

CELLULOSE INSULATION

7404

 $(C_6H_{10}O_5)_n$

MW: 00.00

CAS: 9004-34-6

RTECS: FJ5691460

METHOD: 7404, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 March 2003

PROPERTIES: solid

SYNONYMS: cellulosic fiber loose fill thermal insulation. Cocoon.

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.45- to 1.2-um polycarbonate membrane, 25-mm; conductive cowl on cassette)	TECHNIQUE:	MICROSCOPY, SCANNING ELECTRON (SEM)
FLOW RATE:	1 L/min	ANALYTE:	Fibers, (manual count)
VOL-MIN:	N/A	SAMPLE PREPARATION:	Colloidal graphite paint/carbon disk planchet.
-MAX:		EQUIPMENT:	Scanning electron microscope
SHIPMENT:	Routine (pack to reduce shock)	CALIBRATION:	SEM performance standard
SAMPLE STABILITY:	Stable	RANGE:	Not determined
BLANKS:	2 or 10% field blanks per set	ESTIMATED LOD:	1 confirmed cellulose fiber above 95% of expected mean blank value
ACCURACY		PRECISION:	Not determined
RANGE STUDIED:	Not determined		
BIAS:	Not determined		
OVERALL PRECISION:	Not determined		
ACCURACY:	Not determined		

APPLICABILITY: This method is useful for the quantitative determination of airborne cellulose fibers during insulation installation [1].

INTERFERENCES: Non-fibrous cellulose. Very large cellulose fibers and cellulose fibers with convoluted shapes might interfere with fibers characterization. Other fiber types are typically rare and then only in trace amounts.

OTHER METHODS: The counting rules in this method were derived from Method 7400.

REAGENTS:

1. Colloidal Graphite.*

* (See SPECIAL PRECAUTIONS.)

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three- piece cassette with ca. 50-mm electrically conductive extension cowl and poly-carbonate filter, 0.45- to 1.2-um pore size, and backup pad.

NOTE 1: Analyze representative filters for fiber background before use. This is needed when field blanks contain fibers.

NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.

2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow rate requirements (see step 4), with flexible connecting tubing.
3. Microscope, scanning electron, operated at 15Kv with viewing screen having an inscribed or overlain calibrated scale.
4. Tape, shrink- or adhesive-.
5. Tweezers.
6. 25-mm carbon disk planchets.
7. Colloidal graphite paint.
8. Grounding wire, 22-gauge, multi strand.
9. Sputter coater.

SPECIAL PRECAUTIONS: Colloidal graphite contains isopropanol, which is flammable. Take precautions not to ignite it. Use only in well ventilated area.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward. Wrap joint between extender and monitor body with tape to keep the joint clean and prevent contamination when disassembled. Where possible, especially at low %RH, attach sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least 2 field blanks (or 10% of total samples, whichever is greater) for each set of samples. Remove top covers from field blank cassettes and store top covers and cassettes in a clean area (e.g., a closed bag or box) during sampling. Replace top covers when sampling is completed.
4. Sample at 1L/min.

NOTE: If the cellulose insulation is being applied dry, sample for a shorter period time than if the application is wet. Obtain two personal samples per worker. Sample at same flow rate but for different durations. A minimum duration of 1 minute is appropriate for very dusty environments. The longer duration should be determined by the overall dustiness, size of the area being insulated and other factors as well.

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to minimize jostling or damage.

