the Office during prosecution.”). Moreover, as described above, the Office failed to rely on Hänssgen for any rejection, even though that reference anticipates the Challenged Claims, and failed to appreciate that the combination of Meyers and Deelman discloses each limitation of numerous claims.

PO submitted multiple declarations that misled the Examiner by representing that recreating the synthesis procedures disclosed by Hänssgen and Deelman did not result in the claimed high purity monoalkyltin triamide compositions. *See* §III.B. However, as explained in §IV, a POSITA would have understood that the PO either failed to properly follow the experiment procedures (in the case of Hänssgen) or

adopted an irrelevant synthesis method (in the case of Deelman and Meyers). A POSITA therefore would not have expected PO's flawed experiments to generate the claimed compositions with the requisite impurity level. EX1002, ¶¶105-109.

As described in §IV, Petitioner accurately recreated the synthesis procedures disclosed by Hänssgen and Meyers in view of Deelman and successfully generated the claimed high purity monoalkyltin triamide compositions. This additional evidence demonstrates that PO's inaccurate declarations caused the Examiner to err in allowing the Challenged Claims, and as such, that the prior references should be reconsidered, including in the new combinations presented.

IX. GROUND 1A: Anticipation by Hänssgen

A. Claim 1

1. [1pre]

To the extent that the preamble is limiting, Hänssgen discloses “*a high purity liquid composition.*” Hänssgen discloses “t-Butyl-tris(diethylamino)stannane,” referred to as “compound (I)”, *i.e.*, $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 1, 3, Summary; EX1002, ¶123. Hänssgen describes synthesizing (I) and further explains that “analytically pure I can be obtained by fractiona[l] distillation of the benzene phase[,]” describing the distilled compound as “oil” (*i.e.*, a liquid). EX1005, 2, 4; EX1002, ¶123.

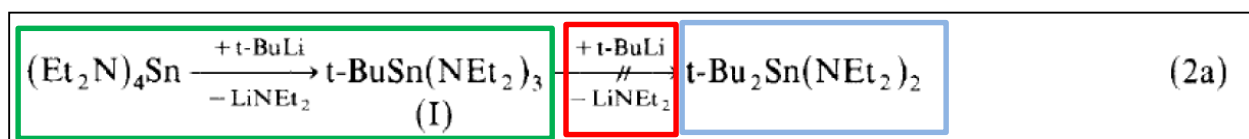
2. [1a]

Hänssgen discloses “*a monoalkyl tin triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$* ” as it teaches the synthesis of compound (I), which

has the chemical formula of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2; EX1002, ¶125. This chemical formula corresponds to a monoalkyl tin triamido compound of $\text{RSn}(\text{NR}'_2)_3$, where R corresponds to the tert-butyl group (*i.e.*, monoalkyl) and R' corresponds to the ethyl group (NEt_2) of the three diethylamido groups (*i.e.*, triamido). EX1002, ¶125.

3. [1b]

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, which yields triamino-t-butylstannane (I) and lithium diethyl amide,” as shown below in green:



EX1005, 2; EX1002, ¶127.

Equation 2a also shows that the chemical reaction does not proceed to generate the dialkyl tin compound, *i.e.*, $\text{t-Bu}_2\text{Sn}(\text{NEt}_2)_2$ (blue), as indicated by the double hash marks through the line above (red annotation). EX1002, ¶128. Hänssgen states equation 2a “proceeds practically quantitatively; there are no indications of secondary reactions.” EX1005, 2; EX1002, ¶128. Thus, a POSITA would have

understood that equation 2a shows there is no or negligible dialkyltin impurity. EX1002, ¶128.

Hänssgen further confirms “analytically pure I [tBuSn(NEt₂)₃] can be obtained by fractiona[l] distillation of the benzene phase,” EX1005, 2, which a POSITA would have understood to mean no or negligible dialkyltin impurity. EX1002, ¶129.

Moreover, Petitioner’s experiments confirmed Hänssgen’s disclosure enabled synthesis of tBuSn(NEt₂)₃ with only 0.17 mole % dialkyltin impurity tBu₂Sn(NEt₂)₂ relative to the total tin amount. §IV; EX1002, ¶130; EX1003, ¶¶17-18.

4. [1c]

Hänssgen discloses “*R is a hydrocarbyl group with 1-31 carbon atoms,*” through the synthesis of tBuSn(NEt₂)₃. EX1005, 2; EX1002, ¶132. Tert-butyl (tBu) is a hydrocarbyl group with four (4) carbon atoms, within the claimed range. EX1002, ¶132.

5. [1d]

Hänssgen discloses “*R’ is a hydrocarbyl group with 1-10 carbon atoms*” through the synthesis of tBuSn(NEt₂)₃. EX1005, 2; EX1002, ¶134. Ethyl (Et) is a hydrocarbyl group with two (2) carbon atoms, in the claimed range. EX1002, ¶134.

B. Claim 2

Hänssgen teaches “*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.” Again, Hänssgen discloses synthesis of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2; EX1002, ¶137. Tert-butyl (tBu) is a branched alkyl ligand R represented by $\text{R}^1\text{R}^2\text{R}^3\text{C—}$ 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 within the claimed range. EX1002, ¶137.

C. Claim 3

Hänssgen teaches “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 synthesis of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2; EX1002, ¶140. Thus, Hänssgen’s R comprises tert-butyl. EX1002, ¶140.

D. Claim 4

Hänssgen teaches “R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.” Hänssgen discloses synthesis of $\text{tBuSn}(\text{NEt}_2)_3$, so Hänssgen discloses R’ comprising ethyl (*Et*). EX1005, 2; EX1002, ¶142.

E. Claim 6

Hänssgen teaches the composition “comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.” Hänssgen teaches synthesis of “analytically pure” $\text{tBuSn}(\text{NEt}_2)_3$ and indicates that the equation does not proceed to generate any dialkyl tin compound, *e.g.*, EX1005, 2, so a POSITA would have understood no or negligible dialkyltin impurity is present in

the compound. EX1002, ¶145. Moreover, Petitioner's experiments confirm there is essentially no dialkyltin compound impurity in the Hänssgen product when measured with an approximate 0.1 mole % detection limit. EX1002, ¶147.

F. Claim 7

Hänssgen discloses a “*solution comprising the composition of claim 1 and an organic solvent.*” Hänssgen teaches a “solution of 3.93 g (10 mmol) [Compound] I in 20 ml dichloromethane;” reaction product (I) is $\text{tBuSn}(\text{NEt}_2)_3$ and meets every limitation of claim 1. EX1005, 4; EX1002, ¶150. Dichloromethane is an organic solvent. EX1002, ¶150. Hänssgen further teaches that t-butanol, which is an alcohol and an organic solvent, is added to the solution. EX1005, 4; EX1002, ¶150.

