UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

ENTEGRIS, INC.,
Petitioner

v.

INPRIA CORP.,
Patent Owner

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

PATENT OWNER'S PRELIMINARY RESPONSE

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LIST OF EXHIBITS

Exhibit No.	Description
2001	Case Docket for *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.)
2002	Order, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del), D.I. 116 (May 3, 2024)
2003	Lam's Initial Invalidity Contentions, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (May 13, 2024)
2004-2027	*Reserved*
2028	Lam-Entegris Strategic Collaboration Announcement, *available at* https://www.entegris.com/en/home/about-us/news/lam-research-entegris-gelest-team-team-up-to-advance-euv-dry-resist-technology-ecosystem-july-2022.html (last visited May 12, 2025), and https://www.prnewswire.com/news-releases/lam-research-entegris-gelest-team-up-to-advance-euv-dry-resist-technology-ecosystem-301584404.html (last visited May 12, 2025)
2029	Amended Complaint, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.), D.I. 43 (Dec. 13, 2023)
2030	Lam's Answer, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.), D.I. 52 (Jan. 12, 2024)
2031	Lam's Initial Invalidity Contentions, Ex. 13, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (May 13, 2024)
2032	Lam's Final Invalidity Contentions, Ex. 903-A, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del) (Feb. 20, 2025)
2033	Lam's Final Invalidity Contentions, Ex. 903-B, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (Feb. 20, 2025)
2034	Lam's Final Invalidity Contentions, Ex. 903-C, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (Feb. 20, 2025)
2035	Lam's Final Invalidity Contentions, Ex. 903-D, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (Feb. 20, 2025)
2036	Lam's Final Invalidity Contentions, Ex. 903-E, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (Feb. 20, 2025)

2037	Lam's Final Invalidity Contentions, Ex. 903-F, *Inpria Corp. v. Lam Research Corp.*, C.A. No. 22-01359-CJB (D. Del.) (Feb. 20, 2025)
2038	U.S. Patent No. 8,198,352
2039	May 9, 2025 email correspondence between counsel for Entegris and counsel for Inpria regarding Lam Common Interest

I. INTRODUCTION

Patent Owner Inpria, Corp. ("PO" or "Inpria") respectfully submits this Preliminary Response to the Petition for *inter partes* review (Paper 1) (the "Petition" or "Pet.") filed by Entegris, Inc. ("Petitioner" or "Entegris") against claims 1-4 and 6-10 ("the challenged claims") of U.S. Patent No. 11,673,903 ("the '903 patent"). First, the Petition should be discretionarily denied for the reasons argued in Patent Owner's Brief Supporting Discretionary Denial, filed concurrently with this Preliminary Response. Second, the Petition should be denied under 35 U.S.C. § 314(a) because Entegris has not met its burden of demonstrating a reasonable likelihood of prevailing with respect to any of the challenged claims.

Accordingly, as discussed in more detail below, institution should be denied.

II. BACKGROUND

The '903 patent is titled "Monoalkyl Tin Compounds with Low Polyalkyl Contamination, Their Compositions and Methods," and issued on June 13, 2023. Ex. 1001 at 1. The claims recite a "high purity liquid composition comprising" a "monoalkyl tin triamide compound" with a maximum allowable impurity of "dialkyltin compounds"—"no more than 0.5 mole %":

> 1. A high purity liquid composition comprising:
>
> a monoalkyl tin triamide compound represented by the chemical formula $RSn(NR'_2)_3$ and

no more than 0.5 mole % dialkyltin compounds as an impurity relative to the total tin amount,

wherein R is a hydrocarbyl group with 1-31 carbon atoms, and

wherein R′ is a hydrocarbyl group with 1-10 carbon atoms.

Ex. 1001 at Claim 1. Claim 6 recites an even stricter purity of "essentially no dialkyltin compounds as measured with an approximate 0.1 mole % detection limit." *Id.* at Claim 6.

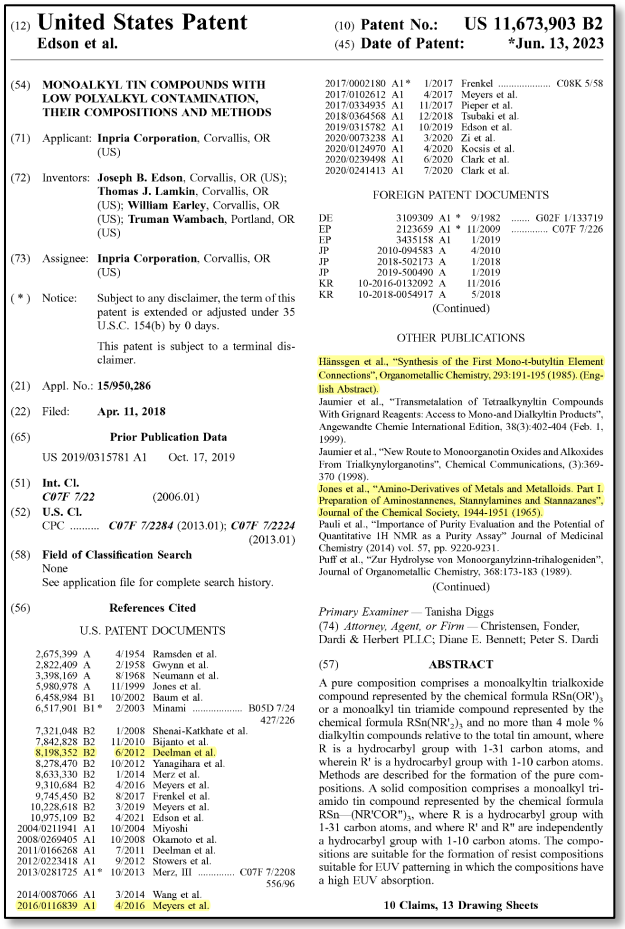
Entegris's Petition relies on four prior art references:

