# Advances in Environmental Measurement Methods for Asbestos

Michael E. Beard and Harry L. Rook, editors

ASTM STP 1342

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ASTM Stock Number: STP1342

ASTM
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959
Printed in the U.S.A.

**Library of Congress Cataloging-in-Publication Data**

Advances in environmental measurement methods for asbestos / Michael E. Beard and Harry L. Rook, editors.
p.cm. — (STP; 1342)
"ASTM Stock Number: STP1342."
Papers presented at a symposium held July 13-17, 1997 in Boulder, Colorado.
Includes bibliographical references and index.
ISBN 0-8031-2616-6
1. Asbestos dust—Measurement—Congresses. 2. Asbestos fibers—Measurement—Congresses. 3. Asbestos—Analysis—Congresses. I. Beard, Michael E., 1940- II. Rook, Harry L. III. ASTM Committee D-22 on Sampling and Analysis of Atmospheres. IV. ASTM special technical publication; 1342.

TD887.A8 A34 2000
666'.72—dc21 99-055148



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Printed in Philadelphia, PA
January 2000

# Foreword

This publication, *Advances in Environmental Measurement Methods for Asbestos,* contains papers presented at the symposium of the same name held 13–17 July 1997 in Boulder, Colorado. The symposium was sponsored by ASTM Committee D-22 on Sampling and Analysis of Atmospheres, and by the Environmental Information Association. The conference chairmen and co-editors of the publication were Michael E. Beard, Consultant, Raleigh, North Carolina, and Harry L. Rook, National Institute of Standards and Technology, Gaithersburg, Maryland.

# Overview

ASTM Committee D 22 on Sampling and Analysis of Atmospheres sponsors a variety of conferences and seminars to promote the exchange of information about monitoring various constituents and properties of air. One such conference is held periodically on the campus of the University of Colorado in Boulder and is known as the ASTM Boulder Conference. The 1997 ASTM Boulder Conference on Advances in Environmental Measurement Methods for Asbestos was held 13–17 July 1997 at the University of Colorado. This conference was co-sponsored by ASTM Committee D-22 and the Environmental Information Association.

The purpose of the conference was to focus on recent advances in research on measurement methods for asbestos in bulk building materials, as well as ambient, indoor, and work place air, water, and settled dust. The program included discussion of measurement methods, monitoring strategies, data interpretation, and quality assurance for asbestos measurements. It was the intent of the program to bring the disciplines of analytical chemistry together with investigators who are assessing exposure to asbestos in the environment and to promote better understanding of their mutual interests, needs, and limitations. The papers presented at the conference have been subjected to peer review, and those accepted are published in this ASTM Special Technical Publication.

Asbestos is a useful material and has been used as a component of many building materials. However, when asbestos fibers become airborne and are inhaled they may produce adverse effects such as asbestosis, lung cancer, and mesothelioma. The U.S. Environmental Protection Agency, the Occupational Safety and Health Administration, and various state and local governments have issued regulations to control exposure to the asbestos fibers. These governmental units have also named analytical methods and procedures that must be used to be in compliance with the regulations. These compliance methods address monitoring asbestos in drinking water, building materials, and in workplace and ambient air.

There are also asbestos-monitoring interests where no government regulation has been promulgated. Such an interest is asbestos in settled dust. While government regulations generally address visible deposits of dust in areas where asbestos-containing materials have been identified, there have been no analytical methods for sampling and analysis of asbestos in this medium. Likewise, there are no regulatory monitoring or control strategies other than requiring that all visible dust should be cleaned. ASTM has addressed these needs by developing draft methods for asbestos in settled dust, and two have become ASTM standards (D 5755: Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations; and D 5756: Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration).

Analytical methods are constantly being reviewed and revised by users to meet new or special analytical needs. ASTM methods are subject to this review process and are required to be re-approved every five years. Governmental compliance monitoring methods for asbestos have proved to be more difficult to amend. Although regulations require periodic review, technical improvements may not be adopted because they may increase the cost of the analysis and thus the burden to the public. While the government will accept results from a more stringent analytical procedure, they are reluctant to require procedures considered burdensome to the public. Where there is no standard method or governmental compliance monitoring procedure, the analytical needs are filled by the so-called "state of the

art procedure." These procedures are commonly used by laboratories to meet the demanding analytical requirements of a wide variety of materials submitted for analysis.

