69511.6 Nail Products Containing Toluene

(a)

"Nail products containing toluene" means nail products containing toluene, including nail coatings and nail polish thinners that contain toluene as an added ingredient, contaminant, or residual. (1) "Nail coating" means any clear or colored paint, polish, lacquer, enamel, or gel product marketed or sold for application to fingernails or toenails, including: (A) "Solvent-based nail coatings." "Solvent-based nail coatings" are clear or colored nail coatings that form a hard coating on nails upon evaporation of their solvents, including: 1. "Nail polish." "Nail polish" is a varnish or paint applied to the fingernails or toenails to color them or make them shiny. 2. "Lacquer." "Lacquer" is a coating that dries by means of solvent evaporation. "Lacquer" is also known as "enamel." 3. "Base coat." "Base coat" is a clear or milky-colored coating that is used before applying other coatings to the nail. It may be marketed for strengthening or protecting the nail, restoring moisture to the nail, or helping other coatings to adhere to the nail. "Base coat" is also known as "undercoat." 4. "Top coat." "Top coat" is a clear coating that is used after applying other coatings to the nail. It may be used to protect underlying coatings or to add shine, gloss, or matte to the nail. 5. "Gel nail polish." "Gel nail polish" is a gel varnish coating with a look and feel similar to UV gel nail coatings but that does not require an ultraviolet (UV) or a light-emitting diode (LED) lamp to dry. Gel nail polish typically contains color but can also be a

clear nail coating. "Gel nail polish" is also known as "gel polish." (B) "UV gel nail coatings." "UV gel nail coatings" are clear or colored gel nail coatings that are cured or hardened on nails using a UV or an LED lamp rather than solvent evaporation, including: 1. "UV gel nail polish." "UV gel nail polish" is a premixed coating that is hardened using a UV or an LED lamp. UV gel nail polish typically contains color but can also be a clear coating. "UV gel nail polish" is also known as "UV gel" or "Gel" or "nail gel." 2. "UV gel base coat." "UV gel base coat" is a clear coating that is used before applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp. 3. "UV gel top coat." "UV gel top coat" is a clear coating that is used after applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp. 4. "Hard gel." "Hard gel" is a premixed coating with high solvent resistance; it is hardened using a UV or an LED lamp. It can be applied directly onto natural nails to provide additional strength or sculptured using nail enhancements. 5. "Shellac." "Shellac" is the brand name for a nail product created by Creative Nail Design. It is a hybrid which is a combination of nail polish and gel. Shellac is applied directly onto natural nails, and it is cured through UV light. (C) "Nail art paint." "Nail art paint" is any decorative paint including various solvent-based or UV gel nail coating overlays of nail polish, UV gel, or hybrid coatings like Shellac or airbrush paint applied to fingernails, toenails, or both by any technique. This definition includes "Airbrush nail art paint," which is a nail art paint that is designed or intended to be sprayed onto the nail by a device using compressed air. This product may also be labeled as ink, polish, paint, or pigment for airbrush nail art. (2) "Nail polish thinner" means any liquid product that is marketed or sold for the use of reducing viscosity of nail coatings. It may be marketed for the use of increasing the fluidity or restoring the consistency of nail coatings.

(1)

"Nail coating" means any clear or colored paint, polish, lacquer, enamel, or gel product marketed or sold for application to fingernails or toenails, including: (A) "Solvent-based nail coatings." "Solvent-based nail coatings" are clear or colored nail coatings that form a hard coating on nails upon evaporation of their solvents, including: 1. "Nail polish." "Nail polish" is a varnish or paint applied to the fingernails or toenails to color them or make them shiny. 2. "Lacquer." "Lacquer" is a coating that dries by means of solvent evaporation. "Lacquer" is also known as "enamel." 3. "Base coat." "Base coat" is a clear or milky-colored coating that is used before applying other coatings to the nail. It may be marketed for strengthening or protecting the nail, restoring moisture to the nail, or helping other coatings to adhere to the nail. "Base coat" is also known as "undercoat." 4. "Top coat." "Top coat" is a clear coating that is used after applying other coatings to the nail. It may be used to protect underlying coatings or to add shine, gloss, or matte to the nail. 5. "Gel nail polish." "Gel nail polish" is a gel varnish coating with a look and feel similar to UV gel nail coatings but that does not require an ultraviolet (UV) or a light-emitting diode (LED) lamp to dry. Gel nail polish typically contains color but can also be a clear nail coating. "Gel nail polish" is also known as "gel polish." (B) "UV gel nail coatings." "UV gel nail coatings" are clear or colored gel nail coatings that are cured or hardened on nails using a UV or an LED lamp rather than solvent evaporation, including: 1. "UV gel nail polish." "UV gel nail polish" is a premixed coating that is hardened using a UV or an LED lamp. UV gel nail polish typically contains color but can also be a clear coating. "UV gel nail polish" is also known as "UV gel" or "Gel" or "nail gel." 2. "UV gel base coat." "UV gel base coat" is a clear coating that is used before applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp. 3. "UV gel top coat." "UV gel top coat" is a clear coating that is used after applying other UV gel coatings to the nail; it is cured using a

UV or an LED lamp. 4. "Hard gel." "Hard gel" is a premixed coating with high solvent resistance; it is hardened using a UV or an LED lamp. It can be applied directly onto natural nails to provide additional strength or sculptured using nail enhancements. 5. "Shellac." "Shellac" is the brand name for a nail product created by Creative Nail Design. It is a hybrid which is a combination of nail polish and gel. Shellac is applied directly onto natural nails, and it is cured through UV light. (C) "Nail art paint." "Nail art paint" is any decorative paint including various solvent-based or UV gel nail coating overlays of nail polish, UV gel, or hybrid coatings like Shellac or airbrush paint applied to fingernails, toenails, or both by any technique. This definition includes "Airbrush nail art paint," which is a nail art paint that is designed or intended to be sprayed onto the nail by a device using compressed air. This product may also be labeled as ink, polish, paint, or pigment for airbrush nail art.

(A)

"Solvent-based nail coatings." "Solvent-based nail coatings" are clear or colored nail coatings that form a hard coating on nails upon evaporation of their solvents, including: 1. "Nail polish." "Nail polish" is a varnish or paint applied to the fingernails or toenails to color them or make them shiny. 2. "Lacquer." "Lacquer" is a coating that dries by means of solvent evaporation. "Lacquer" is also known as "enamel." 3. "Base coat." "Base coat" is a clear or milky-colored coating that is used before applying other coatings to the nail. It may be marketed for strengthening or protecting the nail, restoring moisture to the nail, or helping other coatings to adhere to the nail. "Base coat" is also known as "undercoat." 4. "Top coat." "Top coat" is a clear coating that is used after applying other coatings to the nail. It may be used to protect underlying coatings or to add shine, gloss, or matte to the nail. 5. "Gel nail polish." "Gel nail polish" is a gel varnish coating with a look and feel similar to UV gel nail coatings but that does not require an ultraviolet (UV) or a light-emitting diode (LED) lamp to dry. Gel nail polish typically contains color but can also be a clear nail coating. "Gel nail

polish" is also known as "gel polish."

1.

"Nail polish." "Nail polish" is a varnish or paint applied to the fingernails or toenails to color them or make them shiny.

2.

"Lacquer." "Lacquer" is a coating that dries by means of solvent evaporation. "Lacquer" is also known as "enamel."

3.

"Base coat." "Base coat" is a clear or milky-colored coating that is used before applying other coatings to the nail. It may be marketed for strengthening or protecting the nail, restoring moisture to the nail, or helping other coatings to adhere to the nail. "Base coat" is also known as "undercoat."

4.

