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Field technique for clearing cellulose ester filters used in asbestos sampling

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A field procedure to clear cellulose ester filters used for fiber counting, without the need for boiling or heating acetone as prescribed in the National Institute for Occupational Safety and Health's Method #7400, is presented. The method is simple and requires only that the analyst place the filter wedges/halves on the microscope slide, weight the edges of the filter to prevent curling, place the preparation in a covered dish containing acetone, and allow the filter to clear at room temperature. Filters from previous Proficiency and Analytical Testing (PAT) samples were cleared utilizing the cold acetone clearing technique. Results obtained with the new technique were in good agreement with the reference values and well within the performance limits of the round. Filter clearing time varies, with a suitable result usually obtainable in approximately fifteen minutes. We believe this clearing technique can be substituted for the hot acetone clearing method, when necessary, without degrading analytical precision. **Jankovic, J. T.; Jones, W.; Clere, J.**: Field technique for clearing cellulose ester filters used in asbestos sampling. *Appl. Ind. Hyg. 3*:145-147; 1986.

Introduction

All determinations of airborne asbestos fiber concentrations for purposes of compliance with the Occupational Safety and Health Administration's permissible exposure limits for asbestos are made by phase contrast microscopic counts of fibers collected on a cellulose ester membrane filter. The method is referenced as *NIOSH Analytical Method P&CAM #239 Asbestos Fibers in Air.*[1] Method #239 specifies various parameters for microscope setup, fiber counting rules, and sample preparation; all of these are designed to standardize and improve the analytical reliability of the technique. Various fiber counting rules and techniques as well as their possible biases have been discussed elsewhere.[1-5] The mounting and clearing procedures contained in a method are crucial in determining the accuracy and precision of a fiber count. Method #P&CAM 239 utilizes a blend of dimethyl phthalate and diethyl oxalate in a one-to-one ratio by volume to which is added approximately 50 mg of membrane filter material to thicken the solution. This media does a fair job of clearing a membrane filter, although some graininess is usually evident. A greater problem exists due to fiber floatation and redeposition which results in a non-uniform fiber density. The likelihood of fiber migration is directly related to the analyst's mounting technique. Too much solution and the fibers are floated off the filter. Too little solution and the filter does not clear adequately.

On February 15, 1984, the National Institute for Occupational Safety and Health (NIOSH) issued analytical method #7400 for fiber counting. The new method introduced changes to P&CAM #239, which were designed to improve analytical sensitivity and reproducibility.[6] Some of the major changes included the use of (1) a 25 mm filter and cassette for sampling, (2) a Walton-Beckett graticule for counting, (3) a phase-shift test slide for microscope alignment, (4) modified Central Reference Scheme counting rules, (5) acetone and triacetin for clearing and permanently mounting filters.

The clearing of cellulose ester filters by the hot acetone/triacetin method is rapid and produces good clarity of background and contrast while minimizing the distortion of the filter and fiber floatation.[5] However, the method does require several pieces of equipment such as a hotplate and Guth-type flask, while heating the acetone has some inherent safety hazards. These considerations limit the hot acetone method to a well ventilated laboratory hood, leaving preparations for field examination still to be mounted by the non-permanent technique specified in P&CAM #239.

An alternative to heating the acetone vapor has been developed which allows one to utilize the chemical's useful properties while minimizing the inherent safety hazards.

Procedure

The technique involves placing a filter wedge, or half, on a microscope slide, weighting the edges of the filter to minimize curling, and placing the slide in a covered petri dish containing a small amount of acetone as shown in Figure 1. Acetone vapor clears the exposed por-

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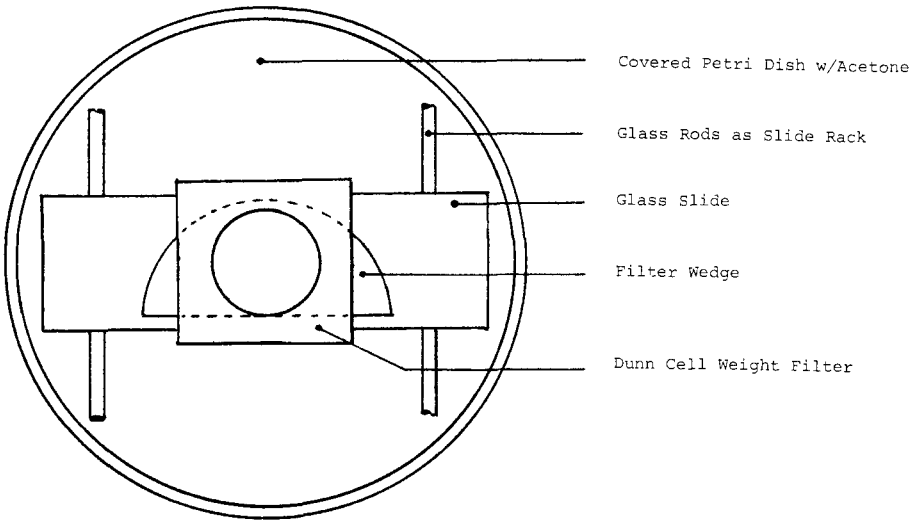

Figure 1—Cellulose filter clearing apparatus.

tion of the filter in approximately 10 to 15 minutes at room temperature, after which time the preparation can be trimmed and fixed with triacetin in preparation for microscopic observation.