G. Claim 8

Hänssgen discloses the “*solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.*” As noted for claim 7, Hänssgen teaches t-butanol, an alcohol solvent, added to the solution including compound (I), so a POSITA would have understood the solvent comprises an alcohol. EX1005, 4; EX1002, ¶153.

Hänssgen teaches a solution concentration within the range from about 0.005M to about 0.5M. Hänssgen discloses a “solution of 3.93 g (10 mmol) [Compound] I in 20 ml dichloromethane.” EX1005, 4; EX1002, ¶154. The molar concentration of Compound I in 20 ml dichloromethane is about 0.5 M (*i.e.*, 10

mmol/20 ml= 0.5 mol/l). EX1002, ¶154. After additional solvent, *i.e.*, 2.2 g (30 mmol) t-butanol, was added to the solution, the molar concentration decreased slightly, *i.e.*, slightly lower than about 0.5M, which is within the claimed range of about 0.005M to about 0.5M. EX1002, ¶154.

H. Claim 9

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** ($\text{CH}_3\text{CH}_2(\text{CH}_3)_2\text{C—}$)⁵, **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.**” Hänssgen teaches the synthesis of $\text{tBuSn}(\text{NEt}_2)_3$. EX1005, 2; EX1002, ¶157. Thus, Hänssgen discloses R comprises tert-butyl and R’ comprises an ethyl group. EX1002, ¶157; *see also* §§IX.C-D.

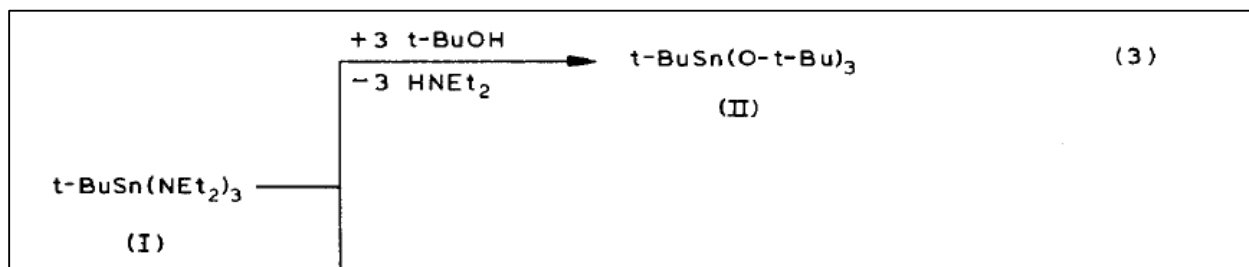
I. Claim 10

1. [10Pre]

To the extent the preamble is limiting, Hänssgen discloses a “*method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.*” Hänssgen teaches synthesis of a monoalkyltin triamide compound (I) with very low dialkyl tin contamination. EX1002, ¶159. Hänssgen further

⁵ See EX1002, n.13 (noting error in tert-amyl chemical formula).

discloses the “alcoholysis” of $t\text{BuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3” to obtain compound (II), shown below:



EX1005, 3; EX1002, ¶159. Hänssgen describes the specific method: “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; EX1002, ¶160.

A POSITA would have understood the alcoholysis reaction impacts only the amido groups, so the reaction does not change the dialkyl tin contamination level. EX1002, ¶161. Thus, the resulting monoalkyltin trialkoxide compound from the alcoholysis reaction has the same low dialkyl tin contamination as the starting monoalkyltin triamide compound. EX1002, ¶161.

2. [10a]

Hänssgen discloses “*reacting the composition of claim 1 with an alcohol represented by the formula HOR in an organic solvent to form $\text{RSn}(\text{OR})_3$* .” As explained for [10pre], Hänssgen teaches reacting the composition of claim 1, $t\text{BuSn}(\text{NEt}_2)_3$, with an alcohol represented by the formula HOR”, specifically t-

butanol, in an organic solvent to form $\text{RSn}(\text{OR}'')_3$, as shown in equation 3 above. EX1002, ¶164. 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 $\text{RSn}(\text{OR}'')_3$. EX1002, ¶165. Hänssgen also teaches the reaction proceeds in an organic solvent that includes dichloromethane. EX1005, 3; EX1002, ¶166.

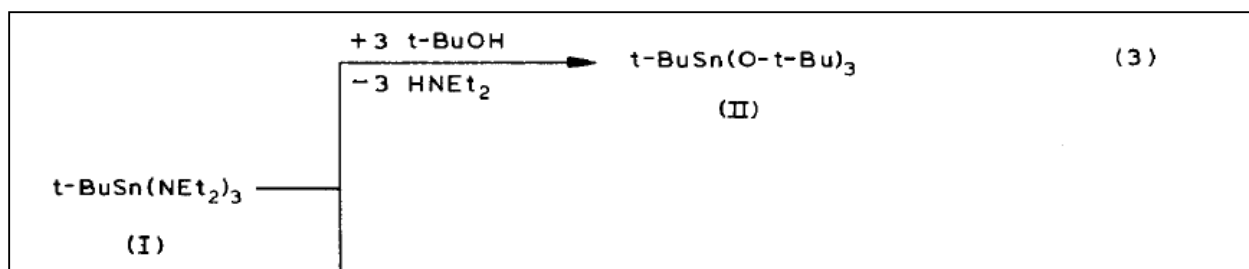
3. [10b]

Hänssgen discloses “*R*” is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition.” 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. EX1005, 4; EX1002, ¶168. In this chemical formula, R'' corresponds to t-butyl, a hydrocarbyl group with 4 carbon atoms. EX1002, ¶168.

4. [10c]

Hänssgen discloses “*the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.*”

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.” §IX.A.3. Hänssgen also teaches “alcoholysis of [compound] (I),” which is the “analytically pure” $\text{tBuSn}(\text{NEt}_2)_3$, with “t-butanol, as described by equation 3:



EX1005, 3; EX1002, ¶171. Because the alcoholysis reaction impacts only the amido groups, the reaction does not change the dialkyl tin contamination level and the resulting monoalkyltin trialkoxide compound has the same very low dialkyl tin contamination—“no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” as the monoalkyltin triamide compound (I). EX1002, ¶¶172-173.

X. GROUND 1B: Obviousness Over Hänssgen and Knowledge of POSITA⁶

A. Reasons to Combine

As described for Ground 1A, Hänssgen (EX1005) explicitly discloses claims 1-4 and 6-10 because it 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

⁶ The defendant in the Delaware Litigation argued that PO disclaimed the synthesis methods taught by Hänssgen and Deelman. EX1012, 43-53. To the extent that construction is advocated or adopted in this proceeding, the Challenged Claims are invalid in view of obvious variants of Hänssgen, as discussed in Ground 1B, and the obviousness combinations involving Deelman in Grounds 2 and 3. See EX1002, n.6.

secondary reactions,” and that “Alcoholysis of I with t-butanol gives the corresponding alkoxystannane t-BuSn(O-t-Bu)₃ (II).” EX1005, 1, 2.