"Top coat." "Top coat" is a clear coating that is used after applying other coatings to the nail. It may be used to protect underlying coatings or to add shine, gloss, or matte to the nail.

5.

"Gel nail polish." "Gel nail polish" is a gel varnish coating with a look and feel similar to UV gel nail coatings but that does not require an ultraviolet (UV) or a light-emitting diode (LED) lamp to dry. Gel nail polish typically contains color but can also be a clear nail coating. "Gel nail polish" is also known as "gel polish."

(B)

"UV gel nail coatings." "UV gel nail coatings" are clear or colored gel nail coatings that are cured or hardened on nails using a UV or an LED lamp rather than solvent evaporation, including: 1. "UV gel nail polish." "UV gel nail polish" is a premixed coating that is hardened using a UV or an LED lamp. UV gel nail polish typically contains color but can also be a clear coating. "UV gel nail polish" is also known as "UV gel" or "Gel" or "nail gel." 2. "UV gel base coat." "UV gel base coat" is a clear coating that is used before applying other UV gel coatings

to the nail; it is cured using a UV or an LED lamp. 3. "UV gel top coat." "UV gel top coat" is a clear coating that is used after applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp. 4. "Hard gel." "Hard gel" is a premixed coating with high solvent resistance; it is hardened using a UV or an LED lamp. It can be applied directly onto natural nails to provide additional strength or sculptured using nail enhancements. 5. "Shellac." "Shellac" is the brand name for a nail product created by Creative Nail Design. It is a hybrid which is a combination of nail polish and gel. Shellac is applied directly onto natural nails, and it is cured through UV light.

1. "UV gel nail polish." "UV gel nail polish" is a premixed coating that is hardened using a UV or an LED lamp. UV gel nail polish typically contains color but can also be a clear coating. "UV gel nail polish" is also known as "UV gel" or "Gel" or "nail gel."
2. "UV gel base coat." "UV gel base coat" is a clear coating that is used before applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp.
3. "UV gel top coat." "UV gel top coat" is a clear coating that is used after applying other UV gel coatings to the nail; it is cured using a UV or an LED lamp.
4. "Hard gel." "Hard gel" is a premixed coating with high solvent resistance; it is hardened using a UV or an LED lamp. It can be applied directly onto natural nails to provide additional strength or sculptured using nail enhancements.
5. "Shellac." "Shellac" is the brand name for a nail product created by Creative Nail Design. It is a hybrid which is a combination of nail polish and gel. Shellac is applied directly onto natural nails, and it is cured through UV light.

(C)

"Nail art paint." "Nail art paint" is any decorative paint including various solvent-based or UV gel nail coating overlays of nail polish, UV gel, or hybrid coatings like Shellac or airbrush paint applied to fingernails, toenails, or both by any technique. This definition includes "Airbrush nail art paint," which is a nail art paint that is designed or intended to be sprayed onto the nail by a device using compressed air. This product may also be labeled as ink, polish, paint, or pigment for airbrush nail art.

(2)

"Nail polish thinner" means any liquid product that is marketed or sold for the use of reducing viscosity of nail coatings. It may be marketed for the use of increasing the fluidity or restoring the consistency of nail coatings.

(b)

Candidate Chemical. For purposes of this chapter, the following Candidate Chemical is identified as the basis for the product defined in subsection (a) being listed as a Priority Product: (1) Toluene, Chemical Abstract Services Registry Number: 108-88-3.

(1)

Toluene, Chemical Abstract Services Registry Number: 108-88-3.

(c)

Hazard traits associated with toluene include: (1) Neurotoxicity, (2) Developmental toxicity, (3) Neurodevelopmental toxicity, (4) Respiratory toxicity, (5) Nephrotoxicity, (6) Dermatotoxicity, (7) Immunotoxicity, (8) Ocular toxicity, and (9) Ototoxicity.

(1)

Neurotoxicity,

(2)

Developmental toxicity,

(3)

Neurodevelopmental toxicity,

(4)

Respiratory toxicity,

(5)

Nephrotoxicity,

(6)

Dermatotoxicity,

(7)

Immunotoxicity,

(8)

Ocular toxicity, and

(9)

Ototoxicity.

(d)

Toxicological endpoints associated with exposure to toluene include:(1) Dizziness, fatigue, headache, decreased manual dexterity, and chronic degenerative brain disorder, (2) Decreased birth weight or retarded skeletal development, (3) Impaired cognitive function, (4) Respiratory tract irritation, (5) Increased kidney weight or metabolic acidosis, (6) Skin irritation, (7) Decreased thymus weight or dose-dependent effects on suppression of antibody response, (8) Vision effects or optic nerve damage, and (9) Hearing loss.

(1)

Dizziness, fatigue, headache, decreased manual dexterity, and chronic degenerative brain disorder,

(2)

Decreased birth weight or retarded skeletal development,

(3)

Impaired cognitive function,

(4)

Respiratory tract irritation,

(5)

Increased kidney weight or metabolic acidosis,

(6)

Skin irritation,

(7)

Decreased thymus weight or dose-dependent effects on suppression of antibody response,

(8)

Vision effects or optic nerve damage, and

(9)

Hearing loss.

(e)

For purposes of this chapter, the Candidate Chemical identified in subsection (b) is designated as the Chemical of Concern for the product defined in subsection (a).

(f)

A responsible entity shall submit a Priority Product Notification within 60 days of the effective date of this regulation or no later than 60 days after the Priority Product is first placed into the stream of commerce in California. The Preliminary Alternatives Analysis Report for this Priority Product shall be submitted within 180 days after the effective date of this regulation, unless the manufacturer submits

an Alternatives Analysis Threshold Notification pursuant to section 69505.3, which may be submitted concurrently with the Priority Product Notification or shall be submitted no later than the due date for the Preliminary Alternatives Analysis Report for the Priority Product.

(g)

The Alternatives Analysis Threshold for toluene in nail products is set at 100 parts per million (ppm).

(h)

A manufacturer submitting an Alternatives Analysis Threshold Notification shall demonstrate that the concentration of toluene in the Priority Product(s) covered by the notification does not exceed the Alternatives Analysis Threshold. A manufacturer that submits an Alternatives Analysis Threshold Notification shall also demonstrate and certify that the manufacturer meets, and will continue to meet, the criteria and conditions required by section 69505.3. The manufacturer shall notify the Department of any change in the product's exemption status within 30 days, pursuant to section 69505.3(c). (1) Notwithstanding section 69505.3(a)(8), a manufacturer shall demonstrate that the conditions for an Alternatives Analysis Threshold exemption have been met by measuring the concentration of toluene in the Priority Product(s) and submitting laboratory testing results or by submitting certificates of analyses from ingredient supplier(s) whose source material contains toluene and are used to make the Priority Product. Further, if a manufacturer provides information from the ingredient supplier(s), the manufacturer shall also submit calculations of the final concentration of toluene for each formulated Priority Product. (2) A manufacturer shall provide the analytical data, laboratory analytical testing methodology, method validation procedure and data, and the quality control and assurance protocols followed to

