

ASBESTOS IDENTIFICATION AND MEASUREMENT

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PROCEEDINGS OF A TOPICAL SYMPOSIUM
APRIL 9, 1979
DES PLAINES, IL.

Sponsored by
THE AMERICAN CONFERENCE OF GOVERNMENTAL INDUSTRIAL HYGIENISTS



The American Conference of Governmental Industrial Hygienists
Cincinnati, Ohio

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The ACGIH, as it is invariably called, is a professional society whose members are primarily industrial hygienists employed by Federal, State, and local governments and universities. Eligible for associate membership are those working for governmental bodies in related activities such as safety. Technical and student memberships are also available. An annual meeting is held as part of the American Industrial Hygiene Conference.

The principal work of the organization is carried out by committees. The best known of these are the Committee on Industrial Ventilation and the Committee on Threshold Limit Values for Chemical Contaminants. The former committee publishes *Industrial Ventilation—A Manual of Recommended Practice*, which is revised biennially, and the latter publishes the annual list of TLV's, with a separate publication documenting the values. The Committee on Air Sampling Instruments publishes and periodically revises their publication, *Air Sampling Instruments*. The sales of publications from these committees supports their activities and also the activities of the many other ACGIH committees, covering virtually every aspect of occupational health.

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ASBESTOS IDENTIFICATION AND MEASUREMENT

April 9, 1979
Des Plaines, IL.

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**THE AMERICAN CONFERENCE
OF
GOVERNMENTAL INDUSTRIAL HYGIENISTS**

David M. Trayer, Chairman
James S. Ferguson
Symposium Program Chairman

Compilation and copy editing by Debra Pinkston
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Asbestos Identification—Proceedings of an ACGIH Topical Symposium 4/9/79.

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Welcoming Remarks

LEE SALTSGAVER

Director, OSHA Training Institute, Des Plaines, IL

I want to take this opportunity to welcome all of you to our Training Institute.

I take it that most of you are members of what we call the "private sector", and I want to let you know that this Institute is open to you at all times. You can come in and take advantage of our library, which is growing by leaps and bounds. I'm sure that from time to time, particularly for those of you who are local, you might find it advantageous to come to our library for research purposes or to talk with any of our instructors or supervisors here at the Institute. In fact, if you get

more than one person into a group, we would be very happy to take you on a conducted tour of the Institute.

I do want to add that we are very happy to cooperate with ACGIH in the presentation of this symposium, and I'm sure that it's going to prove extremely beneficial to everyone concerned.

Again, I want to welcome you all. Our telephone number is 353-2500. We also have a local commercial line—312/293-4810. Any questions you might have? Very good. And, again, welcome!

Opening Remarks

DAVID M. TRAYER

Tennessee Valley Authority Chairman,

The American Conference of Governmental Industrial Hygienists

Good morning. My name is David Trayer, and I am Chairman of the American Conference of Governmental Industrial Hygienists. We welcome you to the ACGIH Symposium on Identification and Measurement of Airborne Asbestos.

We certainly appreciate Jim Ferguson, the Program Chairman, for his efforts in putting the program together. It was Jim, in fact, who first suggested this symposium to ACGIH. We also wish to thank the speakers and participants in today's program and Bill Kelley, Executive Secretary of ACGIH, and Pat Powell, of the ACGIH office staff, for the many arrangements they have handled.

We're especially grateful to the OSHA Training Institute for providing these fine meeting facilities and to Lee Saltsgaver and Tony Touhy of the Training Institute for the great assistance they have provided.

Why is ACGIH sponsoring a symposium on the subject of identification and measurement of airborne asbestos? As a practicing industrial hygienist with responsibilities to protect the health of people who work with asbestos, I can relate to that question. To me it is very important that I have a reliable, practical, and if possible, a rapid and economical method of distinguishing airborne asbestos fibers from other airborne fibers and of sampling and counting air samples to determine worker exposure. The ability to identify conclusively the various chemical and crystalline species known generically as "asbestos" is also of growing importance.

This, along with an effective quality assurance

program to assure final data reliability, is what I need. And I believe that many of you here today have similar interests.

However, serious problems arise in the details of how to achieve this goal. We encounter the problem of dispersion staining versus electron microscopy versus X-ray diffraction. We have to weigh scientific reliability against cost and the time required to obtain results. Will analytical methods that work well for bulk samples also work for airborne samples? Are some changes needed in the standard procedures?

In an attempt to resolve some of these problems, ACGIH felt it was important to bring together some of the leading authorities and specialists in the field.

We like for all ACGIH symposia to be forums for open and free expression. We sincerely invite you to do that. Proceedings will be published and sent to each of you.

James S. Ferguson will chair the program today. Jim is Deputy Director of the NIOSH Division of Training and Manpower Development. Also, he is a microscopist who has suffered from the ravages of the Peter Principle. In fact, he says he has had "the green one" several times and has now risen to the level of his own incompetence.

However, those of us who know Jim, know better, don't we? Thousands of industrial hygienists know Jim Ferguson as a highly competent scientist, industrial hygienist, and educator. It is my pleasure to present him to you. He will outline the objectives of the symposium and introduce the speakers.

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Objectives of the Symposium

JAMES S. FERGUSON

Deputy Director of the Division of Training and Manpower Development, NIOSH,
Cincinnati, OH

Thank you all for coming. I would like to give you a little history of how we came to have this symposium and in the course of the introductions of the speakers, I think you will find out even more about them, what their opinions and ideas are and how they feel about the subject that we are going to talk about today.

Of course we couldn't have a more timely subject, I don't think, to discuss than asbestos; the question is how much longer will it be a timely subject and how much discussion are we going to put into it.

In recent years, we have become much more aware of what some of the problems are, related to asbestos, and we have become much more attentive to various techniques for identifying it. NIOSH has prescribed to OSHA a method which they published in the *Federal Register* as part of the standard technique for counting asbestos fibers, at least those assumed to be asbestos, from air samples. The question of the validity of this method, the reproducibility of it, the equipment used, the operator errors inherent with it and so on have been much cussed and discussed.

There are many techniques for both identifying and evaluating airborne asbestos as well as asbestos from water samples, from food samples, from bulk samples, from almost any source that you care to name.

We didn't know when we planned this symposium, we had no idea that the recent flurry of activity would begin relative to asbestos identification and whether schools have asbestos in them as insulation or not.

EPA has recently announced a program relative to determining whether or not the insulation contains asbestos and then what to do about it, if it does. The information discussed here today is very relevant to this particular problem.

There may be some very intensive opinionated discussions here; I encourage you to state your own opinions and, during the various panel discussions, to ask questions of the panel members. We do have periods set aside for questions and answers, and we hope to hear from all of you.

The speakers are going to be telling you about how they identify asbestos, and perhaps some of their opinions and experiences on other matters related to asbestos. Some are not laboratory workers as much as people who want to discuss the overall problems of identifying asbestos and what the problem of exposures to it might be.

Recently, Dr. Walter McCrone, one of our speakers today, held one of his biennial microscopy meetings. In the United States, these symposia are held in even-numbered years here in Chicago, in the odd-numbered years in England or elsewhere in Europe. The subject of these meetings is microscopy of all types. Last summer

the regular biennial microscopy meeting was held here in Chicago and at that time Walter and I put together a brief presentation with other speaker together a brief presentation with other speakers related to the asbestos problem.

We realize that there are many problems associated with identification of asbestos. Anyone who espouses a technique has many critics of that technique. In many cases, there are more critics than there are proponents of the technique, so that has to tell you something. I think the validity of an analytical procedure can be measured by the number of people who criticize it—it's easy to criticize but not so easy to come up with something better.

In discussions at the meeting in Chicago we talked about the problems from the regulatory side and from the industrial side. There was a representative from the National Crushed Stone Association. We had a representative from the OSHA Solicitor's Office and NIOSH. In addition, Dr. McCrone and Ian Stewart of McCrone Associates, who is also here with us today, talked about identification techniques. So we covered the subject thoroughly, but I don't think we really solved any of the problems associated with identification of asbestos.

Very recently Bill Kelley, the Executive Secretary of ACGIH, called me to say that he had heard from an individual at the Navy Department regarding difficulties in identifying asbestos. The Navy, of course, has been beset recently with considerable problems concerning asbestos. It's not so much a problem of identifying it. They know it's there. The problem is how much of it is there when workers go into ships to repair and recondition them. The Navy was finding that the NIOSH method of evaluating airborne asbestos did not really suit their particular problem or needs.

NIOSH did not originally intend to set it up as a qualitative technique, but it's an evaluation technique in which you assume the fibers there are all asbestos. This was not formerly a problem, because most of the samples that we were collecting at NIOSH in the early days of the technique had been sampled at various plants where asbestos alone was being used. Evaluation of the exposures of workers was based upon the phase contrast microscopy technique. The problems of that technique have been discussed in great degree over the years. When it first began, there wasn't much of an argument about whether or not the fibers found were asbestos.

Today, however, we find ourselves trying to detect asbestos in minute quantities in mixtures of other fibers and materials. So the problems of identification becomes very important and the problem of just quantitative determination of fibers becomes of relatively minor importance. Because the OSHA standards pertain not only to

asbestos alone but also to mixtures of materials containing asbestos and other fibers, it behooves us to know what is asbestos and what is not. The problem facing us today is our need for an asbestos identification procedure adequate in all cases.

