Washington State
Liquor and Cannabis Board

Notice of Permanent Rules

Regarding Amendment WAC 314-55-010 – Definitions; New Section WAC 314-55-550 – Marijuana vapor products; New Section WAC 314-55-1055 – Ingredient disclosure.

This concise explanatory statement concerns the Washington State Liquor and Cannabis Board's (Board) adoption of amendment to section WAC 314-55-010 and adoption of new sections WAC 314-55-550 and WAC 314-55-1055 allowing the Board oversight and regulation of marijuana vapor products.

The Administrative Procedure Act (RCW 34.05.325(6)) requires agencies to complete a concise explanatory statement before filing adopted rules with the Office of the Code Reviser. The concise explanatory statement must be provided to any person upon request, or from whom the Board received comment.

The Board appreciates and encourages your involvement in the rule making process. If you have questions, please contact Casey Schaufler, Policy and Rules Coordinator, at (360) 664-1760 or e-mail at rules@lcb.wa.gov.

Background and reasons for adopting these rules

The adopted rules amend WAC 314-55-010 and add new sections WAC 314-55-550 and WAC 314-55-1055 to implement the directives and requirements of House Bill (HB) 2826 (Chapter 133, Laws of 2020) concerning marijuana vapor products, now codified in RCW 69.50.101, RCW 69,50.327, RCW 69.50.342. HB 2826 provides that the Board may adopt rules prohibiting any type of marijuana vapor product device, or prohibit the use of any type of additive, solvent, ingredient, or compound in the production and processing of marijuana products, including marijuana vapor products.

The adopted rules are necessary to allow the Board to implement marijuana vapor product regulation consistent with HB 2826, and to establish definitions for terms including, but not limited to "characterizing flavor," botanical terpenes," and others.

Rulemaking history for this adopted rule:

CR 101 – filed July 8, 2020 as WSR #20-15-041;
CR 102 – filed December 9, 2020 as WSR #21-01-058.
Public hearing held February 3, 2021.

The effective date of these rules is March 20, 2021.

Public comment received on the rule proposal:

The following comments were received as indicated below, and are presented in their native form, including formatting, text and spelling. A response to each comment is provided, along with an indication regarding whether the comment was reflected in the adopted rule.

1. Email received December 18, 2020:
David Heldreth wrote:
"https://mjbizdaily.com/wp-content/uploads/2020/12/10-11-20-Report-VP-and-ERSA.pdf

I'd like this pdf entered into the record and shared with the WSLCB board."

WSLCB response: The WSLCB appreciates these comments, and the demonstration of meaningful, collaborative participation in the rulemaking process. The WSLCB looks forward to your continued partnership on future policy and rule development projects.

No changes to the rules were requested. Article is presented in full per request and guidelines of original publishers.

2. Email received February 3, 2021:
Ezra Eickmeyer wrote:
Dear Mr. Schaufler,

Attached, please find comments from Producers NW on the draft rules for marijuana vapor products. Thank you. (attachment contents below)

Dear LCB Rules Coordinator,

Producers NW would like to thank LCB for a good overall set of draft rules regarding Marijuana Vapor Products and propose the edits listed below.

We appreciate the need to protect the public from harmful additives and believe these rules will do more than enough to ensure vape product safety. It is also our goal to ensure that the process is streamlined and minimizes costs to the industry. Please contact us anytime with questions. Thank you.

Proposed Changes

- Add definition of marijuana vapor products –

"Marijuana vapor products" means any marijuana concentrate that is intended to be heated into a vapor state and inhaled into the lungs by the consumer. "Marijuana vapor

products" does not include marijuana concentrates or extracts intended to be used as topicals, suppositories, pills, tinctures or any other concentrate-based product that is not intended for inhalation.

- The vapor disclosure form needs to be amended to be less redundant and it is not made clear that all marijuana concentrates for inhalation need to have a form disclosed.
    - We do not support having the WSLCB collecting these forms because of the potential for public records requests making proprietary information public. Instead, we suggest having disclosures kept on site and available for WSLCB audit upon request.

- Characterizing flavor in the definitions section (314-55-010 (4)) needs to be amended in the following way –

  (4) "Characterizing flavor" means a noticeable taste, other than one of cannabis, resulting from an additive or combination of additives including, but not limited to, fruit, spice, herbs, alcohol, candy, or menthol~~, or that is noticeable before or during consumption of the cannabis product~~.

  NOTE - Since WSLCB enforcement is not allowed to consume cannabis this could allow enforcement to write AVN's based upon subjective smell of the product. The smell of a botanical mix can be more fragrant than the actual flavor. This industry should not be in a position where the smell of a cartridge is a determining factor.

- Terpenes (314-55-010 (40)) -

We need to be careful with this section as this definition would apply to the upcoming QA rules that, in their current draft, require the addition of terpenes to require a terpene analysis. If the WSLCB does go through with those rules as drafted, any compound that meets the definition above when added to a cannabis product would then require another test, adding redundant expenses to the cost of the product.

WSLCB Response: The WSLCB appreciates these comments, and the demonstration of meaningful, collaborative participation in the rulemaking process. The WSLCB looks forward to your continued partnership on future policy and rule development projects.

With respect to the definition of marijuana vapor products: The adopted rules do not reflect this suggested revision. WAC 314-55-1055 as adopted distinguishes "ingredients used in the production of marijuana concentrates for inhalation and marijuana-infused extracts for inhalation" as products subject to disclosure and to new section WAC 314-55-550 – Marijuana vapor products.