measure toluene in each Priority Product, and the name and location of the laboratory that conducted the testing. (3) A testing laboratory that conducts the analysis used by a manufacturer to certify the concentration of toluene in a nail product does not exceed the Alternatives Analysis Threshold shall meet the following method performance criteria. If the method performance criteria are not met, the laboratory must take corrective actions until the performance criteria are met. (A) Sample Preparation Criteria: 1. Each nail product shall be gently mixed or shaken prior to taking an aliquot of the product to ensure the aliquot is representative of the contents in the container. 2. A sample may be introduced into the analytical instrument by various techniques including, but not limited to, purge-and-trap, automated static headspace, and direct injection, provided that all other performance criteria are met. (B) Analytical Method Criteria: 1. It is recommended to use a gas chromatograph/mass spectrometer (GC/MS) method designed for analysis of volatile organic compounds in solid and aqueous samples, such as Method 8260D, as described in U.S. EPA SW-846. Any other analytical technique that meets the method performance criteria in section 69511.6(h)(3) may be used for sample analysis and determination of toluene concentrations in nail products. 2. Either toluene-d8 shall be used as the internal standard with chlorobenzene-d5 as a surrogate or chlorobenzene-d5 shall be used as the internal standard and toluene-d8 used as a surrogate. The internal standard shall be added to each sample, prior to introduction into the analytical instrument, at a concentration within the range of the initial calibration for toluene. 3. The limit of quantitation for toluene shall be at or below one-third of the AAT of 100 ppm. (C) Instrument and Calibration Criteria: 1. All study samples shall be analyzed on a properly calibrated instrument that meets instrument manufacturer specifications. If the instrument calibrations or other instrument check requirements (for

example, mass spectrometer tune, mass calibration check, or qualitative identification criteria) are outside the acceptable criteria, standard measures to correct the problem shall be implemented prior to analyzing samples. 2. The use of a gas chromatograph/mass spectrometer is recommended for the separation and fragmentation of analytes for identification. The ratios of qualifier ions to quantitation ions shall be established during calibration and shall be maintained throughout sample analysis to verify the identity of toluene and ensure that there are no interfering peaks. a. Laboratories may use mass spectrometer full scan, selected ion monitoring, or multiple reaction monitoring scanning modes to analyze samples and meet the limit of quantitation requirement. b. The method shall incorporate, at a minimum, one quantitation and one qualifier ion for toluene. i. The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91. c. The method shall incorporate, at minimum, one quantitation ion for internal standards and surrogates. Further, the method may additionally include one or more qualifier ions for internal standards and surrogates.i. The quantitation ions for toluene-d8 shall include 98. ii. The quantitation ions for chlorobenzene-d5 shall include 117. 3. The instrument tune checks shall be done prior to calibration and at the beginning of each 12-hour analytical period. For analysis run by full scan GC/MS, it is recommended to use 4-bromofluorobenzene (BFB) as the tune verification standard. For selected ion monitoring or multiple reaction monitoring scanning modes, it is recommended to follow the manufacturer's instrument tuning criteria. All samples, including quality control and calibration standards, shall be introduced into the GC/MS for analysis within 12 hours of the analysis of the tune verification standard. Samples and quality control standard solutions that are not analyzed within the 12-hour time window cannot be reported. 4. For each analyte and surrogate of interest, prepare

an initial calibration from standards containing toluene, at a minimum of five different non-zero concentrations, and an appropriate solvent.a. The fitted line of the initial calibration shall meet one of these criteria: i. The relative standard deviation of the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or ii. The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99. 5. The concentration of toluene in the lowest calibration standard solution shall be accurate within 50 percent of its true concentration value, meaning the percent recovery shall be within 50 to 150 percent of the expected concentration, and all other toluene calibration levels shall be within 30 percent of their true value, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. 6. After the initial calibration, the retention time of each internal standard shall be within 30 seconds of the retention time of the internal standard at the midpoint of the initial calibration. 7. An initial calibration verification standard solution shall be prepared from a different source than the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range and analyzed immediately following the initial calibration. The calculated concentration of toluene and the surrogate in the initial calibration verification standard solution shall be within 30 percent of their true concentration values, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. No samples shall be run until the initial calibration verification standard solution is analyzed. 8. A continuing calibration verification standard solution shall be prepared from the same source as the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range. The continuing calibration verification standard solution shall be analyzed before the

samples at the beginning of each 12-hour analytical period. The measured concentration of the continuing calibration verification standard solution shall be within 20 percent of its true concentration value, meaning the percent recovery shall be within 80 to 120 percent of the expected concentration. If the calibration verification does not meet the acceptance criteria, standard measures to correct the problem shall be implemented, and another aliquot of the continuing calibration verification standard solution shall be analyzed. If the response of the continuing calibration verification standard solution is still not within 20 percent of its true concentration value, then a new initial calibration shall be conducted. (D) Sample Analysis Criteria:1. An instrument blank shall be analyzed after the continuing calibration verification standard solution, and before the samples, to demonstrate that the total analytical system is free from contaminants. A sufficient number of instrument blanks shall also be inserted between samples to verify no carryover or cross contamination of toluene from one sample to the next. 2. The response of the internal standard for all samples shall be within 50 to 200 percent of the midpoint of the initial calibration or the continuing calibration verification. 3. The retention time of the analyte of interest shall be within 10 seconds of retention time of the midpoint of the initial calibration or within 10 seconds of the continuing calibration verification standard solution analyzed at the beginning of the 12-hour analytical period. (E) Quality Control Criteria:1. All data shall adhere to a quality control protocol for each batch of 20 samples and each type of nail product analyzed, that includes:a. Preparation and analysis of a method blank. The concentration of toluene in each method blank shall not exceed one half of the lower limit of quantitation; b. Preparation and analysis of a duplicate sample; c. Preparation and analysis of a matrix spike and matrix spike duplicate containing a spiked concentration of toluene in the middle range of the

initial calibration; and d. Preparation and analysis of a laboratory control sample and laboratory control sample duplicate, containing a spiked concentration of toluene in the middle range of the initial calibration. 2. Each product sample, method blank, sample duplicate, matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall be spiked with a surrogate standard solution prior to extraction and analysis. 3. Each matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and each spiked quality control sample shall have a spike recovery between 70 to 130 percent of the true value of the spiked concentrations. 4. A laboratory may establish more rigorous internal control limits but shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and a 70 to 130 percent recovery range for known spiked concentrations. 5. The measured concentration of the surrogate standard solution in each product sample, method blank, sample duplicate, laboratory control sample, laboratory control sample duplicate, matrix spike duplicate undergoing analysis shall be within 70 to 130 percent of the spiked concentration. (F) Definitions: 1. "Aliquot" is a measured portion of a total amount of a larger sample solution or suspension. 2. "Certificate of Analysis (COA)" is a quality assurance document that provides the results of laboratory tests or other analyses performed on a product or ingredient. A COA shall contain information on the name of the product or ingredient, including the batch number, and the release date for each batch tested. The COA shall list each test performed including the quality control acceptance limits, the method quantitative limits, and the numerical results obtained. Certificates shall be dated and signed by authorized personnel and shall include the name, address, and telephone number of the

original product or ingredient manufacturer. 3. "Coefficient of Determination (r2)" is a statistical measure of the strength of relationship between two variables and is the squared value of the correlation coefficient. 4. "Continuing Calibration Verification (CCV) Standard" is a mid-range concentration standard analyzed before, during, and at the end of an analytical batch and verifies that the instrument response has not drifted from the initial calibration response. This standard solution contains a known concentration of the target analyte and is typically derived from the same source as the initial calibration standards. 5. "Correlation Coefficient (r)" is a statistical measure of the strength of relationship between two variables. 6. "Duplicate Sample" is a quality control sample which is identical to one of the analytical samples and undergoes the same sample preparation and analytical procedures as the analytical sample. 7. "Initial Calibration (ICAL)" is a determination of the instrument response over a range of known concentrations of an analyte or analytes. A series of standard solutions is prepared from a certified reference material and analyzed on the instrument prior to any samples. Five or more standard solutions containing progressively higher concentrations of the analytes of interest are generally prepared. 8. "Initial Calibration Verification (ICV) Standard" is a certified solution from a source other than used for the initial calibration standards and is used to verify the accuracy of the initial calibration. 9. "Instrument Blanks" are typically analyzed before the sample analysis and following high concentration samples. These blanks are used to assess background contamination or carryover in the analytical system that may lead to reporting of false positives in the subsequent sample analyses. 10. "Internal Standard (IS)" is a chemical substance that is similar, but not identical, to the target analyte and is added to each sample at a known concentration. The internal standard mimics the behavior of the target analyte but has a different