We are reasonably sure that the NIOSH procedure is not the answer. Many people are saying we should replace it. What do you want to replace it with? Replace it with dispersion staining, some voices say. Dispersion staining is a technique which uses light microscopy and visible light for illumination. It's fairly simple and fairly specific, but it has limitations like everything else.

If you want to go further into the physiological problems associated with asbestos exposure or the available techniques and identification problems, you want to be much more specific and pin it down as a fiber, a mineral fiber, or another fiber. Which one is it? Then you have to take considerably more difficult steps. Of course every time you do that, you not only add to your burden as far as labor, but also cost.

Now we find ourselves on the threshold of going into every school in the United States with an army of people who know next to nothing about asbestos. They have only heard it's a terrible thing and they want to find out if any is there. Anything that looks fibrous, you know, is going to be concluded by many to be asbestos.

There are going to be consultants called in who will say yes, we can tell you. They know and we know without singling anyone out that there are many who will not have the foggiest idea of what asbestos looks like. In industry today there are many workers who are exposed to asbestos fibers from some source or another, all the way from the manufacture of hairdryers, where some asbestos had been found recently, to asbestos insulation. So you have everything from a minute quantity of asbestos exposure to the extreme situation, and you must deal with all of those.

The objectives of this symposium are very simple. First is a knowledge of what others are doing and next, some ideas to gain from their particular experiences useful for your own

problems. Finally, the most lofty ideal I have is that we would be able to say, based upon what we are going to hear today, that there is a technique better than phase contrast microscopy. If this group of people who surely have a lot of experience and a lot of reasons to give valid opinions could agree on something like that, we could make such a recommendation to anyone.

We could tell OSHA that a gathering of peers has decided that discussion is over. Here is something that offers a logical, viable alternative to the present procedures recommended for identifying and evaluating asbestos. Do you want to adopt it or don't you? And that would be up to OSHA.

I suppose that's a pretty lofty ideal. I doubt seriously if this many people in the very short time we have could hack out a single conclusion from the whole meeting. But if you go away with some information that you didn't have when you arrived, at least we will have accomplished that much.

A couple of names are not on the program. I would like to go over that for a moment before we start. The agenda you have in the packet is as it says a preliminary one and somewhat incomplete.

The speakers for this morning's program from NIOSH; for the benefit of those who may find themselves involved in evaluating insulation in schools, one speaker is a chemist in NIOSH who has recently developed a technique for the spot test determination of asbestos. Richard Kupel is going to tell you about that, and he brought some copies of the procedure for distribution.

All of these problems began when NIOSH developed a procedure for determining airborne fiber concentrations. It was not proposed as a technique for identifying asbestos. From that point, thirteen to fifteen years ago, there has been much discussion of it and various other techniques. Steve Bayer, a training instructor in the NIOSH Division of Training and Manpower Development in Cincinnati, has worked in sampling and identification of asbestos. He presents a training course on the NIOSH technique and others used in asbestos evaluation. I have asked him to lay a little historical groundwork for you.

A Brief History of the Membrane-Filter Method

STEVEN G. BAYER

**Industrial Hygiene Instructor, Division of Training and Manpower Development,
NIOSH, Cincinnati, OH**

It's a pleasure to be here this morning and renew some old acquaintances. I had planned to take a passive role in this meeting; however, I was asked to present some remarks regarding the history of the membrane-filter method for determining airborne levels of asbestos fibers.

My first experience with the membrane-filter method occurred in March 1967 when I was employed by the Public Health Service, Division of Occupational Health (predecessor of NIOSH).

At that time, the epidemiological study of asbestos industries which started in 1964 was well under way. The study covered about 35 asbestos-consuming industries engaged in the manufacture of brake shoes, clutch facings, cement pipe, and textiles. It focussed on pulmonary-function testing and dust-level determinations. The goal of the study was to develop hygienic criteria which could be used to eliminate asbestosis.

Prior to the formation of OSHA and NIOSH in 1970, airborne-dust levels of asbestos were measured by impinger sampling and particle counting. From 1962 to 1967, the ACGIH TLV for asbestos was 5 mppcf (millions of particles per cubic foot). In 1968, while the TLV remained the same, a notice of intended change to 12 fibers > 5um in length per ml of air was indicated.

Upon passage of the Williams-Steiger Occupational Safety and Health Act of 1970, OSHA and NIOSH were formed. OSHA accepted NIOSH recommendations in the original criteria document: the ceiling limit for asbestos fibers more than 5 um would be 10 fibers/cc, and the 8-hour time-weighted average (TWA) would be 5 fibers/cc. This standard became effective July 7, 1972. In addition, on July 1, 1976, the standard would be reduced to 2 fibers more than > 5um in length/cc based upon an 8-hour TWA.

Prior to 1976, the standards were established to protect workers from asbestosis. However, asbestos was receiving increasing attention as a carcinogen. With this in mind, NIOSH published in December 1976 the *Revised Recommended Asbestos Standard*, wherein it was proposed that the 8-hour TWA would be reduced to 0.1 fiber > 5um in length/cc and a peak limit (of 15 minutes duration) of 0.5 fibers > 5 um in length/cc would limit the upper concentration range.

At any time where the concentration was expressed in fibers/cc, the method of measurement was the counting of fibers deposited on filters using phase-contrast microscopy, at approximately 400 X. The use of the membrane-filter method came to the forefront through the Asbestosis Research Council in Great Britain. During the USPHS study in the early 60's, the British procedure was modified somewhat—primarily by using a different filter-mounting technique.

During the USPHS asbestos study, quite a few different sampling and analytical methods were used for experimental purposes. Those that I recall are listed below.

SAMPLING SYSTEMS

- 1) Gross Sample
 - a) 37 mm filter
 - b) 8" by 10" filter
 - c) midget impinger
- 2) Respirable by Cyclone
 - a) around 1.7 l/m
 - b) around 10 l/m
 - c) around 35 cfm
- 3) Horizontal-plate Elutriators
 - a) converted Cassella Hexhlet
 - b) Cassella Isleworth
 - c) homemade experimental designs
- 4) Various Filters
 - a) cellulose ester
 - b) vinyl metrical
 - c) polyvinyl chloride
 - d) silver membrane
 - e) nuclepore

ANALYTICAL TECHNIQUES

- 1) Gravimetric
- 2) Optical Microscopy
 - a) particle counting in impingers
 - b) fiber counting on filters
- 3) Transmission Electron Microscopy
- 4) Mg Analysis by Atomic Absorption

All these different techniques (and more) were used. However, for a variety of reasons, all were eventually discarded in favor of the membrane-filter method.

Why? Asbestosis was thought to be caused by asbestos fibers. The air in most of the asbestos plants (during the '60's) had an abundance of fibers floating in the air. Using a small battery-powered pump, a sample relative to a worker's personal exposure could be collected on a filter. the filter could then be brought back to a laboratory, mounted on a slide, and the asbestos fibers counted. Actually, it would be more correct to say "fibers counted," since exact differentiation of asbestos based upon appearance is not always possible. But during those years and in those industries, the number of interfering non-asbestos fibers was small, and most professionals agreed that the membrane-filter technique was appropriate.

After working for the U.S. Public Health Service for nearly 13 years and having seen an important first step from particle counting by impinger under 100X light-field microscopy to fiber counting on filters at 400X phase-contrast microscopy, I am now caught in a third possible transition to some unknown better method. As a training instructor, I have maintained (and will maintain until OSHA or NIOSH changes the regulations based upon a new analytical method) the integrity of the membrane-filter method as a "standard method." But there are reasons to formulate a new method which must be an improvement over what is used now. The new method should incorporate the following considerations:

- 1) The disease-causing mechanism must be identified and the analytical method must be able to measure it.
- 2) Since the agent is probably some type, size, and shape of asbestos fiber, the method must demonstrate conclusive identification of the specific asbestos fiber.
- 3) The precision of the method for both counting and identifying the fibers must be appreciably better than the current method. This includes reproducibility between operators and microscopes.

Selected chronological references

1. Grossman, Germain: Counting of Dust Particles by Phase Microscopy. *AMA Archives of Industrial Hygiene and Occupational Medicine* 6: 416-420 (1952).
2. Yaffee, C.D. et al: Preparing Dust Samples for Microscopic Examination—Notes on a New Method. *Am. Ind. Hyg. Assoc. J.* 15:3 (1954).
3. Hall, F.E. et al: Particle Setting Times in Ethyl Alcohol Water Mixtures as Effected by Variables in Impinger Sampling. *Am. Ind. Hyg. Assoc. J.* 26 (September/October 1965).