With respect to marijuana vapor product disclosure forms: The adopted rules do not reflect this suggested revision. There is no proprietary protection afforded to information

required under RCW 69.50.342(1)(n). Additionally, the format and contents of the ingredient disclosure form are outside of the scope of rulemaking.

With respect to the definition for characterizing flavor: The adopted rules do not reflect this suggested revision. It is important to note that the definition includes, "or that is noticeable before or during consumption of the cannabis product." The inclusion of the singular "or" implies that an individual may reasonably determine that a characterizing flavor is present through means other than taste alone, including but not limited to smell or packaging design.

With respect to the definition for terpenes: The adopted rules do not reflect this suggested revision. A definition in concept or proposed language, was not offered.

Public Hearing, February 3, 2021:

There was no oral testimony offered at the public hearing held on February 3, 2021.

Changes from Proposed Rules (CR-102) to the Rules as Adopted:

There were no changes to the proposed rules.

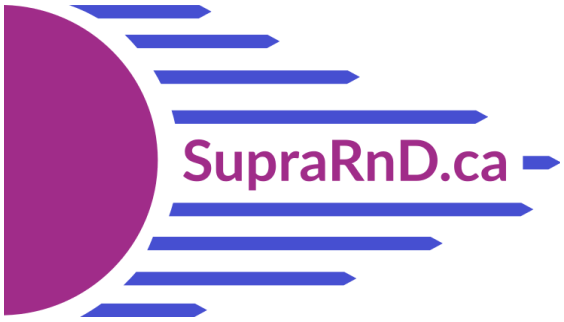
Vaporization Potential and

Effective Residual Solvent Analysis Report

Vitamin E Acetate, Squalane and Squalene

November 10, 2020

Prepared by



Supra Research and Development

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Table of Contents

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

1.0 Introduction

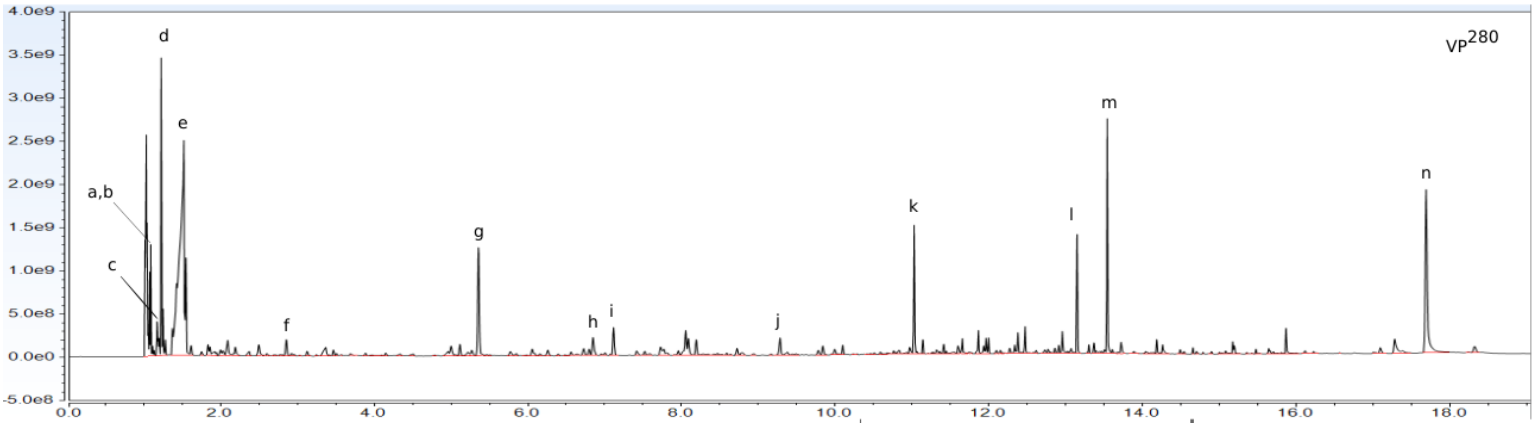
Consumer products that are intended to be consumed by inhalation after high temperature vaporization are a relatively new category of products that require a unique approach to determine the relative risks associated with consumer use. The most significant variable is that at elevated temperatures ingredients can rearrange, react and/or thermally degrade to create new chemical structures that can have fundamentally different chemical properties with different pharmacological consequences of use. This chemical change is dependent not only on the vaporization temperature but also on the composition of the material being vaporized. In some cases, compounds such as Vitamin E acetate which are Generally Regarded as Safe (“**GRAS”**) when introduced to a consumer at room temperature by ingestion may decompose to produce a complex mixture of chemical agents with significant toxicities at high temperatures. Furthermore, the lack of standardization for devices used to generate vapors after high temperature vaporization means that the temperature used is often unknown. Some of the compounds generated at elevated temperatures are themselves reactive and can further react, rearrange or decompose to alternate structures. This type of possible chemical behavior greatly complicated traditional chemical analysis as quantitation standards would also decompose at the temperatures in question. The sampling of vapors produced by devices is a potential approach to determine exposure risk for consumers of devices, however, the diversity of devices used makes determination of the correct devices to use for such studies a significant challenge. Regardless of the challenges, it is critically important to develop approaches to evaluate ingredients that could be used in products that are intended to be consumed by Inhalation after high temperature vaporization so that those materials that have a high likelihood of exposing the consumer to dangerous chemical agents are not used as ingredients. This work will highlight such an approach and apply it to the examination of 3 different potential ingredients, Vitamin E Acetate, Squalane and Squalene.

