NCASI METHOD TERPS-19.01
SELECT TERPENES AND TERPENOIDS IN CONDENSATE SAMPLES BY HEXANE EXTRACTION AND GC/MS ANALYSIS

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NCASI METHOD TERPS-19.01

SELECT TERPENES AND TERPENOIDS TERPENES IN CONDENSATE SAMPLES BY HEXANE EXTRACTION AND GC/MS ANALYSIS

1.0 Scope and Application

1.1 This method is designed to determine concentrations of select terpenes and terpenoids in kraft mill process condensate samples. It involves liquid/liquid extraction of the analytes using hexane and quantification by gas chromatography/mass spectrometry (GC/MS). Quantitation is accomplished using internal standard calibration with n-tridecane.

1.2 These compounds (CAS Registry Numbers are included) can be confirmed or quantitated by GC/MS using NCASI TERPS-19.01:

(-)-alpha-Bisabolol	23089-26-1	(-)-Isopulegol	89-79-2
Borneol	464-45-9	d-Limonene	5989-27-5
Borneol acetate	76-49-3	Linalool	78-70-6
Camphene	79-92-5	beta-Myrcene	123-35-3
delta-3-Carene	13466-78-9	Nerolidol 1 (cis-)	3790-78-1
beta-Caryophyllene	87-44-5	Nerolidol 2 (trans-)	40716-66-3
p-Cymene	99-87-6	ztrans-beta-Ocimene	3338-55-4
Estragole (4-allyl anisole)	140-67-0	cis-beta-Ocimene	13877-91-3
exo-Fenchol	22627-95-8	alpha-Phellandrene	4221-98-1
Geraniol	106-24-1	alpha-Pinene	80-56-8
(-)-Guaiol	489-86-1	(-)-beta-Pinene	18172-67-3
alpha-Humulene	6753-98-6	alpha-Terpinene	99-86-5
Isoborneol	124-76-5	gamma-Terpinene	99-85-4
Isoborneol acetate	125-12-2	alpha-Terpineol	98-55-5
p-Isopropyltoluene (*p*-cymene)	99-87-6	Terpinolene	586-62-9

1.3 This method has been validated at the single laboratory level in kraft mill combined foul condensate, “clean” condensate, and stripped condensate samples. Demonstration of extraction efficiency and method performance for specific matrix types is recommended. This method has not been validated for turpentine product samples.

1.4 The estimated method detection limits (MDLs) were determined as specified in 40 CFR 136 Appendix B (1) using a condensate sample. Calculated MDLs are shown in Section 17, Table 1, and are provided as guidance. Due to improvements in instrumentation and changes in matrix effects, each laboratory should establish its own MDLs. The lower instrument calibration limit (LCL) for the target analytes is 2 mg/L. The concentration range is from 2 to 50 mg/L. Higher concentration ranges can be achieved using sample dilutions.

1.5 GC/MS portions of this method are for use only by analysts experienced with capillary GC/MS or under the close supervision of such qualified persons. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of Method

A 5-mL aliquot of pH 2 preserved condensate is extracted with 5 mL of hexane. A 1.0-mL aliquot of the top hexane layer is transferred to a 2-mL autosampler vial. Tridecane is added as the internal standard and the extract is analyzed by GC/MS.

2.1 Quantitative Analysis

Terpenes are introduced into the GC with a narrow bore fused silica capillary column. The GC column is temperature programmed to separate the target analytes, which are then detected with an MS interfaced to the GC. Identification of target analytes is accomplished by comparing their mass spectra to that of an authentic standard. A compound is identified when its relative retention time and mass spectrum meet the criteria described in Section 12.

2.2 Quality Assurance

Quality is assured through reproducible calibration and testing of the extraction and GC/MS system. A method blank is analyzed with each sample set (samples started through the extraction process on a given day, to a maximum of 20), along with a matrix spike and matrix spike duplicate to ensure quality data. A complete description of quality control procedures, calculations, and method performance criteria are listed in Section 9.