4. Lynch, J.R. and H.E. Ayer: Measurement of Dust Exposures in the Asbestos Textile Industry. *Am. Ind. Hyg. Assoc. J.* 27 (September/October 1966).
5. Edwards, R.G. et al: Dust Counting Variability. *Am. Ind. Hyg. Assoc. H.* 27 (November/December 1966).
6. Lynch, J.R. and J.E. Ayer: Motes and Fibers in the Air of Asbestos Processing Plants and Hygienic Criteria for Airborne Asbestos. Reprint from *Inhaled Particles and Vapors II* (proceedings of an internal symposium organized by the British Occupational Hygiene Society). Pergamon Press, NY: 1966.
7. Crable, J.V.: Quantitative Determination of Chrysotile, Amosite, and Crocidolite by X-Ray Diffraction. *Am. Ind. Hyg. Assoc. J.* 27 (May-June 1966).
8. Lynch, J.R. and H.E. Ayer: Measurement of Asbestos Exposure. *J. of Occupational Medicine* 10 (January 1968).
9. Edwards, G.H. and J.R. Lynch: The Method Used by the USPHS for Enumeration of Asbestos Dust on Membrane Filters. *Am. Occupational Hyg.* 2: 1-6 (Pergamon Press, 1968).
10. Renshaw, F.M. et al: The Use of Midget Impingers and Membrane Filters for Determining Particle Counts. *Am. Ind. Hyg. Assoc. J.* (March/April 1969).
11. Keenan, R.G. and J.R. Lynch: Techniques for the Detection, Identification, and Analysis of Fibers. *Am. Ind. Hyg. Assoc. J.* 31 (September/October 1970).
12. Lynch, J.R. et al: The Interrelationships of Selected Asbestos Exposure Indices. *Am. Ind. Hyg. Assoc. J.* 31 (September/October 1970).
13. *Occupational Exposure to Asbestos*. NIOSH (1972). USGPO #HSM72-10267.
14. LFE Corporation: *Statistical Evaluation of the Procedure for Counting Asbestos Fibers on membrane Filters*. Report submitted to the Asbestos Information Assoc. of North America, Suite 16-11, 22 E. 40th St., New York, NY 10016.
15. *Symposium on Electron Microscopy of Microfibers*. August 1976. USGPO 017-012-00244-7.
16. *Membrane Filter Method for Estimating Airborne Asbestos Dust*. October 1976 Secretary, N.H. & M.R.C., P.O. Box 100, Woden, Canberra, A.C.T. 2606, Australia.
17. *Revised Recommended Asbestos Standard*. NIOSH, December 1976. USGPO NIOSH-DHEW 77-169.
18. Zumwalde, R.D. and J.M. Dement: *Review and Evaluation of Analytical Methods for Environmental Studies of Fibrous Particulate Exposures*. USGPO NIOSH-DHEW 77-204. May 1977.
19. Baron, P.A., Ph.D.: *The Use of Light Scattering for the Detection of Filter Samples of Fibrous Aerosols*. DHEW NIOSH 78-105. January 1978.
20. Levine, R.J., M.D. (editor): *Asbestos: An Information Resource*. DHEW NIH 79-1681. May 1978.
21. Samudra, A.V. et al: *Electron Microscope Measurement of Airborne Asbestos Concentrations—A Provisional Methodology Manual*. EPA 600/2-77-178. Revised June 1978.
22. Gravatt, C.C. et al: *Proceedings of Workshop on Asbestos: Definitions and Measurement Methods*. NBS Special Pub. Number 506, November 1978.
23. Leidel, N.A. et al: *USPHS/NIOSH Filter Method for Evaluating Airborne Asbestos Fibers*. DHEW NIOSH 79-127. February 1979.

Evaluation of Asbestos in Insulation

DR. WALTER C. MCCRONE

McCrone Research Institute, Chicago, IL

The Environmental Protection Agency has undertaken the coordination of a nationwide effort to eliminate a possible threat to the health of school children posed by the past use of asbestos-containing materials in the nation's schools. The purpose, extent, and form of this effort can be best ascertained by contacting the EPA (800/424-9065; in Washington, D.C. call 554-1404). It is therefore important to have the tools and techniques available for rapid and dependable analysis of insulating, sound-proofing, and other materials used in schools for asbestiform minerals.

The task of cleaning up the nation's schools makes Hercules' task of cleaning the Augean stables seem like a Sunday picnic, and the cost will be monumental. Each school and each material in each school must be considered by informed experts who can decide the best solution to the problem. Removal is very costly and could, in some cases, actually increase the hazard. Leaving

well enough alone should be the recommendation unless contraindicated, for example by high friability or a high probability of disturbance. Then encapsulation (sealing in with spray finishes) or isolation (such as installing a lowered ceiling) should be considered. Each situation must, however, be considered individually.

Anyone who must decide what to do must have reliable information on which to base his decision. In particular, he must know the composition of each possible asbestos-containing material. Ideally, he should know:

1. what asbestiform substances are presents;
2. in what proportion;
3. what other substances are present (e.g., cellulosic fibers, mineral wool, other glass fibers, vermiculite, talc, perlite, diatomaceous earth, organic fibers, clays, glass powder, quartz, gypsum, etc.); and
4. size ranges for each substance.

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The third of these points is important because some of these substances are already known to be unhealthful and, the way matters are going, the others may well be declared hazardous in the future. Knowing what is present permits a more intelligent evaluation of the overall situation and may eliminate the need for a later more complete analysis.

Even the chemical analytical problem is formidable and many laboratories will be involved. McCrone Associates' two laboratories in Chicago and London have analyzed thousands of samples of insulation, acoustical tile, wallboard, and other construction and decorative materials. Our staff has sampled hundreds of locations and advised many of the institutions involved as to what we think they should do based on our analyses. Finally, the McCrone Research Institute teaches the methods we feel best serve the analyst who wishes to analyze these materials.

Polarized light microscopy

Our method of choice is polarized light microscopy. There is, in fact, no other method capable of doing the job. Even the minimal job of detecting chrysotile, amosite, and crocidolite can only be done microscopically. Certainly the identification of ground glass, mineral wool, glass wool, diatomaceous earth, micas, clays, perlite, lizardite, antigorite, other amphiboles, cellulosic fibers, organic fibers, etc. requires the microscope.

Part of the reason is that almost the only differences between many substances are microscopic shape or optical features: e.g. lizardite, antigorite, and chrysotile; perlite, diatoms, and quartz; cotton, wood fibers, and linen; or silk, human hair, and horse hair. X-ray diffraction may help in some cases but it does not differentiate between fibrous and nonfibrous varieties of the same minerals. Furthermore, it can't identify amorphous substances such as diatoms, perlite, glass or organic fibers; nor is it useful for particle size measurement. All of these analyses are quickly accomplished microscopically.

Another reason is that asbestos minerals were discovered, characterized, and named before x-ray or electron diffraction and other modern instrumental methods were invented. In fact, only chemical analysis and microscopy were then available; the differentiation of the various amphiboles and the three serpentines can only be quickly, confidently, and conclusively done by polarized light microscopy. The determination of chemical composition is not very practical on single-particles in a mixture even aside from the fact that many different substances can have identical chemical composition, e.g. riebeckite and crocidolite; grunerite or cummingtonite and amosite; lizardite or antigorite and chrysotile; fibrous and nonfibrous tremolite, fibrous and nonfibrous actinolite, or fibrous and nonfibrous ferroactinolite.

Sampling

The first and a major problem faced by the microscopist is sampling. Insulation, especially in microscopic samples, is far from a uniform

material containing a precise percentage of fibers throughout. The wide variation often found in the results from different laboratories or even the same laboratory can in most cases be explained by sample variation. The initial sample should be large enough to be representative and the microscopist must make every effort to take a representative milligram range sample. This is very difficult.

A sampling procedure recommended by the EPA that works very well for friable samples is to press a 35 mm film cannister completely through all layers to yield a core of material. This cannister should be carefully labeled and sent to the laboratory. There each sample should be divided in half (with careful attention to layering if present). One-half (after drying and weighing) should be placed in a small 100 ml beaker with about 50 ml of 10% H_2SO_4 . Only the cementitious components will dissolve, leaving the fibrous and other more inert materials in suspension. The latter can be allowed to settle for several minutes, the solution decanted, and water washed by resuspension and decantation several times. Finally, the washed particles should be resuspended by agitation to give a uniformly mixed composition. A few drops can then be removed by eyedropper and several one-drop samples can be placed on microscope slides. These, after drying, can be used for microscopical identification of the particles present. To give more quantitative results the remaining sample, after removal of the eye-dropper samples, should be filtered, dried, and weighed. This will allow calculation of the % cementitious material and, by microscopy on the residue, the percentages of the remaining components.

When the budget allows, the above procedures would yield more accurate and more reproducible quantitative results. In most cases, the budget does not permit the luxury of doing such a complete job.

Instead, the original ounce sample submitted to the laboratory is usually sampled at 10-20 regularly spaced locations with fine forceps, and the accumulated sample is dispersed on a microscope slide in an appropriate immersion liquid. It is the variation in composition of such samples that often leads to corresponding variations in analytical results. One should expect that repeat analyses made in this way may vary by up to 50%. Any single 1-5 mg sample often contains no asbestos in samples containing as much as 50% of that substance in the overall sample.

Identification

No matter how the final microscope preparation is obtained, we now have the problem of identifying each component. This is best done by classical polarized light methods together with dispersion staining, a specialized petrographic procedure. A well-trained mineralogist may not need dispersion staining although most would find it easier to apply to asbestos identification than classical optical crystallography. Any microscopist not trained in mineralogy will find dispersion staining far simpler and, indeed, the

only method that can be learned in a 3-5 day course or on one's own in any reasonable time.

Are asbestiform minerals present?