signal than the analyte. An internal standard is used for quantitation of target analytes and to account for matrix effects and/or variability in instrument response by normalizing the response of the target analytes and surrogates, thereby decreasing measurement bias. 11. "Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)" is a clean matrix which has been spiked with a known concentration of the target analyte. It is prepared and analyzed in the same analytical batch and in exactly the same manner as the other samples. The laboratory control sample and laboratory control sample duplicate are used to assess general method performance based on the ability of the laboratory to successfully recover target analytes. 12. "Limit of Quantitation (LOQ)" is the lowest measured concentration of the analyte that has gone through extraction or dilution and analysis and meets defined accuracy and precision criteria. 13. "Matrix Spike/Matrix Spike Duplicate (MS/MSD)" are quality control samples that contain known concentrations of target analytes which have been added before extraction and analysis. 14. "Method Blank" is a clean matrix quality control sample that is carried through the entire dilution or extraction and analytical process. Concentrations of the target analyte shall not exceed one half of the lower limit of quantitation. If concentrations exceed this value, it is an indication of contamination of the reagents, glassware, or any part of the extraction and analysis process. 15. "Multiple Reaction Monitoring (MRM)" is a highly sensitive analytical method using a triple quadrupole mass spectrometer. The target analyte is ionized in the ion source which creates specific ions that are characteristic of the target analyte. Selected ions are allowed through the first quadrupole and are then subsequently fragmented into product (quantitation and qualifier) ions in the collision cell. These product ions are selectively passed through the final quadrupole, where they are detected. Multiple product ions can

be detected at once. 16. "Percent Recovery" is the proportion of the concentration of a target analyte measured in a sample relative to the known concentration spiked into a sample, conveyed as a percentage. 17. "Relative Percent Difference (RPD)" is the absolute difference between two measurements divided by their average and converted to a percentage. 18. "Relative Standard Deviation (RSD)" is the standard deviation of a group of measurements in a data set divided by their average and converted to a percentage. Relative standard deviation is an indicator of how a group of measurements in a data set are scattered around the mean. 19. "Response Factor (RF)" is the ratio between a signal produced by an analyte and the concentration of analyte which produced the signal. 20. "Selected Ion Monitoring (SIM)" is a mass spectrometry technique in which a limited set of ions with specific mass-to-charge (m/z) ratios is monitored by the instrument. This technique typically results in increased sensitivity relative to full scan mass spectrometry. 21. "Surrogate" is a compound that is used to monitor extraction and analysis efficiency of the method.

(1)

Notwithstanding section 69505.3(a)(8), a manufacturer shall demonstrate that the conditions for an Alternatives Analysis Threshold exemption have been met by measuring the concentration of toluene in the Priority Product(s) and submitting laboratory testing results or by submitting certificates of analyses from ingredient supplier(s) whose source material contains toluene and are used to make the Priority Product. Further, if a manufacturer provides information from the ingredient supplier(s), the manufacturer shall also submit calculations of the final concentration of toluene for each formulated Priority Product.

(2)

A manufacturer shall provide the analytical data, laboratory analytical testing methodology, method validation procedure and data, and the quality control and assurance protocols followed to measure toluene in each Priority Product, and the name and location of the laboratory that conducted the testing.

(3)

A testing laboratory that conducts the analysis used by a manufacturer to certify the concentration of toluene in a nail product does not exceed the Alternatives Analysis Threshold shall meet the following method performance criteria. If the method performance criteria are not met, the laboratory must take corrective actions until the performance criteria are met. (A) Sample Preparation Criteria: 1. Each nail product shall be gently mixed or shaken prior to taking an aliquot of the product to ensure the aliquot is representative of the contents in the container. 2. A sample may be introduced into the analytical instrument by various techniques including, but not limited to, purge-and-trap, automated static headspace, and direct injection, provided that all other performance criteria are met. (B) Analytical Method Criteria: 1. It is recommended to use a gas chromatograph/mass spectrometer (GC/MS) method designed for analysis of volatile organic compounds in solid and aqueous samples, such as Method 8260D, as described in U.S. EPA SW-846. Any other analytical technique that meets the method performance criteria in section 69511.6(h)(3) may be used for sample analysis and determination of toluene concentrations in nail products. 2. Either toluene-d8 shall be used as the internal standard with chlorobenzene-d5 as a surrogate or chlorobenzene-d5 shall be used as the internal standard and toluene-d8 used as a surrogate. The internal standard shall be added to each sample, prior to introduction into the analytical instrument, at a concentration within the range of the initial calibration for toluene. 3. The limit of quantitation for toluene shall be at or below one-third of the AAT of 100 ppm. (C) Instrument and Calibration Criteria: 1. All

study samples shall be analyzed on a properly calibrated instrument that meets instrument manufacturer specifications. If the instrument calibrations or other instrument check requirements (for example, mass spectrometer tune, mass calibration check, or qualitative identification criteria) are outside the acceptable criteria, standard measures to correct the problem shall be implemented prior to analyzing samples. 2. The use of a gas chromatograph/mass spectrometer is recommended for the separation and fragmentation of analytes for identification. The ratios of qualifier ions to quantitation ions shall be established during calibration and shall be maintained throughout sample analysis to verify the identity of toluene and ensure that there are no interfering peaks. a. Laboratories may use mass spectrometer full scan, selected ion monitoring, or multiple reaction monitoring scanning modes to analyze samples and meet the limit of quantitation requirement. b. The method shall incorporate, at a minimum, one quantitation and one qualifier ion for toluene. i. The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91. c. The method shall incorporate, at minimum, one quantitation ion for internal standards and surrogates. Further, the method may additionally include one or more qualifier ions for internal standards and surrogates.i. The quantitation ions for toluene-d8 shall include 98. ii. The quantitation ions for chlorobenzene-d5 shall include 117. 3. The instrument tune checks shall be done prior to calibration and at the beginning of each 12-hour analytical period. For analysis run by full scan GC/MS, it is recommended to use 4-bromofluorobenzene (BFB) as the tune verification standard. For selected ion monitoring or multiple reaction monitoring scanning modes, it is recommended to follow the manufacturer's instrument tuning criteria. All samples, including quality control and calibration standards, shall be introduced into the GC/MS for analysis within 12 hours of the analysis of the tune verification standard. Samples and quality control standard solutions that are not analyzed within the 12-hour time

window cannot be reported. 4. For each analyte and surrogate of interest, prepare an initial calibration from standards containing toluene, at a minimum of five different non-zero concentrations, and an appropriate solvent.a. The fitted line of the initial calibration shall meet one of these criteria: i. The relative standard deviation of the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or ii. The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99. 5. The concentration of toluene in the lowest calibration standard solution shall be accurate within 50 percent of its true concentration value, meaning the percent recovery shall be within 50 to 150 percent of the expected concentration, and all other toluene calibration levels shall be within 30 percent of their true value, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. 6. After the initial calibration, the retention time of each internal standard shall be within 30 seconds of the retention time of the internal standard at the midpoint of the initial calibration. 7. An initial calibration verification standard solution shall be prepared from a different source than the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range and analyzed immediately following the initial calibration. The calculated concentration of toluene and the surrogate in the initial calibration verification standard solution shall be within 30 percent of their true concentration values, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. No samples shall be run until the initial calibration verification standard solution is analyzed. 8. A continuing calibration verification standard solution shall be prepared from the same source as the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range. The continuing calibration verification standard solution shall be analyzed before the samples at the beginning of each 12-hour