While these "state of the art procedures" may ultimately become the practice of all and be incorporated into the regulations, their adoption may lag in meeting the immediate monitoring needs of the analyst. It is this need that the 1997 ASTM Boulder Conference addresses. Many asbestos-monitoring techniques have been developed for problem materials such as vinyl asbestos floor tiles, bulk samples with less than 10% asbestos content, and asbestos in settled dust. The goal of this conference was to provide a forum for these state-of-the-art improvements and to have them published for wider distribution and dissemination. This Special Technical Publication will provide documentation of this forum and serve as a guide for monitoring asbestos using improved analytical techniques. This publication will be especially useful to those unable to attend the conference and as a foundation for those who are continuing research to meet these analytical needs.

The Conference was organized into technical sessions dealing with four measurement areas: (1) Measurement Methods for Asbestos in Bulk Building Materials; (2) Measurement Methods for Asbestos in Ambient, Indoor, and Workplace Air; (3) Measurement Methods for Asbestos in Water; and (4) Measurement Methods for Asbestos in Settled Dust. Papers describing analytical methods, monitoring strategies, and quality assurance procedures were presented and discussed.

The session on Methods for Asbestos in Bulk Building Materials included discussions concerning polarized light microscopy (PLM), X-ray diffraction (XRD), and transmission electron microscopy (TEM) techniques for analysis of these materials. The performance of regulatory methods in the analysis of a variety of bulk building materials, soils, and paints was presented and discussed. Shortcomings of the regulatory procedures were highlighted, and research to develop improvements, especially for the 1% regulatory statute, was presented.

The session on Asbestos in Ambient, Indoor and Workplace Air included presentations on OSHA, EPA, and ISO methods for monitoring airborne asbestos by either phase contrast microscopy (PCM) or TEM. Interesting research on techniques for determining fiber length/diameter distributions and the depth of penetration of fibers into membrane filters were also presented.

The session on Measurement Methods for Asbestos in Water reviewed EPA and American Water Works Association methods for asbestos in drinking water and research on improved sample preparation techniques. These small fibers dictate the use of TEM for analysis. This session also includes the editor's choice for most interesting title in the conference, namely "Sludge, Crud and Fishguts: Creative Approaches to Non-Standard Asbestos Water Analysis." This title epitomizes the innovative spirit and talent that analysts must exercise in dealing with a wide variety of environmental monitoring needs.

The final session on Measurement Methods for Asbestos in Settled Dust was perhaps the most controversial session in the conference. Analytical methods employing TEM developed by ASTM Subcommittee D 22.07 for monitoring asbestos in settled dust and monitoring strategies and results were presented. Many asbestos in settled dust monitoring efforts have required litigation for final interpretation of datasets. Some of the presentations in this session exemplify the diversity of opinions in this area. Additional studies are needed in this field to determine the effect of human, mechanical, and natural activity on generating asbestos aerosols from settled dusts. Research is also needed to better define the quantity of airborne asbestos that constitutes an environmental exposure hazard.

The 1997 ASTM Boulder Conference on Advances in Environmental Measurement Methods for Asbestos served as a focal point for issues related to the needs for improved monitoring techniques for asbestos. This ASTM Special Technical Publication will serve as a

documentaiton for our collective understanding of these issues as they were at the time of the conference. It is hoped that the papers published here will guide others in understanding these monitoring issues and lead to research for further improvements for us all.

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# Measurement Methods for Asbestos in Bulk Building Materials

Robert L. Perkins[1]

**Analysis of Asbestos in Bulk Materials--1980 to 1997**

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**REFERENCE:** Perkins, R. L., **"Analysis of Asbestos in Bulk Materials—1980 to 1997,"** *Advances in Environmental Measurement Methods for Asbestos, ASTM STP 1342,* M. E. Beard and H. L. Rook, Eds., American Society for Testing and Materials, West Conshohocken, PA, 2000.