NOTE: Do not use untreated polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

7. Mount the entire 25-mm filter directly on a carbon disk planchet by painting the planchet with a colloidal graphite paint and immediately laying the filter, glossy(sample) side up, on the planchet. The sample number is scratched into the back of the planchet prior to mounting the filter.
8. The planchet is then placed in a labeled petri dish and permitted to dry completely.
9. When dry, place sample in a sputter coater and deposit, following manufacturer's instructions, a heavy metal conductive coating on the sample.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturer's instructions. At least once daily use an SEM performance standard, such as the NIST traceable U1011. Record in log book the results of the examination of this standard.
11. If more rigorous magnification calibration is needed, use a diffraction grating replica.
 - a. Insert a mounted diffraction grating replica into the sample chamber.
 - b. Obtain a secondary electron image of the replica and measure the distance (mm) between the same relative position (e.g., between left edges) of two widely-separated lines on the grating replica.
 - c. Measure the distance separated lines on the grating replica. Count the number of spaces between the lines.
 - d. Calculate the true magnification(M)

$$M = \frac{(X)(G)}{Y}$$

where: X = total distance (mm) between the two grating lines;
 G = calibration constant of the grating replica (lines/mm)
 Y = number of grating replica spaces counted.

MEASUREMENT:

12. Use secondary electron detector(at ~15KeV) and scan the filter at low magnification (~100X). Observe the particulate loading. If the filter is not evenly loaded, it should not be analyzed (see note 4 below)
13. Adjust magnification to ~1200X and find the center of the filter using the X-Y manipulators. Fields are examined at regular intervals from the center of the filter along a traverse in one direction.
14. Determine the area of the viewing field at this magnification using the inscribed or overlain calibrated scale.
15. Count fibers in each field; distinguish, based upon morphology, between cellulose and other fiber types and make note of the relative proportion of fibrous to non-fibrous material in the field.
16. Counting rules: (Modified A rules, NIOSH Method 7400).[2]
 - a. Count any fiber longer than 5 µm (see note 2 below for exceptions)
 - i. Count only fibers longer than 5 µm. Measure and record length of fibers.
 - ii. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
 - b. For fibers which cross the boundary of the viewing field:
 - i. Use the X-Y manipulators as needed, to follow and measure the entire length of any fibers that meet the criteria of rule a above. Return to original viewing field before moving to the next field.
 - ii. Reject and do not count all other fibers.
 - c. Count enough viewing fields to yield at least 100 cellulose fibers. Count a minimum of 40 fields.
 - d. When selecting fields, ensure that fields do not contain fibers counted and measured from a previous field.

NOTE 1: When analyzing a viewing field, continuously scan a range of focal planes by moving the focus knob to observe and measure fibers which have do not lie flat on the filter.

- NOTE 2: This method allows for differentiation of fibers based on morphology. Cellulose fibers are easily distinguished from asbestos and glass fibers by morphology [3]. Do not count any fibers that have parallel sides.
- NOTE 3: Do not approach closer than 3 viewing fields from the edge of the filter.
- NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (during dry application), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. In extreme cases, much of the sample may be adhering to the cassette itself [4].

CALCULATIONS:

17. Calculate and report fiber density on the filter, E (fib/mm²), by dividing the average fiber count per viewing field, F/n_f , minus the mean field blank count per viewing field, B/n_b , by the viewing field area, A_f :

$$E = \frac{\frac{F}{n_f} - \frac{B}{n_b}}{A_f}, \text{fibers / mm}^2$$

18. Calculate and report the concentration, C (fib/mL), of cellulose fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm² for a 25-mm filter):

$$C = \frac{(E)(A_c)}{(V)(10^3)}, \text{fibers / mL}$$

19. Calculate and report the fiber length ranges(minimum and maximum) as well as the average length.

EVALUATION OF METHOD:

This method draws on both Methods 7400 for counting procedures and 7402 for instrumentation and setup. The major difference is the counting rules have been adapted from the A rules to allow for the counting and sizing of all cellulose fibers having a 3:1 or greater aspect ratio and a length of at least 5 microns. There are no diameter limits as cellulose fibers are truly 3 dimensional. This is illustrated in the figures below. The complex shapes and constantly varying diameters make even an approximate diameter determination impossible.

Sampling and analysis of both wet and dry cellulose insulation application has shown that there is much more uniform fiber deposition across the filter when the wet process is used. There is also much less fiber lost to the cassette walls.

