Test Method Number 22

Particle Release from Wipers and Other Materials Under Conditions of Moderate Mechanical Stress

1 Scope

1.1 Particles:

After extraction, particle quantification is carried out using a Liquid Particle Counter (LPC).

Two LPCs both manufactured by Particle Measuring Systems (PMS) are being used. One unit, LiQuilaz S02 is used for counting particles from 0.2 µm and up, and the second, LiQuilaz S05 for 0.5 µm and up.

For all practical purposes only the cumulative number of particles is reported, though information on number of particles in different size ranges are available from the instrument. S02 with a channel setting of 0.2, 0.5, 1.0, 1.5 and 2.0 µm will count number of particles of these dimensions, and all particles larger than 2 µm will be included in the 2 µm channel. Similarly with S05 all particles larger than 20 µm will be included in the 20 µm channel.

Quantification of particles in a solution by LPC is limited to concentrations of not more than 10,000 particles per mL in total. Quantification of sample extracts with greater than 10,000 counts per mL can be accomplished through appropriate dilutions.

1.2 Fibers:

Fiber quantification is carried out in two ranges: 20 – 100 µm and > 100 µm. After extraction, fibers are captured on a gridded filter paper, let dry and then counted under an Optical microscope.

Quantification of fibers in a wiper needs to be below 20,000 fibers/m^2. This equates to about 8 fibers/square on the gridded filter. Quantification of samples with fiber concentrations greater than 20,000 fibers/m^2 can be accomplished with appropriate dilutions or smaller sample portions.

PURPOSE

This test method details the extraction and enumeration of particles and fibers released from wipers (or other materials) in a wetted state, under conditions of moderate mechanical stress.

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Reagents and Solutions

Table 2.1 Reagents and Solutions

Particle Size Reference Stock Solutions	0.35 µm and 0.6 µm standards from Duke scientific for S02. 2.0 µm and 5.0 µm for S05.
Reagent-grade water	ASTM D1193, Type III water, filtered through a 0.2 µm-particulate filter
Surfactant solution	A 0.5% v/v solution of Triton X surfactant or equivalent (for glassware cleaning only)
Isopropyl alcohol	Reagent grade or better

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Safety

The toxicity of each reagent used in this method, singularly or in combination has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level. Copies of material safety data sheets for every reagent used in this method are available through TMS. All personnel involved in the chemical analysis should be consulted before handling the chemicals. Proper safety protocols and personal protective equipment (safety eyewear, gloves, and lab coats) must be used in adherence to the Laboratory Chemical Hygiene Plan.

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Equipment

4.1 Labware/Glassware Cleaning[1]

4.1.1 Rinse all labware with hot tap water.

4.1.2 Add sufficient surfactant solution to provide complete wetting of all surfaces. Where possible scrub the interior surfaces with a low particle wiper, or a brush. Be sure not to scratch the interior surfaces of the glassware.

4.1.3 Rinse three times with copious amounts of hot tap water.

4.1.4 Rinse the labware with a small stream of isopropyl alcohol and capture the alcohol rinse for proper disposal.

4.1.5 Rinse with deionized water using the sprayer.

4.1.6 Rinse three times with reagent-grade water.

4.1.7 Fill the labware with reagent-grade, particle-free water to the maximum volume mark. Test the water to ensure that it has a particle count of <10 particles/mL with SO5.