To the extent that PO argues that the purity limitations are not disclosed by Hänssgen, such limitations would have been obvious to a POSITA. EX1002, ¶178. A POSITA would have understood that high purity organotin compounds are desirable, *e.g.*, to minimize side reactions and undesirable impurities, and that achieving the level of purification needed for any given compound or use of a compound is simply a matter of economy and scale, as purification steps take additional manufacturing time and may cost some yield. EX1002, ¶179.

A POSITA would have been motivated to further optimize the process conditions disclosed by Hänssgen, including by use of fractional distillation, as Hänssgen teaches. EX1002, ¶179. Fractional distillation is a well-known effective method to separate a mixture of similar compounds according to their different boiling points, thereby improving the purity of the main product. EX1002, ¶179. Indeed, the '903 Patent describes removal of dialkyl impurity by fractional distillation to purify t-BuSn(NMe₂)₃, a compound with similar properties to t-BuSn(NEt₂)₃ of Hänssgen. *E.g.*, EX1001, 16:54-56 (“This example demonstrates the effectiveness of fractional distillation to purify t-BuSn(NMe₂)₃...”); *see also, e.g.*, EX1001, 3:13-17; EX1002, ¶179 (explaining that any difference between the fractional distillation used in the '903 Patent and conventional fractional distillation

methods would only impact efficiency but not alter whether the mixture is separatable through factional distillation).

Thus, a POSITA would have used the well-known fractional distillation procedure, as needed, to improve the purity of the compound taught by Hänssgen 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. EX1002, ¶180; *see also Cytiva BioProcess R&D AB v. JSR Corp.*, No. 23-2074, 2024 WL 4960327, at *8 (Fed. Cir. Dec. 4, 2024) (no showing of expectation of success is required when a claim “merely recites an inherent property of an otherwise obvious combination”).

B. Claim 1

1. [1pre], [1a], 1[c]-1[d]

See §IX.A.1-2, 4-5.

2. [1b]

Hänssgen discloses “*no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount*” as set forth in §IX.A.3. To the extent PO argues this limitation is not disclosed, it would have been obvious to a POSITA to optimize reaction conditions and/or apply known purification methods to known monoalkyl triamide products to yield predictable results, *i.e.*, to optimize the purity of the monoalkyl tin triamide composition to remove the dialkyltin compounds, including up to the claimed “*no more than 0.5 mole % dialkyltin.*” *See* §X.A.

PO admits many known methods of purification existed prior to the invention, including for monoalkyl tin triamide compounds. EX1001, 3:2-5, 3:13-17 (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.”); EX1002, ¶184-187. Because the boiling points are sufficiently different, fractional distillation could be used to purify the monoalkyl tin triamide compounds, and could be repeated, if needed, to continue increasing the purity to a sufficiently high purity level for the desired application. EX1002, ¶184-187.

Hänssgen 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; EX1002, ¶187. Thus, Hänssgen acknowledges the problem of secondary reactions and would have motivated a POSITA to use the known process of fractional distillation to achieve the desired “analytically pure” compound, with a reasonable expectation of success. EX1002, ¶188.

Thus, to the extent Hänssgen does not explicitly disclose “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,” this limitation would have been obvious to a POSITA given (1) the explicit teaching of Hänssgen to achieve a high purity monoalkyl tin compound, and (2) that such a

process would be nothing more than applying known techniques to the known synthesis process disclosed in Hänssgen to achieve the claimed purity level. See §X.A.

C. Claim 2-4

See §IX.B-D.

D. Claim 6

Hänssgen discloses the composition “*comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.*” See §IX.E. To the extent PO argues Hänssgen does not explicitly disclose this limitation, a POSITA would have found it obvious to purify the monoalkyltin triamide compound to include “essentially no dialkyltin compounds as measured with an approximate 0.1 mole% detection limit” as explained in §X.B.2. See also §X.A.

E. Claims 7-9

See §IX.F-H.

F. Claim 10

1. [10Pre], [10a], [10b]

See §IX.I.1-3.

2. [10c]

Hänssgen discloses “*the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.*” §IX.I.4. To the extent PO argues that Hänssgen does not expressly disclose this limitation, it

would have been obvious for the same or similar reasons discussed in §§IX.I.4, X.A, X.B.2. It requires no more than routine optimization of reaction conditions or conventional fractional distillation already known in the art to arrive at the claimed purity level. EX1002, ¶¶211.

XI. GROUND 2A: Obviousness Over Meyers and Deelman

A. Reasons to Combine

Meyers discloses 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]; EX1002, ¶213. Meyers confirms that ¹H and ¹¹⁹Sn NMR of the distillate obtained from following Meyers’ procedure 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]; EX1002, ¶213. While Meyers notes that high-purity alkyltin compounds are desirable (EX1006, [0063]), Meyers does not explicitly disclose the specific purity levels of the isopropyl tris(dimethylamido)tin compound. EX1002, ¶213.

Deelman, however, explicitly discloses the importance of obtaining “high purity monoalkyltin compounds” with only “minor quantities of di- and/or trialkyltin compounds.” EX1007, Abstract; EX1002, ¶214. Specifically, Deelman teaches

“monoalkyltin compounds of formula $\text{RSn}(\text{T})_3$,” which covers the monoalkyltin triamide compounds disclosed by Meyers, with “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[.]”); EX1002, ¶214.

A POSITA starting with the monoalkyltin triamide compound of Meyers, given Meyers’ explicit teaching that pure monoalkyltin compounds are desirable, would have been motivated to look and would have naturally looked to Deelman to determine a method to increase the purity of the monoalkyltin triamide compound. EX1002, ¶215. The references are in the same field of art and directed to the same types of monoalkyltin compounds, each focused on converting monoalkyltin trihalides into other mono-alkyltin derivatives. EX1002, ¶219. Indeed, a POSITA would have understood that Meyers’ 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 emphasize the need for highly pure monoalkyltin compounds. EX1002, ¶216.

Specifically, Meyers teaches synthesis of an example 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 (EX1006, [0151]); such a selective reaction pathway replacing halide ligands of an organometallic compound with amide ligands was well-known in the art. EX1002, ¶217. Deelman, similarly, teaches “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]; EX1002, ¶218. Accordingly, Deelman teaches the same synthesis strategy disclosed in Meyers for preparing monoalkyltin triamide compound. EX1002, ¶218.

