NCASI METHOD CI/SG/PULP-94.02

CHILLED IMPINGER/SILICA GEL TUBE TEST METHOD AT PULP MILL SOURCES FOR METHANOL, ACETONE, ACETALDEHYDE, METHYL ETHYL KETONE AND FORMALDEHYDE

NCASI
Southern Regional Center
August 1998

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This method was prepared by Dr. MaryAnn Gunshefski, Senior Research Scientist, and Ward Dickens, Research Associate, at the NCASI Southern Regional Center. Other assistance was provided by Terry Bousquet, Senior Research Scientist, with the NCASI West Coast Regional Center.

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CHILLED IMPINGER/SILICA GEL TUBE TEST METHOD AT PULP MILL SOURCES FOR METHANOL, ACETONE, ACETALDEHYDE, METHYL ETHYL KETONE AND FORMALDEHYDE

1.0 Introduction

1.1 This method is intended for the sampling of methanol (CAS # 67-56-1), acetone (CAS # 67-64-1), acetaldehyde (CAS # 75-07-0), methyl ethyl ketone (CAS # 78-93-3) and formaldehyde (CAS # 50-00-0) concentrations in stationary source emissions from pulp and paper mills by using midget impingers and silica gel sorbent tubes. The analysis for methanol, acetone, acetaldehyde and methyl ethyl ketone is performed by gas chromatography/flame ionization detection (GC/FID), and the analysis of formaldehyde is performed by use of the acetylacetone colorimetric procedure. This method was published in Appendix B of NCASI Technical Bulletin 684 as "NCASI Chilled Impinger Train Method for Methanol, Acetone, Acetaldehyde, Methyl Ethyl Ketone and Formaldehyde," and has been rewritten to conform with the contents and format of EPA Air Methods.

2.0 Method Description

2.1 Principle, applicability, interferences and stability

2.1.1 Principle - This method involves collection of an air sample by drawing it through a midget impinger which is filled with water, and then through two 2-section silica gel sorbent tubes. The impinger is kept in an ice water bath during sampling to enhance collection efficiency. The impinger catch is analyzed for methanol, acetone, acetaldehyde and methyl ethyl ketone by direct injection into a gas chromatograph equipped with a flame ionization detector (GC/FID). The silica gel sorbent is desorbed with a 3% (v/v) solution of n-propanol. The desorbate is injected directly into the GC/FID for analysis of methanol, acetone, acetaldehyde and methyl ethyl ketone. To analyze for formaldehyde, the acetylacetone derivatization/spectrophotometric analysis method is used on an aliquot of the impinger solution.

EPA Methods 1-4, or equivalent methods, must be performed in order to obtain mass emissions rates. These methods are not described in this document.

2.1.2 Applicability - The method has been single laboratory validated using the United States Environmental Protection Agency (EPA) Method 301, *Field Validation of Emission Concentrations from Stationary Sources* (Appendix A

to CFR 63). This method was found to be applicable for the measurement of methanol and acetone in pulp mill emissions from recovery furnaces, bleach plant scrubbers, smelt dissolving tank vents, and brownstock washer vents; acetaldehyde in pulp mill emissions from recovery furnaces, smelt dissolving tank vents, and brownstock washer vents; methyl ethyl ketone in pulp mill emissions from recovery furnaces, bleach plant scrubbers, and smelt dissolving tank vents; and the measurement of formaldehyde in pulp mill emissions from bleach plant scrubbers, smelt dissolving tank vents, and brownstock washer vents. From the accuracy section of the Method 301 validation studies correction factors were determined and are given in Section 2.9, Table 1. From the precision section of the Method 301 validation studies it was determined that three samples must be taken at each location to obtain a representative stack concentration.

2.1.3 Interferences - Interferences with the formaldehyde analysis can be caused by the presence of sulfur compounds (i.e. SO_2) in the source gas. This is the reason that this method is not valid for the analysis of formaldehyde in recovery furnace source gas. Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. Clean all glassware by detergent washing with hot water and rinsing with tap water. The glassware should then be drained dry and baked at greater than 100^{o}C for over 2 hours.