4.1.8 If the resulting solution has counts >10 particles/mL at ≥0.5 µm, repeat the cleaning process.

Table 4.1 Equipment

Liquid Particle Counter	Particle Measuring System, LiQuilaz S02 Unit for 0.2 to 2 µm and LiQuilaz SO5 for 0.5 to 20 µm
Autosampler for the LPC	Particle Measuring Systems, LS200 Autosampler
Laminar flow workstation	To meet ISO Class 5 or better
Mechanical stirring plate	Having a mixing surface area of at least a 5" diameter and variable speeds
Extraction Vessels	1000, and 2000 mL capacity Erlenmeyer flasks, Pyrex® glass
Graduated Cylinders	Various size cylinders
Volumetric Flasks	Various size flask, class A
Biaxial Shaker	Capable of providing a 10 to 15 mm vertical and horizontal displacement a 500 rpm
Filtration Apparatus	Membrane filter holder with 47 mm diameter glass base (such as Millipore type XXI004700 or equivalent)
Vacuum Source	Pump or aspirator capable of producing a vacuum sufficient to produce a flow through the filter of 25 mL/min
Filters	Gridded membrane filters 47 mm in diameter with a 0.45 µm pore size, such as Millipore type HABG 04700 or equivalent)
Filter holders	Plastic petri dishes for 47 mm filters, such as Millipore PD2004700, PD1504700
Desiccating cabinet	Suitably sized for the drying of membranes using silica gel
Deionized reagent-grade and particle-free water system	System capable of producing at least ASTM Type III water, with a 0.2 µm-particulate filter and capable of producing water with 10 particles/mL at 0.5 µm A Barnstead Diamond System is sufficient
Tweezers	Duckbill type
Micro-pipette with washed tips	100 – 5000 µL electronic pipette, Eppendorf Research Pro or similar
Latex Cleanroom Gloves	Rinsed with reagent-grade water
Aluminum foil	Rinsed with reagent-grade water
Objective Micrometer	1 mm micrometer with graduations of 0.01 mm

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Facilities

Sample preparation for extraction must be conducted in a Class 5 particle hood. During extraction, samples must be contained in a sealed environment that minimizes the possibility of contamination. The sample filtration unit, liquid particle counter (LPC), and sampler can be housed in a laminar flow particle hood capable of producing a Class 5 environment.

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Procedures

6.1 The LPC instrument and the software (Sampler Sight) are all installed strictly according to the Manufacturer's Instructions.

Typical Set up for the LiQuilaz SO2/SO5 and LS200 autosampler are as follows:
(Done through the Sample Configuration Dialog Box)

	SO5	SO2
Sample volume	10 mLs	10 mLs
Fill Speed	20 mLs/min	20 mLs/min
Drain Speed	50 mLs/min	50 mLs/min
Repeat Count	3	3
Threshold settings	0.5, 0.6, 0.8, 1.0, 1.2, 1.5, 1.8, 2.0, 5.0, 10, 12, 15, 18, 20	0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 1.0, 1.5, 2.0
Count Mode	Cumulative and cts/mL	Cumulative and cts/mL

6.2 LPC Operation

When the LPC is set as outlined above, the unit will withdraw a 10 mL aliquot of fluid from the beaker (using the liquid sampling system that is part of the LPC) and determine the concentration of particles. The unit will repeat this process three more times. The software will discard the first reading and average the second, third and fourth results and report accumulative and differential particles per mL for all the established threshold (channel) ranges.

1. Turn on the LPC unit and allow it to warm up at least 30 minutes.
2. Ensure the unit is set up in accordance with Section 6.1, above.
3. Transfer 100 mL sample in a clean 250 mL beaker.
4. Add a clean stirring bar to the 250 mL beaker.
5. Place the beaker onto the magnetic stirrer of the LPC sampler and adjust the speed so that the solution just mixes, without causing bubbles or turbulence in the beaker.
6. Place end of the tube into the beaker of sample, and initiate the analysis (Using the mouse select the green "Go" button in the Sampler Sight).
7. After the analysis is complete, rinse the auto sampler using clean particle-free water.
8. The cumulative and differential counts are displayed.

6.3 Biaxial Shaker Sample Extraction

6.3.1 Method Blank Preparation

1. Transfer ~200 mL of the Reagent water to a clean 400 mL beaker and confirm that the particle counts in the water are <10 particle/mL using S05 (and 300 cts/mL for SO2).
2. In the Class 5 workstation, add exactly 600 mL of Reagent water to a cleaned 2-liter flask. Using gloved hands and clean forceps, cover the flask with aluminum foil that has been rinsed with deionized water.
3. Mount the covered flask onto the biaxial shaker, secure it in place using the necessary cushions to prevent the flask from breaking or chipping, and seal by compression. Start the shaker and shake the flask for five minutes.
4. Decant 100 mL into a clean 250 mL beaker for LPC extraction. Reserve the remaining 500 mL sample portion for the Fiber Analysis Method Blank. Cover the beaker with foil that has been rinsed with particle-free water. See Section 6.4 for preparation of the sample for fibers analysis.
5. LPC Method Blanks must be prepared and proven to be acceptable, before processing any samples. The method blank must have less than 10 particle counts/mL for SO5, (and 300 cts/mL for SO2) or less than one-tenth the particle counts of the sample(s) whichever is more. If the method blank does not meet acceptance criteria, all labware must be cleaned again and a new method blank extracted. No sample processing can be performed until the extraction system is proven to be sufficiently clean[2].

