Applied Industrial Hygiene

ISSN: 0882-8032 (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/uaph20

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Paul Andrew Baronw & Geoffrey C. Pickford

To cite this article: Paul Andrew Baronw & Geoffrey C. Pickford (1986) An Asbestos Sample Filter Clearing Procedure, Applied Industrial Hygiene, 1:4, 169-171, DOI: 10.1080/08828032.1986.10390517

To link to this article: https://doi.org/10.1080/08828032.1986.10390517

An asbestos sample filter clearing procedure

PAUL ANDREW BARON[A] and GEOFFREY C. PICKFORD[B]
[A]National Institute for Occupational Safety and Health, 4676 Columbia Parkway, Cincinnati, OH 45226; [B]James Hardie Industries Limited, 65 York Street, Sydney, N.S.W. 2000, Australia

An acetone clearing technique for cellulose ester or nitrate membrane filters is presented. This technique has the advantages of increased safety and more consistent clearing than ones previously recommended in other methods. The technique involves the use of a heated metal block in which a small amount of acetone is rapidly vaporized and ducted to the filter. This prevents spattering of acetone liquid on the filter, reduces the amount of acetone required, and gives even and rapid clearing of the filter. Construction and operational details of the block and associated heating apparatus are presented. **Baron, P.A.; Pickford, G.C.:** An asbestos sample filter cleaning procedure. *Appl. Ind. Hyg. 4*:169-171; 1986.

Introduction

The method for analyzing filter sample concentrations for asbestos fibers involves chemically collapsing the cellulose ester or nitrate filter to provide a clear background against which to count the individual fibers with an optical microscope. The currently recommended method for accomplishing the filter clearing, both in the United States (NIOSH Method 7400)[1] and in a number of other countries,[2] is to use a stream of acetone vapor to clear the filter. This involves using a flask of boiling acetone to provide sufficient vapor to rapidly collapse the filter structure without distorting the dust deposit on the filter. This procedure has been criticized as being a potential fire hazard, especially in situations outside the laboratory. Another acetone clearing technique has been recommended by the U.K. Health and Safety Executive that consists of a boiling acetone reservoir with a condensing coil to prevent acetone escape.[3] This technique is safer but still uses a reservoir of hot acetone and requires the use of circulating water for cooling. A technique for clearing filters will be presented that allows rapid clearing with the use of a minimum amount of acetone liquid or vapor.

The basis for the technique is the rapid vaporization of a small amount of injected acetone liquid, approximately 0.20 ml, in a heated metal block. The vapor is then transported through the metal block and condensed on the filter. The condensation is rapid (the entire injection and clearing process takes less than 5 seconds) and occurs over the entire surface of the filter so that clearing is gentle and complete without shrinkage of the filter surface.

Equipment and procedures

Figure 1 shows the overall layout of the apparatus currently in use. An SCR voltage controller (Cole Parmer R-2603-00) provides power to two 2" × 5" 50 watt insulated heating pads (Cole-Parmer R-3125-20). The heating pads and a surface thermometer (Fisher 15-170-10A) are attached with silicone glue to a machined aluminum block. The cross section of the aluminum block is shown in Figures 2 and 3. The ¼-20 screw plugs the end of the connecting hole between the vaporization chamber and the condensation chamber to prevent vapor escape. An insulating Teflon plug with a small inlet allows the insertion of the tip of an adjustable micropipette (such as the Eppendorf Digital). Acetone is injected into the vaporization chamber directly below the Teflon plug. The vapor is ducted over to the filter where it condenses on the cooler filter and slide surfaces. The ducting has no direct path for liquid droplets to reach the filter while allowing the vapor to expand and diffuse so that the filter is exposed to a

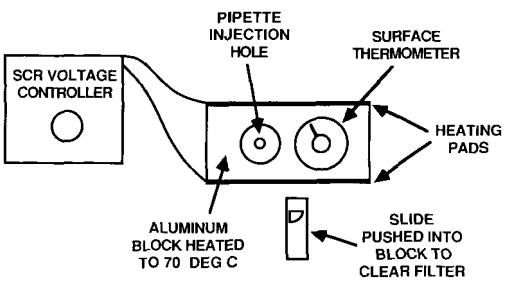

Figure 1—Diagram of filter clearing system.

Editor's note: One of the peer reviewers of this article routinely presents NIOSH Course 582, Sampling and Evaluating Air Evaluating Airborne Asbestos Dust. He was impressed with the potential of the technique described in this article and conducted his own evaluation of the procedure. His views are presented in the *Letters to the Editor* section of this issue.