Generally, this means—Is chrysotile, amosite (fibrous cummingtonite or grunerite), or crocidolite (fibrous riebeckite) present? The actual analysis proceeds by adding a Cargille refractive index liquid (nD 1.550, high dispersion) to one of the slide samples after complete drying. The particles should be dispersed in this liquid by tearing aggregates apart, using two fine needles before covering with a coverslip. In this liquid, chrysotile has distinctive shape—very fine flexible fibrils (often curly) plus straight bundles of such fibrils—and distinctive dispersion staining colors*—usually blue-magenta parallel to the fiber axis and blue perpendicular. Different samples of chrysotile, however, may vary somewhat in λ_0 , the wavelength at which particle and liquid have the same refractive index. Parallel to the length λ_0 ranges from about 440-560 nm and perpendicular to the length from about 560-660 nm, $\Delta\lambda_0$ for $n_{\perp} - n_{\parallel}$ is usually close to 100-120 nm.

The presence of chrysotile and other fibrous particles is often obscured by a covering of large numbers of other particles. This is especially troublesome because most such particles are so different in refractive index from the mounting liquid that they appear bright white with the central stop. Trying to see the asbestos is like trying to see while driving at night with an oncoming stream of cars with high-beam headlights. Often one can be pretty sure the particles are covering obscured fibers because of their pattern. I have described this as the milky way effect. Sometimes a stray fibril may poke its way out into the liquid to show dispersion staining colors. It is always a help in such situations to examine the "milky way" with crossed polars since the underlying fibers often then become visible and recognizable as fibers. Crossed polars also help to locate smaller fibers and small percentages of fibers. Every sample should be quickly scanned with crossed polars before the absence of asbestos is reported.

Amosite and crocidolite are both very pale yellow to white by central stop dispersion staining in liquid 1.550, because their refractive indices are so much higher. Crocidolite, of course, also usually shows a blue absorption color. If no anisotropic fibers with refractive indices much higher than 1.550 are present, the analysis is completed.

If higher index anisotropic fibers are present, another sample is mounted in Cargille liquid nD = 1.680 and examined with dispersion staining. Most amosites used in buildings will show a λ_0 of about 460 nm (golden yellow) parallel to the length and about 600 nm (blue-magenta) $\perp$ 660 nm (pale blue) perpendicular to the length depending on fiber orientation. A perpendicular λ_0 of >660 nm corresponds to the α vibration direction which usually shows oblique extinction of about 15° ; a λ_0 600 nm perpendicular to the length corresponds to β on the view showing parallel extinction. Higher or lower values for any one of the three vibration directions should mean higher or lower values for all three. As with most silicate minerals,

substitutional solid solution—in this case Fe^{2+} with Mg^{2+} —can cause α , β , and γ to vary.

Although the refractive indices of amosite and other amphiboles may vary over a wide range, most of these minerals are not from commercial sources. Nearly all amosite actually used in insulation commercially has the optical properties given above.

If, in liquid nD 1.680 low birefringent fibers show lower λ_0 colors close together in the yellow to golden magenta, crocidolite is strongly indicated. If they show a negative sign of elongation (higher λ_0 parallel to the length) and blue absorption colors with pleochroism (blue parallel, gray-blue perpendicular), crocidolite is present. Further confirmation can be obtained by mounting a third sample in Cargille refractive index liquid nD = 1.70. Crocidolite will show λ_0 colors close to 485 nm (golden magenta) parallel to the length and about 455 (golden yellow) perpendicular. Again, some parallel movement of the α , β , γ colors should be expected for crocidolites from different sources.

We should emphasize that dispersion staining is a method for rapid refractive index determination. To be certain the colors observed mean a particular asbestos is present one must be certain the dispersion staining data are consistent with particle size and shape as well as the relationship between the optical properties and the crystallographic axes. With amosite, for example, the crosswise index must be α for oblique extinction views and β for views showing parallel extinction.

Are other asbestiform minerals present?

If, during the above examination, anomalous results were observed, that is, highly fibrous with

λ_0 colors in other than the prescribed ranges for chrysotile, amosite, and crocidolite, then fibrous tremolite, fibrous actinolite, or anthophyllite may be present. These are rarely found, however, in insulation. When these anomalous results were obtained, you should have characterized the fibers in those liquids as to refractive indices relative to those liquids and extinction angles. If all of the fibers show parallel extinction, they are anthophyllite, if the possibility of organic fibers is first eliminated.

Tremolite will show strong colors in all orientations in 1.605 high dispersion liquid. The oblique extinction view (ca. $15-20^\circ$) will show α perpendicular (λ_0 ca. 440 nm; yellow) and γ nearly parallel (λ_0 ca. 680 nm; pale blue). The parallel extinction view shows γ' parallel to the length (λ_0 ca. 460 nm; golden yellow) and β perpendicular (λ_0 ca. 530 nm; red magenta).

Actinolite has similar morphology and optics except that the indices are all higher than tremolite. Actinolite may also show pleochroism (green to colorless). It is best studied in high dispersion liquid 1.630 in which α and γ on the oblique extinction view show magenta (λ_0 ca. 565 nm) and golden yellow (λ_0 ca. 445 nm), respectively. On the parallel extinction view γ' (lengthwise) shows λ_0 ca. 470 nm or golden magenta and β (perpendicular) shows λ_0 ca. 495 nm, also golden magenta although with more red.

Tremolite, actinolite, and ferroactinolite are

*The dispersion staining colors mentioned throughout this paper are those obtained by using the central stop rather than the annular.

parts of a continuous solid solution series in the same manner as the amosite minerals, cummingtonite and grunerite. The name to use for a given amphibole depends directly on the optical properties, as shown in Table 1.

Identification of interfering substances

A few common substances show dispersion staining colors similar to those of chrysotile, and some are elongated as well. These include antigorite and lizardite (polymorphs of chrysotile), quartz, talc, paper fibers, and hairs. All of these show dispersion staining colors in 1.550 and all except talc and paper fibers show colors similar to chrysotile. Antigorite in 1.550 high dispersion liquid shows λ_o for γ , parallel to the length, about 465 nm (golden yellow), λ_o for α and β crosswise are ca. 500 nm (golden magenta) and 520 nm (red-magenta), respectively. Lizardite is platelike (often a lamellar aggregate) with α (λ_o ca. 700 nm, pale blue) perpendicular to the plate; it generally shows undulose extinction. The β and γ indices lie in the plane of the plate and both show s near 510 nm in the 1.550 high dispersion liquid (red magenta).

Quartz, although usually glassy flakes, shows blue and magenta central stop colors very similar to chrysotile in the 1.550 liquid. The shape is very different, however, and some isotropic views show only blue.

Talc fibers are derived by cleavage from large talc plates; I have never seen rolled talc plated as fibers. The dispersion staining colors are therefore always very pale yellow (λ_o ca. 360 nm) parallel to the length. They may also show $\lambda_o = 360$ nm for the perpendicular direction but, if on edge, they will show $\lambda_o = 645$ nm (blue-green). Tapping gently on the coverslip with a needle will usually bounce these needles from the 360-360 nm view to the 360-645 nm view. This is a very useful technique for quartz and other mineral grains as well.

Animal and human hair may also have refractive indices in the same range as chrysotile, and if finely fibrillated, by electric razor for example, can be confusingly similar. Such fibrillated fibers are, however, rare; they also usually show melanin pigment particles and sufficiently different indices to avoid confusion. Paper fibers also show a crosswise index close to 1.55 (but lower) and the lengthwise index is much higher, hence shows a yellow color. Usually also the morphology of paper fibers is distinctive.

Identification of other asbestos substitutes

A number of substances are often used as asbestos substitutes, e.g. wollastonite, glass wool, mineral wool, polyester and paper fibers. Wollastonite, a low birefringent (0.014) mineral, is triclinic and therefore shows oblique extinction in all views. It has refractive indices in the same range as tremolite and anthophyllite but, fortunately, the β index is nearly parallel to the length, hence some views show positive and some negative signs of elongation (anthophyllite and tremolite always show a positive sign). The dispersion staining colors for wollastonite in 1.605 liquid are: parallel, 429 nm (yellow); perpendicular, 410 nm (pale yellow), and 532 nm (red-magenta).

Glass fibers, usually as mineral wool, are often found in insulation. Different samples vary widely in refractive index and may show dispersion staining colors in any of the standard liquids from 1.55 to 1.68. Generally, however, mineral wool has low indices < 1.55, and is often coated with a colored (yellow, orange to red) resin. It is always isotropic or very slightly birefringent due to strain.

Another unusual constituent found in several recent samples is a polyester fiber. The very high birefringence, uniform cylindrical crosssection, and considerable length make this easy to distinguish. The indices are about 1.53

TABLE I
Optical Properties of the Amphiboles

Mineral	Refractive indices		$\gamma - \alpha$ (aver.)	Extinction angle	Sign		$2V$ $^\circ$
	α	γ			elonga'n	optic	
Tremolite	1.603-1.620	1.627-1.642	0.023	19-21	+	-	86-80
Actinolite	1.620-1.667	1.642-1.686	0.020	15-19	+	-	80-70
Ferroactinolite	1.667-1.683	1.686-1.702	0.019	10-15	+	-	70-65
Cummingtonite	1.633-1.664	1.654-1.687	0.022	15-21	+	+	103-92
Grunerite	1.664-1.686	1.687-1.729	0.033	10-15	+	+,-	92-82
Riebeckite	1.654-1.698	1.666-1.712	0.014	10-20	-	-	40-90
Anthophyllite	1.606-1.648	1.626-1.670	0.021	0	+	+,-	68-120

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perpendicular (less than chrysotile) and 1.71 parallel (greater than crocidolite).