Furthermore, the combination requires no more than applying known techniques to achieve a high purity compound using a known reaction pathway, with predictable results. EX1002, ¶220. Deelman teaches that high purity monoalkyltin trihalides are desirable (EX1007, [0010]) and can be achieved through previously existing means, including “distillation of monoalkyltin halides from thermal redistribution mixtures containing mono-, di-, and tri-alkylhalides[]” (EX1007, [0028]), as well as through the reactions disclosed in Deelman that selectively form monoalkyltin halides without multi-alkyl impurities (EX1007, [0031]-[0033]). EX1002, ¶220. Thus, Deelman provides a specific motivation 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. EX1002, ¶220. Indeed, a POSITA would have a reasonable expectation of success in maintaining the purity of the monoalkyltin compounds because such a reaction is a well-defined exchange pathway in which the highly nucleophilic amide ion displaces the halide ion. EX1002, ¶221. When properly controlled, a POSITA would have expected production of high purity monoalkyltin triamides. EX1002, ¶221.

Petitioner's experiments 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 $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$. EX1002, ¶222; EX1003, ¶¶46-47. 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 at the time of the alleged invention (April 11, 2018) would have expected the combination to generate the claimed high purity monoalkyltin triamide compounds at that time as well because all of Petitioner's reaction conditions were available to a POSITA at that time. EX1002, ¶222.

Finally, even if the purity was not maintained, methods to subsequently improve the purity of monoalkyltin triamide compounds, *i.e.*, fractional distillation, were well-known prior to the alleged invention, and a POSITA would have had a

reasonable expectation of success at increasing the purity to the extent further purification was necessary to obtain the purity that Deelman teaches is desirable and achievable. EX1002, ¶223.

B. Claim 1

1. [1pre]

To the extent the preamble is limiting, Meyers in view of Deelman (“Meyers/Deelman”) renders obvious “*a high purity liquid composition.*” Meyers discloses preparation of “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]; EX1002, ¶225. After several steps, the product was a “pale yellow liquid[.]” EX1006, [0151]; EX1002, ¶225. Meyers further teaches that high-purity monoalkyltin precursors are desired. EX1006, [0063]; EX1002, ¶225.

Deelman likewise 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 RSn(T)₃.” EX1007, [0013]; EX1002, ¶226.

2. [1a]

Meyers/Deelman renders obvious “*a monoalkyl tin triamide compound represented by the chemical formula RSn(NR'₂)₃.*” Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃),” which is a monoalkyl tin

triamide compound represented by the chemical formula $\text{RSn}(\text{NR}'_2)_3$ where “R” is iso-propyl and “R’” is methyl. EX1006, [0150]-[0151]; EX1002, ¶229.

3. [1b]

Meyers/Deelman renders obvious *“no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.”*

Meyers teaches synthesis of an exemplary 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 . EX1006, [0151]; EX1002, ¶232. Meyers discloses that high-purity monoalkyltin precursors are desired. EX1006, [0063]; EX1002, ¶232. 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.”* EX1002, ¶232. Deelman, however, teaches that *“high purity monoalkyltin trihalides can be converted to other mono-alkyltin derivatives”* and that such 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, [0010], [0013]; EX1002, ¶233. Deelman further discloses *“[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]; EX1002, ¶233.

Accordingly, a POSITA would have desired to make Meyers' monoalkyltin triamide compound with high purity and would have been motivated to use Deelman's high purity monoalkyltin trihalide in the same synthesis process disclosed by Meyers. EX1002, ¶234; §XI.A. Given the well-known reaction pathway (replacing halide groups of a high purity monoalkyltin trihalide with amide groups) a POSITA would have had a reasonable expectation of success to arrive at "no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount," consistent with the teachings of Deelman. EX1002, ¶234; §XI.A. Moreover, a POSITA would have conducted fractional distillation as needed to further improve the purity of the target compound. EX1002, ¶223.

Petitioner's experiments confirmed that the steps described in Meyers, with adjustment to the solvent (as disclosed by Deelman), successfully and predictably yielded a monoalkyltin triamide ($i\text{-PrSn}(\text{NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$, which falls within the scope of "no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount." EX1002, ¶235; EX1003, ¶¶46-47.

4. [1c]

Meyers/Deelman renders obvious "*wherein R is a hydrocarbyl group with 1-31 carbon atoms.*" Meyers teaches preparation of "isopropyl tris(dimethylamido)tin, ($i\text{-PrSn}(\text{NMe}_2)_3$)." EX1006, [0150]-[0151]. Isopropyl is a hydrocarbyl group with 3

carbon atoms, which is within the claimed range of 1-31 carbon atoms. EX1002, ¶238.

5. [1d]

Meyers/Deelman renders obvious “*R*’ is a hydrocarbyl group with 1-10 carbon atoms.” 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, within the claimed range. EX1002, ¶241.

C. Claim 2

Meyers/Deelman renders obvious “*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.” 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. EX1002, ¶245.

D. Claim 3

Meyers/Deelman renders obvious “*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*.” Meyers teaches preparation of

“isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]; EX1002, ¶249. Thus, Meyers discloses R comprises isopropyl. EX1002, ¶249.

E. Claim 4

Meyers/Deelman renders obvious “*R’ comprises a methyl group, ethyl group, isopropyl group, or t-butyl group.*” Meyers teaches preparation of “isopropyl tris(dimethylamido)tin, (i-PrSn(NMe₂)₃).” EX1006, [0150]-[0151]; EX1002, ¶253. Accordingly, Meyers discloses R’ comprises a methyl group. EX1002, ¶253.

F. Claim 6

Meyers/Deelman renders obvious the composition “*comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.*”

Meyers teaches 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]; EX1002, ¶257. While Meyers teaches that high purity compounds are desirable, it does not explicitly disclose the purity level of the monoalkyltin triamide compound i-PrSn(NMe₂)₃. EX1002, ¶257.

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]; EX1002, ¶258. Deelman explains 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]; EX1002, ¶258. 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]; EX1002, ¶258.

Accordingly, as explained in §§XI.A and XI.B.3, a POSITA would have desired to make Meyers’ monoalkyltin triamide compound with high purity and would have been motivated to use Deelman’s high purity monoalkyltin trihalide in the Meyers synthesis process to arrive at a high purity monoalkyltin triamide composition. EX1002, ¶259; see §§XI.A, XI.B.3.

Indeed, Petitioner’s experiments confirmed that the steps described in Meyers, with adjustment to the solvent (as disclosed by Deelman), successfully and predictably yielded a monoalkyltin triamide ($\text{i-PrSn(NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $\text{}^i\text{Pr}_2\text{Sn(NMe}_2)_2$,” within the claimed scope of “essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit.” EX1002, ¶260; EX1003, ¶¶46-47. Moreover, a POSITA would have used known purification techniques including fractional distillation, as needed, to further purify the composition. EX1002, ¶223.