1. Hänssgen – An article published in the Journal of Organometallic Chemistry in 1985 titled "Synthesis of the First Mono-t-Butyltin Element Compounds." Ex. 1005 at 1.
2. Meyers – A publication of an Inpria patent application publication that was published on April 28, 2016, entitled "Organometallic Solution Based High Resolution Patterning Compositions and Corresponding Methods." Ex. 1006 at 1.
3. Deelman – A publication of a patent application titled "High Purity Monoalkyltin Compounds and Uses Thereof," published on July 7, 2011. Ex. 1007 at 1.
4. Jones – A publication in the Journal of the Chemical Society in 1965 titled "Amino-derivatives of Metals and Metalloids. Part I. Preparation

of Aminostannanes, Stannylamines, and Stannazanes." Ex. 1009 at 1, 3.

Pet. at 7, 17-22.

Each of these prior art references were presented to the Office during prosecution and are shown on the cover of the '903 patent[1]:



(12) **United States Patent**
Edson et al.

(10) **Patent No.: US 11,673,903 B2**
(45) **Date of Patent: *Jun. 13, 2023**

(54) **MONOALKYL TIN COMPOUNDS WITH LOW POLYALKYL CONTAMINATION, THEIR COMPOSITIONS AND METHODS**

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(72) Inventors: **Joseph B. Edson**, Corvallis, OR (US); **Thomas J. Lamkin**, Corvallis, OR (US); **William Earley**, Corvallis, OR (US); **Truman Wambach**, Portland, OR (US)

(73) Assignee: **Inpria Corporation**, Corvallis, OR (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **15/950,286**

(22) Filed: **Apr. 11, 2018**

(65) **Prior Publication Data**

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CPC ***C07F 7/2284*** (2013.01); ***C07F 7/2224*** (2013.01)

(58) **Field of Classification Search**
None
See application file for complete search history.

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(57) **ABSTRACT**

A pure composition comprises a monoalkyltin trialkoxide compound represented by the chemical formula $RSn(OR')_3$ or a monoalkyl tin triamide compound represented by the chemical formula $RSn(NR'_2)_3$ and no more than 4 mole % dialkyltin compounds relative to the total tin amount, where R is a hydrocarbyl group with 1-31 carbon atoms, and wherein R' is a hydrocarbyl group with 1-10 carbon atoms. Methods are described for the formation of the pure compositions. A solid composition comprises a monoalkyl triamido tin compound represented by the chemical formula $RSn—(NR'OR'')_3$, where R is a hydrocarbyl group with 1-31 carbon atoms, and where R' and R'' are independently a hydrocarbyl group with 1-10 carbon atoms. The compositions are suitable for the formation of resist compositions suitable for EUV patterning in which the compositions have a high EUV absorption.

10 Claims, 13 Drawing Sheets

[1] Entegris's Petition relies on a publication of the Deelman application that matured into the Deelman patent listed on the face of the '903 patent.

Ex. 1001 at 1. Hänssgen, Meyers, and Deelman were all expressly incorporated by reference into the '903 patent. Ex. 1001 at 3:32-36, 3:55-62, 4:8-16. Jones was cited in an IDS and listed on the cover of the '903 patent. Ex. 1004 at 1149.

Hänssgen and Meyers describe t-BuSn$(NEt_2)_3$ and i-PrSn$(NMe_2)_3$ compounds, respectively, and procedures for synthesizing those compounds. Ex. 1005 at 2, 4; Ex. 1006 at ¶¶ 150-151 (Example 7). Neither reference describes the purity of the desired compound or the amount of undesired dialkyl compound resulting from the synthesis. Because of this, Entegris conducted laboratory experiments purporting to replicate the prior art procedures (or modified versions of the procedures) in attempting to prove the inherent result of the procedures fell within the purity restrictions of the '903 patent claims.

III. LEVEL OF ORDINARY SKILL

For purposes of this Preliminary Response, Inpria adopts Entegris's definition of a person of ordinary skill in the art ("POSITA"). Inpria reserves the right to propose an alternative definition of a POSITA should the Board grant institution.