3.0 Definitions

3.1 These definitions are specific to this method but conform to common usage as much as possible.

3.1.1 mg/L – milligrams per liter

3.1.2 µg – micrograms

3.1.3 May – this action, activity, or procedural step is neither required nor prohibited

3.1.4 May not – this action, activity, or procedural step is prohibited

3.1.5 Must – this action, activity, or procedural step is required

3.1.6 Should – this action, activity, or procedural step is suggested but not required

3.1.7 GC/MS – gas chromatograph with mass spectrometer

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute analytical interferences resulting in misinterpretation of chromatograms. Run method blanks initially and with each subsequent sample set to demonstrate that they are free from interferences under the conditions of the method. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

4.2 Interferents co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

4.3 Contamination by carryover can occur when samples containing high concentrations of the target analytes are analyzed in sequence with low concentration samples. Whenever unusually concentrated samples are encountered, they should be followed by injections of a solvent blank to check for cross contamination prior to analysis of additional samples.

5.0 Safety

5.1 The toxicity or carcinogenicity of the compounds or reagents used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to a level protective of human health. This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses.

5.2 Hexane may be harmful if inhaled or absorbed through the skin and is a suspected carcinogen. Use it in a laboratory fume hood or wear an approved respirator and avoid contact by wearing chemical-resistant gloves, eye protection, and other protective clothing.

5.3 As with all samples, precautions should be taken to avoid exposure to potentially toxic, caustic, or nuisance odor compounds. Samples should be handled with gloves and opened in a fume hood.

6.0 Equipment and Supplies

Note: *Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

6.1 Do not use glassware with any star fractures, cracks, or severe scratches. All fittings should be snug, and clamps and springs should be in good working order. All glassware should be washed with detergent, rinsed with tap water, then rinsed with reagent-grade

water. If blank contamination is observed, the glassware may be solvent rinsed or baked prior to use.

6.2 Sampling Equipment

6.2.1 Use of glass containers, Teflon™ tubing, or stainless steel is recommended for sample collection. Use amber glass bottles equipped with Teflon-lined screw caps to store all samples.

6.2.2 Automatic sampling equipment that comes in contact with a sample should be constructed of glass, Teflon, or stainless steel.

6.3 Equipment for Sample Extraction (per sample)

6.3.1 One 15-mL glass centrifuge tube (Cole Parmer #UX-34534-10 or equivalent)

6.3.2 One 5¾-inch disposable Pasteur pipette

6.3.3 One 2-mL glass autosampler vial with Teflon-lined cap

6.4 Other Apparatus

6.4.1 Vortex

6.4.2 Rotary shaker table

6.4.3 Gas chromatograph – must be equipped with a mass spectrometer and a splitless injection port for capillary column and have the capacity to run the temperature program and performance specifications outlined in Sections 9.2 and 10.1

6.4.4 Gas chromatographic column – 30 m x 0.25 mm i.d., 0.25 µm film thickness, 5% phenyl, 95% methyl (DB-5 or equivalent)

6.4.5 Mass spectrometer – 70 eV electron impact ionization; must repetitively scan from 42 to 360 AMU in 4.4 scans/second and must produce a unit resolution (valley between m/z 441-442 less than 10% of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet; spectrum must meet the mass intensity criteria listed in Section 9.2 and Section 17, Table 2; mass spectrometer must be interfaced to the GC via a directly coupled column with a heated transfer line in accordance with the manufacturer's specifications; all portions of the column that connect the GC to the ion source must remain at or above oven temperature during analysis to preclude condensation of less volatile compounds

6.4.6 The GC data system should collect and record GC data, process and store GC/MS data, generate reports, and compute and record response factors.

7.0 Reagents and Standards

7.1 Solvents

7.1.1 Hexane supplied by Burdick & Jackson or equivalent high purity solvent suitable for gas chromatography and pesticide residue analysis; acetone is distilled prior to use for preparation of standards

7.2 Standards

7.2.1 Restek (State College, PA) supplies a Certified Reference Material standard containing 19 terpenes at 2500 µg/mL each in isopropanol (part number 34095); it contains these terpenes: (-)-alpha-Bisabolol, Camphene, delta-3-Carene, beta-Caryophyllene, (-)-Guaiol, alpha-Humulene, (-)-Isopulegol, d-Limonene, Linalool, beta-Myrcene, Nerolidol, Ocimene, alpha-Pinene, (-)-beta-Pinene, alpha-Terpinene, gamma-Terpinene, Terpinolene

7.2.2 alpha-Phellandrene, Estragole, and d26-Dodecane (surrogate) can be purchased from Sigma Aldrich; borneol acetate (containing 20% isoborneol acetate) can be purchased from TCI; Borneol and Isoborneol can be purchased from SPEX Certiprep; and alpha-Terpineol can be purchased from Restek. An equivalent supplier may replace any of the listed suppliers. If standards have a chemical purity <96%, correct all calculations, calibrations, and matrix spikes for the difference in purity.