XII. GROUND 2B: Obviousness Over Meyers and Deelman and Hänssgen

A. Reasons to Combine

As explained for Ground 2A, a POSITA would have combined Meyers' teaching of 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 that same type of compound to begin with the same starting compound (*i.e.*, a monoalkyltin trihalide compound) and achieve a high purity monoalkyltin triamide composition, with a minimal amount of dialkyltin compound as an impurity. See §XI.A.

A POSITA would have been further motivated to combine the high purity monoalkyltin triamide composition of Meyers/Deelman with Hänssgen ("Meyers/Deelman/Hänssgen") to arrive at the solutions of claims 7-10 of the '903 Patent. Hänssgen also 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' [6] **typical** for aminostannanes to trialkoxystannane (II)[.]" EX1005, 3; EX1002, ¶266. A POSITA would have been motivated to introduce the monoalkyltin triamide composition of Meyers/Deelman into a solution including an organic solvent, *i.e.*, an alcohol, as in Hänssgen, to

synthesize a high purity monoalkyltin trialkoxide, another well-known compound, the usefulness and desirability of which would have been understood by a POSITA. EX1002, ¶267; *see, e.g.*, EX1014 (describing uses of tin alkoxides).

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. EX1002, ¶268. Moreover, alcoholysis of organotin amides was well-known in the art before the alleged invention, and Hänssgen discloses a typical alcoholysis reaction of monoalkyltin triamide compound to derive monoalkyltin trialkoxides, another useful class of compounds. EX1002, ¶¶267-268. 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). EX1002, ¶269.

The combination of Meyers/Deelman/Hänssgen requires no more than applying a known chemical reaction (alcoholysis) to a known class of compounds (monoalkyltin triamide compounds) to arrive at a known-beneficial compound (monoalkyltin trialkoxide), with predictable results. EX1002, ¶270; *Cytiva*, 2024 WL 4960327, at *8. A POSITA would have had a reasonable expectation of success applying Hänssgen's alcoholysis reaction to the high purity monoalkyltin triamide composition taught by Meyers/Deelman because Hänssgen teaches the alcoholysis

of a monoalkyltin triamide compound ($\text{tBuSn}(\text{NEt}_2)_3$) (§§V.A, IX.A), which is similar to the monoalkyltin triamide compound of Meyers/Deelman (a high purity isopropyl tris(dimethylamido)tin, $(\text{i-PrSn}(\text{NMe}_2)_3)$). EX1002, ¶271. Moreover, the alcoholysis reaction of Hänssgen impacts only the amido groups and not the alkyl group (displacing the amido group with the alkoxyl group). EX1002, ¶271. As such, a POSITA would have understood that the reaction would not change the dialkyl tin contamination level of the monoalkyltin triamide compound and 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. EX1002, ¶271.

Accordingly, it would have been obvious to a POSITA interested in deriving monoalkyltin trialkoxides to subject the high purity monoalkyltin triamide composition of Meyers/Deelman to Hänssgen's alcoholysis reaction with predictable results and a reasonable expectation of success. EX1002, ¶272.

B. Claims 1-4 and 6

See §§XI.A-F.

C. Claim 7

Meyers/Deelman/Hänssgen renders obvious “*a solution comprising the composition of claim 1 and an organic solvent.*” First, Meyers/Deelman/Hänssgen discloses the composition of claim 1. §§XI.A-B. Hänssgen further teaches creating

a solution of “tBuSn(NEt₂)₃” in “dichloromethane.” EX1005, 4; EX1002, ¶287. Dichloromethane is an organic solvent. EX1002, ¶287. Hänssgen further teaches that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4; EX1002, ¶287. A POSITA would have understood that the solvent comprises an alcohol, as t-butanol is added into the solution, and a POSITA would have understood that the high purity monoalkyltin triamide composition of Meyers/Deelman could be easily dissolved in an organic solvent (like the t-butanol disclosed in Hänssgen) to create the claimed solution with predictable results. EX1002, ¶288.

D. Claim 8

Meyers/Deelman/Hänssgen renders obvious “*the solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.*” Meyers/Deelman/Hänssgen discloses the solution of claim 7. §XII.C. Hänssgen also 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; EX1002, ¶291. Hänssgen further teaches the solution concentration from about 0.005M to about 0.5M. See §IX.G; EX1002, ¶291. A POSITA would have understood that the high purity monoalkyltin triamide composition of Meyers/Deelman would similarly be dissolved in 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 results. EX1002, ¶292; §XII.A.

E. Claim 9

Meyers/Deelman/Hänssgen renders obvious “*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,” for the reasons described in XI.D; XI.E; EX1002, ¶295.*

F. Claim 10

1. [10Pre]

To the extent the preamble is limiting, Meyers/Deelman/Hänssgen renders obvious “*a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.*” Hänssgen teaches a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination. See IX.I.1; EX1002, ¶298. A POSITA would have been motivated to combine the high purity monoalkyltin triamide compositions of Meyers/Deelman with the method to form a monoalkyltin trialkoxide compound taught by Hänssgen as set forth in §XII.A.

2. [10a]

Meyers/Deelman/Hänssgen renders obvious “*reacting the composition of claim 1 with an alcohol represented by the formula HOR in an organic solvent to*

form RSn(OR'')₃." Meyers/Deelman/Hänssgen discloses the composition of claim 1. See §§XI.B, XII.B. Hänssgen teaches reacting a composition like the high purity monoalkyltin triamide composition of Meyers/Deelman with an alcohol represented by the formula HOR'' in an organic solvent to form RSn(OR'')₃. Specifically, Hänssgen teaches "alcoholysis of" tBuSn(NEt₂)₃, with "t-butanol" which "results in the 'displacement reaction' [6] typical for aminostannanes to trialkoxystannane (II)," resulting in t-BuSn(O-t-Bu)₃, which can be represented by the formula RSn(OR'')₃. See IX.I.2; EX1005, 3; EX1002, ¶303. A POSITA would have been motivated to react the high purity monoalkyltin triamide Meyers/Deelman composition 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. See §XII.A; EX1002, ¶304.

3. [10b]

Meyers/Deelman/Hänssgen renders obvious "*R is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition,*" for the reasons described in IX.I.2-3. EX1002, ¶306; *see also* §XII.A.

4. [10c]

Meyers/Deelman/Hänssgen renders obvious "*the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.*" As explained, Meyers/Deelman 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. See §XI.B.3.

Alcoholysis of organotin amides were well-known in the art, and Hänssgen expressly teaches alcoholysis of a monoalkyltin triamide compound with t-butanol in an organic solvent. §§IX.I.2-4, V.A; EX1002, ¶309. A POSITA would have been motivated to combine the high purity monoalkyltin triamide composition of Meyers/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. §XII.A; EX1002, ¶309.

Moreover, it would have been obvious to a POSITA 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 synthesis of monoalkyltin triamide compositions and known methods of performing the alcoholysis reactions on those compositions to yield predictable results, *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. EX1002, ¶310; §XII.A.