Identification of other possible nonfibrous constituents of insulation

Possible additional constituents of insulation etc. include ground glass, perlite (a heat-expanded volcanic lava), diatomaceous earth, vermiculite, and mica. Of these, ground glass, perlite, and diatoms are isotropic and characteristically shaped. Vermiculite, a clay, and the micas are very thin, flat plates often nearly isotropic but with turned-up higher birefringent edges. Vermiculite will have indices less than 1.55 and the micas above 1.55 and often above 1.605. If colored brownish-gray the mica is biotite with higher indices than colorless muscovite, another common mica.

Other materials may well be found as time goes on but the observations made during the analytical procedure described above should uncover such substances, since they will not fit the data given here.

All things considered, a careful microscopist, confident with dispersion staining, should have no difficulty in identifying asbestos and most other substances associated with it.

Acknowledgement

Although fully responsible personally for the ideas expressed here, the author acknowledges with gratitude the constructive appraisal by his colleagues at McCrone Associates: John Delly, Lucy McCrone, Mark Palenik, and Ian Stewart.

Asbestos and the Electron Microscope

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I first learned about this meeting two weeks ago, when I saw the provisional program, and my immediate reaction was that the ACGIH was going to wish on us in electron microscopy the same "raw deal," if I may call it that, as they had wished on us with light microscopy. I noted that there was to be a paper on the use of the scanning electron microscope to characterize asbestos but not one on transmission electron microscopy. I felt that this was similar to using phase contrast microscopy to tackle a mineralogical problem which is more appropriately tackled by a petrographic microscope, so I called Mr. Kelley to ask him what he was doing to us. He very kindly suggested that I come along and present my views in the general discussion at this meeting and so I came prepared with one or two slides to do this. When I arrived, however, Jim Ferguson told me that I was to present my views a little more formally, so here they are.

Criteria for Asbestos identification

Let me say from the outset that I should not be regarded as an anti-scanning electron microscope man. There are many situations in which the scanning electron microscope is the appropriate tool to use, but I do not believe that the asbestos situation is necessarily one of them. My reasons for saying this are that, like Dr. McCrone, I believe that the main criteria by which one must identify asbestos are crystallographic with chemistry as a secondary consideration, and the scanning electron microscope is unfortunately lacking in its ability to give crystallographic characterization from particles of the sizes which are going to be of interest to us.

An additional problem in many scanning electron microscopes is that of resolution. Although manufacturers currently will claim that resolutions are better than 100 Å (10nm) for their microscopes, they do reserve the right to select the specimens on which they demonstrate this resolution, and their samples are generally those

with very clearly defined features and very high contrast. This situation does not normally prevail with asbestos fibers down to unit fibril dimensions.

Having brought in the term "unit fibril," let me define it. This term is applied generally to chrysotile asbestos. It is the smallest chrysotile fiber which can exist as a single entity and has an approximate diameter of 300-350 Å (30-35 nm). Fibers of these dimensions are quite common in environmental samples but one does generally see larger fibers in the workplace. However, as control procedures improve, it is to be hoped that the larger fibers will be less prevalent in the workplace, in which case we will have to concentrate on the very fine fibers which are not visible and thus cannot be characterized by the light microscope.

How then do we go about characterizing these fine fibrils with the electron microscope? As I have mentioned, the principal criterion is a crystallographic one. However, one may summarize the three main criteria for identification of a fiber as asbestos under three headings: morphology, crystallography, and chemistry.

Morphology

Clearly, since you are interested in controlling asbestos fibers, the particle in question must have the morphology of a fiber. I will not at the present time go into the various semantics of defining a fiber. But I will say that, at this time, everyone concerned with this problem is utilizing (I almost said accepting) the federal definition of a fiber as a particle with an aspect ratio greater than 3:1. It is quite possible that this may stimulate some discussion. However, though one may argue the semantics of a fiber from a mineralogist's point of view, the final criterion to decide what will be called a fiber for regulatory purposes must only be the biological significance of the particles's aspect ratio. This is a subject on which I am not qualified

to talk; therefore I will continue to use definitions that have been laid down in federal publications.

Crystallography

Fortunately, in the transmission electron microscope it is possible to obtain a diffraction pattern of individual particles present in the sample. In doing so, one examines the back focal plane of the objective lens and obtains a pattern related to the crystal lattice of the mineral in question. These diffraction patterns are characteristic "fingerprints" and can be used as a basis for identification of the mineral species present. It is thus possible to differentiate between individual particles which may look extremely similar in morphology—typically one refers to the similarity between chrysotile and some of the fibrous clays such as attapulgite and halloysite—and so ensure that only asbestos is being considered.

Chemistry

In modern analytical transmission electron microscopes, the chemical composition of the particles can be deduced from the x-ray fluorescence which they produce when excited by the high energy electron beam. The energies of this x-ray fluorescence can be measured by using an energy-dispersive spectrometer, or in some instruments the wavelength of these x-rays can be measured by using wavelength spectrometers.

The energy-dispersive spectrometer is cheaper, faster, and generally the more favored technique employed.

This chemical information cannot be substituted for crystallographic information but is used to supplement it. It is generally only necessary when one is considering the amphibole asbestiform minerals and is used to differentiate between the different amphibole species. In the case of chrysotile asbestos, the information is valid but not necessary, since the diffraction pattern of chrysotile asbestos is sufficiently distinctive on its own to permit an identification to be made.

Summary

Bearing in mind that the principal criterion for the identification of a mineral fiber as asbestos is its crystallography, only the transmission electron microscope—of the electron microscopical methods available—will provide the crystallographic information to enable a positive identification. The scanning electron microscope does not provide such information, although it does or can be made to provide the morphological and chemical information which in some cases of larger fibers may be sufficiently definitive.

I thank you for your attention and for the opportunity to present my views on this subject.

Characterization and Identification of Asbestos

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A combination of four microscopical techniques that have been utilized for characterization and identification of asbestos at the ASRC Laboratory for Environmental Particulates Analysis will be discussed. Fiber concentrations on a sample are obtained by phase contrast microscopy in accordance with the recommendations and criteria of NIOSH. A computer-based energy dispersive x-ray micro-analyzer (EDXA) interfaced to a scanning electron microscope (SEM) is employed for rapid determination of gross physical properties (size, shape, ratio of diameter to length, etc.). Ratios of elemental compositions from EDXA measurements are performed; the data obtained serves as the basis of selection for the proper index of refraction liquids for positive asbestos identification by dispersion staining. For sub-micron particles, a transmission electron microscope (TEM) with high magnification and resolution is used, applying the technique of selected area electron diffraction (SAED) for crystal structure identification. Classifications, sources, and selected micrographs of asbestos from the "ASRC Atmospheric Particulates Atlas" will be presented.

Procedure for asbestos identification

I. Scanning electron microscopy

The sample is prepared for examination under

SEM and energy dispersive x-ray analyzer (EDXA).

A. SEM analysis

Visual identification by morphological analysis (including length to diameter ratio of 3:1 or better).

B. SEM-EDXA analysis

1. Elemental composition, in particular Na, Mg, Si, Ca, and Fe as elemental variables, will determine if the sample is serpentine, amphibole, or non-asbestos, as specified in Table I.

2. Key to EDXA identification:

a. Chrysotile exhibits peaks of Mg, and Si.

If chrysotile is suspect go to II-A.

b. Anthophyllite shows Mg and Si.

c. Actinolite contains Mg, Si, Ca, and Fe.

d. Tremolite exhibits peaks of Mg, Si, and

Ca.

e. Amosite contains Mg, Si, and Fe.

f. Crocidolite shows S, Fe, and Na.

If b-f are suspect proceed to II-B.

II. Optical microscopy: dispersion staining

In order to confirm asbestos and its recognized varieties, we employ dispersion staining (2).

A. Chrysotile identification

If chrysotile is suspected from SEM-EDXA analysis, a sample is prepared in 1.545 high dispersion Cargille refractive index liquid. A positive test is indicated by blue and red magenta colors crosswise and lengthwise respectively.

1. If these colors are displayed, repeat in adjacent refractive index liquids for corresponding color display; if all check, the sample is positively identified as chrysotile.

2. If these colors are not observed, proceed to II.

B. Amphibole identification

SEM-EDXA may narrow possibilities; use this as a guide. Each amphibole if tested as procedure indicates; use corresponding liquids from Table II. For example, an EDXA spectrum of Si, Na, and Fe may be crocidolite.

With liquid 1.695, magenta and blue colors should be noted.

1. If these colors are seen, observation in adjacent liquids is advisable for definite identification.

2. If these colors are not observed, each liquid from Table II is prepared and tested.

III. Other analytical methods

1. If the sample is suspected of containing sub-micron fibers, an alternative is employed:

transmission microscopy,(2), coupled with selected area electron diffraction. This method requires a longer preparation time, along with knowledge of crystallography.

2. Counting procedures are accomplished by phase contrast microscopy (3) or polarized light microscopy utilizing diffraction staining.

References

1. McCrone, Walter: Detection and Identification of Asbestos by Microscopical Dispersion Staining. *Environmental Health Perspectives* 9: 57-61 (1974).
2. Langer, A., A.D. Mackler, and F. Pooley: Electron Microscopical Investigation of Asbestos Fibers. *Environmental Health Perspectives* 9: 63-80 (1974).
3. U.S. Dept. of Health, Education, and Welfare, Public Health Service Center for Disease Control, NIOSH: *Revised Recommended Asbestos Standard*. December 1976.