2.1.4 Stability - The stability of acetaldehyde in the impinger catch was found to be 10 days, with refrigeration at approximately 4^{o}C. The stability of acetone, methyl ethyl ketone, methanol and formaldehyde was found to be 21 days, with refrigeration at approximately 4^{o}C. The stability of acetaldehyde, acetone, methyl ethyl ketone, and methanol on the silica gel sorbent tubes was found to be approximately 10 days, with refrigeration at approximately 4^{o}C. Once desorbed in 3% n-propanol, these same compounds are stable for up to 21 days, with refrigeration at approximately 4^{o}C.

2.2 Apparatus

2.2.1 Sampling apparatus - A diagram of the sampling train is shown in Figure 1.

2.2.1.1 Probe/sampling line - The probe is made from Teflon tubing or stainless steel, which is then attached to the first impinger.

2.2.1.2 Impinger train - Two 30 mL capacity midget impingers are connected in series to the sampling probe. The impingers should have regular tapered stems. All impinger train connectors should be glass and/or Teflon.

2.2.1.3 Sorbent tubes - Two 2-section silica gel sorbent tubes (SKC #226-15 GWS) are placed in line after the impingers.

2.2.1.4 Rotameter - A 1000 mL/min capacity rotameter should be placed in line after the silica gel sorbent tubes for a visual flow check during sampling and leak checking. The rotameter is not used to determine the actual flow rate through the impingers.

2.2.1.5 Critical orifice - A 400 ± 50 mL/min critical orifice should be used for flow control.

2.2.1.6 Vacuum pump - The critical orifice is followed by a pump capable of providing a vacuum of about 18 inches of Hg. (Pump capacity should be sufficient to obtain and maintain critical conditions at the orifice.)

2.2.1.7 Pressure gauges - One pressure gauge is placed before the critical orifice, and one pressure gauge is placed before the pump, and both are used when leak checking the sample train. The pressure gauge downstream of the critical orifice provides a check for critical flow conditions at the orifice.

2.2.1.8 On/off valve - An on/off valve is placed between the critical orifice and the second pressure gauge, and is used when leak checking the sample train.

2.2.1.9 Flowmeter - A bubble tube flowmeter is used to measure flow at the sampling line tip prior to and after sampling. Alternatively, a dry gas meter may be used.

2.2.1.10 Thermometer - An accurate thermometer is used to measure ambient temperature.

2.2.1.11 Barometer - A barometer is used to measure barometric pressure.

2.2.1.12 Sample storage bottles - Glass (i.e., 40 mL VOA vials) or polyethylene bottles can be used to store the impinger catch sample after stack sampling is complete.

2.2.2 GC/FID analysis apparatus

2.2.2.1 Laboratory glassware - Volumetric pipets, volumetric flasks, autosampler vials, syringes, and cuvettes necessary for standards preparation and analysis.

2.2.2.2 Gas chromatography system - Gas chromatography/flame ionization detector system. Gas chromatography analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required

accessories including syringes, analytical columns and gases. Note that we suspect systems with EPC are not designed to handle aqueous injections, and as a result the FID flame may begin to go out during the runs. This could be due to the water which builds up in the GC system after several injections on any type of GC. Bakeouts are necessary for any type of GC system, but more frequent bakeouts of a system with EPC may need to be performed.

2.2.2.3 Column - 30 m x 0.53 mm x 1 µm bonded phase DB-WAX fused silica capillary column (J&W Scientific or equivalent), 30 m x 0.32 mm x 0.25 µm bonded phase DB-WAX fused silica capillary column (J&W Scientific or equivalent) or 30 m x 0.53 mm x 3 µm bonded phase DB-624 fused silica capillary column (J&W Scientific or equivalent) or other column shown to be capable of separating methanol, acetone, acetaldehyde, methyl ethyl ketone and n-propanol.

2.2.2.4 GC detector - Flame ionization detector with appropriate data system.

2.2.3 Formaldehyde analysis apparatus

2.2.3.1 Spectrophotometer - A spectrophotometer capable of measuring absorbance at 412 nm.

2.3 Reagents

2.3.1 Water - Deionized water is to be used as the impinger collection liquid, and in the preparation of all standard and spike solutions.