2.0 Vaporization Potential

Supra Research and Development (“**SUPRA**”) has developed an approach to determine the profile of the diverse range of thermally generated compounds generated by ingredients that are intended to be used in vaporizers. Rather than try and develop a standardized device for producing vapors, we use an analytical instrument that can heat a sample in a controlled manner and then collect and analyse the byproducts. The instrumentation we are using is called Headspace - Gas Chromatography Mass Spectrometry. In this approach a small quantity of sample is accurately heated in hermetically sealed glass vials to a series of well defined temperatures. At each temperature, a sample of the gas phase vapour, also called the “HeadSpace”, is collected and analysed. This analysis involves separation of individual chemical components in a Gas Chromatograph followed by detection in a Mass Spectrometer. The Mass Spectrometer allows for both identification of individual components as well as relative quantitation. The

information can be graphically displayed as a chromatogram where individual compounds are displayed as 'peaks'. A sample chromatogram is presented in Figure 1 below;

Figure 1: Vaporization Potential Chromatogram of Vitamin E Acetate collected at 280ºC



The Chromatograph shows the range of thermal degradation vaporization byproducts that are generated at a given temperature. We have defined this profile of products that can be produced at a given temperature as the Vaporization Potential ("**VP**"). This profile is temperature dependent and so to further define the profile we use the nomenclature **VP^{xyz}** where the number "xyz" is the temperature that the profile was gathered, for example **VP^{280}** is the Vaporization Potential profile collected at 280ºC.

The **VP** profiles are representative of the gas phase above a vaporized sample and thus the profile of chemical agents that would be delivered to the consumer when the user draws in this vapor when using a heated device. This information is critical to understanding the potential pharmacological consequences of inhaling the chemical profile generated at a specific temperature from a specific composition from a vaporized sample. However, at the current time there are no established regulatory limits to the quantity of chemical agents a user can safely be exposed to when using a vaporized product. The development of these types of regulatory standards and the universal acceptance of such standards would require a lengthy and potential contentious legal and scientific based process. Although, we fundamentally agree that this type of process has significant merit, there is also merit in finding an alternate approach that could identify additives, such as Vitamin E Acetate, that have been clearly linked to adverse health events, specifically the **EVALI** hospitalizations and deaths observed in late 2019 and 2020. **EVALI** is the name given by the US Centers for Disease Control and Prevention ("**CDC**") to the dangerous, newly identified lung disease linked to vaping. The name **EVALI** is an acronym that stands for e-cigarette or vaping product use-associated lung injury.

In order to develop an approach for screening ingredients and mixtures intended to be used in vaporization devices for their potential to produce dangerous chemical agents, we have developed an alternate approach we refer to as Equivalent Residual Solvent Analysis ("**ERSA**").

3.0 Equivalent Residual Solvent Analysis

Most finished consumer products intended for human consumption which could include exposure to solvents as extraction agents or chemical cleaning agents are required to be tested for Residual Solvents. This Residual Solvent Analysis is a well established approach and section 467 of the US Pharmacopeia ("**USP<467>**") outlines limits for a variety of potential residual solvents. These limits are universally accepted as levels that consumer products should not exceed in order to be safe. We have observed that many of the chemical agents observed when collecting VP data are in fact included on the residual solvent list. Given this we developed a testing protocol where we place a test sample in an hermetically sealed glass headspace vial, then heat this to a defined test temperature, say 240ºC, hold it for 5 minutes, then cool it to room temperature and then analysed this material using a validated Residual Solvent Analysis method. The validated Residual Solvent Analysis method we employ is also a Headspace-GCMS method, however, in this case the vial is only heated to 95ºC and an external calibration curve is used to quantify the observed residual solvents generated from the heated incubation step. We refer to this approach as Equivalent Residual Solvent Analysis ("**ERSA**"). If the residual solvent analysis indicates that a sample would fail, then we conclude that the material should not be used in any product intended for inhalation that heats the material at a temperature above the temperature at which it failed.

Even though the stated approach will work at any temperature, we have found that as the temperature approaches 300ºC almost all materials we have examined fail and for practical considerations we have selected a temperature of 280ºC as the highest test point in this study. Furthermore, we also consider 240ºC to be the highest temperature that any vaporization device should be set as, as above this the concentrations of problematic thermal degradation products increase drastically. Given that, we typically recommend that a **VP^{240}** be the test temperature for routine screening and the **ERSA** analysis at 240ºC be used as the definitive pass fail test criteria. We have also observed that 180ºC is a temperature where Cannabinoids, typical Terpenes and Nicotine and related chemical compounds are effectively vaporized with little or no thermal degradation. Although we have observed a few problematic compounds begin to thermally degrade at temperatures as low as 210ºC, most do not begin to degrade until the temperature exceeds 220ºC. With this in mind we can imagine a public health message that strongly discourages any vaporization above 420ºF or 215.6ºC.

4.0 Summary of results for Vitamin E Acetate, Squalane and Squalene

In this report 3 ingredients have been examined: Vitamin E Acetate, Squalane and Squalene. Each of these have "failed" the **ERSA** assessment at 240ºC.

4.1 Vitamin E Acetate

The chemical structure of Vitamin E Acetate is presented below. This compound was a known additive in e-juice and Cannabis concentrates associated with many of the **EVALI** hospitalizations and deaths observed in late 2019 and 2020. It has been suggested that this compound is responsible for many of the adverse health effects in the **EVALI** event.