**ABSTRACT:** Federally sponsored asbestos proficiency testing programs have operated continuously in the United States since 1980. The U. S. Environmental Protection Agency published a test method for the analysis of asbestos in bulk materials in 1982, commonly referred to as the "Interim Method." Major revisions were made to the method and the revised version was published in 1993. Polarized light microscopy , supplemented by x-ray diffraction, is the primary analytical technique presented in the 1982 and 1993 test methods. The 1993 version of the test method also recommends additional analytical techniques such as gravimetric sample reduction, transmission electron microscopy, and the use of bulk calibration standards. Research Triangle Institute's more than 17 years' experience evaluating the test methods and characterizing thousands of proficiency testing samples for testing programs indicates that the available analytical techniques can provide very accurate results for qualitative analysis of asbestos-containing materials and reasonably accurate results for quantitation of asbestos concentrations. The quality of the results being produced by the asbestos laboratory community appears to be most influenced by the skill level of the analysts and the degree of employment of the available analytical techniques.

**KEYWORDS:** proficiency testing, test method, polarized light microscopy, quantitation, laboratory performance

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## Introduction

Federally sponsored proficiency testing (PT) programs for asbestos laboratories have operated continuously since 1980 when the U. S. Environmental Protection Agency's (EPA's) Bulk Sample Quality Assurance Program was initiated. The EPA program was replaced by the National Institute of Standards and Technology's (NIST's), National Voluntary Laboratory Accreditation Program (NVLAP) in 1989 as mandated by

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the Asbestos Hazard Emergency Response Act (AHERA) [*1*].

There were approximately 100 laboratories enrolled in the initial round of the EPA program in 1980 and approximately 1 100 enrolled in the 18th and final round conducted in 1988. Enrollment in the NVLAP has ranged from 661 laboratories in the initial test round conducted in 1989 to a high of 707 laboratories in 1990. There are currently 350 laboratories enrolled in the program. In addition to the NVLAP, Research Triangle Institute (RTI) conducts two other PT programs for polarized light microscopy (PLM) asbestos laboratories, namely the American Industrial Hygiene Association (AIHA) program with 280 laboratories and the U.S. Navy program with 87 laboratories. There have been significant changes in the enrollment in these three programs (Figure 1) over the past several years.

## The Test Method

Although PT of asbestos laboratories was initiated in 1980, the EPA test method, the so-called Interim Method [*2*] , was not published until 1982. This method designated PLM as the method of choice for analysis of asbestos in bulk building materials; the method also included a section on analysis of asbestos using x-ray diffraction (XRD) as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other analytical methods.

The revised EPA test method was published in 1993 [*3*]. PLM, supplemented by XRD, is the primary analytical technique presented in the revised method. The revised method was expanded from the 1982 version to include additional analytical techniques such as gravimetric sample reduction, transmission electron microscopy (TEM), and the formulation and employment of bulk asbestos calibration standards.

## Effectiveness of the EPA Test Method

As stated previously, the primary analytical technique for analysis of asbestos in bulk building materials is PLM. After nearly 20 years of employing this analytical technique for the analysis of asbestos, RTI is in a position to comment on the adequacy of the method and the ability of asbestos laboratories to successfully utilize the test method.

### *Qualitative Analysis*

In addition to extensive evaluation by EPA, the test method has been utilized to characterize test samples used in the various PT programs for the past 17 years. Unlike analytical methods that rely on sophisticated equipment to provide analytical results, PLM depends greatly on the skill and experience of the analyst to provide accurate and complete results. Qualitative analysis of asbestos in bulk materials requires that the analyst be able to accurately determine the optical properties of fibrous particles. Accurate measurement of refractive indices (RIs), angles of extinction, birefringence, and so on is necessary to preclude the occurrence of false positives attributed to incorrect identification of asbestos look-alikes such as polyethylene fibers, wollastonite, and fibrous talc.