IV. CLAIM CONSTRUCTION

The Board need not construe any claim term because no construction is needed to resolve the parties' dispute. *See Toyota Motor Corp. v. Cellport Sys., Inc.*, IPR2015-00633, Paper 11, at 16 (Aug. 14, 2015) (citing *Vivid Techs., Inc. v. Am. Sci. & Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999)). Under any reasonable

construction of the claim terms, Entegris has failed to demonstrate that the prior art discloses or suggests the challenged claims.

V. ARGUMENT

A. Legal Standard

1. Inherent Anticipation

Claims are unpatentable for inherent anticipation only "when a single prior art reference discloses each and every limitation of the claim." *Glaxo Inc. v. Novopharm Ltd.*, 52 F.3d 1043, 1047 (Fed. Cir. 1995); 35 U.S.C. § 102. "Prior art that must be modified to meet the disputed claim limitation does not anticipate the claim." *Enplas Display Device Corp. v. Seoul Semiconductor Co., Ltd.*, 909 F.3d 398, 405 (Fed. Cir. 2018). "Such modifications are permissible only in an obviousness analysis." *Apple Inc. v. Corephotonics, Ltd.*, 861 F. App'x 443, 450 (Fed. Cir. 2021).

A claim is inherently anticipated only when the "missing descriptive matter is necessarily present in the [prior art]." *Cont'l Can Co. USA v. Monsanto Co.*, 948 F.2d 1264, 1268 (Fed. Cir. 1991). But proving inherency is a high burden—it "may not be established by probabilities or possibilities." *In re Oelrich*, 666 F.2d 578, 581 (C.C.P.A. 1981) (internal quotation omitted). Instead, "[t]he inherent result must inevitably result from the disclosed steps." *In re Montgomery*, 677 F.3d 1375, 1380 (Fed. Cir. 2012).

Inherency of a claimed physical property may be proven through extrinsic evidence, such as experimental testing of the prior art, but the tests must faithfully follow the prior art procedures, and the claimed property must be the natural result of the experiments. *See, e.g.*, *Purdue Pharma L.P. v. Epic Pharma, LLC*, 811 F.3d 1345, 1357 (Fed. Cir. 2016). Experimental testing is not probative of inherency when the tests deviate from the procedures described in the prior art. *See In re Armodafinil Pat. Litig. Inc.*, 939 F. Supp. 2d 456, 478-82 (D. Del. 2013). Moreover, "limited testing and selection of variables, where reasonable alternatives were available" does not show that a claimed limitation "is the necessary and inevitable result" of procedures described in the prior art. *Id.* at 486.

2. Inherent Obviousness

The use of inherency, "a doctrine originally rooted in anticipation, must be carefully circumscribed in the context of obviousness." *PAR Pharm., Inc. v. TWI Pharms., Inc.*, 773 F.3d 1186, 1195 (Fed. Cir. 2014). "'[T]o rely on inherency to establish the existence of a claim limitation in the prior art in an obviousness analysis,' [the patent challenger] must show that [the missing claim] limitation is 'necessarily present' or 'the natural result of the combination of elements explicitly disclosed by the prior art.'" *LBT IP I LLC v. Apple Inc.*, No. 2022-1613, 2023 WL 3914920, at *3 (Fed. Cir. June 9, 2023) (alteration in original) (quoting *PAR Pharm.*, 773 F.3d at 1195-96). Indeed, the "mere possibility [of meeting the claim limitation]

is not enough" to show inherency in the obviousness context, which "may not be established by probabilities or possibilities," and requires a showing that "*the natural result flowing* from the operation as taught would result in the performance of the questioned function." *PersonalWeb Techs., LLC v. Apple, Inc.*, 917 F.3d 1376, 1382 (Fed. Cir. 2019) (emphasis in original).

Like with inherent anticipation, experimental testing is not probative of inherent obviousness when the tests deviate from the procedures described in the prior art. *See Excelixis, Inc. v. MSN Labs. Priv. Ltd.*, No. 22-228-RGA, 2024 WL 4491176, at *26-27 (D. Del. Oct. 15, 2024). Furthermore, conclusory assertions or reliance on only common sense are insufficient to establish inherency in the obviousness context. *See SSI Techs., LLC v. Dongguan Zhengyang Elec. Mech. Ltd.*, No. 20-cv-19-jdp, 2024 WL 2383268, at *7-8 (W.D. Wis. May 23, 2024).

B. Each of Entegris's Grounds Rely on Experimental Testing

The arguments in Entegris's Petition are substantively based on inherency. This is demonstrated by, *inter alia*, Entegris's assertions as to experiments it conducted allegedly following the prior art and its conclusory analysis with respect to reasonable expectation of success. *See* Pet. at 29, 33-34 (Ground 1A), 37, 39-40 (Ground 1B), 43-44, 46, 49 (Ground 2A), 51-52, 56 (Ground 2B), 59-61, 64 (Ground 3A), 70-72 (Ground 3B). Indeed, Entegris cites case law concerning inherent

properties. Pet. at 37, 51 (citing *Cytiva BioProcess R&D AB v. JSR Corp.*, 122 F.4th 876, 889 (Fed. Cir. 2024)).