Tables

I—Classification of Asbestos

TYPE	COMPOSITION	CRYSTAL STRUCTURE
SERPENTINE Chrysotile	$3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$	monoclinic
AMPHIBOLES Tremolite	$2\text{CaO} \cdot 5\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	monoclinic
Actinolite	$2\text{CaO} \cdot 4\text{MgO} \cdot \text{FeO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	monoclinic
Anthophyllite	$7\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	orthorhombic
Amosite	$5.5\text{FeO} \cdot 1.5\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	monoclinic
Crocidolite	$\text{Na}_2\text{O} \cdot \text{Fe}_2\text{O}_3 \cdot 3\text{FeO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	monoclinic

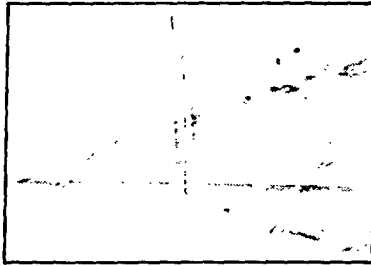
II—Color Chart for Dispersion Staining

ASBESTOS TYPES	REFRACTIVE INDEX LIQUID	CENTRAL SCREENING	CENTRAL SCREENING	CENTRAL SCREENING
SERPENTINE Chrysotile	1.545 (H.D.)	Golden-magenta	Red-magenta	Blue
AMPHIBOLE Tremolite	1.600 (H.D.)	Bright yellow	Red-magenta	Blue-magenta to blue
Actinolite	1.640 (H.D.)	Red-magenta to magenta	Green to yellow-green	Blue-green
Anthophyllite	1.615 (H.D.)	Golden yellow	Red-magenta to magenta	Bright green
Amosite	1.670	Bright yellow	Gold	Blue-magenta
Crocidolite	1.695	Golden magenta	Magenta	Blue-green

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FIGURE I

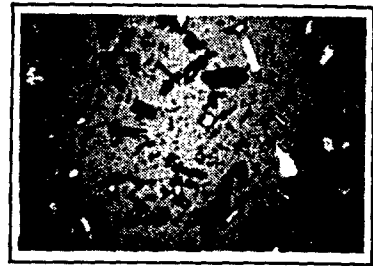
Asbestos observed under a polarized microscope (500X)



A. Chrysotile



B. Tremolite



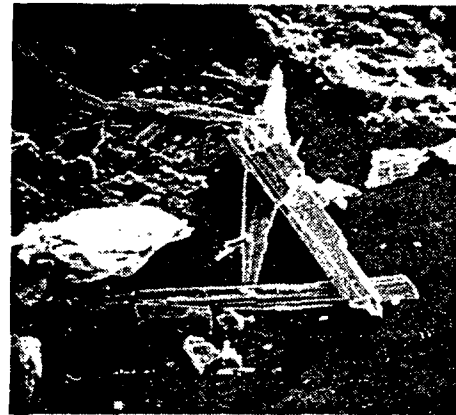
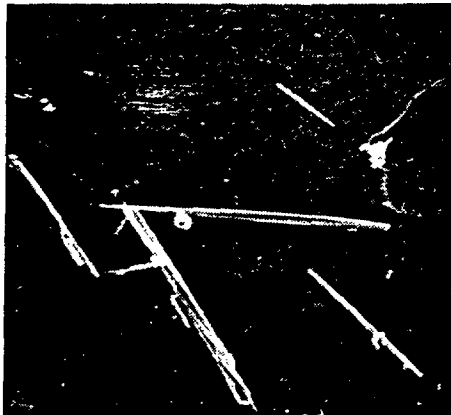
C. Actinolite

FIGURE 2.



Asbestos examined by a phase-contrast microscope (1000X)

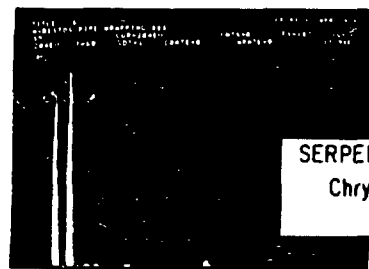
FIGURE 3



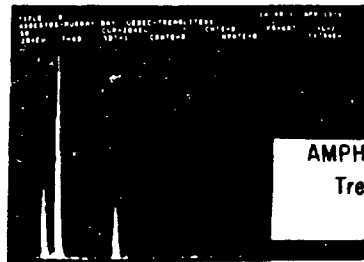
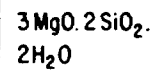
Scanning electron micrographs of asbestos
chrysotile and actinolite (3000X)

FIGURE 4

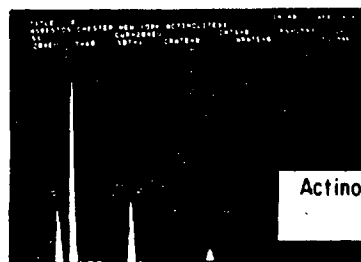
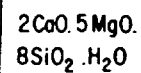
Classification of Asbestos



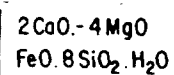
SERPENTINE
Chrysotile



AMPHIBOLES:
Tremolite



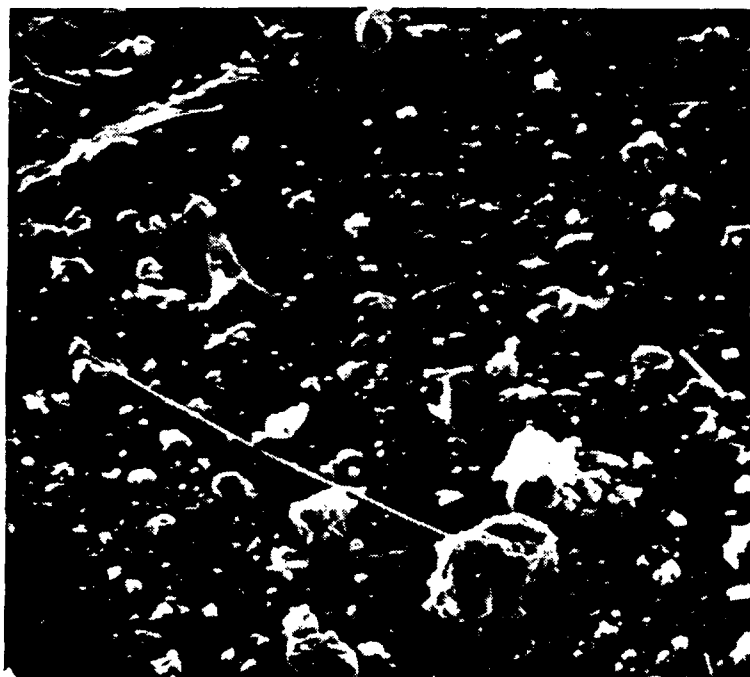
Actinolite



Energy dispersive x-ray spectra of asbestos

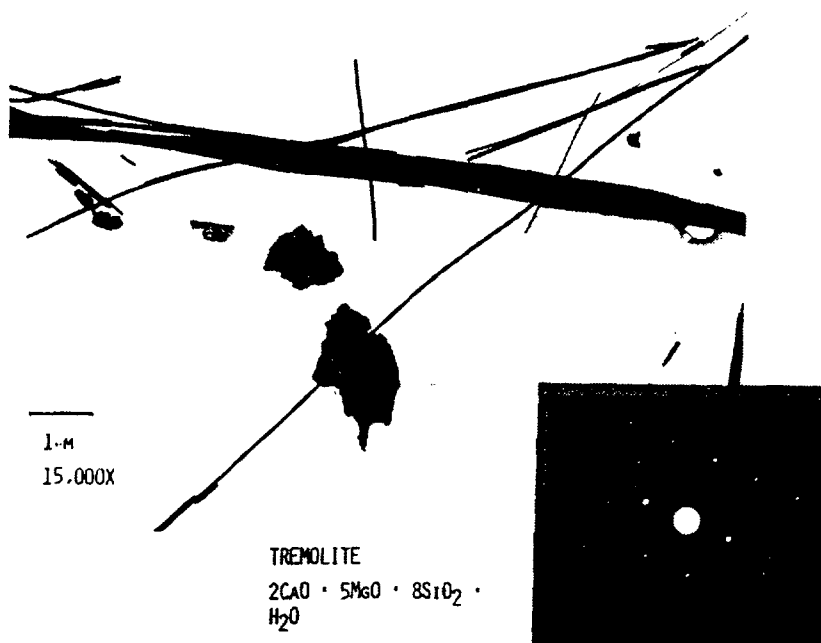
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FIGURE 5



More than 20 asbestos fibers 5m or less are observed in this scanning electron micrograph (3000X).

FIGURE 6



TREMOLITE
 $2\text{CaO} \cdot 5\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$

Transmission electron micrograph of asbestos (15,000X) tremolite with its selected area electron diffraction pattern.

A COMPARISON OF THE OPTICAL MICROSCOPE AND ANALYTICAL ELECTRON MICROSCOPE FOR ASBESTOS ANALYSIS

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Light and electron microscopy can both be used effectively in asbestos analysis, but each is best applied to different types of samples.

In asbestos analysis there are two fundamental types of samples—bulk samples, such as insulation and industrial talc, and dispersed samples, such as air and water samples.