7.2.3 Prepare working terpene standards of the target analytes (Section 1.2) in hexane at a concentration of 200 ±0.1 mg/L. Place solutions into glass vials with Teflon-lined caps.

7.2.4 Prepare the surrogate working standard (d26-dodecane) at a concentration of 200 ±0.1 mg/L. Place solutions into glass vials with Teflon-lined caps.

7.2.5 N-Tridecane, 99+% pure, is available from Aldrich or an equivalent supplier. Prepare an internal standard working stock solution of 1 mg/mL in hexane.

7.2.6 Prepare a five-point calibration curve encompassing the sample concentration range of approximately 2.0 to 50 mg/L:

- 2.0 mg/L calibration level – spike 10 µL of the 200 mg/L working terpenes standard and 10 µL of the 200 mg/L working surrogate standard into 980 µL of hexane in a 2-mL autosampler vial.
- 5.0 mg/L calibration level – spike 25 µL of the 200 mg/L working terpenes standard and 25 µL of the 200 mg/L working surrogate standard into 950 µL of hexane in a 2-mL autosampler vial.
- 10.0 mg/L calibration level – spike 50 µL of the 200 mg/L working terpenes standard and 50 µL of the 200 mg/L working surrogate standard into 900 µL of hexane in a 2-mL autosampler vial.

- 25.0 mg/L calibration level – spike 125 µL of the 200 mg/L working terpenes standard and 125 µL of the 200 mg/L working surrogate standard into 750 µL of hexane in a 2-mL autosampler vial.
- 50.0 mg/L calibration level – spike 250 µL of the 200 mg/L working terpenes standard and 250 µL of the 200 mg/L working surrogate standard into 500 µL of hexane in a 2-mL autosampler vial.

Add 20 µL of the 1000 µg/mL n-tridecane internal standard to all blank and calibration standards.

7.2.7 Decafluorotriphenylphosphine (DFTPP) for GC/MS confirmation analyses can be purchased from Supelco or an equivalent supplier as a 25,000 µg/mL solution in dichloromethane (DCM). Prepare a working stock solution in hexane at a concentration of 50 µg/mL. Store in the dark in autosampler vials with Teflon-seal crimp caps until used.

7.2.8 Stock solutions of all standards must be stored under refrigeration (4°C) and should be checked for signs of concentration or formation of precipitates prior to preparation of calibration or performance test standards. Replace stock solutions if a change in concentration is indicated by the inability to meet criteria specified in Sections 9.2 and 10.3.

8.0 Sample Collection, Preservation, and Storage

8.1 Sample Collection

Collect grab samples in glass containers with Teflon-lined screw caps. Composite samples may be collected using automatic sampling equipment. Parts of automatic sampling equipment that come in contact with the sample should be constructed of glass, Teflon, or stainless steel. Composite samples should be refrigerated during the sampling period.

8.2 Sample Preservation

Preserve all samples in the field by acidification at pH 2 to pH 3 using 1:4 sulfuric acid, then refrigerate. This should be done as soon as possible after sample collection. Ship samples in iced containers as quickly as possible.

8.3 Sample and Extract Storage

Results of storage stability studies indicate that samples and sample extracts may be stored for up to 30 days in the refrigerator (4°C) prior to analysis.

9.0 Quality Control

9.1 Each laboratory that uses this method should operate a formal Quality Assurance Program. Minimum requirements consist of an initial demonstration of laboratory capability and ongoing analyses of standards and blanks as a test of continued

performance. Laboratory performance is compared to established performance criteria to determine if results of analyses meet the performance characteristics of the method.