Glass rods are placed in the petri dish to serve as a rack for the slide preparation in order to keep it above the acetone liquid. Some filters require a longer period to clear satisfactorily. This is believed to be related to the amount of particulate loading and the nature of the gaseous contaminants which may have been drawn through the filter during sampling. In such cases, the preparation can be inverted to speed the clearing once the preparation had adhered to the slide. A standard bacterial counting cell (Dunn Cell) is used to weight the edges of the filter, although a flat metal washer would work equally well for this purpose. The Dunn Cell is removed after the filter has cleared by applying a twisting motion to separate the cell from the filter. This will prevent the inadvertent separation of the cleared filter from the slide.

Prior to adding the triacetin and coverslip, any curled edges should be trimmed from the cleared filter with a scalpel. This will help to prevent trapping of air bubbles between the coverslip and preparation. While trimming, care should be exercised to prevent the cleared portion of the filter from breaking loose from the slide. Warming of the slide preparation, after mounting, can be used to both speed and enhance the clearing process.

Clearing a cellulose ester filter with acetone instead of the dimethyl phthalate/diethyl oxalate mounting solution offers the advantages of speed, clarity of the preparation, and little if any particle floatation and migration on the surface of the filter. Using acetone in a covered petri dish, as opposed to hot acetone vapor, reduces the safety hazard and makes the technique field worthy.

Discussion

As the modified clearing technique involves essentially the same process as the hot acetone method, one would expect no significant disagreement between counts made from preparations by either technique. To test this hypothesis, filters from six NIOSH Proficiency and Analytical Testing (PAT) rounds were mounted using the modified acetone/triacetin mounting technique and counted using the P&CAM #239 counting rules. An additional trial was conducted utilizing a laboratory-prepared set of exposed filters from which three sets of slides were prepared for counting utilizing hot acetone vapor, cool acetone vapor, and standard clearing solution.

Results

For the first trial, all counts were well within the performance limits specified for the PAT round (Table I). For the second trial, the mean of all three method counts was taken as the true reference value. The percent deviation from the mean was then calculated by subtracting the method specific count from the mean count and dividing the difference by the mean count (Table II). The regression of each individual clearing method about the mean count is also presented for purposes of comparison (Table III). The agreement between all three methods appears to be reasonable for optical counting when one considers that OSHA uses a sampling and analytical error estimate of +/− 0.25.

Recommendations

Clearing a cellulose ester filter with

TABLE I
PAT fiber count comparison with counts from cool acetone cleared PAT samples

	Fibers per mm²		
Filter no.	Reference value	Performance limits	Lab count
A-73-1	292.4	39.6– 778.4	527.8
A-73-3	361.0	70.5– 876.1	236.6
A-73-4	519.8	116.6–1218.0	430.6
A-77-1	912.0	252.4–1981.1	970.0
A-77-2	707.0	191.5–1547.6	540.5
A-77-3	174.2	19.9– 480.9	370.2
A-77-4	478.7	112.3–1099.5	481.0
A-79-1	181.4	29.4– 462.6	229.0
A-79-2	529.9	161.2–1111.5	891.0
A-79-3	274.2	33.1– 748.5	465.0
A-79-4	523.4	128.3–1185.4	833.0
A-80-1	934.5	352.6–1794.3	1013.0
A-80-2	198.8	61.7– 413.7	101.6
A-80-3	699.6	240.2–1398.7	620.0
A-80-4	370.9	126.5– 743.6	426.1
A-82-1	588.0	219.9–1133.6	439.0
A-82-2	900.0	353.8–1696.6	721.0
A-82-3	719.3	208.2–1537.4	844.0
A-82-4	143.0	28.8– 362.5	162.0
A-83-1	254.0	37.5– 663.0	262.6
A-83-2	839.2	311.8–1622.4	667.0
A-83-3	959.1	325.4–1927.2	1176.3
A-83-4	392.8	115.7– 834.0	351.8

TABLE II
Internal comparison of counts by clearing technique

Fibers per mm²			
Hot acetone	Cool acetone	Clearing sol'n	Mean
201.4 (−15.8)*	253.7 (+6.1)	262.6 (+9.8)	239.2
273.4 (−6.9)	255.9 (−12.9)	351.8 (+19.8)	293.7
608.5 (−15.9)	895.5 (+23.7)	667.6 (−7.8)	723.9
1190.2 (+2.6)	916.8 (−21.0)	1373.3 (+18.4)	1160.1

*|(Count-Mean Count) ÷ Mean Count | X 100

TABLE III
Comparison of regression equations by method

Method	m	b	r
Hot acetone	1.04	−61	0.9918
Cool acetone	0.80	95.5	0.9179
Clearing solution	1.15	−33.5	0.9833

X = Mean Count or True Value Y = Method Specific Count

acetone instead of the dimethyl phthalate/diethyl oxalate mounting solution offers the clear advantage of speed. Visually, the clarity of the preparations and absence of particle floatation and migration on the surface of the filters suggests an improvement over the standard clearing technique. However, no differences were demonstrated by the data. Using acetone in a covered petri dish, as opposed to hot acetone vapor, reduces the safety hazard and makes the technique suitable for field use.

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Drug and alcohol abuse costs employers over $164 million per day

The U.S. Chamber of Commerce estimates that drug and alcohol abuse costs employers $60 billion a year—the total tab for lost productivity, accidents, higher medical claims, increased absenteeism and theft of company property (the means by which many workers finance their drug habits). (Source: *Newsweek,* May 5, 1986, p. 50)