In most bulk samples asbestos occurs in relatively large bundles which can be easily seen and identified on the light microscope. A sample can be qualitatively analyzed in a few minutes and an experienced light microscopist can make a reasonable estimate of the quantity of asbestos in the sample.

On the other hand, if dispersed samples are to be analyzed, the light microscope generally runs into major problems. In order to analyze asbestos by microscopy one must be able to see the asbestos fibers and also identify them. To observe the fibers, adequate resolution and contrast are necessary, then a technique must be applied to unambiguously identify the fibers.

The resolution of the light microscope using a 40X/9.65 N.A. objective will be able to resolve particles down to approximately 0.5 μm in diameter. The transmission electron microscope (TEM) can usually resolve down to at least 0.001 μm . This difference is critical when the size of asbestos fibrils are taken into account. Chrysotile fibrils average about 0.03 μm in diameter and are easily observed on the TEM. The light microscope would need a bundle of approximately 17 fibrils in diameter in order to be resolved (figure 1).

In air, water, and other dispersed samples asbestos often exists as single fibrils or small bundles of fibrils that cannot be seen by the light microscope. (Figure 2) Even those particles that can be seen may be difficult to unambiguously identify because of their small widths. The optical parameters used to identify particles (e.g., refractive indices, extinction angles, optic axial angles, etc.) can be difficult or impossible to measure on particles less than about 1 μm in diameter.

The analytical electron microscope (AEM), which is simply a TEM equipped with scanning and X-ray detector and electron diffraction can determine the chemical composition and some of the crystal structure parameters using an energy dispersive X-ray detector and electron diffraction. In addition it is capable of easily resolving even the smallest asbestos fibers. Because of these capabilities, the AEM is capable of unambiguously analyzing most asbestos fibers from dispersed samples.

The AEM does have some disadvantages. The analysis time is about 2-4 hours for one sample

and the turn-around time is up to two days depending on the method of sample preparation that is used. The purchase and maintenance costs are many times that of the light microscope. However, the AEM is currently the *only* instrument that can provide information on the quantity of asbestos over the entire size distribution from the single fibril upwards.

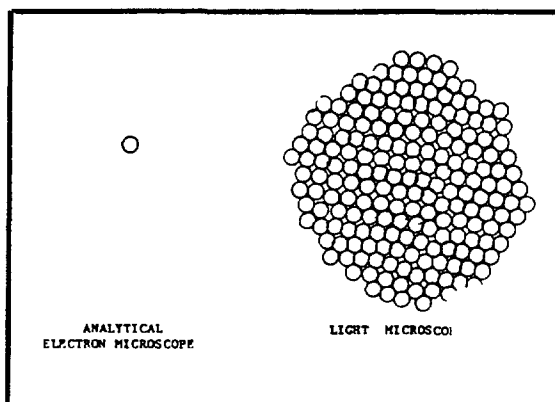


FIGURE 1. Schematic showing a cross-section of a 300 Å asbestos fiber which is easily resolved on the TEM, while only the cross-section of the large bundle approximately 17 fibrils in diameter can be resolved on the light microscope. This means that tremendous numbers of fibers can be missed by the light microscope.

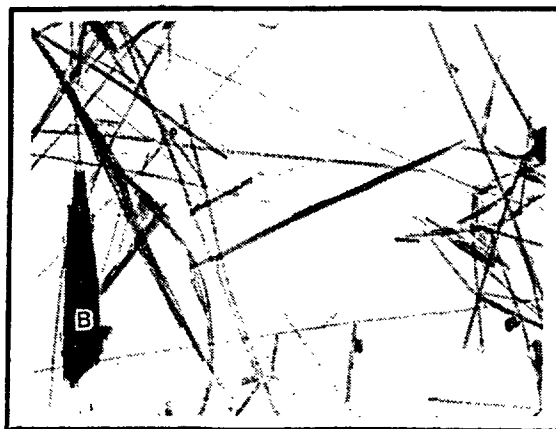


FIGURE 2. Transmission electron micrograph of chrysotile fibers collected on an air filter. Only the large bundle (B) would be seen on the light microscope and its small size would probably prohibit an unambiguous identification.

DR. ERIC STEEL: I think most of my talk has been given so I will go through it very briefly and try to give a little better idea of where the advantages are in the TEM. When we look at asbestos, we are trying to do one of two things: we are looking at bulk type specimens such as ceiling tile or insulation materials with the light microscope. It may very easily be the most efficient way because you can be very sure when you have before you bundles, large bundles.

It will be easily seen under the light microscope and can be identified very fast using refractive index measurements.

On the other hand, if we look at dispersed samples, such as air samples or water samples, and we want to quantify the number of fibers in that sample, we have a definite problem.

We need two things for measurement. We first need to resolve the particle and we need adequate contrast in the system to be able to see the fibers, also.

The contrast resolution is the problem with the light microscope on many asbestos samples and contrast can be a problem on the SEM as well as resolution, although it's marginal, as has already been mentioned.

Identification is another separate problem and resolution is a problem largely for chrysotile and to a lesser extent on the amphiboles which have generally a larger diameter. You will see many more of the amphiboles but have a much greater problem in identifying them.

So, look at the resolution problems first, the light microscope using the 40-X objective. You will be able to resolve a particle that is approximately 5,000 angstroms wide or in diameter. That's approximate.

The transmission electron microscope should be able to see down to 10 angstroms or less. Chrysotile having average dimensions of 300 angstroms wide, would need to have a bundle of some 17 fibrils thick in order to resolve it compared to the TEM having one fibril which is this kind of comparison.

On dispersed samples in air and water, we would expect a wide variety of fibers, from the single fibril up to 17 fibrils and beyond that to much thicker bundles.

One study carried out in Canada in association with the ASTM and the Quebec Asbestos Mining Association showed that the TEM found approximately 50 times the number of fibers that the light microscope found. That's a considerable difference in quantity.

Here the electron micrograph with .5 micron scale shows a fiber at about the resolution limit of the light microscope using the 40-X objective which means you would see this particle here.

The rest of the fibers on this picture you would not see on the light microscope, although you often can't see, although you cannot focus particles that are below the resolution limit of the light microscope.

Another problem is the identification. Now, this is critical for amphiboles and somewhat for chrysotile, also.

We have seen the methods using the dispersion staining in the light microscope and these are good as long as you can see the particle and you don't have particles that have interfering properties, such as refractive indices or other optical properties.

For chrysotile this is not too much of a problem, but for the amphiboles, let's say, in the industrial text, where we may have a small amount of tremolite in addition to wollastonite which have almost identical refractive indices and optical properties, they can simply not be told apart on a particle by particle basis on a filter sample using the light microscope.

The TEM and/or analytical electron microscope can first see the particle. In chrysotile, you can often see the morphological factor you can obviously not see on the light microscope.

We have an X-ray spectrum which you have seen several of before and an electron refraction pattern so you can identify the crystal structure and the chemical composition which are the fundamental parameters which define a solid.

By contrast you can see individual fibers here which you simply could not see, again, on the light microscope and very probably on the SEM you would have a great deal of trouble.

One problem on the SEM, which again has been mentioned for chrysotile, in particular, is the polymers of the same mineral chrysotile appears in several forms, antigorite, lizardite, and chrysotile. They are all serpentine. They all have the same chemical formula, many of them.

Antigorite often occurs in 3:1, 10:1 morphologies and by just using the dispersive X-ray spectrum, you cannot distinguish the two. You have to have the crystal structure of the hollow tube structure or some other indication to say it's chrysotile.

Now, one of the things that has not been mentioned is the drawbacks to the analytical electron microscope which somebody will be asking questions about and that is time.

It does take a fair amount more time to do sample preparation for counting than it does on a light microscope, but at the same time you can be assured that you can see all the fibers and you can identify each and every individual fiber.

The cost of the instrument is tremendously expensive. You are talking about hundreds of thousands of dollars and you have to have experienced operators, more so than just counting by morphology.

The EPA round robin study was mentioned. This is a graphic example of why a standard method would have to be used for sample preparation and identification techniques.

There are different techniques to prepare the sample and different ways of identifying positively the fibers which goes a long way toward explaining the wide variety of results that were obtained. Thank you.

MR. KELLEY: Are there some questions at this point?

VOICE FROM THE FLOOR: What kind of TEM are you using?

DR. STEEL: Brands?

VOICE FROM THE FLOOR: Yes.

DR. STEEL: There are several brands in the field and you can use —

VOICE FROM THE FLOOR: What kind of AEM are you using?

DR. STEEL: What kind of AEM are we using? We are in the process of buying one right now. There are several brands that are made. They have the TEM capability. It doesn't really matter on the brand so much.

There are some technical differences that may make a difference in your quantitative analysis of the X rays, but most of the AEMs available commercially will do asbestos sufficiently.

VOICE FROM THE FLOOR: Do you feel that dispersion staining and x-ray diffraction is needed in the research analysis or does x-ray diffraction suffice in identifying?

DR. STEEL: Dispersion staining depends on the sample. For chrysotile, it's less critical to have the X-ray data especially if you can see the hollow tube and get the electron diffraction pattern, for the amphiboles it's more important because you have a wide variety in chemistry. You have many similar species. If you have an indexed pattern, rather than just measuring the $5.3\text{\AA}$ spacing, you have a better chance of positive identification. The $5.3\text{\AA}$ is very common in the mineral world and could not be used as conclusive evidence for the mineral type.