The combination of low asbestos concentration, small fiber size, and interfering

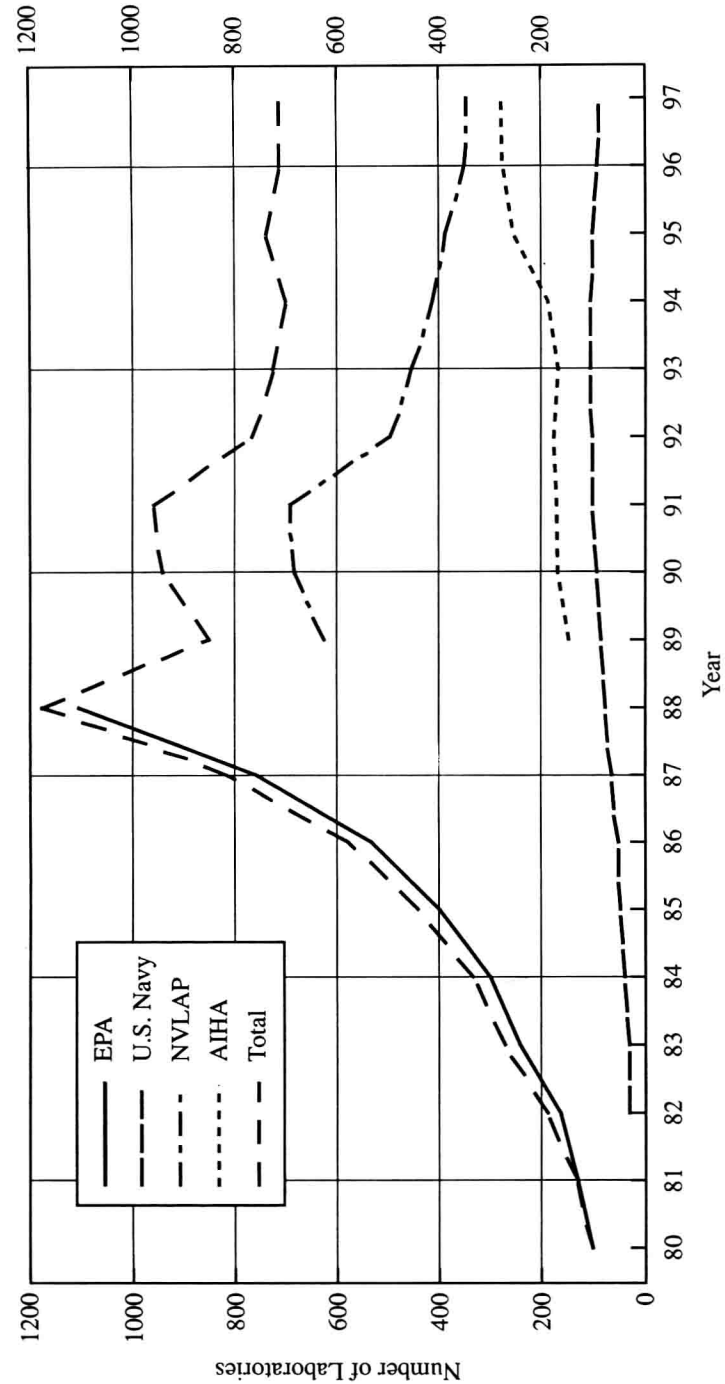


Figure 1 - *Average annual laboratory enrollment, by program.*

binder/matrix causes some bulk materials to be very difficult to analyze by PLM. Analysis of such samples may be facilitated by employment of additional analytical techniques such as gravimetric sample reduction by ashing and/or acid washing. Samples such as vinyl flooring materials, asphaltic roofing materials, and plasters may be gravimetrically reduced. This procedure removes the interfering matrix/binder and concentrates the asbestos, providing greater opportunity to detect and verify it.

The detection limit for PLM asbestos analysis is sample-dependent, but for the majority of bulk samples, this value is <1 %. RTI analysts have never failed to detect asbestos in a positive sample (with negative samples verified by TEM).

*Quantitative Analysis*

Although PLM is a very effective method for the qualitative analysis of bulk materials, quantitation of asbestos content using this technique is less certain. The only options available to the PLM analyst for determining the asbestos concentration are: 1) visual estimation and 2) point counting.

Visual estimation may be performed using a stereomicroscope at 10-40x magnification. The analyst estimates the relative volume proportions of asbestos and matrix components, resulting in an asbestos concentration expressed as a percent volume. Visual estimation may also be performed on slide mounts using PLM, resulting in an asbestos concentration expressed as a percent area.