9.2 GC/MS Performance and Calibration Verification

9.2.1 Verify GC/MS instrument performance by conducting a DFTPP tune prior to analyzing any samples, blanks, or standards. Analyze the tune check just prior to calibration standard analyses and confirm that it meets specifications listed in Section 17, Table 2.

9.2.2 Determine that the GC/MS system is operating within acceptable parameters by conducting a calibration check before each set of samples (samples started through the extraction process on a given day, to a maximum of 20) is analyzed. The calibration check involves reanalyzing one of the extracts used in the calibration curve (Sections 7.2.6 and 10.2). Response factors determined for the calibration check should not deviate by more than ±15% from the average response factors determined for the calibration curve. The analytes may be sensitive to GC/MS instrument conditions such as contamination of the injection port, detector, and/or column. If the calibration check fails to meet the ±15% acceptance criterion, appropriate GC/MS maintenance is necessary. Reanalyze the calibration verification upon completion of all necessary maintenance. If all recommended instrument maintenance fails to correct calibration verification difficulties, the calibration curve should be reprepared and reanalyzed.

9.3 Frequency

One sample per analytical batch of no more than 20 samples of similar matrix type should be allocated for quality control. A representative sample from each new or untested source or sample matrix should be treated as a quality control sample. Laboratory replicates and fortifications (matrix spikes) should be conducted on each quality control sample to document method performance as indicated by precision and recovery.

9.4 Blanks

9.4.1 Demonstrate that the analytical system is free of contamination by preparing and analyzing a blank with each sample set. Prepare a method blank using the procedure outlined in Section 11 utilizing reagent grade water as the sample.

9.4.2 If any of the compounds of interest (Section 17, Table 1) or any potentially interfering compounds are found in the blank at greater than 10% of the MDL or the LCL (assuming a response factor of 1 relative to the internal standard n-tridecane for compounds not listed in Section 17, Table 1), halt analysis of samples until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

9.5 Surrogate and Matrix Spikes

9.5.1 Spike all samples with the surrogate compound to monitor surrogate recovery. Compute recovery of the surrogate compound as the ratio of concentration found to concentration spiked, using Equation 1.

Equation 1

$$Percent\ recovery = \frac{Concentration\ found\ x\ 100}{Concentration\ spiked}$$

Performance criterion for acceptable surrogate recovery of ±20% was determined during a single laboratory validation of this method (n=25), as presented in Section 17, Table 3. If recovery is outside the acceptable range, action should be taken to resolve the problem and samples should be reextracted and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate and Duplicate Sample Precision

Spike 5% of samples with the matrix spike compound in duplicate to monitor method performance. Compute recovery of the surrogate compound as the ratio of concentration found to concentration spiked, using Equation 1. Calculate the relative percent difference in concentrations for each sample and duplicate pair (matrix spike and spike duplicate pair) using Equation 2.

Equation 2

$$Relative\ Percent\ Difference = \frac{(Highest\ concentration - Lowest\ concentration)\ x\ 100}{Average\ concentration\ of\ the\ sample\ and\ duplicate}$$

A summary of the precision determined in a single laboratory is provided in Section 17, Table 4 for ten condensate samples.

9.7 Field Replicates and Field Spikes

Depending on specific program requirements, field replicates and field spikes of the analytes of interest into samples may be required to assess precision and accuracy of sampling and sample transporting techniques.

10.0 Calibration and Standardization

10.1 Assemble the GC/MS and establish the operating conditions outlined herein. Optimize GC conditions as specified by the criteria outlined in Section 9.2. Once operating conditions are optimized, use the same operating conditions to analyze all samples, blanks, calibration curves, calibration verification samples, and matrix spikes.

GC/MS Operating Conditions for NCASI Method TERP-19.01

Injector Temperature:	270°C
Splitless Valve Time:	0.8 min
Carrier Gas:	Helium @ 30-35 cm/sec and 130°C
Injection Volume:	1 µL
Temperature Program	
Initial:	35°C for 2 min
Ramp:	35°C to 150°C @ 5°C/min
Ramp 2:	150°C to 290°C @ 15°C/min
Post Run:	290°C for 0.1 min
Oven Equilibration:	0.50 min
Run Time:	34.3 min
Interface Temperature:	290°C
MS Conditions	
Scan Start Time:	5.00 min
Scan Range:	42 to 360 AMU
Scans/Sec:	4.4

10.2 Internal Standard Quantitation

10.2.1 Analyze the calibration standards (Section 7.2.6) using the procedure described in Section 11.5. Compute relative response factors (RRFs) using Equation 3.