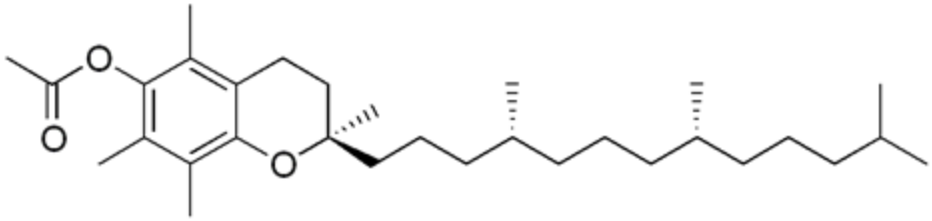


Figure 2: Chemical structure of Vitamin E Acetate

The VP profiles at a series of temperatures for Vitamin E Acetate is presented in Figure A.1 of Appendix A. The most dominant Oxidation products are Acetic acid and Formic acid and these are observed at sufficient quantities to have the compound fail the **ERSA** screening approach at 240ºC. This data is presented in Table A.2 presented in Appendix A.

4.2 Squalane

The chemical structure of squalene is presented in Figure 3 below. This is a possible ingredient that could be used in vaporization devices.

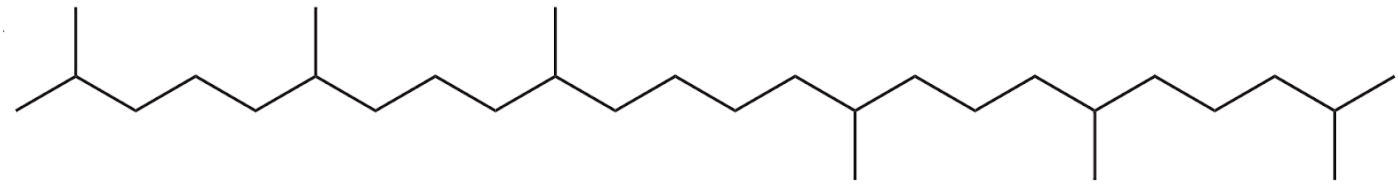


Figure 3: Chemical structure of squalane

The VP profiles at a series of temperatures for this compound is presented in Figure B.1 of Appendix B. The most dominant Oxidation products are Acetone, Methanol and Acetic acid and these are produced at sufficient quantities to have the compound fail the **ERSA** analysis at 240ºC. This **ERSA** data is presented in

Table B.2 presented in Appendix B. There are a diverse number of thermal degradation and oxidation products produced by squalane and based on this and the very high concentration of Acetone, Methanol and Acetic Acid we speculate that this additive would produce more diverse and adverse health effects as Vitamin E Acetate does.

4.3 Squalene

The chemical structure of squalene is presented in Figure 4 below. This is also a possible ingredient that could be used in vaporization devices.

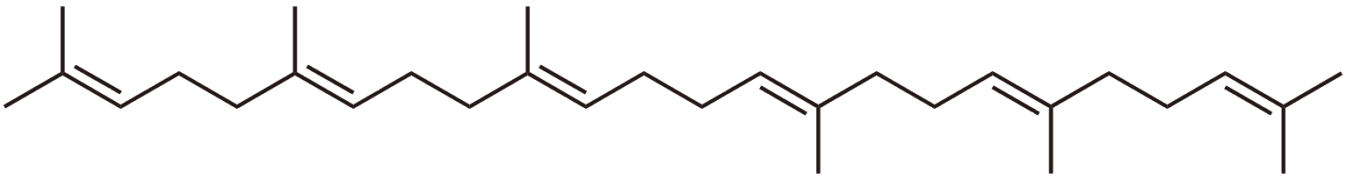


Figure 4: Chemical structure of squalene

The VP profiles at a series of temperatures for this compound is presented in Figure C.1 of Appendix C. There are a large number of Oxidation products generated including Acetone, Methanol, Acetic acid and Formic Acid that are produced at sufficient quantities to have the compound fail the **ERSA** analysis at 240ºC. This **ERSA** data is presented in Table C.2 presented in Appendix C. The diverse number of thermal degradation and oxidation products produced by squalene is of significant concern, especially, because this degradation begins at much lower temperature, 180ºC, than observed for other ingredients that we have studied previously. It is speculated that Squalene would produce more adverse health effects as Vitamin E Acetate does and that these adverse effects could begin at much lower vaporization temperatures.

5.0 Conclusion

The three compounds that we have examined in this report, Vitamin E Acetate, Squalane and Squalene each have failed the **ERSA** assessment protocol we have defined at 240ºC. Vitamin E Acetate has been identified as a problematic ingredient associated with **EVALI** hospitalizations and deaths. The data presented here suggests that Squalane and Squalene thermally degrade in a manner that produces higher levels of chemical agents than we observed for Vitamin E Acetate. From this, we speculate that these compounds could be more problematic than Vitamin E Acetate. However, it should be noted that these are speculations based on assumptions and this opinion is provided for discussion purposes only and is not intended to be a definitive statement on the safety of a given product or ingredient.

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Appendix A: Sample Results for Vitamin E Acetate

Client ID: n.a.