6.3.2 Sample Extraction

1. After thorough rinsing of 2L flask, select a representative wiper from the package and insert it into the refilled flask and cover the flask with aluminum foil that has been rinsed with particle-free water.
2. Follow the same steps as the method blank, but leave the wiper in the flask when decanting the 100 mL extract into the beaker.
3. With the remaining 500 mL extract, decant into TM22 filter apparatus. Water is filtered so fibers may be quantified.
4. Once all the extract has been poured off, remove the wiper from the flask, measure the width and length, and calculate the area in square meters. Record in the extraction worksheet.

6.4 Optical Microscope Determination for Fibers

1. Under the laminar flow hood, preclean the 47 mm membrane filter by holding it with forceps and gently rinsing the surface with a stream of deionized water. Carefully place the filter on the fritted glass base so that it is centered and lies flat, then clamp the funnel in place.
2. Transfer the sample portion from the biaxial shaker extraction slowly from the beaker into the funnel of the filtration apparatus. Apply a vacuum to the filtration apparatus and continue to transfer the remainder of the extract from the beaker into the funnel until all of the extract is filtered.

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Procedures (cont.)

3. After filtration is complete, turn off the vacuum and remove the filter from the support base with forceps. Place the filter in a 47 mm Petri dish cover and allow the filter to dry overnight in a particle-free zone, such as the laminar-flow hood or desiccator.
4. Prepare the microscope for use. Set a magnification of 40x and establish a size reference through the eyepiece by focusing on to the Objective Micrometer, and aligning with the eyepiece reticle. Normally 1 reticle division is equal to 25 microns at 40x.
5. On the dried sample filter, locate the square directly in the center of the filter's grid and make an identification mark. This is area of interest – square # 6/17. See Figure 6.5.
6. Mount the petri cover into place on the microscope stage so that area of interest (square 6/17) is centered on the stage. Adjust the microscope focus and lamp intensity to obtain maximum fiber definition.
7. Scan the entire surface of the filter for uniformity of particle distribution. If the particles are not uniformly distributed over the filtered area of the filter, a new sample must be prepared. Uniformity of particle distribution over the filter is a must for accurate quantification.
8. Count the fibers in the ranges of 20 – 100 µm and > 100 µm on square #6 and record the data into the appropriate box on Optical Microscopy Worksheet. Repeat this procedure until all 30 identified squares have been counted.

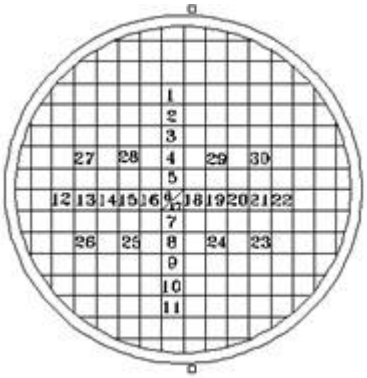

Figure 6.5

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Calculations

7.1 LPC Calculation Overview

With the LPC set up in default mode the unit will withdraw three aliquots of extract from the sample beaker (using the liquid sampling system that is part of the LPC) and determine the concentration of particles. The LPC will discard the first aliquot as a rinse, average the two remaining results and report accumulative and differential particles per mL across the threshold ranges. Calculate the particle concentration for the wiper using the formula below. Calculate, to two significant figures, the number of particles per m^2.

7.1.1 LPC Sample Calculation

Formula 7.1.1: $\text{Particles/m}^2 = (((S_C * df) - B_C) * V) / A$

Where:

- S_C = Averaged cumulative sample counts at the size range of interest
- df = Dilution factor for any external dilution of the sample extract
- B_C = Averaged cumulative method blank counts at the size range of interest
- V = Total volume of the sample extract
- A = Area of the wiper in m^2

7.1.2 Example Calculation:

Particle Range	0.2 – 300 µm (0.5 – 300 µm)
Particle Channels	0.2 – 2 µm (0.5 – 20 µm)
Area of wiper:	0.230 m x 0.230 m
Average Count/mL	300
Total Volume	600 mL
Blank:	10 particles/mL
External Dilution	none

$$\frac{((300 * 1) - 10) * 600}{0.230 \times 0.230} = 3.3 \times 10^6 \text{ particles/m}^2$$

