Method no.:	ID-178SG
Matrix:	Air
OSHA Standard:	Oil Mist not presently part of the cotton dust standard (1910.1000) Oil Mist Std = 5 mg/m^3 (1910.1043) Cotton Dust Std = 200 µg/m^3 Yarn = 750 µg/m^3 Slash = 500 µg/m^3 Other
Validation Level:	0.04 mg/mL to 2.0 mg/mL
Collection Procedure:	Preweighed 37-mm PVC filters are used with vertical elutriator cotton dust sampler at flowrate of 7.4 L/min and 2.7 m^3 sample volume.
Recommended air volume and sampling rate studied:	2.7 m^3 at 7.4 L/min
Analytical procedure:	1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113) extraction followed by IR scan from 3400 to 2600 cm^{-1}.
Detection limit or reliable quantitative limit:	0.04 mg/mL
Precision	CV:1 = 0.079 (analytical)

1. Introduction

1.1 Scope

1.1.1 This method describes the separation of oil mist from cotton dust and the analysis of the oil mist by infrared spectrophotometry.

1.1.2 This method is used for analysis of oil mist in the presence of cotton dust. The oil mist from lubricating oils used in weaving machines contributes to the cotton dust concentration. The weight of oil mist found in a cotton dust sample is subtracted from the total weight of the sample to obtain a corrected cotton dust measurement.

1.1.3 This work was done using equipment and operating parameters similar to those used for the oil mist studies with carbon tetrachloride as the extraction solvent (References 7.1 and 7.3).

1.2 Advantages and Disadvantages

1.2.1 The analytical method is simple and fast. It can be used for most lubricating mineral oils which are soluble in Freon 113.

1.2.2 The instrumentation required is available in most laboratories.

1.2.3 While the method is not specific for a particular oil, it will give a good indication of total oil mist exposure in locations where several oils are in use (Reference 7.1).

1.3 Principle

1.3.1 The samples are collected using preweighed 37 mm PVC filters in a vertical elutriator cotton dust sampler (Reference 7.2).

1.3.2 The filters are weighed to determine the total weight of oil and cotton dust. The oil is then extracted from the filters with Freon 113.

1.3.3 The Freon 113 solution is analyzed for oil content using infrared spectrophotometry to measure the C-H absorbance near 2940 reciprocal centimeters (cm^{-1}).

1.3.4 The weight of oil is then subtracted from the total weight of mist plus dust in order to determine the weight of the cotton dust.

2. Range and Detection Limit

2.1 The analytical range is 0.04 mg/mL to 2.0 mg/mL oil in solution. The detection limit is about 35 µg/mL. This detection limit may vary depending on the oil and the path length of the IR cell. A detection limit of 2.0 µg/mL can be obtained using a 1.0 cm IR cell (Reference 7.1). The upper analytical range can be extended by diluting the sample.

3. Precision and Accuracy

3.1 Eighteen samples were spiked with known amounts of oil, six at each of three levels, corresponding to 1/2, 1, and 2 times the PEL of 0.750 mg/m^3 for cotton dust slashing and weaving operations. One blank filter was used as a lab and reagent control, it was determined to be below the detection limit of the method.

Precision and accuracy data for spiked lab samples were determined. The mean recovery was 94.5%

with a pooled coefficient of variation of 0.079. This data represents the precision and accuracy for the analytical part of the procedure.

4. Interferences

4.1 Filters must be handled with care to avoid any oil contamination since this method is sensitive to any C-H (methyl group) containing compound.

Although the method is sensitive to hydrocarbon compounds, this should not cause a serious interference since most hydrocarbons are volatile and would not be collected on a membrane filter.

4.2 Tobacco smoke is a possible interference and workers should not be allowed to smoke while the sample is being collected (Reference 7.1).

5. Sampling (Samples are taken using the procedure described in 29 CFR Part 1910.1043)

5.1 Apparatus

5.1.1 A vertical elutriator cotton dust sampler capable of operating at a sampling rate of 7.4 ± 0.2 L/min is used. The pump must be properly monitored during sampling so that the volume of air sampled can be determined accurately from the flow rate and time. It is important that the samplers be cleaned prior to sampling.

5.1.2 A filter unit consists of a 37-mm PVC filter with 5 micron pore size supported by a backup pad and 37-mm 3-piece cassette filter holder secured with tape or a shrinkable band.

5.1.3 An analytical balance capable of weighing to the nearest 0.01 mg.

5.2 Procedure

5.2.1 Sampling is done in accordance with current instructions contained in OSHA directives to the industrial hygienist and 29 CFR 1910.1043.

5.2.2 The sample is collected on a preweighed 37 mm PVC filter with a pore size of 5 microns. The sample is collected with a vertical elutriator cotton dust sampler using a flow rate of 7.4 ± 0.2 liters per minute. A 2.7 m^3 air sample is recommended.

5.2.3 Ship the samples to the laboratory as soon as possible. With each batch of samples, at least one filter, labeled as a blank, must be submitted. No air should be drawn through this filter.