For Grounds 1A and 1B, Entegris's analysis focuses on Hänssgen's discussion of the "t-BuSn(NEt_2)$_3$" compound (also described as Compound ("I")), which is produced according to Hänssgen's Equation (2a). *See* Petition at 27-29 (citing Ex. 1005 at 2); Ex. 1002 at ¶¶ 113-118; Ex. 1020; Ex. 1003 at ¶¶ 9-25.

$$(Et_2N)_4Sn \xrightarrow[-LiNEt_2]{+\text{t-BuLi}} \underset{(I)}{\text{t-BuSn}(NEt_2)_3} \xrightarrow[-LiNEt_2]{+\text{t-BuLi}} \text{t-Bu}_2\text{Sn}(NEt_2)_2 \qquad (2a)$$

Ex. 1005 at 2.

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

To a solution of 4.08 g (10 mmol) $(Et_2N)_4Sn$ in 50 ml diethyl ether, at -78°C under agitation, 6.2 ml of a 1.6 *M* solution of t-BuLi was added by drops into n-pentane. It was allowed to come to room temperature and agitated for an additional 2 h. The ether was distilled off and the residue was dissolved in 30 ml benzene. The precipitated $LiNEt_2$ was filtered out and the benzene phase was fractionated in a vacuum.

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

IR (Film, $\bar{\nu}$ from selection): 2958vs, 2920s, 2840s, 1460m, 1440m 1368s, 1282m, 1170vs, 1090w, 1062w, 1000s, 874m, 782m, 570m, 450m. ^{1}H-NMR (δ(ppm), C_6D_6): 1.13 (t, 18H, $NCCH_3$); 3.13 (q, 12H, NCH_2); 1.28 (s, 9H, Sn-t-Bu); $^3J(^1HCN^{117/119}Sn)$ 91/95 Hz; $^3J(^1HCN^{117/119}Sn)$ 37.5/39 Hz. MS: Only rearrangement peaks were recorded. Relative molar mass (cryoscopic benzene): 367.

Precip.: C, 48.70; H, 9.90; N, 10.90. $C_{16}H_{39}N_3Sn$ (392.2) Rep.: C, 49.00; H, 10.02; N, 10.17%.

Ex. 1005 at 4.

In describing this reaction, Hänssgen states that

> The reaction of $(Et_2N)_4Sn$ with equimolar quantities of lithium-t-butyl, by splitting a tin-nitrogen bond according to equation 2a, yields triamino-t-butylstannane (I) and

> lithium diethyl amide. The reaction proceeds practically quantitatively; there are no indications of secondary reactions. Lithium diethyl amide can be separated from the reaction mixture by precipitation with benzene and analytically pure I can be obtained by fractionated distillation of the benzene phase (yield 93%).

Ex. 1005 at 2.

For Grounds 2A and 2B, Entegris's analysis focuses on Meyers's discussion of the "iPrSn(NMe_2)$_3$" compound (called "iso-propyl tris(dimethylamido)tin"), whose synthesis is described at the beginning of Example 7. *See* Pet. at 40-44 (citing Ex. 1006 at ¶¶ 150-151); Ex. 1002 at ¶¶ 119-122; Ex. 1019; Ex. 1003 at ¶¶ 26-47.

Example 7

Preparation of an Iso-Propyltin Hydroxide Oxide Hydrolysate Via an Iso-Propyl Tris(dimethylamido)tin Precursor

[0150] A water-reactive precursor, isopropyl tris(dimethylamido)tin, (i-PrSn(NMe_2)$_3$), was synthesized under inert atmosphere and subsequently hydrolysed by two methods using 1) atmospheric moisture and 2) liquid H_2O, to form an i-PrSn$O_{(3/2-x/2)}(OH)_x$ hydrolysate.

[0151] Under argon, a 1 L Schlenk-adapted round bottom flask was charged with $LiNMe_2$ (81.75 g, 1.6 mol, Sigma-Aldrich) and anhydrous hexanes (700 mL, Sigma-Aldrich) to form a slurry. A large stir bar was added and the vessel sealed. An addition funnel under positive argon pressure was charged with i-PrSnCl_3, (134.3 g, 0.5 mol, Gelest) via syringe and attached to the reaction flask. The flask was cooled to −78° C. and the i-PrSnCl_3 added dropwise over a period of 2 hours. The reaction was allowed to come to room temperature overnight while stirring, and the solid precipitates allowed to settle. After settling, the reaction solution was filtered under positive argon pressure through an in-line cannula filter. The solvent was removed under vacuum and the residue distilled under reduced pressure (50-52° C., 1.4 mmHg) to give a pale yellow liquid (110 g, 75% yield). ^{1}H and ^{119}Sn NMR spectra of the distillate in a C_6D_6 solvent collected on a Bruker DPX-400 (400 MHz, BBO probe) spectrometer are shown in FIGS. **22** and **23**, respectively. Observed ^{1}H resonances, as shown in FIG. **22**, (s, 2.82 ppm, —N(CH_3)$_2$; d 1.26 ppm, —CH_3; m, 1.60 ppm, —CH) match the predicted spectra for ^{i}PrSn(NMe_2)$_3$. The primary ^{119}Sn resonance, as shown in FIG. **23**, at −65.4 ppm is consistent with a major product with a single tin environment, with a chemical shift comparable to reported monoalkyltin amido compounds.