Point counting using PLM is a systematic procedure that involves traversing a slide mount and recording the type of particle(s) directly under the intersection of the reticle cross lines or the points of the Chalkley point array. A minimum of 400 occupied points is recommended for each sample. The asbestos concentration determined by this technique will be a projected area percent concentration.

There are shortcomings to each of these techniques. Visual estimation, with stereomicroscopy or with PLM, is subject to analyst bias. Test results submitted by laboratories enrolled in the PT programs directed by RTI indicate a continuing tendency of laboratories to overestimate the asbestos content and also a continuing reliance by most laboratories on visual estimation for determining asbestos concentrations. Visual estimation is a viable quantitative technique only if: 1) asbestos fibers/bundles are visible by microscopy and 2) the analyst has been "calibrated" through the use of asbestos standards or reference materials containing known concentrations of asbestos. Not only should the analyst receive training with such materials, but reference materials should be included in the quality assurance/quality control (QA/QC) program as blind samples. This provides documentation of analyst bias and also information on the accuracy and precision of the analyst's quantitative results.

Results obtained by point counting should not be affected by analyst bias as greatly as results obtained by visual estimation if the technique is performed properly. As with quantitation by visual estimation, asbestos fibers/bundles must be visible,( i.e., the microscope set up should maximize fiber visibility). As is the requirement for all quantitative techniques using microscopy, the sample should be homogeneous to ensure that the small subsamples used for the quantitative procedure(s) are representative of the total sample. Assuming optimum conditions, an individual analyst's precision, and the

precision between and among analysts, should be very good. As would be expected, precision and accuracy should increase with the increasing number of points counted.

Point counting precision and accuracy may be illustrated (Table 1) by examining the results produced from the point counting of formulated (by weight percent) asbestos calibration standards by two experienced analysts. The following observations may be made about these point-counting data: 1) the concentration of chrysotile determined by point-counting tended to be lower than the true weight-percent concentration; 2) the concentrations of amphibole asbestos types tended to be higher than the actual weight-percent concentrations; 3) point counting of samples containing less than 1 % chrysotile always resulted in mean values of less than 1 %, and 4) the point counting values for samples containing less than 1 % amosite were always 1 % or greater. These relationships are explained in the discussion that follows.

Quantitative results may also be biased by sample composition and relative particle thickness [*4*]. Building materials may contain a variety of components having a wide range of densities. For example, a sample could contain cellulose (specific gravity of 0.9), perlite (0.4), and chrysotile (2.6). The asbestos concentration determined for such a sample would be expected to be biased low (as compared to the true weight-percent concentration) because of discrepancies in the relative volumes of the components.

Visual area estimates and point counting are really measurements of the relative projected areas of particles as viewed on a microscope slide. Such estimates may be biased by differences in particle thicknesses. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clays or calcite, the asbestos concentration measured by the projected area (and volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin asbestos bundles/fibers, the asbestos content would likely be overestimated. It is apparent from these two simple examples that particle thickness is an important factor when relating area percent to volume percent and that the ideal situation would involve quantitation of materials having components of uniform particle thickness and similar densities. It is recommended that asbestos concentration be reported as volume percent, weight-percent, or area percent depending on the method of quantitation used. A weight percent concentration **cannot be determined** without knowing the relative densities and volumes of the sample components.

As stated previously, the employment of gravimetric sample reduction may greatly improve quantitative results. Difficult samples such as floor tiles, plasters, and roofing materials may be reduced greatly (greater than 85 % for some samples) by ashing and/or acid washing. Removal of interfering matrix material, resulting in a concentration of the asbestos, should greatly improve the accuracy of quantitative results.

There are some sample types for which the asbestos concentration cannot be determined adequately by PLM, (e.g., vinyl floor tiles). Although ashing followed by acid washing generally removes 80 % or more of the sample, the remaining residue usually contains a considerable amount of titanium dioxide ($TiO_2$), a pigment material that coats the asbestos fibers very effectively. This negates quantitation by PLM because few fibers are visible. For such samples, analysis by TEM or XRD is recommended.