5.2.4 An unused, undiluted oil bulk (5-10 mL is sufficient) of the oil being used must be collected and sent to the laboratory for use as a reference standard in the analysis. To prevent contamination of the air samples, this bulk oil should be shipped in a separate container.

6. Analytical Procedure

6.1 Apparatus

6.1.1 Infrared spectrophotometer: Perkin-Elmer Model 567 or equivalent.

6.1.2 Liquid sample cells: Matched 1-mm reference and sample cells with NaCl or BaF_2 windows.

6.1.3 Scintillation vials with Teflon-lined caps for desorption of samples.

6.1.4 Volumetric flasks: 5, 10, 25, and 50-mL Class A glassware.

6.1.5 Glass volumetric pipettes: 1, 2, 3, 5, and 10-mL Class A.

6.1.6 Hamilton #1001 Gastight 1-mL syringe or equivalent.

6.2 Reagents

6.2.1 1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113) Fisher reagent grade or equivalent. There have been reports of spectral problems when using a poor grade of this reagent. Therefore, ensure the quality of new lots of this reagent before using it in the analyses by analyzing a reagent blank.

6.3 Precautions

6.3.1 When handling Freon 113, gloves and safety glasses should be worn. Care should be observed to avoid splashing or spilling. All work with Freon 113 should be done with adequate ventilation and all waste solutions should be collected, labeled, and disposed of properly.

6.4 Sample Preparation

6.4.1 Clean all glassware by washing in detergent solution and rinse with deionized water. Let dry, then rinse with Freon 113.

6.4.2 Extract each filter as described below with Freon 113. Use tweezers to transfer sample and blank filters into individual glass sampling vials exercising care to avoid contaminating the filters with oil from hands. The use of gloves is strongly advised.

Add 5 or 10 mL Freon 113 depending on the sample weight and cap the vial. Allow vials to stand for 30 minutes with occasional agitation. At this point the oil has been extracted from the filter and the sample extracts are ready for analysis.

6.5 Standard Preparation

6.5.1 Stock Solution, 1000 µg/mL: Weigh 50.0 mg of the bulk oil into a 50 mL volumetric flask and dissolve with Freon 113. Dilute to volume with Freon 113.

6.5.2 Prepare a series of working standards in the analytical range of 40 to 2000 µg oil/mL by making the following serial dilutions. Add the appropriate aliquots of the oil standard solution (6.5.1) to the appropriate volumetric flask using glass volumetric pipettes and dilute to volume with Freon 113. Use these dilutions only as a guide.

Aliquot of 100 µg/mL Standard	Dilution Volume (mL)	Final Standard (µg/mL)
0.5	10.	50
1.0	10.	100
5.0	10.	500
-	-	1000

6.6 Analysis

6.6.1 Fill the sample cell with the sample and the reference cell with Freon 113 and scan the region from 3400 to 2700 cm^{-1}. All scans should be done in absorbance mode, if available. Otherwise, data must be converted to absorbance units.

6.6.2 Analyze the reagent blank, standards, and the sample(s). A standard should be reanalyzed after every 5 or 10 samples to bracket the sample concentrations.

6.6.3 The absorbance at the wavenumber of the peak used to plot the calibration curve (generally about 2940 cm^{-1}) is measured and used to determine the oil concentration in the samples. The calibration curve is prepared from the oil bulk standards.

6.6.4 Use any available least square regression program to plot a calibration curve of peak height or absorbance vs. concentration (in µg/mL) of the standards.

6.6.5 If the baseline is not even, draw a baseline which connects points on the spectrum from 3000 and 2700 cm^{-1}.

6.6.6 The instrument parameters used for a Perkin-Elmer 567 infrared spectrophotometer are as follows: time constant of -1, scan time MED, slit N (normal), and liquid sample cells which have 1-mm path length and BaF_2 windows.

6.7 Calculations

6.7.1 A calibration curve is obtained by measuring the peak height or absorbance of the standards and plotting these values vs the concentration in µg/mL.

6.7.2 The concentration of oil in the air sampled can be calculated as mg oil/m^3 as follows.

$C = [(K - B) \times V] / V_s$

where: K = concentration of oil from calibration curve in µg oil/mL.
B = concentration of oil in blank in µg oil/mL.
V = final volume (mL) of sample analyzed including any dilution factor.
V_s = volume of air in liters sampled at 25°C and 760 mmHg.
Note: for C in µg/m^3, multiply by 1000

Then the final concentration of cotton dust in µg/m^3 C will be calculated as follows:

$C_{cd} = C_t - C$

where C_t = total weight of dust and oil after sample collection, as determined in 29 CFR 1910.1043 in µg/m^3
C = concentration of oil mist in µg/m^3

7. References

7.1 Oil Mist, P&CAM 283, NIOSH Manual of Analytical methods, Second Edition, Volume 4, US Department of Health, Education, and Welfare, 1978.

7.2 OSHA Safety and Health standards, 29 CFR Part 1910.1043, Revised December 14, 1985.

7.3 Method used by Burlington industries, submitted to AIHA for publication in "Recommended Practice for Cotton Dust Sampling and Analysis, Volume 1", Textile Industrial Hygeine Roundtable, Editor- John D. Neefus, Phd.