2.3.2 Pure compounds - Reagent grade methanol, acetone, acetaldehyde, methyl ethyl ketone and 37% formaldehyde solution (formalin) for preparation of standard and spike solutions.

2.3.3 GC/FID calibration primary stock solution - Prepare stock solution by diluting 0.126 mL of pure methanol, 0.127 mL of pure acetone, 0.128 mL of pure acetaldehyde and 0.124 mL of pure methyl ethyl ketone in 100 mL volumetric flask with DI water (1000 mg/L).

2.3.4 GC/FID calibration and matrix spike solutions - Prepare standard solutions by serial dilutions of the stock solution. The recommended calibration range is 0.5 to 1000 mg/L. It has been found that the linear range can be extended up to 10,000 mg/L. Prepare matrix spike solutions by calculating the concentration of analytes desired and diluting the primary stock solution.

2.3.5 GC/FID internal standard primary spiking solution (if used) - Prepare primary stock solution by adding 0.312 mL cyclohexanol and diluting to 100 mL with DI water in a 100 mL volumetric flask (3 mg/mL cyclohexanol). Another internal standard material could be used if it is demonstrated that it does not interfere with the analyte peaks in the chromatogram.

2.3.6 n-propanol - Prepare a 3% (v/v) n-propanol solution for desorption of the analytes from the silica gel sorbent tubes.

2.3.7 Acetylacetone reagent - Prepare by dissolving 15.4 g of ammonium acetate in about 50 mL of DI water in a 100 mL volumetric flask. Add 0.20 mL of acetylacetone to this solution, along with 0.30 mL of glacial acetic acid. Mix thoroughly and dilute to 100 mL with DI water. Store reagent in a brown glass bottle in the refrigerator. Reagent is stable for at least two weeks.

2.3.8 Formaldehyde analysis primary stock solution - Prepare stock solution by diluting 2.7 mL of formalin in a 1000 mL volumetric flask with DI water (1000 mg/L formaldehyde).

2.3.9 Formaldehyde analysis calibration standard solution - Prepare standard solution by diluting 1.0 mL of primary stock solution in a 100 mL volumetric flask with DI water (10 mg/L formaldehyde).

2.3.10 Formaldehyde analysis calibration solutions - A series of calibration standards are made from the standard solution by adding 0, 0.1, 0.2, 0.4, 1.0 and 1.5 mL of the standard solution to individual screw-capped vials. The volume in each vial is adjusted to 2.0 mL with DI water. This corresponds to 0, 0.5, 1, 2, 5 and 7.5 mg/L calibration solutions. To each vial, 2.0 mL of the acetylacetone reagent is added, and the procedure described in Section 2.4.4.4 is then followed.

2.4 Procedure

2.4.1 Sample bottle preparation - Determine the number of sample bottles required for the sampling trip. Weigh each bottle and record the pre-sampling weight on the bottle.

2.4.2 Sampling - A sample field data sheet is shown in Figure 2.

2.4.2.1 Measure and record ambient temperature and barometric pressure.

2.4.2.2 Preparation of collection train - Measure 20 mL of DI water into the second impinger and connect probe, impingers, silica gel sorbent tubes, rotameter, critical orifice and pump as in Figure 1.

2.4.2.3 Leak and flow check procedure - Make sure that the on/off valve is in the on position, plug the sampling line inlet tip and turn on pump to draw a vacuum. When the vacuum reading is approximately 25 inches of Hg, turn the on/off valve to the off position, then record time and pressure reading on first pressure gauge. A leak is indicated by a flow of bubbles in the impinger, liquid being drawn into the stem of the impinger or a loss of vacuum. If a leak is present, tighten fittings, connections and impingers, and restart the leak check procedure. After 2 minutes, record the pressure reading on the first pressure gauge again. The leakage rate should not be in excess of 1 inch Hg (vacuum) in 2 minutes. Slowly and carefully remove the plug from the end of the probe, and turn the on/off valve back to the on position. Next, check the flow rate at the probe inlet with a bubble flowmeter. The flow rate should be comparable to the flow rate of the critical orifice with the impingers off-line. Record five measurements of the flow rate and turn off the pump.