Supra Details: α-Tocopheryl acetate (Vitamin E acetate) (Sigma-Aldrich PN#R1030 Lot#LRAC1696)

Batch ID: 201022_VP-RS-quant-Oregon

Submission Date: 2020 October 15

Reporting Date: 2020 November 12

Analysis Date: 2020 October 22

Analyst: RJH / SRS

Authorized By: Ryan Hayward

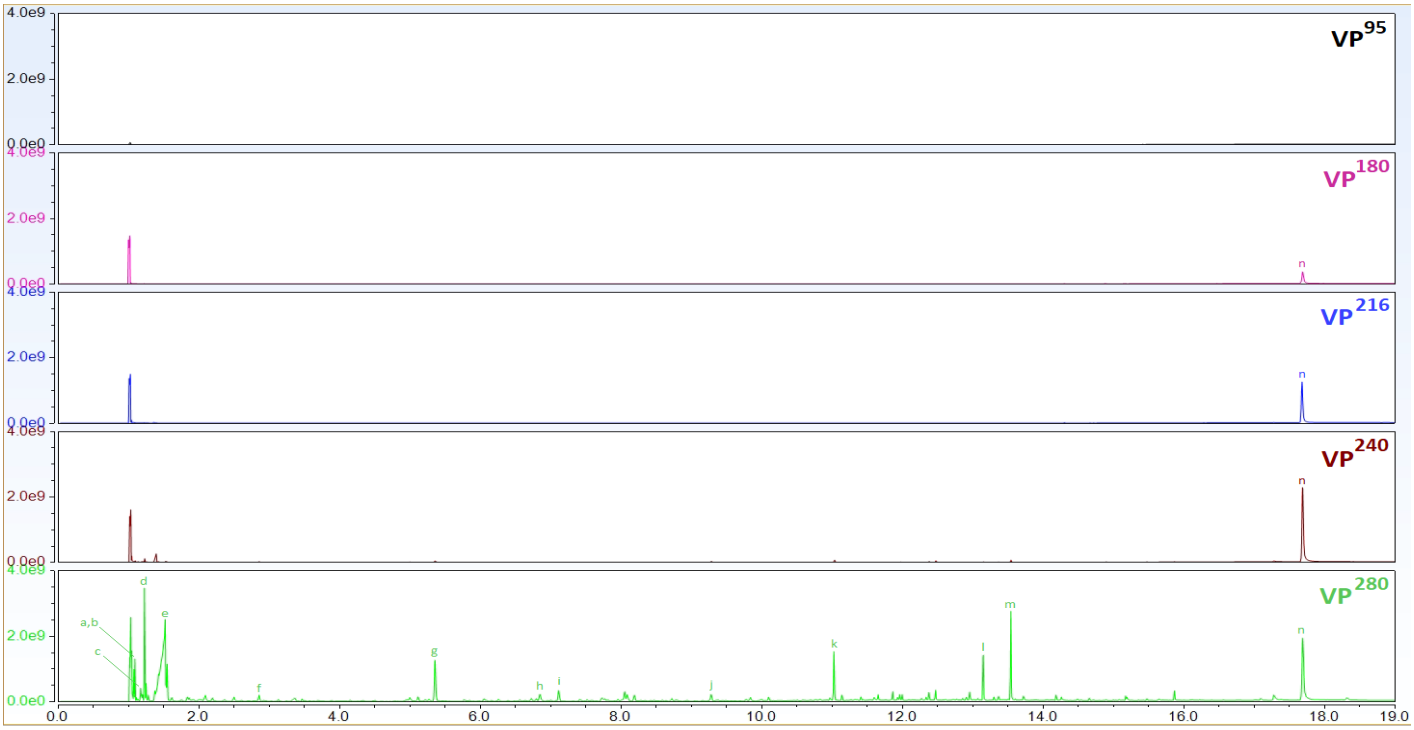
Job Function: Laboratory Manager

Date Authorized: 2020 November 10

Signature: 

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Figure A.1: Vaporization Potential Chromatograms For Vitamin E Acetate



Total Ion Chromatograms (TICs) of VP^{95}, VP^{180}, VP^{216}, VP^{240} and VP^{280} of vitamin E acetate. The chromatograms are scaled to the same *y*-axes.

Table A.1: Identified peaks for Vitamin E Acetate (qualitative profile)

Compound	Retention time (min)	Chromatogram label
methanol	1.07	a
acetaldehyde*	1.09	b
oxalic acid*	1.17	c
acetone	1.23	d
formic acid	1.50	e
hexanal*	2.86	f
6-methyl-2-heptanone*	5.36	g
2-nonanone*	6.85	h
4-methyl-3-pentenoic acid*	7.13	i
4,8-dimethylnonanol*	9.28	j
6,10-dimethyl-2-undecanone*	11.04	k
6,10,14-trimethyl-2-pentadecanone*	13.15	l
3-formyl-4-hydroxy-2,5,6-trimethylphenyl acetate*	13.54	m
vitamin E acetate	17.69	n

List of identified compounds in thermally-treated samples (see Figure 1 for labelled chromatograms). Compounds marked with an asterisk (*) were identified using NIST library matching (>800 SI and RSI). All other compounds were identified using analytical standards

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Table A.2: Equivalent Residual Solvent Analysis at 240ºC Vitamin E Acetate

	USP limit	VP^{240}
2-Butanone	5000	< 1000
2-Propanol	5000	nd
Acetone	5000	< 1000
Acetonitrile	410	nd
Benzene	2	nd
Cyclohexane	3880	nd
Ethanol	5000	< 1000
Ethyl formate	5000	nd
Hexane	290	nd
Isobutanol	5000	< 1000
Isopropyl acetate	5000	< 1000
Methanol	3000	< 600
Methylcyclohexane	1180	nd
n-Pentane	5000	< 1000
Acetic acid*	5000	> 10000
Formic acid*	5000	> 10000

Table 2 Description: Quantitated concentrations (parts-per-million [ppm] relative to original sample mass [Table 3]) of degradation products identified for each sample treatment at 240 ºC. Values were calculated using a full evaporation technique (FET) headspace method calibrated with residual solvent standards. Calibration ranges were 0.2x to 2x each analyte's USP limit. Results outside the calibration range are reported as greater than (>) or less than (<) the respective upper or lower limits of calibration. A semi-quantitative calibration was performed for formic acid and acetic acid. These compounds have been marked with an asterisk (*) and their results should be treated as estimates. Shaded values indicate failures.