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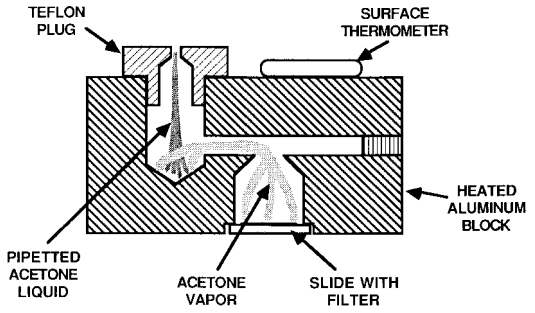

Figure 2—Cross section of heating block during operation.

uniform plume of acetone. A slot in the bottom of the block allows the analyst to push a slide with the filter on it directly under the condensation chamber. The height of the slot is sufficient to allow filters or filter segments that are not completely flat to enter without touching the top of the slot. Recessed areas in the base of the block reduce heat loss to the surface on which the block is resting.

The voltage controller is turned on to begin operation and set to maximum for about five minutes to reach the operating temperature of 70°C ± 5°C. Care should be taken not to allow the block to overheat during this period since the block will then require additional time to cool off. At this time, the controller can be set at the final voltage setting. This setting should be determined for each operational system and location but is approximately half the maximum voltage. When the block has reached operating temperature, filter clearing can begin. The block is placed on a smooth, clean, chemically unreactive surface (e.g., a glass plate) so that the slide holding the filter segment can be pushed in and out of the slot at the bottom of the block with ease. A slide with a filter or filter segment placed at the appropriate location is positioned at the entrance to the slot. A blank slide can be marked to act as a template showing the correct location of the filter segment.

When clearing one-fourth of a 25 mm filter, the pipette is loaded with 0.2 ml acetone and positioned at the hole in the Teflon plug. The slide is pushed into the slot and the acetone is then immediately injected into the vaporization chamber. Note that if the uncleared filter is exposed to the heat longer than several seconds, it will start to curl up and the cleared filter may be distorted. The pipette is kept in the hole in the Teflon plug to prevent acetone vapor from being released through the injection hole. The slide is left in the slot for approximately 3 to 5 seconds to allow complete clearing of the filter and is then removed.

Discussion

The design details of the apparatus seem to be open to modification as long as the basic considerations of safety and even exposure of the filter to acetone vapor are observed. The use of a temperature controller and thermistor sensor has been used to give better temperature control and eliminate the need to watch the device during warm-up. However, since the temperature of the aluminum block does not seem to be particularly critical, the added expense of temperature control may not be warranted. The apparatus has been operated at temperatures between 65° to 75°C with good results. At higher temperatures, the acetone boils more violently (and may even decompose) upon injection, and liquid droplets can impact on the surface of the filter. At lower temperatures, the acetone tends to stay on the filter and slide for a longer time, and the surface tension of the liquid draws dissolved filter material and particles away from the edge of the filter.

The amount of acetone needed for complete and consistent clearing varies with the size of filter segment being cleared. An injection of 0.15 to 0.30 ml works well for one quarter of a 25 mm filter. For larger or smaller segments, the amount should be increased or decreased and tested on blank filter segments.

It was found that one batch of filters produced cleared filters that were somewhat mottled in appearance. For this reason, it is suggested that filters from each batch be tested before sampling for asbestos.

Experience with hundreds of filters cleared with this technique has produced samples that were consistently flat, unwrinkled, fully cleared, and without air bubbles. This was not the case using the previous method, with which problems in these areas often necessitated preparation of additional slides.

After clearing the filter, final preparation of the sample involves covering the filter with triacetin and a cover slip, then waiting overnight to allow maximum clearing of the filter surface. An additional technique to speed up the preparation of acetone-cleared asbestos filter samples has recently been described.[2] This technique involves heating the acetone-fixed, triacetin-covered filter for 15 minutes at 50°C to speed the clearing process. If only a few slides are being prepared, it is possible to place the triacetin covered sample slide on the aluminum block to obtain the increased clearing speed. The combination of the flash vaporization clearing technique and the one described here will allow safer and more rapid sample preparation of asbestos filter samples.