7.2 Fibers Calculation Overview

There is 960 mm^2 of filtration area on the filter membrane. This represents approximately 107 square grid areas of interest on the filter. The 30 grids that are examined represent 28.1% of this total area and are accounted for in the formula by the view factor of 3.6 (107/30 = 3.6). Two length ranges of fibers are counted, 20 to 100 µm and >100 µm. Count and quantify the fibers in each of these ranges separately. The calculation for each fiber range is the same.

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Calculations (cont.)

Fibers Sample Calculation

Formula 7.2.1: Fibers /m^2 = $\frac{(((S_C * df) - B_C) * 3.6) * (V_t/V_f))}{A}$

Where:

- S_C = Total fiber count for the sample in the specific size range
- df = Any external dilution factor
- B_C = Method blank fiber count in the specific size range
- V_t = Total extraction volume (normally 600 mL)
- V_f = Volume of the extract filtered
- A = Area of the wiper extracted in m^2

Example Calculations

Fiber Range:	20-100 µm
Area of wiper:	0.230 m by 0.230 m
Sample Counts for the 30 squares:	215
Total Extract Volume:	600 mL
Volume of extract filtered:	500 mL
Blank Counts for the 30 squares:	6 particles/mL
External Dilution:	none

Fibers/m^2 = (((215 * 1) – 6) * 3.6) * (600/500)) / (0.23 * 0.23)
= (752.4 * 1.2) / 0.0529
= 17,067.67
= 17,100

Fiber Range:	>100 µm
Area of wiper:	0.230 m by 0.230 m
Sample Counts for the 30 squares:	170
Total Extract Volume:	600 mL
Volume of extract filtered:	500 mL
Blank Counts for the 30 squares:	3 particles/mL
External Dilution:	none

Fibers/m^2 = (((170 * 1) – 3)*3.6) * (600/500)) / (0.23 * 0.23)
= (701.4 * 1.2) / 0.0529
= 13638
= 13600

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Quality Assurance Criteria

8.1 LPC Unit Calibration

The liquid particle counter (LPC) is to be calibrated annually by a qualified provider. It is preferred that the LPC unit be returned to Particle Measuring System Inc. for calibration and servicing. The provider of this service must provide written documentation of the calibration and acceptance criteria. This documentation must be kept on file in accordance with the documentation control standard operating procedure.

8.2 Periodic Analysis of Certified Standards

Prepare particle size standard solutions of the 0.35µm and 0.6 µm for S02 (2.0 and 5.0 um for S05) by dilution of the Duke stock reference solutions. Standards should be prepared in accordance with the supplier's directions. Select a stock standard and dilution that will produce a final concentration in the 1000 to 5000 particles/mL range. Fill the volumetric flask to about two-thirds particle free DI water, add the required amount of stock standard using a micropipette, and bring to final volume with DI water. Gently, invert the stoppered flask three times to mix the standard. Run analysis to ensure proper sizing is being determined.

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References and Notes

Institute of Environmental Sciences and Technology (IEST), IEST-RP-CC004.3, *Evaluating Wiping Materials Used in Cleanrooms and Other Controlled Environments*

1. For apparatus that is not already scrupulously clean, it is sometimes helpful to fill the vessel with deionized water and place them in an ultrasonic bath for several minutes. This is effective for removing stubborn surface particles that may not be removed using the standard washing procedure and that might otherwise be dislodged during the testing of the wiper.

2. Periodic cleaning of the capillary column may be necessary if DC voltage goes above 1.0mV reference instrument manual for proper cleaning.

ITW Texwipe®

North America
300B Route 17 South
Mahwah, NJ 07430
Tel (800) TEXWIPE ext 120
(201) 684-1800 ext 120
Fax (201) 684-1801
www.texwipe.com
info@texwipe.com

Europe/Middle East
Skejby Nordlandsvej 307
DK-8200 Aarhus N
Denmark
Tel +45 87 400 220
Fax +45 87 400 222

Asia/Pacific
50 Tagore Lane
#02-01 Entrepreneur Centre
Singapore 787494
Tel +65 6468 9433
Fax +65 6468 6772