2.4.2.4 Sample collection - Insert the probe into the stack and secure it. Start the pump, recording the time and the flow reading on the rotameter. End the sampling after 60 minutes. Record the time and remove the tubing from the vent. Recheck the sample flow rate at the probe inlet and turn off the pump. If the flow rate has changed significantly, redo sampling with fresh capture water. A slight variation (< 5%) in flow can be averaged. With the probe inlet end of the line elevated above the impinges, add about 5 mL of water into the inlet tip to rinse the line into the first impinger.

2.4.3 Sample recovery - Transfer the contents of the impingers into an appropriately labeled and pre-weighed sample storage bottle. The contents of both impingers can be combined into one bottle. If a large amount of water was collected in the dropout impinger, two bottles can be used. Remove the silica gel tubes from the sampling train, cap ends (tape caps on if necessary), and label. Store both impinger and sorbent tube samples in a cooler with ice until they can be stored in a laboratory refrigerator at approximately 4°C.

2.4.4 Sample analysis

2.4.4.1 Preparation of impinger samples - Remove bottles from refrigerator. Weigh the sample bottles and record weights on the bottle. Transcribe initial and final bottle weight to sample field data sheet. Bottles do not need to be at room temperature before weighing. Remove an aliquot of sample and place in the

sampler vial, add 10 µL of internal standard solution (if using internal standard calibration curve), and cap vial.

2.4.4.2 Preparation of sorbent tube samples - Remove sorbent tubes from refrigerator. Remove end caps and score glass to remove the silica gel from one section. Each section of the silica gel tube is analyzed separately. Pour into a 4.0 mL screw-capped vial and add 3.0 mL of a 3% (v/v) n-propanol/water desorption solution. Allow to sit for 30 minutes, with occasional light shaking. Vigorous shaking causes the silica gel particles to adhere to the cap and walls of the vial. Remove an aliquot of the desorption solution and place in an autosampler vial. Add 10 µL of internal standard solution (if using internal standard calibration curve) and cap vial.

2.4.4.3 GC/FID analysis - Analysis is performed by direct aqueous injection into the GC/FID. Representative conditions for the GC/FID analysis are given in Tables 2, 3 and 4. Other chromatographic columns and conditions may be used if it has been established that the compounds are separated and quality control parameters are met. Once the GC/FID system is optimized for analytical separation and sensitivity, the sample operating conditions must be used to analyze all samples, blanks, calibration standards and quality assurance samples. Note that constant injections of aqueous samples can cause water to build up in the system. This will cause the retention times to shift, and the peaks to broaden. It is recommended that after approximately 50 injections a bakeout of the system be performed. This should consist of heating the injector to 250°C, the oven to over 200°C and the detector to 275°C for at least several hours.

2.4.4.4 Formaldehyde sample analysis - Remove a 2.0 mL aliquot of the impinger sample and transfer to a screw-capped vial. Add 2.0 mL of the acetylacetone reagent and mix thoroughly. Place vial in a water bath at 60°C for 10 minutes. Allow vials to cool to room temperature. Transfer the solution to a cuvette and measure the absorbance at 412 nm. If the sample solution concentration is above the calibration curve, dilute original sample and repeat entire procedure. Do not dilute colored (derivitized) samples.

2.4.5 Quality assurance/quality control - Each field sampling program or laboratory that uses this method is required to operate a formal quality assurance program. Laboratory or field performance is compared to established criteria

to determine if the results of analyses meet the performance criteria of the method.

2.4.5.1 Field blank samples - A field blank sample of water must be prepared to assure that the water being used in the impingers is not contaminated. It is made in the field by filling a 40 mL VOA vial or polyethylene bottle with the same water being used to fill the impingers. A blank silica gel tube sample must be prepared to assure that any analytes that might be present on the silica gel or in the desorption solution are accounted for. It is made by breaking the ends of a silica gel tube in the field, capping it, and sending it with the samples to be analyzed.

2.4.5.2 Field spike sample - A field spike sample should be prepared by spiking the impinger with a known amount of analyte before sampling. After the impinger is spiked, a sample bottle containing DI water should also be spiked. This provides a check of the spiking solution and spiking procedure. The impinger spiking may be done on a duplicate sampling train if the equipment is available or may be done during a normal sampling run. This type of spiking is performed when a check of the complete sampling procedure, sample storage and sample analysis is desired.