Ex. 1006 at ¶¶ 150-151 (highlighting by Entegris); *see also* Pet. at 40 (citing these paragraphs); Ex. 1002 at ¶ 120 (pointing to "Example 7 of Meyers").

For Grounds 3A and 3B, although Entegris's grounds only facially recite "Deelman and Jones" or "Deelman and Jones and Hänssgen," Pet. at 7, 57-74, the analysis relies on the Meyers experiments that were relied upon for Grounds 2A and 2B. Specifically, for Ground 3A, Entegris relies on the experiments in its "Reasons to Combine" section. Pet. at 59-61, 64 (referring to "Petitioner's experiments" regarding "i-PrSn(NMe_2)$_3$"). For Ground 3B, Entegris provides no new analysis for independent claim 1 or dependent claims 2-4 and 6, simply referring back to Ground 3A which, as described above, relies on "Petitioner's experiments." Pet. 70; *see also id.* at 73 (relying on "§XIII.B.3," which is the section of Ground 3A relying on "Petitioner's Experiments," Pet. at 64).

As described above, all of Entegris's unpatentability grounds rely on its experimental testing. Because this experimental testing was faulty, for the reasons described below, Entegris has not shown a reasonable likelihood of success in showing unpatentability of any of the claims challenged in Entegris's petition.

C. Entegris's Experimental Tests Are Flawed and Fail to Meet the Heavy Burden of Evidencing Inherency

Entegris does not meet the heavy burden to show inherency and cannot support a reasonable likelihood of prevailing with respect to any challenged claim. *See In re Montgomery*, 677 F.3d at 1380. Inherency "may not be established by

probabilities or possibilities." *In re Oelrich*, 666 F.2d at 581 (internal quotation omitted). Entegris's Petition should be denied because it includes unexplained modifications to the prior art in attempting to prove inherency through experimental testing—which undermines all of its unpatentability arguments. In addition to making unexplained modifications, Entegris also fails to provide sufficient data to show its tests reliably meet the heavy burden of proving inherency.

1. Entegris Fails to Support Its Experimental Results with Sufficient Facts or Data to Show Reliability

Entegris and its experts fail to disclose underlying bases and data from its experiments to show reliability of the results. For example, Entegris does not provide the laboratory notebooks that would have been created by Dr. Hood in performing the experiments. And even though the critical metric from the testing was a purity value derived from NMR data, Entegris fails to include any of the basic NMR data (Free Induction Decay data) or certain conditions used to collect the data (such as relaxation values)—instead only providing screenshots of the NMR spectra and limited information in Dr. Hood's declaration. *See* Ex. 1003 at ¶¶ 10, 16-18, 23-25, 35-38, 45-47.

For starting materials, even though Entegris's experts conducted several, different NMR tests (^{1}H, ^{13}C, and ^{119}Sn NMR) on the resulting products of its experiments, it failed to characterize *any* of the starting materials with any NMR testing or provide any basic data about those starting materials (such as providing

material safety data sheets or other standardized documentation), other than a generic statement that "[r]eagents for the reactions were purchased from Sigma Aldrich" for the Hanssgen experiments. Ex. 1003 at ¶ 9. Notably, as shown below, Entegris conspicuously omits even this basic information from the Meyers experiment description:

> **II. Synthesis of $^{t}BuSn(NEt_2)_3$**
>
> **a. General methods and materials**
>
> 9. All manipulations were performed in the absence of light under a dry nitrogen atmosphere inside a mBRAUN glovebox or using Schlenk glassware and standard air free techniques. Reagents for the reactions were purchased from Sigma Aldrich. Solvents were taken from a solvent purification system and stored in the glovebox over 4 Å molecular sieves.

Ex. 1003 at ¶ 9 (highlighting added).

> **III. Synthesis of i-PrSn(NMe$_2$)$_3$**
>
> **a. General methods and materials**
>
> 26. All manipulations were performed in the absence of light under a dry nitrogen atmosphere inside a mBRAUN glovebox or using Schlenk glassware and standard air free techniques. Solvents were taken from a solvent purification system and stored in the glovebox over 4 Å molecular sieves. All ^{1}H, ^{13}C and ^{119}Sn NMR

Ex. 1003 at ¶ 26 (annotation added).

All of this undermines Entegris's unpatentability challenges based on inherency, which carries a high burden.

2. Entegris's Hänssgen Experiments are Flawed

Entegris alleges that "Petitioner's experiments confirmed Hänssgen's disclosure enabled synthesis of $tBuSn(NEt_2)_3$ with only 0.17 mole % dialkyltin impurity $tBu_2Sn(NEt_2)_2$ relative to the total tin amount." *See* Pet. at 29 (citing Ex. 1002 at ¶ 130; Ex. 1003 at ¶¶ 17-18). In addition to being unreliable for the reasons above, Entegris's experiments modify the prior art process described in Hänssgen and accordingly cannot confirm the inherent content of Hänssgen's disclosure.