Table A.3: Experimental details Vitamin E Acetate

After accurate weighing (Table 3), all samples were incubated in gas-tight headspace vials fitted with PTFE-lined silicone septa for temperatures ranging from 95 - 280 °C (*n* = 1/temperature). All incubations were performed for five minutes and included a blank vial alongside client formulations.

	Vaporization Potential ($VP^{°C}$)				
	VP^{95}	VP^{180}	VP^{216}	VP^{240}	VP^{280}
Vitamin E acetate (g)	0.0104	0.0098	0.0111	0.0111	0.0105

Masses of materials used for each temperature treatment. Samples were incubated at their designated temperature for five minutes to achieve an equilibrated headspace, from which 1 mL was sampled for analysis. Sampling was performed directly from the incubated vial to reflect delivery of volatiles into the headspace at respective temperatures.

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Appendix B: Sample Results for Squalane

Client ID: n.a.

Sample Details: Squalane (Sigma-Aldrich PN#PMR1417 Lot#LRAC4099)

Batch ID: 201022_VP-RS-quant-Oregon

Submission Date: 2020 October 15

Reporting Date: 2020 November 12

Analysis Date: 2020 October 22

Analyst: RJH / SRS

Authorized By: Ryan Hayward

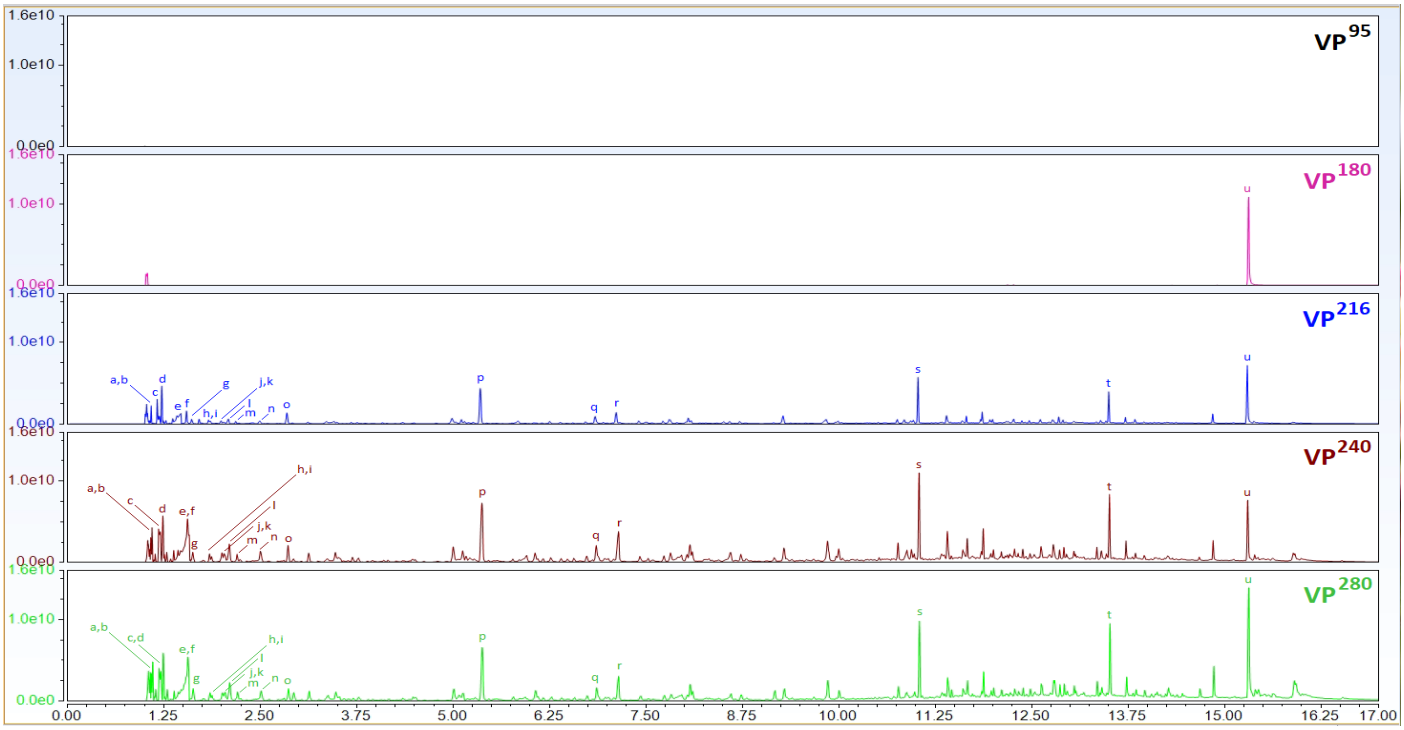
Job Function: Laboratory Manager

Date Authorized: 2020 November 10

Signature: 

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Figure B.1: Vaporization Potential Chromatograms For Squalane



Total Ion Chromatograms (TICs) of VP^{95}, VP^{180}, VP^{216}, VP^{240} and VP^{280} of squalane. See Table 1 for peak labels. The chromatograms are scaled to the same *y*-axes.