REFERENCES:

- [1] NIOSH [2001]. Hazard evaluation and technical assistance report: Cellulose Insulation Applicators: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, NIOSH HETA Report No. 2000-0332-22827
- [2] NIOSH [1994]. Asbestos and Other Fibers by PCM: Method 7400. In: Eller PM, Cassinelli ME, eds. NIOSH manual of analytical methods, 4th ed. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [3] McCrone W and Dely J [1973]. "The Particle Atlas," Ann Arbor, MI: Ann Arbor Science.
- [4] Baron P, Deye G [1990]. "Electrostatic Effects in Asbestos Sampling," Parts I and II Amer. Ind. Hyg. Assoc. J., 51, 51-69.

METHOD WRITTEN BY:

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FIGURES:

The figures below are examples of cellulose insulation fibers and accompanying non-fibrous cellulose.

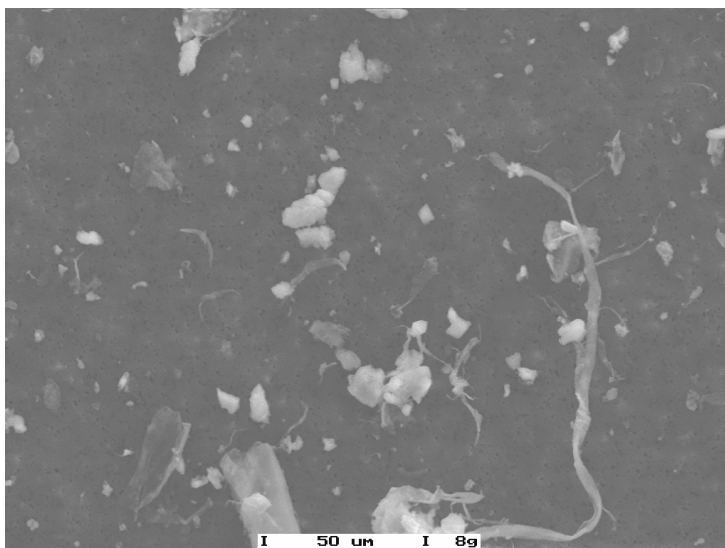


FIGURE 1. Figure shows many of the shapes and sizes of both fibrous and non-fibrous cellulose insulation.

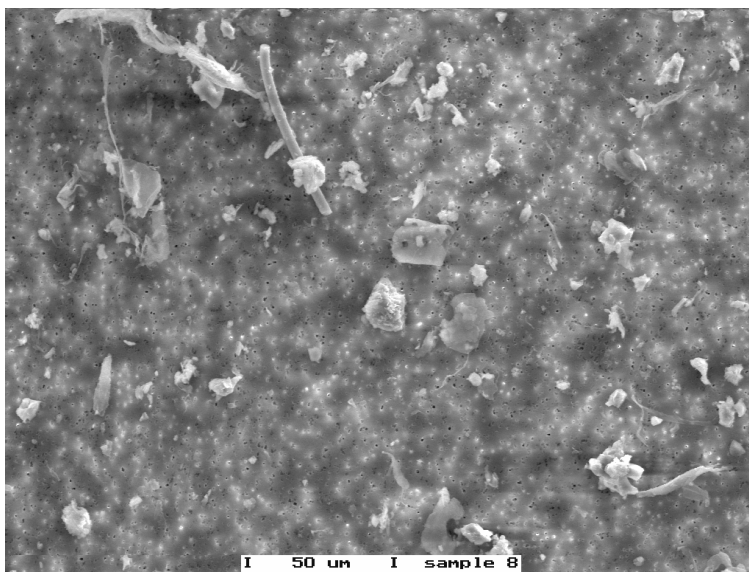


FIGURE 2. Figure shows the variety of shapes and sizes, as in Figure 1, as well as a glass fiber left over from the previous attic insulation.