2.4.5.3 Laboratory blank sample - A laboratory blank sample should be analyzed with each batch of samples. A batch is considered no more than 10 samples of similar matrix type.

2.4.5.4 Laboratory duplicates - A replicate injection of one sample in the analytical batch should be performed. The results of the duplicate analysis should be within 10% of the mean of the original and duplicate sample analysis.

2.4.5.5 Laboratory matrix spike samples - A laboratory matrix spike sample may be prepared with each group of similar matrix type. Using the mean concentration determined by the replicate analyses or the background level determined from a single measurement, determine the spiking level which will give one to four times the background. If the background sample does not have detectable levels of analytes, spike the sample at approximately five times the lowest calibration level of the instrument. Spike the sample with the determined amount of the calibration standard/matrix spike solution and proceed to analyze the sample in the normal manner. The results can be considered acceptable if the calculated spike recovery is 70 to 130%. In cases where multiple analytes are present, the analyte

with the highest concentration should govern the acceptance criteria.

2.5 GC/FID analysis of calibration standards

2.5.1 Internal standard calibration

2.5.1.1 Inject 1 µL of a methanol, acetone, acetaldehyde, methyl ethyl ketone calibration solution containing the internal standard and determine the retention time of the analytes relative to the internal standard. Each analyst should optimize the temperature program or instrument conditions, as necessary, to establish distinct separate peaks.

2.5.1.2 Calculate the relative response factor for the analytes (RRF_M) using Equation 1. If the average of the relative response factor for the analytes is constant, i.e., exhibits a coefficient of variation less than 20%, the calibration is acceptable and the average RRF_M can be used in all subsequent calculations; otherwise, the calibration curve solutions must be reanalyzed and reevaluated. It may be necessary to perform instrument maintenance prior to reanalysis. If reanalysis also fails to produce a linear curve, new calibration standards must be prepared and analyzed.

2.5.1.3 Analyze and calculate the relative response factor of a mid-range calibration standard daily, prior to each sample set, using Equation 2 to verify the calibration. The relative response factors must be within an acceptable range. If they are not, either prepare a new standard or perform instrument maintenance. If necessary, re-calibrate the instrument.

Equation 1

$$RRF_M = \left[\frac{A_M}{A_{IS}} x \frac{C_{IS}}{C_M} \right]$$

Where:

A_M = *area of analyte peak*
A_{IS} = *area of internal standard peak*
C_M = *concentration of analyte injected*
C_{IS} = *concentration of internal standard injected*

Equation 2

$$Concentration\,(mg/L) = \left(\frac{A_S \times C_{IS}}{A_{IS} \times RRF_M} \right)$$

Where:

A_s = *Area of the analyte peak in the sample*
C_{IS} = *Concentration of the internal standard (mg/L)*
A_{IS} = *Area of the internal standard peak*
RRF_M = *Relative response factor of analyte (Section 2.5)*

2.5.2 External standard calibration

2.5.2.1 Inject 1 µL of a methanol, acetone, acetaldehyde, methyl ethyl ketone calibration solution and determine the retention time of each analyte. Each analyst should optimize the temperature program or instrument conditions, as necessary, to establish distinct separate peaks.

2.5.2.2 Measure and plot the response of each analyte vs. concentration. If the correlation coefficient of the graph is greater than 0.99, the calibration is acceptable and the equation of the line can be used in all subsequent calculations; otherwise, the calibration curve solutions must be reanalyzed and reevaluated. It may be necessary to perform instrument maintenance prior to reanalysis. If reanalysis also fails to produce a linear curve, new calibration standards must be prepared and analyzed.

2.5.2.3 Analyze and calculate the concentration of a mid-range calibration standard daily, prior to each sample set, to verify the calibration. The recovery should be between 70 and 130%. If it is not, either prepare a new standard or perform instrument maintenance. If necessary, re-calibrate the instrument.

2.6 Analytical range and minimum calibration level

2.6.1 Demonstrate that the calibration curve is linear (relative response factors exhibit a coefficient of variation less than 20%, or correlation coefficient greater than 0.99) throughout the range of the calibration curve.