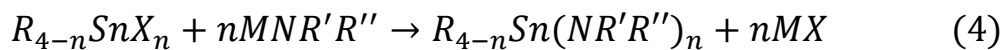
XIII. GROUND 3A: Obviousness Over Deelman and Jones

A. Reasons to Combine

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], [0023].

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 alkene to selectively produce mono-alkyltin trihalides, which can “be easily converted to mono-alkyltin compound of formula RSn(T)_3 .” EX1007, [0031]-[0033].

While Deelman 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 $NR'R''$, where R' and R'' are alkyl or aryl groups. EX1009, 4. In the same field of organotin synthesis, Jones teaches 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 illustrates this “transmetallation” reaction as follows:



where “ R , R' , and R'' ” = alkyl or aryl, $X = Cl$ or Br , $M = Li$, and $n = 1, 2, 3$, or 4 .” EX1009, 4 (modified, *see supra* n.4). Jones’s “[r]eaction (4) invariably proceeded smoothly.” EX1009, 4. This is consistent with the knowledge of a POSITA that the amide group replaces the halide ion on the metal center, leaving lithium halide as a byproduct. EX1002, ¶314. Jones further provides numerous examples of “Transmetallation with Lithium Salts of Secondary Amines” including monoalkyltin triamide compounds, such as $MeSn(NMe_2)_3$, $PhSn(NMe_2)_3$, $Bu^nSn(NMe_2)_3$. EX1009, Tables 1 and 2.

A POSITA would have been motivated to combine the teachings of Deelman regarding the need for high purity monoalkyltin compounds of the formula $RSn(T)_3$ with the disclosure of Jones that the T could be an amino group with a reasonable

expectation of success (“Deelman/Jones”). A POSITA would have looked to Jones because Deelman and Jones are in the same field of art and directed to the same types of monoalkyltin compounds. EX1002, ¶316. A POSITA would have understood that incorporation of an amino group from Jones as “T” would be nothing more than a simple substitution of a known ligand for another, involving known chemistry and known benefits with predictable results. *See* EX1009, 4 (“major advance in SnN chemistry was the preparation of an aminostannane...by way of the lithium salt of the amine[]” in 1960); EX1002, ¶316.

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, successful results. EX1002, ¶317. 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 converting alkyltin halides into alkyltin amides by reacting the organotin halides with lithium amide salts. EX1009, 4; EX1002, ¶317. 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. EX1002, ¶317. A POSITA would have had a reasonable expectation of success maintaining the level of purity of the monoalkyltin trihalide as disclosed by Deelman through Jones's reaction with the lithium amide salt to derive a high purity monoalkyltin triamide 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. EX1002, ¶318.

Petitioner's experiments 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 $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$. EX1002, ¶319; EX1003, ¶¶46-47. A POSITA would have understood that the same type of reaction could generate the specific monoalkyltin triamide compounds taught by Deelman/Jones. EX1002, ¶319. Moreover, while Petitioner's experiments were conducted recently, a POSITA at the time of the alleged invention would have expected the combination to generate the claimed high purity monoalkyltin triamide compounds at that time as well by conducting the same experiment, as Petitioner's reaction conditions were available at that time. EX1002, ¶319.

Finally, even if the purity was not maintained, methods to further improve the purity of monoalkyltin triamide compounds, *i.e.*, fractional distillation, were well known and a POSITA would have had a reasonable expectation of success at increasing the purity of the resulting monoalkyltin triamide compounds to the extent further purification was necessary. *E.g.*, §§X.A, X.B.2; EX1002, ¶320.

B. Claim 1

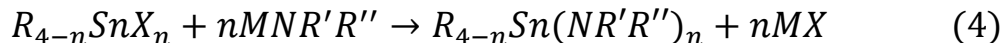
1. [1pre]

To the extent the preamble is limiting, Deelman/Jones renders obvious “*a high purity liquid composition.*” 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 C_1 - C_{20} alkyl, preferably C_1 - 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 [0196]-[0198]; EX1002, ¶322.

2. [1a]

Deelman/Jones renders obvious “*a monoalkyl tin triamide compound represented by the chemical formula $R\text{Sn}(NR'_2)_3$.*” Deelman teaches “mono-alkyltin compounds of formula $R\text{Sn}(T)_3$.” EX1007, [0031]. Deelman further teaches “hydrostannylation of 1-alkene” to selectively produce mono-alkyltin trihalides, and acknowledges that mono-alkyltin trihalides can “be easily converted to mono-

alkyltin compound of formula $R\text{Sn}(T)_3$.” EX1007, [0031]-[0033]; EX1002, ¶325. A POSITA would have been motivated to combine Deelman, which does not expressly teach an amino group as a preferred embodiment of ligand T, with Jones, which does expressly teach 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; §XIII.A. 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, $\text{X}=\text{Cl}$ or Br , $\text{M} = \text{Li}$, and $n=1, 2, 3$, or 4 .” EX1009, 4 (modified, *see supra* n.4). When $n=3$, reaction 4 of Jones, teaches reacting monoalkyltin trihalide (RSnX_3) with lithium amine salts to form monoalkyltin triamide compounds ($\text{R}\text{Sn}(\text{N}\text{NR}'\text{R}'')_3$), consistent with the teachings of Deelman. EX1002, ¶326. When R' and R'' are the same alkyl group and $n=3$, the disclosed $\text{R}_{4-n}\text{Sn}(\text{NR}'\text{R}'')_n$ product corresponds to a monoalkyl tin triamide compound represented by the chemical formula $\text{R}\text{Sn}(\text{NR}'_2)_3$. This is further supported by compound examples disclosed in Jones. *E.g.*, EX1009, Tables 1 and 2 (disclosing the synthesis of monoalkyltin triamide compounds represented by the chemical formula $\text{R}\text{Sn}(\text{NR}'_2)_3$, including $\text{PhSn}(\text{NMe}_2)_3$ and $\text{Bu}^n\text{Sn}(\text{NMe}_2)_3$); EX1002, ¶326.

3. [1b]

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

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 ” overlaps the claim scope of no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.

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

A POSITA would have been motivated to follow the approach taught by Deelman to obtain high purity monoalkyltin trihalides, and further to adopt the well-known reaction pathway taught by Jones to convert the high purity monoalkyltin

trihalides into high purity monoalkyltin triamides. See §XIII.A; EX1002, ¶331. Given the well-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 “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.” See §XIII.A; EX1002, ¶331.

Petitioner’s experiments confirmed that 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 ($i\text{-PrSn}(\text{NMe}_2)_3$) that contains only 0.19 mole % dialkyltin impurity $i\text{-Pr}_2\text{Sn}(\text{NMe}_2)_2$, which falls within the scope of “no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount.” EX1002, ¶332; EX1003, ¶¶46-47; see also EX1007 (identifying “iso-propyl” as a preferred embodiment of R in $\text{RSn}(\text{T})_3$); EX1009, 4 (identifying dimethylamino group as a suitable ligand for monoalkyltin triamides).