Table B.1: Identified peaks for Squalane (qualitative profile)

Compound	Retention time (min)	Chromatogram label
methanol	1.07	a
acetaldehyde*	1.09	b
oxalic acid*	1.17	c
acetone	1.23	d
acetic acid	1.50	e
2-butanone	1.55	f
4-methyl-3-pentenal*	1.62	g
3-methylbutanal*	1.85	h
3-methyl-2-butanone*	1.87	i
2-methylheptane*	2.00	j
2,2-dimethyltethrahydrofuran	2.04	k
2-pentanone*	2.10	l
acetol*	2.19	m
2-hexanone*	2.50	n
hexanal*	2.86	o
6-methyl-2-heptanone*	5.36	p
2-nonanone*	6.85	q
4-methyl-3-pentenoic acid*	7.14	r
6,10-dimethyl-2-undecanone*	11.04	s
2-nonadecanone*	13.52	t
squalane	15.31	u

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

List of identified compounds in thermally-treated samples (see Figure B.1 for labelled chromatograms). Compounds marked with an asterisk (*) were putatively identified using NIST library matching (>800 SI and RSI). All other compounds were identified using analytical standards.

Table B.2: Equivalent Residual Solvent Analysis at 240ºC Squalane

	USP limit	VP^{240}
2-Propanol	5000	nd
Acetone	5000	> 10000
Acetonitrile	410	< 82
Benzene	2	nd
Cyclohexane	3880	< 776
Ethanol	5000	< 1000
Ethyl formate	5000	nd
Hexane	290	nd
Isobutanol	5000	nd
Isopropyl acetate	5000	nd
Methanol	3000	> 6000
Methylcyclohexane	1180	< 236
n-Pentane	5000	< 1000
Acetic acid*	5000	> 10000
Formic acid*	5000	< 1000

Quantitated concentrations (parts-per-million [ppm] relative to original sample mass [Table B.3]) of degradation products identified for each sample treatment at 240 ºC. Values were calculated using a full evaporation technique (FET) headspace method calibrated with residual solvent standards. Calibration ranges were 0.2x to 2x each analyte's USP limit. Results outside the calibration range are reported as greater than (>) or less than (<) the respective upper or lower limits of calibration. A semi-quantitative calibration was performed for formic acid and acetic acid. These compounds have been marked with an asterisk (*) and their results should be treated as estimates. Shaded values indicate failures.

Table B.3: Experimental details Squalane

After accurate weighing (Table B.3), all samples were incubated in gas-tight headspace vials fitted with PTFE-lined silicone septa for temperatures ranging from 180 - 300 °C (*n* = 1/temperature). All incubations were performed for five minutes and included a blank vial alongside client formulations.

	Vaporization Potential ($VP^{°C}$)				
	VP^{95}	VP^{180}	VP^{216}	VP^{240}	VP^{280}
Squalane (g)	0.0100	0.0094	0.0094	0.0103	0.0099

Masses of materials used for each temperature treatment. Samples were incubated at their designated temperature for five minutes to achieve an equilibrated headspace, from which 1 mL was sampled for analysis. Sampling was performed directly from the incubated vial to reflect delivery of volatiles into the headspace at respective temperatures.

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Appendix C: Sample Results for Squalene

Client ID: n.a.

Supra Details: Squalene (Sigma-Aldrich PN#S3626 Lot#MKCJ2769)