analytical period. The measured concentration of the continuing calibration verification standard solution shall be within 20 percent of its true concentration value, meaning the percent recovery shall be within 80 to 120 percent of the expected concentration. If the calibration verification does not meet the acceptance criteria, standard measures to correct the problem shall be implemented, and another aliquot of the continuing calibration verification standard solution shall be analyzed. If the response of the continuing calibration verification standard solution is still not within 20 percent of its true concentration value, then a new initial calibration shall be conducted. (D) Sample Analysis Criteria:1. An instrument blank shall be analyzed after the continuing calibration verification standard solution, and before the samples, to demonstrate that the total analytical system is free from contaminants. A sufficient number of instrument blanks shall also be inserted between samples to verify no carryover or cross contamination of toluene from one sample to the next. 2. The response of the internal standard for all samples shall be within 50 to 200 percent of the midpoint of the initial calibration or the continuing calibration verification. 3. The retention time of the analyte of interest shall be within 10 seconds of retention time of the midpoint of the initial calibration or within 10 seconds of the continuing calibration verification standard solution analyzed at the beginning of the 12-hour analytical period. (E) Quality Control Criteria:1. All data shall adhere to a quality control protocol for each batch of 20 samples and each type of nail product analyzed, that includes:a. Preparation and analysis of a method blank. The concentration of toluene in each method blank shall not exceed one half of the lower limit of quantitation; b. Preparation and analysis of a duplicate sample; c. Preparation and analysis of a matrix spike and matrix spike duplicate containing a spiked concentration of toluene in the middle range of the initial calibration; and d. Preparation and analysis of a laboratory control sample and laboratory control sample duplicate, containing a spiked concentration of toluene in the

middle range of the initial calibration. 2. Each product sample, method blank, sample duplicate, matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall be spiked with a surrogate standard solution prior to extraction and analysis. 3. Each matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and each spiked quality control sample shall have a spike recovery between 70 to 130 percent of the true value of the spiked concentrations. 4. A laboratory may establish more rigorous internal control limits but shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and a 70 to 130 percent recovery range for known spiked concentrations. 5. The measured concentration of the surrogate standard solution in each product sample, method blank, sample duplicate, laboratory control sample, laboratory control sample duplicate, matrix spike duplicate undergoing analysis shall be within 70 to 130 percent of the spiked concentration. (F) Definitions: 1. "Aliquot" is a measured portion of a total amount of a larger sample solution or suspension. 2. "Certificate of Analysis (COA)" is a quality assurance document that provides the results of laboratory tests or other analyses performed on a product or ingredient. A COA shall contain information on the name of the product or ingredient, including the batch number, and the release date for each batch tested. The COA shall list each test performed including the quality control acceptance limits, the method quantitative limits, and the numerical results obtained. Certificates shall be dated and signed by authorized personnel and shall include the name, address, and telephone number of the original product or ingredient manufacturer. 3. "Coefficient of Determination (r2)" is a statistical measure of the strength of relationship between two variables and is the squared value of the correlation coefficient. 4. "Continuing Calibration Verification (CCV) Standard" is a mid-range concentration standard

analyzed before, during, and at the end of an analytical batch and verifies that the instrument response has not drifted from the initial calibration response. This standard solution contains a known concentration of the target analyte and is typically derived from the same source as the initial calibration standards. 5. "Correlation Coefficient (r)" is a statistical measure of the strength of relationship between two variables. 6. "Duplicate Sample" is a quality control sample which is identical to one of the analytical samples and undergoes the same sample preparation and analytical procedures as the analytical sample. 7. "Initial Calibration (ICAL)" is a determination of the instrument response over a range of known concentrations of an analyte or analytes. A series of standard solutions is prepared from a certified reference material and analyzed on the instrument prior to any samples. Five or more standard solutions containing progressively higher concentrations of the analytes of interest are generally prepared. 8. "Initial Calibration Verification (ICV) Standard" is a certified solution from a source other than used for the initial calibration standards and is used to verify the accuracy of the initial calibration. 9. "Instrument Blanks" are typically analyzed before the sample analysis and following high concentration samples. These blanks are used to assess background contamination or carryover in the analytical system that may lead to reporting of false positives in the subsequent sample analyses. 10. "Internal Standard (IS)" is a chemical substance that is similar, but not identical, to the target analyte and is added to each sample at a known concentration. The internal standard mimics the behavior of the target analyte but has a different signal than the analyte. An internal standard is used for quantitation of target analytes and to account for matrix effects and/or variability in instrument response by normalizing the response of the target analytes and surrogates, thereby decreasing measurement bias. 11. "Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)" is a clean matrix which has been spiked with a known concentration of the target analyte. It

is prepared and analyzed in the same analytical batch and in exactly the same manner as the other samples. The laboratory control sample and laboratory control sample duplicate are used to assess general method performance based on the ability of the laboratory to successfully recover target analytes. 12. "Limit of Quantitation (LOQ)" is the lowest measured concentration of the analyte that has gone through extraction or dilution and analysis and meets defined accuracy and precision criteria. 13. "Matrix Spike/Matrix Spike Duplicate (MS/MSD)" are quality control samples that contain known concentrations of target analytes which have been added before extraction and analysis. 14. "Method Blank" is a clean matrix quality control sample that is carried through the entire dilution or extraction and analytical process. Concentrations of the target analyte shall not exceed one half of the lower limit of quantitation. If concentrations exceed this value, it is an indication of contamination of the reagents, glassware, or any part of the extraction and analysis process. 15. "Multiple Reaction Monitoring (MRM)" is a highly sensitive analytical method using a triple quadrupole mass spectrometer. The target analyte is ionized in the ion source which creates specific ions that are characteristic of the target analyte. Selected ions are allowed through the first quadrupole and are then subsequently fragmented into product (quantitation and qualifier) ions in the collision cell. These product ions are selectively passed through the final quadrupole, where they are detected. Multiple product ions can be detected at once. 16. "Percent Recovery" is the proportion of the concentration of a target analyte measured in a sample relative to the known concentration spiked into a sample, conveyed as a percentage. 17. "Relative Percent Difference (RPD)" is the absolute difference between two measurements divided by their average and converted to a percentage. 18. "Relative Standard Deviation (RSD)" is the standard deviation of a group of measurements in a data set divided by their average and converted to a percentage. Relative standard deviation is an indicator of

how a group of measurements in a data set are scattered around the mean. 19. "Response Factor (RF)" is the ratio between a signal produced by an analyte and the concentration of analyte which produced the signal. 20. "Selected Ion Monitoring (SIM)" is a mass spectrometry technique in which a limited set of ions with specific mass-to-charge (m/z) ratios is monitored by the instrument. This technique typically results in increased sensitivity relative to full scan mass spectrometry. 21. "Surrogate" is a compound that is used to monitor extraction and analysis efficiency of the method.

(A)

Sample Preparation Criteria: 1. Each nail product shall be gently mixed or shaken prior to taking an aliquot of the product to ensure the aliquot is representative of the contents in the container. 2. A sample may be introduced into the analytical instrument by various techniques including, but not limited to, purge-and-trap, automated static headspace, and direct injection, provided that all other performance criteria are met.

1.

Each nail product shall be gently mixed or shaken prior to taking an aliquot of the product to ensure the aliquot is representative of the contents in the container.

2.

A sample may be introduced into the analytical instrument by various techniques including, but not limited to, purge-and-trap, automated static headspace, and direct injection, provided that all other performance criteria are met.