4. [1c]

Deelman/Jones renders obvious “*R is a hydrocarbyl group with 1-31 carbon atoms.*” Deelman discloses synthesis of a monoalkyl tin triamide compound: “monoalkyltin compounds of formula $\text{RSn}(\text{T})_3$.” EX1007, [0031]. Deelman teaches “R is a linear, branched or cyclic alkyl radical having from **1 to 20** ($\text{C}_1\text{-C}_{20}$ alkyl), **preferably from 1 to 10 carbon atoms** ($\text{C}_1\text{-C}_{10}$ alkyl).” EX1007, [0021]. And

“[a]ccording to a preferred embodiment,” R can be methyl, iso-propyl, n-butyl, and phenyl groups. EX1007, [0021]-[0022]; EX1002, ¶335.

5. [1d]

Deelman/Jones renders obvious “*R*’ is a hydrocarbyl group with 1-10 carbon atoms.” Deelman discloses synthesis of “monoalkyltin compound[s] 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], [0031]. Deelman further 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].

Deelman does not expressly teach an amino group as a preferred embodiment of ligand T. EX1002, ¶338. A POSITA would have been motivated to combine Deelman and Jones to use an amino group for ligand T, including amino groups with the formula $\text{NR}'\text{R}''$, where R’ and R’’ are alkyl or aryl groups. EX1009, 4; §XIII.A. Starting with an alkyltin trihalide, as Deelman does, Jones further teaches 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 synthesis of a monoalkyltin triamide of the formula $\text{RSn}(\text{NR}'_2)_3$. Jones further disclose several exemplary monoalkyltin

triamide compounds, such as $\text{MeSn}(\text{NMe}_2)_3$, $\text{PhSn}(\text{NMe}_2)_3$, $\text{Bu}^n\text{Sn}(\text{NMe}_2)_3$. EX1009, Tables 1 and 2. The “methyl” groups in Jones’s exemplary compounds fall within the claim scope as they are a hydrocarbyl group with one (1) carbon atom. EX1002, ¶339.

C. Claim 2

Deelman/Jones renders obvious “*R is a branched alkyl ligand represented by $R^1R^2R^3\text{C—}$, 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.*” Deelman discloses synthesis of “mono-alkyltin compounds of formula $\text{RSn}(\text{T})_3$,” where “R is a linear, **branched** or cyclic alkyl radical having from **1 to 20** ($\text{C}_1\text{--C}_{20}$ 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 $R^1R^2R^3\text{C—}$, where R^1 and R^2 are independently an alkyl group with one (1) carbon atom each, and R^3 is hydrogen. EX1002, ¶343.

D. Claim 3

Deelman/Jones renders obvious “*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” for the same reasons as [1c], as Deelman’s iso-propyl group is within the scope of claim 3. See §XIII.B.4.*

E. Claim 4

Deelman/Jones renders obvious “*R*’ comprises a methyl group, ethyl group, isopropyl group, or *t*-butyl group” for the same reasons as claim [1d], as the “methyl” groups in Jones’s exemplary compounds fall within the claim scope. See §XIII.B.5.

F. Claim 6

Deelman/Jones renders obvious the composition “*comprising essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit*” for the same reasons as claim [1b]. See §§XIII.A, XIII.B.2.

A POSITA would have been motivated to follow the approach taught by Deelman to obtain high purity monoalkyltin trihalides, and further to adopt the well-known reaction pathway taught by Jones to convert the high purity monoalkyltin trihalides into high purity monoalkyltin triamides. Indeed, given the well-known reaction pathway, replacing halide groups of a high purity monoalkyltin trihalide with amide groups, a POSITA would have had a reasonable expectation of success to arrive at “essentially no dialkyltin compounds as measure with an approximate 0.1 mole % detection limit.” EX1002, ¶358. To the extent necessary, a POSITA would have used fractional distillation to further reduce the dialkyltin impurity. *Id.*

XIV. GROUND 3B: Obviousness Over Deelman and Jones and Hänssgen

A. Reasons to Combine

As described for Ground 3A, 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. §XIII.A.

While Deelman/Jones does not expressly teach monoalkyltin trialkoxide, reactions for converting monoalkyltin triamide into monoalkyltin trialkoxide were well-known in the art, as expressly taught by Hänssgen, and tin alkoxides were known to provide benefits for a number of applications. EX1002, ¶¶30, 363. 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’ [6] **typical** for aminostannanes to trialkoxystannane (II).” EX1005, 3.

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. EX1002, ¶363.

The references are all in the same field of art, directed to the same type of monoalkyltin compounds with the same generic chemical formula. EX1002, ¶365. Moreover, alcoholysis of organotin amides was well-known in the art to produce tin alkoxides, useful for a number of applications including forming electrodes. EX1002, ¶30, 365. Because of these known benefits, a POSITA would have been motivated, for example, to convert the monoalkyltin triamide of Deelman/Jones into a monoalkyltin trialkoxide as taught by Hänssgen. EX1002, ¶365.

The combination of Deelman/Jones with Hänssgen (“Deelman/Jones/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 predictably successful results. EX1002, ¶366. A POSITA would have a reasonable expectation of success applying the known alcoholysis reaction to the high purity monoalkyltin triamide composition taught by Deelman/Jones because Hänssgen teaches the alcoholysis typical of monoalkyltin triamide compounds (*e.g.*, $\text{tBuSn}(\text{NEt}_2)_3$, which is a very similar compound to the monoalkyltin triamide compounds taught by Deelman/Jones). EX1002, ¶366. Thus, because Hänssgen discloses 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. EX1002, ¶366.

Moreover, as explained in §IX.I.4, 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 does not change the dialkyl tin contamination level of the monoalkyltin triamide compound and 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 of Deelman/Jones. EX1002, ¶367.

Accordingly, a POSITA interested in deriving monoalkyltin trialkoxides would find it obvious to subject the high purity monoalkyltin triamide composition of Deelman/Jones to Hänssgen's typical alcoholysis reactions with predictable results and a reasonable expectation of success. EX1002, ¶368.

B. Claims 1-4, 6

See §XIII.A-F.

C. Claim 7

Deelman/Jones/Hänssgen renders obvious “a solution comprising the composition of claim 1 and an organic solvent.” Hänssgen teaches creating a solution of an alkyltin triamide compound, “tBuSn(NEt₂)₃,” in “dichloromethane.”

EX1005, 4; EX1002, ¶383. Dichloromethane is an organic solvent. EX1002, ¶383. Hänssgen further teaches that t-butanol, an alcohol and an organic solvent, is added to the solution. EX1005, 4; EX1002, ¶383. A POSITA would have understood that the solvent comprises an alcohol, as t-butanol is added into the solution. EX1002, ¶383.