2.6.2 Demonstrate that the analytes are detectable at the minimum levels using the lowest level calibration curve solution.

2.7 Calculations

2.7.1 Nomenclature and calculations - Perform the calculations as follows:

Equation 3

Calculation of sample flow rate corrected to a dry basis:

$$S_C = S_U \left(\frac{BP - PW}{760} \right) \left(\frac{293}{273 + t} \right)$$

where:

S_C = *Corrected (dry standard) sampling flow rate, L/min*
S_U = *Uncorrected sampling flow rate, L/min*
BP = Barometric pressure at time of sampling, mm Hg
PW = Saturated partial pressure of water vapor, mm Hg at t
t = Ambient temperature at time of sampling, °C

Equation 4

Calculation of stack concentration:

$$C_S = \left(\frac{g_X}{MW_X} \right) \left(\frac{24.04}{S_C \, x \, S_T} \right) x \, CF \, x \, 10^6$$

where:

C_S = *Stack concentration, ppmv*
g_X = *Total amount of analyte collected in impingers and on sorbent tubes, grams*
MW_X = *Molecular weight of analyte, grams/mole*
S_C = *Corrected (dry standard) sampling flow rate, L/min*
S_T = *Sampling time, min*
CF = Correction Factor from Table 1

2.8 Alternative procedures - Not applicable to this method.

2.9 References

United States Environmental Protection Agency (EPA) Method 301, *Field Validation of Emission Concentrations from Stationary Sources* (Appendix A to CFR 63).

2.10 Tables, diagrams, flowcharts and validation data

Table 1. Method 301 Validation Results

Pollutant	Brownstock Washer Hood		Bleach Plant Scrubber Inlet		Smelt Dissolving Tank		Recovery Furnace	
	Validated	CF	Validated	CF	Validated	CF	Validated	CF
Methanol	Yes	None	Yes	1.0	Yes	1.0	Yes	None
Acetone	Yes	1.3	Yes	1.2	Yes	1.1	Yes	1.2
Acetaldehyde	Yes	None	No	---	Yes	None	Yes	None
Methyl Ethyl Ketone	No	---	Yes	1.3	Yes	1.2	Yes	1.3
Formaldehyde	Yes	1.1	Yes	1.2	Yes	None	No	---

Table 2: GC/FID Operating Conditions for Methanol, Acetaldehyde, Acetone and Methyl Ethyl Ketone Analysis-DB-WAX Column

Injection:	Direct
Injector Temperature:	150°C
Injection Volume:	1 µL
FID Detector Temperature:	250°C
Carrier Gas:	Helium
Column:	DB-WAX, 30 m x 0.53 mm id x 1 micron fused silica capillary column
Temperature Program °C:	
Initial:	18°C for 8 min
Ramp 1:	3°C/min to 20°C for 2 minutes
Ramp 2:	50°C/min to 220°C
Ramp 3:	
Final Hold Time:	5 minutes
Retention Time Order:	acetaldehyde, acetone, methyl ethyl ketone, methanol, n-propanol, cyclohexanol

Table 3: GC/FID Operating Conditions for Methanol, Acetaldehyde, Acetone and Methyl Ethyl Ketone Analysis-DB-WAX Column

Injection:	Direct
Injector Temperature:	170°C
Injection Volume:	1 µL
FID Detector Temperature:	275°C
Carrier Gas:	Helium
Column:	DB-WAX, 30 m x 0.32 mm id x 0.25 micron fused silica capillary column
Temperature Program °C:	
Initial:	0°C for 3 min
Ramp 1:	5°C/min to 50°C for 4 minutes
Ramp 2:	70°C/min to 100°C for 10 min
Ramp 3:	70°C/min to 200°C
Final Hold Time:	4 minutes
Retention Time Order:	acetaldehyde, acetone, methyl ethyl ketone, methanol, n-propanol, cyclohexanol

Table 4: GC/FID Operating Conditions for Methanol, Acetaldehyde, Acetone and Methyl Ethyl Ketone Analysis-DB-624 Column