Equation 3

$$RRF = [(A_S / A_{IS}) \times (C_{IS} / C_S)]$$

where:
A_S = *area of the target compound in the calibration standard*
A_{IS} = *area of the internal standard in the calibration standard*
C_{IS} = *concentration of the internal standard in the calibration standard*
C_S = *concentration of the target compound in the calibration standard*

10.2.2 If the average of RRFs calculated across the calibration range is constant (i.e., less than 20%) the calibration is acceptable and the average RRF can be used in all target analyte quantifications. Otherwise, evaluate the problem, undertake appropriate remedial action, and reanalyze the calibration curve extracts. If remedial actions and reanalysis fail to produce a constant RRF, prepare new calibration curve extracts and analyze. Statistics for response factors determined during a single laboratory validation of this method are included in Section 17, Table 1.

10.3 Verify calibration prior to analysis of each set of samples (Section 9.2). Analyze one of the calibration standards (Section 7.2.6) prior to analysis of each set of samples. It is recommended that the selected calibration standard vary over time in order to verify calibration over the range of the method. Recalibrate if RRFs for the target compounds in the analyzed calibration verification point differ by ±15% from the RRF determined for that calibration point in the current calibration curve. Calculate the percent difference between the calibration curve and calibration verification RRFs using Equation 4.

Equation 4

$$Percent\ Difference = [(RRF_{AVG} - RRF_V)/RRF_{AVG}] * 100$$

where:
RRF_{AVG} = *the average relative response factor from the initial calibration curve*
RRF_V = *the relative response factor from the calibration verification*

10.4 Process a blank with the curve to confirm that glassware, reagents, and other components are free from contamination. Prepare the blank using the same procedure used to prepare the calibration standards, omitting addition of the target analytes and surrogates (Section 7.2.6).

10.5 Demonstrate that the target analytes are detectable at the minimum level using the lowest level calibration curve standard.

11.0 Procedure

11.1 This section includes the procedure used to extract condensate samples. The extraction procedure is used for all types of samples and method blanks.

11.2 Remove the sample, surrogate working stock (Section 7.2.4), internal standard working stock (Section 7.2.5), and appropriate working terpene stock solution (Section 7.2.3) from the refrigerator and bring to room temperature.

11.3 Extraction of Samples

11.3.1 Shake the condensate sample to ensure homogeneity and immediately measure a 5-mL portion into a 15-mL glass centrifuge tube. For method blanks, measure 5 mL of reagent grade water.

11.3.2 Pour a 5-mL portion of hexane into the 15-mL glass centrifuge tube containing the sample. Cap the tube and vortex for 30 seconds. Place the tube on a shaker table for a minimum of 60 minutes.

Note: *Methylene chloride may be used as the extraction solvent instead of hexane to extend this method to more polar analytes (e.g., furfural). This solvent has a greater tendency to form emulsions when extracting condensate samples*

and a drying step (using anhydrous sodium sulfate) after extraction will be required. Surrogate recoveries will have to be closely monitored to ensure adequate method performance.

11.3.3 Allow the phases to separate for a minimum of 10 minutes, then transfer 1 mL of the top hexane layer to a 2-mL autosampler vial.

11.3.4 Add 20 µL of the n-tridecane internal standard (Section 7.2.5) to each sample, mix thoroughly, and cap the vial with a Teflon-lined cap. If extracts are not analyzed immediately, store at 4°C. Always allow the extract to come to room temperature prior to GC/MS analysis.

11.4 GC/MS Analysis

11.4.1 GC/MS conditions should be set according to the criteria described in Section 10.1.

11.4.2 Bring the DFTPP tune solution to room temperature. Perform the DFTPP tune as outlined in Section 9.2.1.

11.4.3 Bring the daily calibration solution to room temperature. Perform the daily calibration verification as outlined in Section 9.2.2.