A POSITA would have understood that the high purity monoalkyltin triamide composition of Deelman/Jones would easily dissolve in an organic solvent, like the dichloromethane and t-butanol, as disclosed in Hänssgen, to create the claimed solution with predictable results. EX1002, ¶384.

D. Claim 8

Deelman/Jones/Hänssgen renders obvious “[t]he solution of claim 7 having a concentration from about 0.005M to about 0.5M and wherein the solvent comprises an alcohol.” 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; EX1002, ¶387. Hänssgen further teaches the solution concentration from about 0.005M to about 0.5M as described in §IX.G. A POSITA would have understood that the high purity monoalkyltin triamide composition of Deelman/Jones could be easily dissolved in 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 results. EX1002, ¶388; §XIV.A.

E. Claim 9

Deelman/Jones/Hänssgen renders obvious “*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*” as explained for claims [1c] and [1d]. See §§XIII.B.4-5.

F. Claim 10

1. [10Pre]

To the extent the preamble is limiting, Deelman/Jones/Hänssgen renders obvious “*a method to selectively form a monoalkyltin trialkoxide compound with low dialkyl tin contamination.*” Hänssgen teaches “a method to selectively form a monoalkyltin trialkoxide compound” as explained in §IX.I.1. A POSITA would have been motivated to combine the high purity monoalkyltin triamide compositions of Deelman/Jones with the method to form a monoalkyltin trialkoxide compound taught by Hänssgen. See §XIV.A.

2. [10a]

Deelman/Jones/Hänssgen renders obvious “reacting the composition of claim 1 with an alcohol represented by the formula HOR” in an organic solvent to form

RSn(OR'')₃.” Hänssgen teaches reacting a composition like the high purity monoalkyltin triamide composition of Deelman/Jones with an alcohol represented by the formula HOR'' in an organic solvent to form RSn(OR'')₃. Specifically, Hänssgen teaches “alcoholysis of” tBuSn(NEt₂)₃, with “t-butanol” “results in the ‘displacement reaction’ [6] **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. A POSITA would have been motivated to react the high purity monoalkyltin triamide composition of Deelman/Jones with the alcohol solvent disclosed by Hänssgen, t-butanol, or any other alcohol, to obtain a monoalkyltin trialkoxide of the formula RSn(OR'')₃. See §XIV.A.

3. [10b]

Deelman/Jones/Hänssgen renders obvious “*R is independently a hydrocarbyl group with 1-10 carbon atoms to form a product composition*” for the reasons set forth in [1d]. See §XIII.B.5.

4. [10c]

Deelman/Jones/Hänssgen renders obvious “*the product composition has no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total amount of tin.*” As explained in §XIII.B.3, Deelman/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. EX1002, ¶404.

Furthermore, alcoholysis of organotin amides was well-known in the art, and Hänssgen expressly teaches alcoholysis of a monoalkyltin triamide compound with t-butanol in an organic solvent. EX1002, ¶405. A POSITA would have been motivated to combine the high purity monoalkyltin triamide composition of Deelman/Jones with 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. EX1002, ¶405; §XIV.A.

It also would have been obvious to a POSITA 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 synthesis of monoalkyltin triamide compositions and known methods of performing the alcoholysis reactions on those compositions to yield predictable results, *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. EX1002, ¶406; §XIV.A.

XV. SECONDARY CONSIDERATIONS

Petitioner is aware of no evidence of secondary considerations that would meaningfully rebut a finding of obviousness.

PO contended during prosecution that the purported invention met a long-felt need, citing a patent application of Petitioner that allegedly showed an “unmet need

for highly pure monoalkyl tin triamide compounds.” EX1004, 549 (citing EX1013). There is no nexus between Petitioner’s patent and the Challenged Claims. Petitioner’s patent stated there was a need for an “improved *methodology* for manufacturing” the organotin compounds “in highly pure forms for use in the deposition of highly pure tin oxide films,” not the composition of the organotin compounds themselves (as recited in the Challenged Claims). EX1013, [0004]; EX1002, ¶409. Moreover, PO’s argument regarding Petitioner’s later-filed patent application is illogical; if PO’s earlier-filed patent application had fulfilled the supposed unmet need for “highly pure monoalkyl tin triamide compounds,” there would be no need for Petitioner’s patent application. Indeed, there was no unmet need for “highly pure monoalkyl tin triamide compounds,” as PO asserts, because the prior art already disclosed methods to synthesize such compounds, as shown herein.

XVI. CONCLUSION

Petitioner respectfully requests institution of this IPR and cancellation of the Challenged Claims.

Respectfully submitted,
BAKER BOTTS L.L.P.

December 12, 2024

Date

/ Eliot D. Williams /

Eliot D. Williams (Reg. No. 50,822)
LEAD COUNSEL FOR PETITIONER

CERTIFICATE OF COMPLIANCE

1. Pursuant to 37 C.F.R. §42.24(d), the undersigned certifies that the foregoing Petition, exclusive of the exempted portions as provided in 37 C.F.R. §42.24(a), contains 13,824 words which is no more than 14,000 words and therefore complies with the type-volume limitations of 37 C.F.R. §42.24(a). The word count was calculated by starting with Microsoft Word's total document word count and subtracting the words for the Table of Contents, the Exhibit List, Claims List, the Mandatory Notices, the Certificate of Compliance, and the Certificate of Service.

2. This Petition complies with the general format requirements of 37 C.F.R. § 42.6(a) and has been prepared using Microsoft® Word in 14-point Times New Roman.

Respectfully submitted,
BAKER BOTTS L.L.P.

December 12, 2024

Date

/ Eliot D. Williams /

Eliot D. Williams (Reg. No. 50,822)
LEAD COUNSEL FOR PETITIONER

CERTIFICATE OF SERVICE

In accordance with 37 C.F.R. §§42.6(e) and 42.105, the undersigned certifies that on the 12th day of December, 2024, a complete and entire copy of the PETITION FOR *INTER PARTES* REVIEW (“petition”), Petitioner’s power of attorney, and related Exhibits were served on Patent Owner via FedEx or by means at least as fast and reliable as Express Mail at the correspondence address of record for the subject patent.

Douglas Christensen
Christensen, Fonder, Dardi & Herbert PLLC
11322 86th Ave. N.
Maple Grove, MN 55369

Counsel of record in the Delaware proceeding below was served via email.

Naveen Modi
Paul Hastings LLP
2050 M Street NW
Washington, DC 20036.
naveenmodi@paulhastings.com

Respectfully submitted,
BAKER BOTTS L.L.P.

December 12, 2024

Date

/ Eliot D. Williams /

Eliot D. Williams (Reg. No. 50,822)
LEAD COUNSEL FOR PETITIONER