Injection:	Direct
Injector Temperature:	170°C
Injection Volume:	1 µL
FID Detector Temperature:	275°C
Carrier Gas:	Helium
Column:	DB-624, 30 m x 0.53 mm id x 3 micron fused silica capillary column
Temperature Program °C:	
Initial:	0°C for 3 min
Ramp 1:	5°C/min to 50°C for 0 minutes
Ramp 2:	70°C/min to 105°C for 17 min
Ramp 3:	70°C/min to 220°C
Final Hold Time:	3 minutes
Retention Time Order:	acetaldehyde, methanol, acetone, n-propanol, methyl ethyl ketone, cyclohexanol

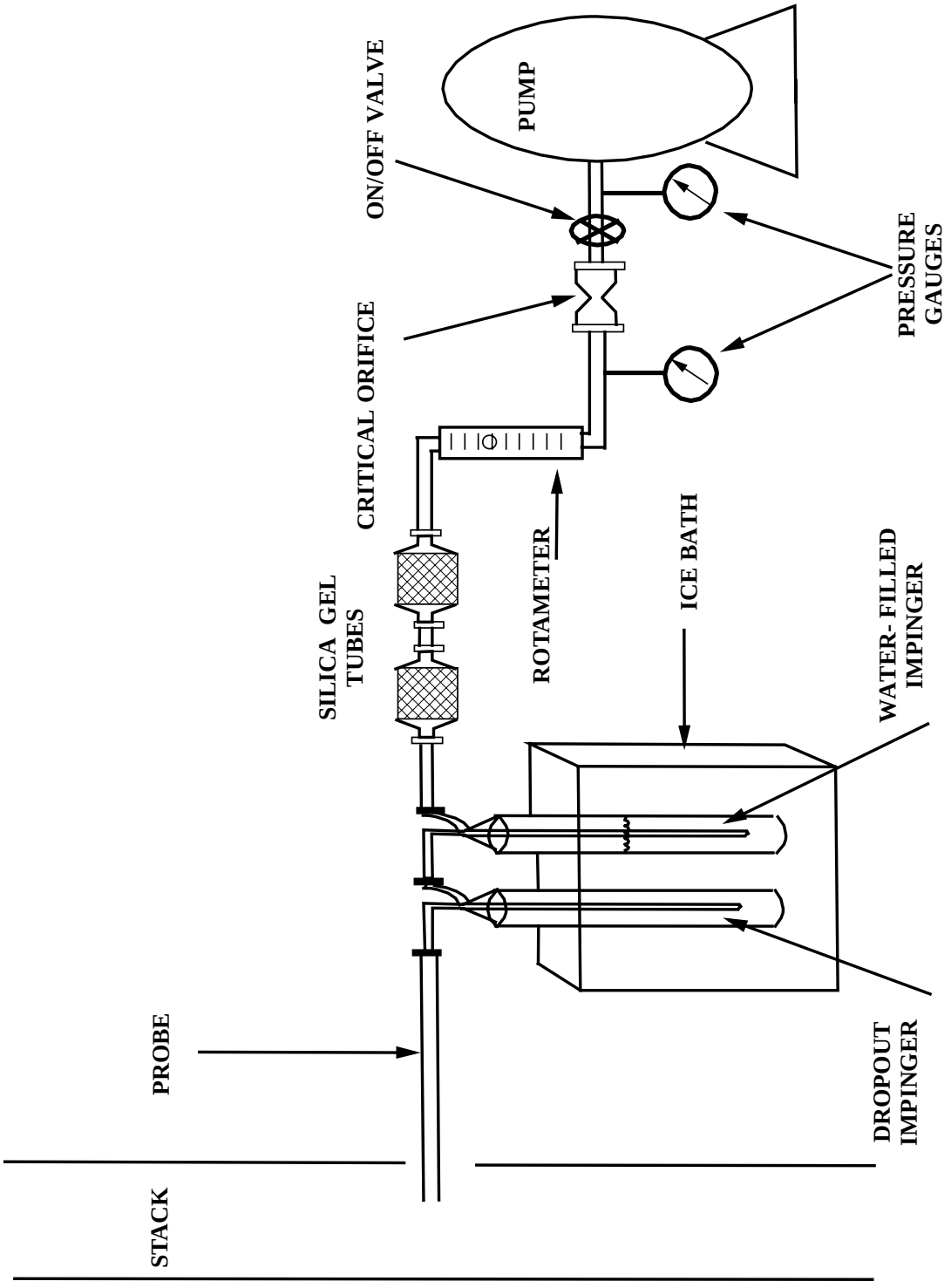

Figure 1. Chilled Impinger/Silica Gel Tube Sampling Train

NCASI Chilled Impinger/Silica Gel Tube Test Method At Pulp Mill Sources For Methanol, Acetone, Acetaldehyde, Methyl Ethyl Ketone And Formaldehyde