(B)

Analytical Method Criteria: 1. It is recommended to use a gas chromatograph/mass spectrometer (GC/MS) method designed for analysis of volatile organic compounds in solid and aqueous samples, such as Method 8260D, as described in U.S. EPA SW-846. Any other analytical technique that meets the method performance criteria in section 69511.6(h)(3)

may be used for sample analysis and determination of toluene concentrations in nail products. 2. Either toluene-d8 shall be used as the internal standard with chlorobenzene-d5 as a surrogate or chlorobenzene-d5 shall be used as the internal standard and toluene-d8 used as a surrogate. The internal standard shall be added to each sample, prior to introduction into the analytical instrument, at a concentration within the range of the initial calibration for toluene. 3. The limit of quantitation for toluene shall be at or below one-third of the AAT of 100 ppm.

1.

It is recommended to use a gas chromatograph/mass spectrometer (GC/MS) method designed for analysis of volatile organic compounds in solid and aqueous samples, such as Method 8260D, as described in U.S. EPA SW-846. Any other analytical technique that meets the method performance criteria in section 69511.6(h)(3) may be used for sample analysis and determination of toluene concentrations in nail products.

2.

Either toluene-d8 shall be used as the internal standard with chlorobenzene-d5 as a surrogate or chlorobenzene-d5 shall be used as the internal standard and toluene-d8 used as a surrogate. The internal standard shall be added to each sample, prior to introduction into the analytical instrument, at a concentration within the range of the initial calibration for toluene.

3.

The limit of quantitation for toluene shall be at or below one-third of the AAT of 100 ppm.

(C)

Instrument and Calibration Criteria: 1. All study samples shall be analyzed on a properly calibrated instrument that meets instrument manufacturer specifications. If the instrument calibrations or other instrument check requirements (for example, mass spectrometer tune, mass calibration check, or qualitative identification criteria) are outside the acceptable criteria, standard measures to correct the problem shall be implemented prior to analyzing

samples. 2. The use of a gas chromatograph/mass spectrometer is recommended for the separation and fragmentation of analytes for identification. The ratios of qualifier ions to quantitation ions shall be established during calibration and shall be maintained throughout sample analysis to verify the identity of toluene and ensure that there are no interfering peaks. a. Laboratories may use mass spectrometer full scan, selected ion monitoring, or multiple reaction monitoring scanning modes to analyze samples and meet the limit of quantitation requirement. b. The method shall incorporate, at a minimum, one quantitation and one qualifier ion for toluene. i. The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91. c. The method shall incorporate, at minimum, one quantitation ion for internal standards and surrogates. Further, the method may additionally include one or more qualifier ions for internal standards and surrogates.i. The quantitation ions for toluene-d8 shall include 98. ii. The quantitation ions for chlorobenzene-d5 shall include 117. 3. The instrument tune checks shall be done prior to calibration and at the beginning of each 12-hour analytical period. For analysis run by full scan GC/MS, it is recommended to use 4-bromofluorobenzene (BFB) as the tune verification standard. For selected ion monitoring or multiple reaction monitoring scanning modes, it is recommended to follow the manufacturer's instrument tuning criteria. All samples, including quality control and calibration standards, shall be introduced into the GC/MS for analysis within 12 hours of the analysis of the tune verification standard. Samples and quality control standard solutions that are not analyzed within the 12-hour time window cannot be reported. 4. For each analyte and surrogate of interest, prepare an initial calibration from standards containing toluene, at a minimum of five different non-zero concentrations, and an appropriate solvent.a. The fitted line of the initial calibration shall meet one of these criteria: i. The relative standard deviation of the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or ii. The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99. 5. The

concentration of toluene in the lowest calibration standard solution shall be accurate within 50 percent of its true concentration value, meaning the percent recovery shall be within 50 to 150 percent of the expected concentration, and all other toluene calibration levels shall be within 30 percent of their true value, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. 6. After the initial calibration, the retention time of each internal standard shall be within 30 seconds of the retention time of the internal standard at the midpoint of the initial calibration. 7. An initial calibration verification standard solution shall be prepared from a different source than the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range and analyzed immediately following the initial calibration. The calculated concentration of toluene and the surrogate in the initial calibration verification standard solution shall be within 30 percent of their true concentration values, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. No samples shall be run until the initial calibration verification standard solution is analyzed. 8. A continuing calibration verification standard solution shall be prepared from the same source as the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range. The continuing calibration verification standard solution shall be analyzed before the samples at the beginning of each 12-hour analytical period. The measured concentration of the continuing calibration verification standard solution shall be within 20 percent of its true concentration value, meaning the percent recovery shall be within 80 to 120 percent of the expected concentration. If the calibration verification does not meet the acceptance criteria, standard measures to correct the problem shall be implemented, and another aliquot of the continuing calibration verification standard solution shall be analyzed. If the response of the continuing calibration verification standard solution is still not within 20 percent of its true concentration value, then a new initial calibration shall be conducted.

1.

All study samples shall be analyzed on a properly calibrated instrument that meets instrument manufacturer specifications. If the instrument calibrations or other instrument check requirements (for example, mass spectrometer tune, mass calibration check, or qualitative identification criteria) are outside the acceptable criteria, standard measures to correct the problem shall be implemented prior to analyzing samples.

2.

The use of a gas chromatograph/mass spectrometer is recommended for the separation and fragmentation of analytes for identification. The ratios of qualifier ions to quantitation ions shall be established during calibration and shall be maintained throughout sample analysis to verify the identity of toluene and ensure that there are no interfering peaks. a. Laboratories may use mass spectrometer full scan, selected ion monitoring, or multiple reaction monitoring scanning modes to analyze samples and meet the limit of quantitation requirement. b. The method shall incorporate, at a minimum, one quantitation and one qualifier ion for toluene. i. The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91. c. The method shall incorporate, at minimum, one quantitation ion for internal standards and surrogates. Further, the method may additionally include one or more qualifier ions for internal standards and surrogates.i. The quantitation ions for toluene-d8 shall include 98. ii. The quantitation ions for chlorobenzene-d5 shall include 117.

a.

Laboratories may use mass spectrometer full scan, selected ion monitoring, or multiple reaction monitoring scanning modes to analyze samples and meet the limit of quantitation requirement.

b.

The method shall incorporate, at a minimum, one quantitation and one qualifier ion for toluene. i. The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91.

i.

The quantitation ions for toluene shall include 92 and the qualifier ions shall include 91.

c.

The method shall incorporate, at minimum, one quantitation ion for internal standards and surrogates. Further, the method may additionally include one or more qualifier ions for internal standards and surrogates.i. The quantitation ions for toluene-d8 shall include 98. ii. The quantitation ions for chlorobenzene-d5 shall include 117.

i.

The quantitation ions for toluene-d8 shall include 98.

ii.

The quantitation ions for chlorobenzene-d5 shall include 117.

3.

The instrument tune checks shall be done prior to calibration and at the beginning of each 12-hour analytical period. For analysis run by full scan GC/MS, it is recommended to use 4-bromofluorobenzene (BFB) as the tune verification standard. For selected ion monitoring or multiple reaction monitoring scanning modes, it is recommended to follow the manufacturer's instrument tuning criteria. All samples, including quality control and calibration standards, shall be introduced into the GC/MS for analysis within 12 hours of the analysis of the tune verification standard. Samples and quality control standard solutions that are not analyzed within the 12-hour time window cannot be reported.

4.

For each analyte and surrogate of interest, prepare an initial calibration from standards containing toluene, at a minimum of five different non-zero concentrations, and an appropriate solvent.a. The fitted line of the initial calibration shall meet one of these criteria: i. The relative standard deviation of the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or ii. The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99.

a.