Entegris's expert, Dr. Parkin, testifies that he "closely follow[ed] the detailed experimental conditions disclosed in Hänssgen, including using the same amounts of reagents. *Compare* EX1020 *with* EX1005." Ex. 1002 at ¶ 115. However, comparing Hänssgen (Ex. 1005) with the procedures from Dr. Parkin (Ex. 1020) shows that Dr. Parkin silently added—without providing *any* basis in the prior art or a POSA's knowledge—that "the reactants and the products … should also be protected from ambient light to prevent any possible decomposition from prolonged exposure." Ex. 1020. Dr. Hood appears to have complied with this unexplained directive from Dr. Parkin by ensuring that the "flask containing $Sn(NEt_2)_4$ was then

wrapped in aluminum foil to prevent light exposure," which is further corroborated by an image found in Dr. Hood's declaration:

by a keck clamp. The flask containing $Sn(NEt_2)_4$ was then wrapped in aluminum foil to prevent light exposure (Figure 1).

Figure 1. Air free addition setup with $Sn(NEt_2)_4$ in the flask and ^tBuLi in the addition funnel.

Ex. 1003 at ¶¶ 11, 19.

Entegris points to nothing in any prior art (let alone Hänssgen itself) or the knowledge of a POSA mentioning or suggesting this modification. Dr. Parkin does not provide any rationale or cited reference to justify this modification to Hänssgen's prior art process. Because Entegris does not provide any prior-art supported

rationale for wrapping the reaction vessel in aluminum foil, the results of Entegris's testing are irrelevant for inherency of the prior art.

Entegris's discussion of Hänssgen in Ground 1A of the Petition also ignores portions of Hänssgen which provide caution that "***disubstitutions*** on the dimethyl aminostannane $(Me_2N)_4Sn$ are accordingly ***thermodynamically favored over a mono-substitution***. ***That may also be true*** for the diethyl amino substitution product $(Et_2N)_4Sn$." Ex. 1005 at 3 (emphases added). Hänssgen goes on to discuss how "reactions of $(Et_2N)_4Sn$ with lithium-t-butyl ***in excess under moderate conditions*** always yield only the mono-substitution product I. If the components are heated, the result is product mixtures of undetermined genesis." *Id.* (emphasis added). In other words, Hänssgen qualified the conditions which led to the production of Compound I in Equation 2(a).

Entegris does not address these qualifications from Hänssgen in the Petition. Dr. Parkin, in his declaration, cherry picks the word "excess" as a basis for instructing Dr. Hood to conduct an additional reaction with "excess amount of lithium t-butyl." Ex. 1002 at ¶¶ 113-114. Hänssgen does not define what is intended by "in excess," and Dr. Parkin without explanation or literature support instructs Dr. Hood to use an arbitrary amount of "1.2eq of tBuLi" (or 20% excess). Ex. 1002 at ¶¶ 114. Dr. Parkin creates a new "Procedure 2" stating that "[i]nstead of employing 10 mmol of t-BuLi, use an excess amount of t-BuLi (e.g., by 20%) in

Step 2." Ex. 1020. Dr. Hood appears to have complied with this unexplained directive from Dr. Parkin to increase the amount of tBuLi to 1.2eq. *See* Ex. 1003 at ¶¶ 19-25. Because Entegris provides no prior-art supported rationale for constructing the new "Procedure 2," the results of Entegris's testing of procedure 2 are irrelevant for inherency of the prior art.

To the extent Entegris suggests the modification is supported by the bare words "in excess" in Hänssgen, Ex. 1005 at 3, Entegris's single experiment fails to show the "necessary and inherent result" of "in excess." Entegris's testing of only one arbitrary amount of lithium t-butyl does not confirm that a POSA following Hänssgen's process would necessarily and inevitably produce "no more than 0.5 mole % dialklyltin compounds as an impurity relative to total tin amount" as claimed. *See In re Armodafinil*, 939 F. Supp. 2d at 486-87 (finding no inherency where challenger provided "limited testing and selection of variables, where reasonable alternatives were available"). And as discussed below, the results indicate that achieving results within the claimed purity limits would not be inherent.

The results of this additional reaction show that only a 20% increase in the amount of lithium t-butyl reactant doubled the amount of dialkyltin impurity. Ex. 1002 at ¶¶ 118, 130 (reporting 0.34 mol % $tBu_2Sn(NEt_2)_2$); *see also* Ex. 1003 at ¶¶ 24-25. Dr. Parkin does not request additional testing or explain what amount of dialkyltin impurity would be produced with an even greater (>20%) excess of

lithium t-butyl, which would arguably be within Hänssgen's ambiguous statement of "excess amount."