Field Sampling Data Sheet

Mill Name:_____ Date: _____

City,State: _____ Sampler's Name: _____

Source Name/Description: _____

_____

Run Number: _____

Start Time: _____ Stop Time: _____

Ambient Temp at Start: _____ Ambient Temp at Stop: _____

Barometric Pressure: _____

Leak Test

Time:_____ Initial Measurement (in Hga): _____

Time: _____ Final Measurement (in Hga): _____

Leak Check Criteria- Must not lose more than 1 inch of Hg (vacuum) in 2 minutes.

Meets Criteria? Yes No

System Flow Rate Measurement

Average of 5 flow measurements for Pre-Sample FlowRate:

1._____ 2._____ 3._____ 4._____ 5._____ Avg: _____

Average of 5 flow measurements for Post-Sample Flow Rate:

1._____ 2._____ 3._____4._____ 5._____ Avg: _____

Overall Average Sample Flow Rate: (indicate units!)

Avg: _____

Rotameter Readings

Time: _____ Flow: _____
Time: _____ Flow: _____
Time: _____ Flow: _____
Time: _____ Flow: _____
Time: _____ Flow: _____
Time: _____ Flow: _____

QA/QC Measures

Train Spike Conducted?	Yes	No
Duplicate Conducted?	Yes	No
Spiked Duplicate Made?	Yes	No
Field Blank Made?	Yes	No
Field Spike Made?	Yes	No

Sample Bottle Weight(s):

Bottle 1: Initial Weight: _____ Bottle 2: Initial Weight: _____

Final Weight:_____ Final Weight:_____

Notes/Comments

_____

Figure 2. Field Sampling Data Sheet

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

AUG 12 1997

OFFICE OF
AIR QUALITY PLANNING
AND STANDARDS

Ms. Mary Ann Gunshefski
NCASI
Southern Regional Center
P.O. Box 141020
Gainesville, Florida 32614-1020

Dear Ms. Gunshefski:

We have reviewed your report entitled, "Method 301 Validation of the NCASI Chilled Water Impinger/Silica Gel Tube Test Method at Selected Pulp Mill Sources for Methanol, Acetone, Acetaldehyde, Methyl Ethyl Ketone, and Formaldehyde." We agree with your conclusion that the NCASI Chilled Water Impinger/Silica Gel Tube Test Method (NCASI Impinger Method) met Method 301 criteria for the pollutants and sources that are summarized in the enclosed table. The NCASI Impinger Method may be used for determining compliance with the proposed emission limits in 40 CFR Part 63, Subpart S.

If you have any questions about our comments or you would like to meet to discuss them, please contact Gary McAlister of my staff at (919) 541-1062.

Sincerely,

William F. Hunt, Jr.
Director
Emissions, Monitoring and
Analysis Division

cc: Penny E. Lassiter (MD-13)
Stephen A. Shedd (MD-13)
Jeffrey A. Telander (MD-13)

Enclosure

Figure 3. EPA Approval Letter - Page 1

Pollutant	Brownstock Washer Hood		Bleach Plant Scrubber		Smelt Dissolving Tank		Recovery Furnace	
	Validated	Correction Factor	Validated	Correction Factor	Validated	Correction Factor	Validated	Correction Factor
Methanol	Yes	None	Yes	1.0	Yes	1.0	Yes	None
Acetone	Yes	1.3	Yes	1.2	Yes	1.1	Yes	1.2
Acetaldehyde	Yes	None	No	-	Yes	None	Yes	None
Methyl ethyl ketone	No	_	Yes	1.3	Yes	1.2	Yes	1.3
Formaldehyde	Yes	1.1	Yes	1.2	Yes	None	No	-

Figure 4. EPA Approval Letter - Page 2