Precautions

Note that in spite of the reduction of acetone vapor release into the environ-

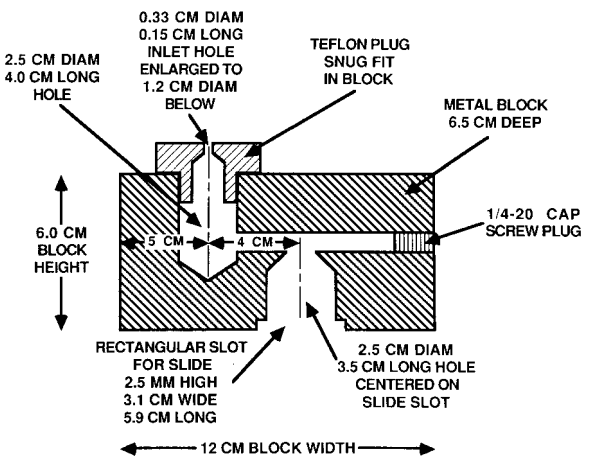

Figure 3—Dimensions of heating block.

ment with this technique, the presence of liquid acetone is still a fire hazard and should be treated with a great deal of respect. If this technique is performed outside of a laboratory, it should be carried out in a well ventilated area in the absence of flames and spark sources. The volume of acetone liquid in current use should be kept to a minimum (less than 20 ml) and the liquid kept covered when not in immediate use.

Several considerations need to be observed if other heating systems are used. The voltage or temperature controller should be solid state to eliminate the high temperature and sparking of contacts in non-solid state controllers. The heating element should be well insulated electrically in order to prevent shorting to the metal block and be in good contact with the block surface to prevent overheating of the element. The wires should also be well insulated and secure to reduce electrical shock hazard to the operator. In areas where only 240 V line power is available, a reduced voltage control system is recommended.

Recommendations

The use of flash vaporization to limit the amount of acetone consumed during filter clearing provides a significant safety advantage. In addition to the increased safety, the wider and more homogeneous plume of acetone vapor impinging on the filter clears the filter in a more complete and consistent fashion than methods previously recommended. It is recommended that this technique be included in a future revision of NIOSH Method 7400 and in other methods involving cellulosic membrane filter collapse for sample preparation.

Disclaimer

Mention of commercial products does not constitute endorsement by the National Institute for Occupational Safety and Health.

References

1. **NIOSH**: Fibers, Method 7400 (Revision 1). *NIOSH Manual of Analytical Methods*, 3rd ed. NIOSH, Cincinnati, OH (1985).
2. **Asbestos International Association**: *Reference Method for the Determination of Airborne Asbestos Fibre Concentrations at Workplaces by Light Microscopy.* AIA Health and Safety Publication, Recommended Technical Method No. 1 (1979).
3. **Health and Safety Executive**: *Method for the Determination of Hazardous Substances 39. Asbestos Fibres in Air.* HSE, Occupational Medicine and Hygiene Laboratory, London (1984).

Received 1/02/86; review/decision 2/11/86; revision 4/22/86; accepted 5/01/86

Ford-UAW National Joint Committee on Health and Safety

Announcement of request for proposals

The Ford Motor Company and the United Auto Workers Union have established a jointly-administered fund of $750,000 for occupational health and safety research. The Ford-UAW National Joint Committee on Health and Safety is requesting proposals by scientific investigators for studies of health effects and control of exposures which arise from processes in UAW-represented facilities of the Ford Motor Company.

Ford Motor Company facilities include foundry, forge, stamping, machining, and plastics fabrication plants, as well as vehicle assembly plants, and use a wide variety of industrial processes and chemicals. The areas of interest for which proposals are requested are:

1. Injury epidemiology;
2. Auditory and other effects of high noise environments;
3. Systematic review of mortality data;
4. Neurological effects of solvent exposure;
5. Techniques for control of
   (a) formaldehyde emissions in foundry coremaking,
   (b) vehicle emissions in drive-off areas in vehicle assembly plants, or
   (c) cutting fluid mist and vapor exposure in machining operations;
6. Design parameters and evaluation of effectiveness for supplied-air islands;
7. Design of protocols for evaluation of effectiveness of high velocity-low volume exhaust systems;
8. Ventilation requirements for confined spaces.

Studies may include laboratory and field phases. The Ford Motor Company and the UAW will cooperate in making facilities and information available to investigators for the conduct of this work.

Prospective applicants should indicate which of the research areas listed above they are interested in and obtain the full text of the relevant Request for Proposal from either John J. Whelan (Co-Chairman, National Joint Committee on Health and Safety, Ford Motor Company, World Headquarters, Room 367, The American Road, Dearborn, MI 48121) or William M. White (Co-Chairman, National Joint Committee on Health and Safety, Ford Department, United Auto Workers, 8000 East Jefferson, Detroit, MI 48214). Applications should be received by November 21, 1986.