Batch ID: 201022_VP-RS-quant-Oregon

Submission Date: 2020 October 15

Reporting Date: 2020 November 12

Analysis Date: 2020 October 22

Analyst: RJH / SRS

Authorized By: Ryan Hayward

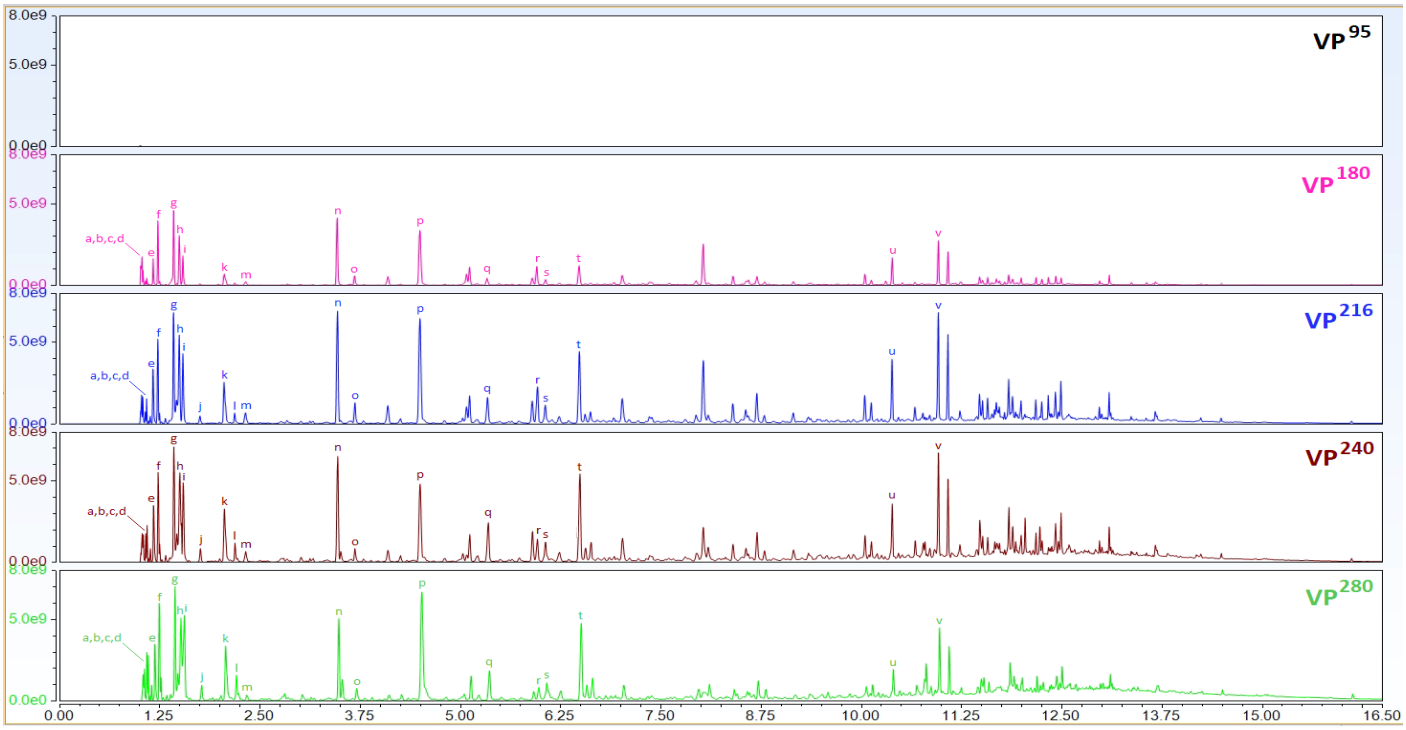
Job Function: Laboratory Manager

Date Authorized: 2020 November 10

Signature: 

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

Figure C.1: Vaporization Potential Chromatograms For Squalene



Total Ion Chromatograms (TICs) of VP^{95}, VP^{180}, VP^{216}, VP^{240} and VP^{280} of squalene. See Table 1 for peak labels. The chromatograms are scaled to the same *y*-axes.

Table C.1: Identified peaks for Squalene (qualitative profile)

Compound	Retention time (min)	Chromatogram label
methanol	1.07	a
acetaldehyde*	1.09	b
glyoxal*	1.10	c
ethanol	1.13	d
oxalic acid*	1.17	e
acetone	1.23	f
methacrolein*	1.42	g
2-methyl-3-buten-2-ol*	1.50	h
3-buten-2-one*	1.54	i
3-hydroxy-3-methyl-2-butanone*	1.76	j
3-ethyl-2,2-dimethyloxirane*	2.06	k
1-hydroxy-2-propanone*	2.18	l
1-ethyl-5-methylcyclopentene*	2.32	m
3-methyl-2-butenal*	3.46	n
4-hydroxy-2-butanone*	3.69	o
3-methylcyclopentyl acetate*	4.50	p
4,4,5-trimethyl-1,3-dioxan-5-ol*	5.36	q
2,3-dimethyl-3-buten-2-ol*	5.95	r
6-methyl-5-hepten-2-one*	6.06	s
1-(1-butenyloxy)pentane*	6.49	t
citral*	10.04	u
3,6-dimethyloctan-2-one*	10.96	v

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

List of identified compounds in thermally-treated samples (see Figure C.1 for labelled chromatograms). Compounds marked with an asterisk (*) were putatively identified using NIST library matching (>800 SI and RSI). All other compounds were identified using analytical standards.

Table C.2: Equivalent Residual Solvent Analysis at 240ºC Squalene

	USP limit	VP^{240}
2-Propanol	5000	< 1000
Acetone	5000	> 10000
Acetonitrile	410	nd
Benzene	2	0.4
Cyclohexane	3880	nd
Ethanol	5000	1382
Ethyl formate	5000	< 1000
Hexane	290	136
Isobutanol	5000	< 1000
Isopropyl acetate	5000	< 1000
Methanol	3000	> 6000
Methylcyclohexane	1180	< 236
n-Pentane	5000	< 1000
Acetic acid*	5000	> 10000
Formic acid*	5000	> 10000

Quantitated concentrations (parts-per-million [ppm] relative to original sample mass [Table 3]) of degradation products identified for each sample treatment at 240 ºC. Values were calculated using a full evaporation technique (FET) headspace method calibrated with residual solvent standards. Calibration ranges were 0.2x to 2x each analyte's USP limit. Results outside the calibration range are reported as greater than (>) or less than (<) the respective upper or lower limits of calibration. A semi-quantitative calibration was performed for formic acid and acetic acid. These compounds have been marked with an asterisk (*) and their results should be treated as estimates. Shaded values indicate failures.

Table C.3: Experimental details Squalene

After accurate weighing (Table 3), all samples were incubated in gas-tight headspace vials fitted with PTFE-lined silicone septa for temperatures ranging from 95 - 280 °C (*n* = 1/temperature). All incubations were performed for five minutes and included a blank vial alongside client formulations.

	Vaporization Potential ($VP^{°C}$)				
	VP^{95}	VP^{180}	VP^{216}	VP^{240}	VP^{280}
Squalene (g)	0.0096	0.0095	0.0102	0.0103	0.0111

Masses of materials used for each temperature treatment. Samples were incubated at their designated temperature for five minutes to achieve an equilibrated headspace, from which 1 mL was sampled for analysis. Sampling was performed directly from the incubated vial to reflect delivery of volatiles into the headspace at respective temperatures.

Supra Research and Development Inc.
106-2293 Leckie Rd, Kelowna B.C. V1X 6Y5
http://suprarnd.ca/ | inquire@suprarnd.ca | 1-855-437-8772

END OF REPORT

Results reported by Supra Research and Development are representative of the materials as provided by the client.

Results are provided for information only and are not intended to comment on the safety of a given product.

Supra Research and Development shall retain all reports in a secure manner to prevent unauthorized access. Supra reserves the right to charge for the reissuance of reports in some instances.

Supra Research and Development will retain samples for a minimum period of 45 days following the release of the report. After 90 days following the release of the report samples are subject to disposal.