11.4.4 Bring the sample extract or standard to room temperature and verify that any precipitate has redissolved. Inject a 1-µL volume of the standard solution or extract, using a splitless injection.

12.0 Data Analysis and Calculations

12.1 GC/MS Data Analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound that has been previously stored in a mass spectral library. Refer to Section 17, Table 2 for a list of characteristic ions. Identification of a compound is confirmed when the criteria here are met.

12.1.1 Verify that the selected ions specified in Section 17, Table 2 are present and maximize within the same two consecutive scans.

12.1.2 The relative percent abundance of the ions designated in Section 17, Table 2 must agree within ±20% of those observed for the mid-point calibration curve standard during the most current calibration curve analysis.

12.1.3 The m/z present in the mass spectrum from components in the samples that are not present in the reference spectrum should be accounted for by contamination or background ions. If the experimental mass spectrum is contaminated or if identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the compound.

12.2 Internal Standard Quantitation

12.2.1 The n-tridecane internal standard is used to quantitate the corresponding terpenes. Calculate concentrations of the target compound in the sample according to Equation 5.

Equation 5

$$Concentration\ of\ target\ (\mu g/L) = [(A_S \times C_{IS})/(A_{IS} \times RRF_{AVE})]$$

where:
A_S = *area of the compound being measured*
C_{IS} = *concentration (µg/L) of the tridecane internal standard in the sample*
A_{IS} = *area of the internal standard*
RRF_{AVE} = *averaged relative response from the initial calibration curve*

12.3 Data Review Requirements

12.3.1 Review data for accuracy of identification, GC problems, interferences, and bias. Correct any problems prior to reporting analytical results.

12.3.2 Manually review the chromatograms to confirm internal standard and analyte identification and area integrations. As part of this review, assess the need for sample/extract dilutions. The procedure for conducting extract dilution and reanalysis is described in Section 12.4.

12.3.3 Visually inspect the total ion chromatogram for obvious problems that might result in poor internal standard recoveries or false negatives/false positives. The presence of non-target species can become apparent from this review.

12.3.4 Resolve any inconsistencies between duplicate analyses (i.e., if a compound shows up in one replicate but not the other) and attempt to determine the reason.

12.3.5 Generate a GC/MS report that includes the retention time of the compound, area of the compound, width of the peak, and calculated concentration of the target compound detected. If review of the data shows any problems that could affect subsequent analyses, discontinue analyses until the problems are resolved.

12.4 Results Outside the Calibration Range

If the calculated concentration of any of the target analytes exceeds the concentration of the highest calibration point, dilute an aliquot of the extract with hexane to bring the concentration within the calibration range of the method and reanalyze.

13.0 Method Performance

Single laboratory performance for this method is detailed in Section 17, Tables 3 and 4. Acceptance criteria were established from single laboratory use of the draft method.

14.0 Pollution Prevention

Pollution prevention approaches have not been evaluated for this method. It is recommended that the laboratory check state and local requirements to determine if pollution prevention equipment, such as solvent recovery devices, are required or recommended in its area. Use of these devices to reclaim solvents can be part of a pollution prevention program to reduce air emissions.

15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.2 Instructions for Sample and Waste Handling and Disposal

15.2.1 Dispose of all waste solvents as required by federal, state, and local regulations.

15.2.2 Neutralize the aqueous portion of extracted samples to pH 7 and pour down the drain with copious amounts of water.

15.3 For further information on waste management, the Environmental Protection Agency suggests you consult *The Waste Management Manual for Laboratory Personnel* and *Less is Better: Laboratory Chemical Management for Waste Reduction*. Both are available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC, 20036.

16.0 References

Federal Register. 2016. Appendix B to Part 136 – Definition and procedure for the determination of the method detection limit, revision 2.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Table 1. Retention Times, Primary Ion, Secondary Ions, Relative Response Factors (RRF), and Method Detection Limits (MDL) for Condensates[a]