The fitted line of the initial calibration shall meet one of these criteria: i. The relative standard deviation of

the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or ii. The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99.

i.

The relative standard deviation of the average response of the target analyte, expressed as a percentage, shall not exceed 20 percent; or

ii.

The linear fit shall have a correlation coefficient (r) greater than or equal to 0.995 or a coefficient of determination (r2) greater than 0.99.

5.

The concentration of toluene in the lowest calibration standard solution shall be accurate within 50 percent of its true concentration value, meaning the percent recovery shall be within 50 to 150 percent of the expected concentration, and all other toluene calibration levels shall be within 30 percent of their true value, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration.

6.

After the initial calibration, the retention time of each internal standard shall be within 30 seconds of the retention time of the internal standard at the midpoint of the initial calibration.

7.

An initial calibration verification standard solution shall be prepared from a different source than the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range and analyzed immediately following the initial calibration. The calculated concentration of toluene and the surrogate in the initial calibration verification standard solution shall be within 30 percent of their true concentration values, meaning the percent recovery shall be within 70 to 130 percent of the expected concentration. No samples shall be run until the initial calibration verification standard solution is analyzed.

8.

A continuing calibration verification standard solution shall be prepared from the same source as the initial calibration standard solution, with a toluene concentration at or near the midpoint of the calibration range. The continuing calibration verification standard solution shall be analyzed before the samples at the beginning of each 12-hour analytical period. The measured concentration of the continuing calibration verification standard solution shall be within 20 percent of its true concentration value, meaning the percent recovery shall be within 80 to 120 percent of the expected concentration. If the calibration verification does not meet the acceptance criteria, standard measures to correct the problem shall be implemented, and another aliquot of the continuing calibration verification standard solution shall be analyzed. If the response of the continuing calibration verification standard solution is still not within 20 percent of its true concentration value, then a new initial calibration shall be conducted.

(D)

Sample Analysis Criteria:1. An instrument blank shall be analyzed after the continuing calibration verification standard solution, and before the samples, to demonstrate that the total analytical system is free from contaminants. A sufficient number of instrument blanks shall also be inserted between samples to verify no carryover or cross contamination of toluene from one sample to the next. 2. The response of the internal standard for all samples shall be within 50 to 200 percent of the midpoint of the initial calibration or the continuing calibration verification. 3. The retention time of the analyte of interest shall be within 10 seconds of retention time of the midpoint of the initial calibration or within 10 seconds of the continuing calibration verification standard solution analyzed at the beginning of the 12-hour analytical period.

1.

An instrument blank shall be analyzed after the continuing calibration verification standard solution, and before the samples, to demonstrate that the total analytical system is free from contaminants. A

sufficient number of instrument blanks shall also be inserted between samples to verify no carryover or cross contamination of toluene from one sample to the next.

2.

The response of the internal standard for all samples shall be within 50 to 200 percent of the midpoint of the initial calibration or the continuing calibration verification.

3.

The retention time of the analyte of interest shall be within 10 seconds of retention time of the midpoint of the initial calibration or within 10 seconds of the continuing calibration verification standard solution analyzed at the beginning of the 12-hour analytical period.

(E)

Quality Control Criteria:1. All data shall adhere to a quality control protocol for each batch of 20 samples and each type of nail product analyzed, that includes:a. Preparation and analysis of a method blank. The concentration of toluene in each method blank shall not exceed one half of the lower limit of quantitation; b. Preparation and analysis of a duplicate sample; c. Preparation and analysis of a matrix spike and matrix spike duplicate containing a spiked concentration of toluene in the middle range of the initial calibration; and d. Preparation and analysis of a laboratory control sample and laboratory control sample duplicate, containing a spiked concentration of toluene in the middle range of the initial calibration. 2. Each product sample, method blank, sample duplicate, matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall be spiked with a surrogate standard solution prior to extraction and analysis. 3. Each matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and each spiked quality control sample shall have a spike recovery between 70 to 130 percent of the true value of the spiked concentrations. 4. A laboratory may establish more rigorous internal control limits but shall demonstrate a relative percent

difference less than or equal to 20 percent for duplicate samples and a 70 to 130 percent recovery range for known spiked concentrations. 5. The measured concentration of the surrogate standard solution in each product sample, method blank, sample duplicate, laboratory control sample, laboratory control sample duplicate, matrix spike duplicate undergoing analysis shall be within 70 to 130 percent of the spiked concentration.

1.

All data shall adhere to a quality control protocol for each batch of 20 samples and each type of nail product analyzed, that includes:a. Preparation and analysis of a method blank. The concentration of toluene in each method blank shall not exceed one half of the lower limit of quantitation; b. Preparation and analysis of a duplicate sample; c. Preparation and analysis of a matrix spike and matrix spike duplicate containing a spiked concentration of toluene in the middle range of the initial calibration; and d. Preparation and analysis of a laboratory control sample and laboratory control sample duplicate, containing a spiked concentration of toluene in the middle range of the initial calibration.

a.

Preparation and analysis of a method blank. The concentration of toluene in each method blank shall not exceed one half of the lower limit of quantitation;

b.

Preparation and analysis of a duplicate sample;

c.

Preparation and analysis of a matrix spike and matrix spike duplicate containing a spiked concentration of toluene in the middle range of the initial calibration; and

d.

Preparation and analysis of a laboratory control sample and laboratory control sample duplicate, containing a spiked concentration of toluene in the middle range of the initial calibration.

2.

Each product sample, method blank, sample duplicate, matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall be spiked with a surrogate standard solution prior to extraction and analysis.

3.

Each matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and each spiked quality control sample shall have a spike recovery between 70 to 130 percent of the true value of the spiked concentrations.

4.

A laboratory may establish more rigorous internal control limits but shall demonstrate a relative percent difference less than or equal to 20 percent for duplicate samples and a 70 to 130 percent recovery range for known spiked concentrations.

5.

The measured concentration of the surrogate standard solution in each product sample, method blank, sample duplicate, laboratory control sample, laboratory control sample duplicate, matrix spike duplicate undergoing analysis shall be within 70 to 130 percent of the spiked concentration.

(F)

Definitions: 1. "Aliquot" is a measured portion of a total amount of a larger sample solution or suspension. 2. "Certificate of Analysis (COA)" is a quality assurance document that provides the results of laboratory tests or other analyses performed on a product or ingredient. A COA shall contain information on the name of the product or ingredient, including the batch number, and the release date for each batch tested. The COA shall list each test performed including the quality control acceptance limits, the method quantitative limits, and the numerical results obtained. Certificates shall be dated and signed by authorized personnel and shall include the name, address, and telephone number of the original product or ingredient manufacturer. 3. "Coefficient of Determination (r2)" is a

statistical measure of the strength of relationship between two variables and is the squared value of the correlation coefficient. 4. "Continuing Calibration Verification (CCV) Standard" is a mid-range concentration standard analyzed before, during, and at the end of an analytical batch and verifies that the instrument response has not drifted from the initial calibration response. This standard solution contains a known concentration of the target analyte and is typically derived from the same source as the initial calibration standards. 5. "Correlation Coefficient (r)" is a statistical measure of the strength of relationship between two variables. 6. "Duplicate Sample" is a quality control sample which is identical to one of the analytical samples and undergoes the same sample preparation and analytical procedures as the analytical sample. 7. "Initial Calibration (ICAL)" is a determination of the instrument response over a range of known concentrations of an analyte or analytes. A series of standard solutions is prepared from a certified reference material and analyzed on the instrument prior to any samples. Five or more standard solutions containing progressively higher concentrations of the analytes of interest are generally prepared. 8. "Initial Calibration Verification (ICV) Standard" is a certified solution from a source other than used for the initial calibration standards and is used to verify the accuracy of the initial calibration. 9. "Instrument Blanks" are typically analyzed before the sample analysis and following high concentration samples. These blanks are used to assess background contamination or carryover in the analytical system that may lead to reporting of false positives in the subsequent sample analyses. 10. "Internal Standard (IS)" is a chemical substance that is similar, but not identical, to the target analyte and is added to each sample at a known concentration. The internal standard mimics the behavior of the target analyte but has a different signal than the analyte. An internal standard is used for quantitation of target analytes and to account for matrix effects and/or variability in instrument response by normalizing the response of the target analytes and surrogates, thereby decreasing measurement bias. 11. "Laboratory Control Sample/Laboratory Control Sample Duplicate