Further undermining Entegris's experiments, Dr. Parkin never explains why the initial reaction went so poorly—yielding only 86.72% of the desired $tBuSn(NEt_2)_3$ with 12.93% of starting material left over after the reaction. *See* Ex. 1003 at ¶ 18.[2] This is significantly different from the yield and starting material left over from the "Procedure 2" reaction performed with a 20% excess of t-BuLi, which showed a 98.74% yield of $tBuSn(NEt_2)_3$ with 0.79% of starting material left over after reaction. *See* Ex. 1003 at ¶ 25. Entegris's failure to explain this glaring discrepancy, or conduct more than its limited set of experiments to resolve the issue, undermines its argument that the claimed purity is the necessary and inevitable result from practicing Hänssgen.

3. Entegris's Meyers Experiments are Flawed

Entegris's Grounds 2 and 3 rely on experimental results from a modified version of Meyers. But because the rationale that Entegris's expert gives for modifying Meyers's procedure is hindsight and because Entegris fails to explain the motivation for the specific way in which it modified Meyers's procedure or why a

[2] None of Entegris's other experiments, even for other grounds, resulted in such a low yield or any leftover starting material. *See* Ex. 1003 at ¶¶ 37, 47.

POSA would have had a reasonable expectation of success with the specific modifications Entegris made, its experimental results are irrelevant to prove inherency.

Entegris admits that "Meyers does not explicitly disclose the specific purity levels of the isopropyl tris(dimethylamido)tin compound." Pet. at 40 (citing Ex. 1002 at ¶ 213). Nevertheless, Entegris attempted to prove inherency by "first replicat[ing] the synthesis procedures described in Example 7 of Meyers." Ex. 1002 at ¶ 120; Ex. 1019. But, as with Entegris's Hänssgen testing, Dr. Parkin's procedure silently adds the same unexplained modification that "the reactants and the products … should also be protected from ambient light to prevent any possible decomposition from prolonged exposure," Ex. 1019, without providing any basis in the prior art or a POSA's knowledge. Again, Dr. Hood complied with this unexplained directive by ensuring the "entire reaction setup was … covered in aluminum foil to prevent exposure to light," as shown in the images in Dr. Hood's declaration:

Figure 22. Air free addition setup attached to the Schlenk line with the $LiNMe_2$ suspension being cooled in an isopropanol liquid nitrogen bath (-78 ºC).

Ex. 1003 at ¶ 28. Due to this unexplained modification alone, Entegris's experiments are irrelevant to prove inherency.

Even with this unexplained modification, Entegris still failed to meet the claim limitations. And it was not close. As Dr. Hood's results show, the reaction resulted in 93.32% of the desired monoalkyl product with 6.16% of the undesired dialkyl impurity. Ex. 1003 at ¶¶ 36-37. Dr. Parkin attempts to downplay this failure, calling it a "small quantity of dialkyltin diamide impurity," Ex. 1002 at ¶ 120, but this experiment resulted in more than twelve times the allowable limit in claim 1 of

the ’903 patent, Ex. 1001 at 17:44-45 (“no more than 0.5 mole % dialkyltin compounds”).

Entegris nonetheless alleges that a POSA “would have naturally looked to Deelman to determine a method to increase the purity of the monoalkyltin triamide compound.” Pet. at 41. But the Petition does not give a rationale for why a POSA would have been looking to “increase” the purity of Meyers’s procedures. Instead, they point to a single paragraph of Meyers describing “high-purity alkyltin compounds” for performing the synthesis approaches “[a]s described herein.,” Ex. 1006 at ¶ 63; Pet. at 20, 40, but Entegris provides no explanation whatsoever for why Meyers’s disclosure of such synthetic approaches (*e.g.*, in Example 7) would have been viewed by a POSA as deficient. Entegris’s Petition merely provides high-level rationales regarding the purported compatibility of Meyers and Deelman but does not describe the modification Entegris actually makes to Meyers other than stating it involved “adjustment to the solvent (as disclosed by Deelman).” Pet. at 43.

Although Entegris’s Petition does not describe the details of the modification, Dr. Parkin does. Ex. 1002 at ¶¶ 119-122. Dr. Parkin first describes Entegris’s initial attempts to meet the claims by testing a modified version of Meyers, as described above, but admits that experiment failed to achieve the claim limitation. Ex. 1002 at ¶ 120. Dr. Parkin then leverages this failure of Meyers to produce the claimed

purity as his basis for modifying the Meyers procedures. After explaining that the first experiment failed, Dr. Parkin concludes that "[i]t is apparent that Example 7 of Meyers was not optimized for achieving high purity." Ex. 1002 at ¶ 120. In the next paragraph he explains, "[a]ccordingly, I instructed Dr. Hood to modify the synthesis process." *Id.* at ¶ 121. In other words, Dr. Parkin used the results of an experiment conducted in 2024—that would not have been in the knowledge of a POSA—as a basis to modify the experimental procedures described in Meyers in an attempt to prove inherency.[3] This is impermissible hindsight. *Honeywell Int'l Inc. v. Mexichem Amanco Holding S.A. DE C.V.*, 865 F.3d 1348, 1353-54 (Fed. Cir. 2017). This is a second reason why Entegris's experimental testing of Meyers should be rejected, which undermines each of Grounds 2A, 2B, 3A, and 3B that rely on this second Meyers experiment. *See* Pet. at 43, 46, 49, 52, 60, 64, 70.