Compound	RT (min)	Primary Ion	Secondary Ions	RRF	MDL (mg/L)
Tridecane (IS)	20.112	57	43, 71	1.000	---
d26-Dodecane (SURR)	16.791	66	50, 82	0.710	---
alpha-Pinene	9.057	93	121, 136	0.680	0.12
Camphene	9.563	93	121, 136	0.465	0.15
beta-Pinene	10.437	93	121, 136	0.662	0.12
beta-Myrcene	10.860	93	69,41	0.559	0.12
alpha-Phellandrene	11.355	93	77, 136	0.638	0.10
3-Carene	11.420	93	121, 136	0.617	0.23
alpha-Terpinene	11.692	121	93, 136	0.530	0.12
p-Cymene	11.938	119	134, 91	1.495	0.11
D-Limonene	12.079	68	93, 107	0.463	0.20
trans-beta-Ocimene	12.316	93	79, 121	0.152	0.16
cis-beta-Ocimene	12.645	93	79, 121	0.318	0.10
gamma-Terpinene	12.994	93	121, 136	0.640	0.13
alpha-Terpinolene	13.829	93	121, 136	0.386	0.15
Linalool	14.300	71	55, 93	0.314	0.22
Fenchol, exo-	14.871	81	95, 121	0.879	0.10
Isopulegol	15.692	55	67, 81	0.446	0.33
Isoborneol	15.745	95	110, 121	0.118	0.18
Borneol	16.452	95	110, 121	0.760	0.15
alpha-Terpineol	17.136	59	93, 121	0.507	0.19
Estragole (4-Methyl An...	17.221	148	146, 121	0.593	0.12
Geraniol	18.716	69	91, 123	0.132	0.17
Borneol Acetate	19.654	95	121, 136	0.673	0.15
Isobornyl Acetate	19.718	95	121, 136	0.157	0.37
beta-Caryophyllene	23.257	93	123, 161	0.210	0.11
alpha-Humulene	24.165	93	121, 147	0.651	0.11
Nerolidol 1	25.869	69	93, 107	0.136	0.39
Nerolidol 2	26.414	69	93, 107	0.192	0.20
Guaiol	26.989	161	59, 189	0.426	0.18
alpha-Bisabolol	28.113	43	109, 119	0.268	0.24

[a] MDLs determined using 40 CFR 136, Appendix B, Revision 2; *Federal Register* 2016

Table 2. DFTPP Criteria for TERPS-19.01

m/z	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
69	10-100% of mass 198
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-60% of mass 198
365	1-100% of mass 198
441	10-100% of mass 443
442	50-100% of mass 198
443	15-24% of mass 442

Table 3. Surrogate Recovery in Condensates during NCASI TERP-19.01 Method Validation Studies (n=25)

	Average Percent Recovery (%)	Standard Deviation	RPD
d26-Dodecane (SURR)	100.2	6.5	6.5

Table 4. Matrix Spike Recoveries in Condensates during NCASI TERP-19.01 Method Validation Studies (n=10)

	Percent Recovery (%)	Standard Deviation	RPD
alpha-Pinene	98.3	8.3	8.4
Camphene	103.0	5.0	4.9
beta-Pinene	99.4	6.0	6.0
beta-Myrcene	92.6	8.2	8.9
alpha-Phellandrene	95.2	8.0	8.4
3-Carene	96.8	7.1	7.3
alpha-Terpinene	98.1	3.7	3.8
p-Cymene	95.9	3.9	4.1
D-Limonene	97.4	8.2	8.5
trans-beta-Ocimene	92.0	7.3	8.0
cis-beta-Ocimene	96.6	7.7	7.9
gamma-Terpinene	98.2	5.8	5.9
alpha-Terpinolene	98.4	7.1	7.2
Linalool	99.3	3.9	3.9
Fenchol, exo-	105.2	2.6	2.5
Camphor	95.5	3.4	3.5
Isopulegol	100.4	9.8	9.8
Geraniol	99.3	11.6	11.7
alpha-Terpineol	94.7	15.4	16.3
Estragole (4-Allyl Anisole)	105.4	11.0	10.5
Borneol Acetate	98.3	4.9	5.0
Isobornyl Acetate	95.7	4.2	4.4
beta-Caryophyllene	97.4	6.1	6.3
alpha-Humulene	97.5	6.6	6.8
Nerolidol 1	95.0	3.4	3.6
Nerolidol 2	95.9	4.7	4.9
Guaiol	98.3	4.8	4.9
alpha-Bisabolol	98.7	5.6	5.7