(LCS/LCSD)" is a clean matrix which has been spiked with a known concentration of the target analyte. It is prepared and analyzed in the same analytical batch and in exactly the same manner as the other samples. The laboratory control sample and laboratory control sample duplicate are used to assess general method performance based on the ability of the laboratory to successfully recover target analytes. 12. "Limit of Quantitation (LOQ)" is the lowest measured concentration of the analyte that has gone through extraction or dilution and analysis and meets defined accuracy and precision criteria. 13. "Matrix Spike/Matrix Spike Duplicate (MS/MSD)" are quality control samples that contain known concentrations of target analytes which have been added before extraction and analysis. 14. "Method Blank" is a clean matrix quality control sample that is carried through the entire dilution or extraction and analytical process. Concentrations of the target analyte shall not exceed one half of the lower limit of quantitation. If concentrations exceed this value, it is an indication of contamination of the reagents, glassware, or any part of the extraction and analysis process. 15. "Multiple Reaction Monitoring (MRM)" is a highly sensitive analytical method using a triple quadrupole mass spectrometer. The target analyte is ionized in the ion source which creates specific ions that are characteristic of the target analyte. Selected ions are allowed through the first quadrupole and are then subsequently fragmented into product (quantitation and qualifier) ions in the collision cell. These product ions are selectively passed through the final quadrupole, where they are detected. Multiple product ions can be detected at once. 16. "Percent Recovery" is the proportion of the concentration of a target analyte measured in a sample relative to the known concentration spiked into a sample, conveyed as a percentage. 17. "Relative Percent Difference (RPD)" is the absolute difference between two measurements divided by their average and converted to a percentage. 18. "Relative Standard Deviation (RSD)" is the standard deviation of a group of measurements in a data set divided by their average and converted to a percentage. Relative standard deviation is an indicator of how a group of measurements in a data set are scattered around the mean. 19.

"Response Factor (RF)" is the ratio between a signal produced by an analyte and the concentration of analyte which produced the signal. 20. "Selected Ion Monitoring (SIM)" is a mass spectrometry technique in which a limited set of ions with specific mass-to-charge (m/z) ratios is monitored by the instrument. This technique typically results in increased sensitivity relative to full scan mass spectrometry. 21. "Surrogate" is a compound that is used to monitor extraction and analysis efficiency of the method.

1.

"Aliquot" is a measured portion of a total amount of a larger sample solution or suspension.

2.

"Certificate of Analysis (COA)" is a quality assurance document that provides the results of laboratory tests or other analyses performed on a product or ingredient. A COA shall contain information on the name of the product or ingredient, including the batch number, and the release date for each batch tested. The COA shall list each test performed including the quality control acceptance limits, the method quantitative limits, and the numerical results obtained. Certificates shall be dated and signed by authorized personnel and shall include the name, address, and telephone number of the original product or ingredient manufacturer.

3.

"Coefficient of Determination (r2)" is a statistical measure of the strength of relationship between two variables and is the squared value of the correlation coefficient.

4.

"Continuing Calibration Verification (CCV) Standard" is a mid-range concentration standard analyzed before, during, and at the end of an analytical batch and verifies that the instrument response has not drifted from the initial calibration response. This standard solution contains a known concentration of the target analyte and is typically derived from the same source as the initial calibration standards.

5.

"Correlation Coefficient (r)" is a statistical measure of the strength of relationship between two variables.

6.

"Duplicate Sample" is a quality control sample which is identical to one of the analytical samples and undergoes the same sample preparation and analytical procedures as the analytical sample.

7.

"Initial Calibration (ICAL)" is a determination of the instrument response over a range of known concentrations of an analyte or analytes. A series of standard solutions is prepared from a certified reference material and analyzed on the instrument prior to any samples. Five or more standard solutions containing progressively higher concentrations of the analytes of interest are generally prepared.

8.

"Initial Calibration Verification (ICV) Standard" is a certified solution from a source other than used for the initial calibration standards and is used to verify the accuracy of the initial calibration.

9.

"Instrument Blanks" are typically analyzed before the sample analysis and following high concentration samples. These blanks are used to assess background contamination or carryover in the analytical system that may lead to reporting of false positives in the subsequent sample analyses.

10.

"Internal Standard (IS)" is a chemical substance that is similar, but not identical, to the target analyte and is added to each sample at a known concentration. The internal standard mimics the behavior of the target analyte but has a different signal than the analyte. An internal standard is used for quantitation of target analytes and to account for matrix effects and/or variability in instrument response by normalizing the response of the target analytes and surrogates, thereby decreasing measurement bias.

11.

"Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)" is a clean matrix which has been spiked with a known concentration of the target analyte. It is prepared and analyzed in the same analytical batch and in exactly the same manner as the other samples. The laboratory control sample and laboratory control sample duplicate are used to assess general method performance based on the ability of the laboratory to successfully recover target analytes.

12.

"Limit of Quantitation (LOQ)" is the lowest measured concentration of the analyte that has gone through extraction or dilution and analysis and meets defined accuracy and precision criteria.

13.

"Matrix Spike/Matrix Spike Duplicate (MS/MSD)" are quality control samples that contain known concentrations of target analytes which have been added before extraction and analysis.

14.

"Method Blank" is a clean matrix quality control sample that is carried through the entire dilution or extraction and analytical process. Concentrations of the target analyte shall not exceed one half of the lower limit of quantitation. If concentrations exceed this value, it is an indication of contamination of the reagents, glassware, or any part of the extraction and analysis process.

15.

"Multiple Reaction Monitoring (MRM)" is a highly sensitive analytical method using a triple quadrupole mass spectrometer. The target analyte is ionized in the ion source which creates specific ions that are characteristic of the target analyte. Selected ions are allowed through the first quadrupole and are then subsequently fragmented into product (quantitation and qualifier) ions in the collision cell. These product ions are selectively passed through the final quadrupole, where they are detected. Multiple product ions can be detected at once.

16.

"Percent Recovery" is the proportion of the concentration of a target analyte measured in a sample

relative to the known concentration spiked into a sample, conveyed as a percentage.

17.

"Relative Percent Difference (RPD)" is the absolute difference between two measurements divided by their average and converted to a percentage.

18.

"Relative Standard Deviation (RSD)" is the standard deviation of a group of measurements in a data set divided by their average and converted to a percentage. Relative standard deviation is an indicator of how a group of measurements in a data set are scattered around the mean.

19.

"Response Factor (RF)" is the ratio between a signal produced by an analyte and the concentration of analyte which produced the signal.

20.

"Selected Ion Monitoring (SIM)" is a mass spectrometry technique in which a limited set of ions with specific mass-to-charge (m/z) ratios is monitored by the instrument. This technique typically results in increased sensitivity relative to full scan mass spectrometry.

21.

"Surrogate" is a compound that is used to monitor extraction and analysis efficiency of the method.