Even putting aside the fact that Entegris fails to provide a motivation to combine Meyers with Deelman and the fact that Entegris's expert expressly uses hindsight as the motivation, Entegris and its expert still fail to support the specific modification that was made to the experimental procedure. Dr. Parkin "instructed

[3] Notably, Dr. Parkin did not even bother to create a second procedure (akin to "Procedure 2" in Hänssgen) for his modification of Meyers's experimental process. *See* Ex. 1019 (no mention of addition of THF or dilution of $iPrSnCl_3$).

Dr. Hood" to "first dilute i-$PrSnCl_3$ with tetrahydrofuran ('THF')" before adding that diluted material to the reaction mixture. Ex. 1002 at ¶ 121. Entegris and Dr. Parkin fail to support both the selection of THF and the specific way it was added.

For the selection, Dr. Parkin offhandedly remarks that THF is a "known good solvent for monoalkyl tin trihalide," but only points to a stray paragraph in Deelman as supposedly "disclosing THF as a suitable solvent for monoalkyltin trihalide." *Id.* (citing Ex. 1007 at ¶ 52). In that paragraph, however, Deelman generally describes broad categories of "organic, aprotic, or even protic solvents" as "preferred," with "aromatic solvents, chloroaromatic solvents, alkanes, ethers and alcohols" being "especially" preferred. Ex. 1007 at ¶ 52. Deelman then lists THF as one of three merely "suitable solvents." *Id*. Neither Entegris nor Dr. Parkin give a rationale for plucking THF from this list. Nor do they give any reason why a POSA would have reasonably expected success in "increas[ing] the purity" of Meyers's synthesis of its monoalkyl tin triamide compound (Entegris's stated basis for the combination, Pet. at 41) by using THF. Instead, Dr. Parkin merely alleges this was the predictable result without any supporting rationale. Ex. 1002 at ¶¶ 121 (stating without support that "[a]s expected, the modified process minimized secondary reactions"), 222 (stating "adjustment to the solvent (as disclosed by Deelman) … predictably yielded

a monoalkyltin triamide … that contains only 0.19 mol % dialkyltin impurity"); Pet. at 43 (citing Ex. 1002 at ¶ 222).

For the specific manner in which the THF is added to the experiment, Entegris and Dr. Parkin provide absolutely no rationale whatsoever for choosing this technique. The only explanation from Dr. Parkin is he instructed Dr. Hood to "instead of directly adding the pure monoalkyl tin trihalide (i-$PrSnCl_3$) into the $LiNMe_2$ hexane suspension as described in Example 7 of Meyers, I asked Dr. Hood to first dilute i-$PrSnCl_3$ with tetrahydrofuran ('THF') … and then add the i-$PrSnCl_3$ THF solution to the reaction mixture." Ex. 1002 at ¶ 121. In other words, Dr. Parkin told Dr. Hood what to do, but he provided no reason why. Dr. Hood took this instruction and then arbitrarily chose to use "23 mL of THF" to "dilute the i$PrSnCl_3$" without any rationale as to why that specific amount was chosen. Ex. 1003 at ¶ 40. As such, Entegris entirely fails to provide a motivation for the specific modification that they chose, let alone prove a reasonable expectation of success in their stated goal of "increas[ing] the purity" of Meyers's synthesis of its monoalkyl tin triamide compound using this specific technique. Pet. at 41. Entegris cannot reconcile why a POSA would have focused on Meyers's synthesis process as a target for increasing purity through an unexplained modification with Deelman's THF solvent. This is yet another reason to reject Entegris's grounds that rely on this flawed experimental testing (Grounds 2A, 2B, 3A, and 3B).

In sum, Entegris's reliance on an alleged confirmatory experiment constructed from the hindsight combination of Meyers and Deelman is insufficient to show inherent obviousness. Entegris's conclusory modification of Meyers with Deelman is far from a modification guided by the prior art and does not evidence the "natural result" flowing from the combination of references. *See LBT IP I LLC*, 2023 WL 3914920, at *3; *PersonalWeb Techs., LLC*, 917 F.3d at 1382.

VI. CONCLUSION

For these reasons, Inpria respectfully requests that the Board deny the Petition for *inter partes* review of the '903 patent.[4]

Respectfully submitted,

Dated: May 12, 2025

By: /Naveen Modi/
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Counsel for Patent Owner

[4] If trial is instituted, Inpria reserves the right to raise additional arguments as to why Entegris has failed to carry its burden and why the challenged claims should be confirmed.

CERTIFICATE OF COMPLIANCE

Pursuant to 37 C.F.R. § 42.24(d), the undersigned certifies that the foregoing Patent Owner's Preliminary Response contains, as measured by the word-processing system used to prepare this paper, 4,429 words. This word count does not include the items excluded by 37 C.F.R. § 42.24.

Respectfully submitted,

Dated: May 12, 2025

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CERTIFICATE OF SERVICE

I hereby certify that on May 12, 2025, I caused a true and correct copy of the foregoing Patent Owner's Preliminary Response to be served via email on Petitioner at the following addresses:

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