VOICE FROM THE FLOOR: I want to come back to time and practicability again because this is what I am really interested in.

We want the accuracy of course. Assuming that you have a very simple situation which in the United States we do have, as far as occupation problems are concerned. This is an OSHA symposium. Assume we are concerned with occupational problems. Could the electron microscopy technique that you have been describing be programmed so that they would be looking at chrysotile alone to give quick answers, quantitative answers over brief periods of time?

This is, I realize, a rather theoretical question but you made a statement earlier which I read before which is very significant that this is easily recognizable because of its structure.

What we have to bear in mind, all of us notwithstanding this is that 95% of all the asbestos sold in this country is chrysotile. The rest of it we can throw into the dust bin, for that matter, as far as occupational health is concerned for this discussion. Ninety-five percent is chrysotile. That's what the exposure is.

How quickly can we do a chrysotile analysis, how many samples can we do in a day, in a week or in a month, even, given this problem? I am trying to hone in on the target.

DR. STEEL: I mentioned the hollow tube morphology which I think is probably what you are referring to. The hollow tube morphology.

You will not see that in all chrysotile fibers. You will see it in most of them.

VOICE FROM THE FLOOR: Addressing chrysotile, itself, now, okay, chrysotile?

DR. STEEL: If you can zap in electron diffraction pattern and use the hollow tube morphology, you can count the chrysotile very fast.

VOICE FROM THE FLOOR: How fast is very fast?

DR. STEEL: I depends on the samples again. In this case it becomes very similar to counting, you are adding the electron--

VOICE FROM THE FLOOR: Is it sample an hour, a sample a day?

DR. STEEL: You shouldn't fail to do a couple of sample a day probably. Again, it depends on the samples.

VOICE FROM THE FLOOR: What do you want to include?

MR. KELLEY: Hold it. Let's put the questions so that the stenographer can hear them and so that all benefit from them, please.

That's a very important question that he is asking.

VOICE FROM THE FLOOR: Well, we have come up with some estimates here, but I am not sure that what he is asking is what he is answering.

VOICE FROM THE FLOOR: There is a lot of sample preparation to get it into the electron microscope. There is a lot of data counting, enumeration of the fibers, the sizing of them, putting them in size categories, then there is a lot of data reduction to present somebody with a report. I don't know what he is including in his estimate of two a day.

DR. STEEL: It really does depend on the sample. Sample prep takes a long time. It doesn't necessarily take that many man hours depending on which technique you use.

VOICE FROM THE FLOOR: I don't want you to think that I am needling you. I am trying to, as I say, hone in on the bull's eye here. You are speaking as an expert and I respect you for it. I was going to ask the question of the expert over here to my right, but I am asking you these questions.

Assuming that we begin with that, do you believe, given that you have a human element, that we can really rationalize such an approach that we can do more than one or more than two samples in a day?

Do you believe that we can improve on this so it becomes a practical approach for the engineer who needs a resolution of a problem?

DR. STEEL: If your samples were well characterized, came from the same occupational settlement and were characterized so that you knew almost all or all your fibers were chrysotile, after a few weeks of counting and identifying them on the TEM, then, it may be that you could make it much more rapid because less identification would be needed, but that would involve a very good characterization of that workplace, using that same material, et cetera, in the workplace.

VOICE FROM THE FLOOR: Along the same line when we talk about fiber, at some point someone has to add to the question the size.

If you talk about particles larger than five microns in length with a greater than three to one, obviously you are talking about an instrument other than you could take about something, all fibers including the particles that have to be counted.

So, either your question relates to mass per unit volume that determines one set of procedures or your question is addressed to fibers and then a deeper technique and a deeper approach, almost research approach is required.

So, as we go on, keep those two goals in mind. The two just don't go together with one single instrument.

VOICE FROM THE FLOOR: I came back to this asbestos analysis by AEM. When you go into this, you now have two factors to consider, the first one dealing with total amount of time expended per sample, but the other problem you get into that is different from optical microscopy is the fact that

you don't get your answers as quickly.

The time frame, for instance, the time expended, the effort and time expended to do a sample is separated.

You can run samples from the same place that come out the same. You can run two per day, but you can't do them by taking a sample in the morning and having the answer the next day at noon.

You are going to get your answer, if you get your samples on Monday, you will get your answer the next Monday because of the time lag due to the necessary operations.

DR. STEEL: That's very possible.

VOICE FROM THE FLOOR: But, we do routine samples this way and you can do them for about two to three per day through the entire process on a cost basis.

On a time basis it still takes a week or better to get your answer.

K² Test for Screening Asbestos

RICHARD KUPEL

Division of Physical Sciences and Engineering, NIOSH, Cincinnati, OH

We all know that chrysotile asbestos, as we have seen this morning, consists of magnesium and silicon, and that crocidolite and amosite have the same with a little bit of iron tossed in.

We thought, why can't we use this information to assist us in at least looking at the first bulk sample taken to find out if there is any asbestos there at all? In the first place, my boss said, "Don't call it asbestos. You're not looking for asbestos, you're looking for magnesium and iron." He said we'd go that route to find out if any magnesium or iron-bearing compounds are present.

We have looked at about 150 samples so far, and Dr. McCrone has looked at a large number of them received from Mr. Chiljean of the New York City school system. We first looked at a number of the field samples and as yet we do not have a false negative—not one out of about 150 samples.

What we are saying is that you can detect asbestos—it has to be somewhere above 3%, maybe less than that, if we can get some known samples done in that range. We know we can see it down to 3% in the bulk sample, but we're not saying absolutely that it's chrysotile, crocidolite, or amosite. We're saying that the possibility is there. In the first place, if you took a bulk sample, you would have to send it to the laboratory to get it analyzed anyway.

If you can show that no magnesium or iron-bearing compounds are present, you can eliminate that sample, just as we have eliminated some of the samples we received from the New York City schools. We got 31 samples and did not know the analyses of them. We found that two had chrysotile and one had amosite. We had four false positives; we don't know why these were positive. We think we're getting a positive from magnesium, but we had 25 true negatives and

these were confirmed by Dr. McCrone. They already had their results and when we gave them ours, they confirmed what we had found.

It's a relatively simple test; it takes just a few reagents. We take a sample of the bulk and analyze for magnesium first. Calcium does interfere with this test, so we have to get rid of the calcium.

Place a 25-50mg sample in a small beaker, add about 5 drops of glycerine, stir it well, grind it into the glycerine, and filter using a syringe filter system; the sample is washed into the syringe from the small beaker. Pass the washings through the filter and wash five times with distilled water. Take the filter out and put it through the procedure which amounts to adding one drop of concentrated phosphoric acid, two drops of 10N sodium hydroxide, then ten drops of the magnesium reagent and the blue color that is formed is due to the presence of magnesium.

It's a very sensitive test. We're looking at large quantities of asbestos and talking about anywhere from 3% up to 90% or even 100% asbestos. It's important, if you're going to survey according to the method EPA requests or will require, that you take a bulk sample about every 5000 square feet. So in a large school system you could have thousands of samples, and if you could cut this number down in any way it would be well worthwhile.

If we get a positive for magnesium, it will have to be analyzed by other means. It could be chrysotile asbestos; it could also be one of the amphiboles that has magnesium in it.

We then go through the iron test. We add a drop of HF to the sample, and next five drops of the iron reagent. If it turns a bright red, iron is present. It could be either crocidolite or amosite. So it is

possible to make these tests, very simple, very reasonable. We have never tried to estimate how much it's going to cost to do these. My colleague and I did about 35 of them in two and a half hours, so it can be done very quickly.

As I said, we have now done about 150 samples and have had no false negatives. We have had false positives since talc will give you the same reaction as will magnesium and chrysotile, but you have to send any positives to the lab for further analysis.

I don't know what else to say except the fact that it works. We have sent copies of this procedure to our ten regional offices. To my knowledge, none of them have tried it yet. Our industrial hygienists have performed the test a couple of times in the field. It has worked well for them.

We sent a kit with one of our industrial hygienists into Kentucky, where he was going to look at a number of schools. He did some tests in the field and brought samples back to the laboratory. We confirmed his tests and also had them analyzed with the electron microscope, which confirmed those results.

We have enough data now, we think, to show that the test does work. We also have another 90 samples, I believe, coming from Mr. Chiljean—which I think you have already analyzed, Dr. McCrone—and we will hopefully agree with those results as we did in the past.

I will conclude with a detailed description of the test.

K² Test for Screening Asbestos

I. SCOPE AND APPLICATION

This colorimetric test is applicable to the detection of magnesium (II) and iron (II) from asbestos in bulk samples. These samples include sprayed-on asbestos as well as ceiling tiles. The K² test is simple and can be readily used in the field to screen for the presence or absence of asbestos.

II. PRINCIPLE

The K² test is based upon the formation of color complexes with Mg⁺² and Fe⁺² released from asbestos. The Mg⁺² from chrysotile is complexed with p-nitrobenzeneazo- α -naphthol. The Fe⁺² from crocidolite and amosite is complexed with 1, 10-phenanthroline. A positive test is indicated by the formation of colored complex for Mg⁺² and/or Fe⁺², and it indicates possible presence of asbestos.

III. INTERFERENCES

The K² test is a colorimetric test for Mg⁺² and Fe⁺², and is not specific for asbestos.

The bulk sample may contain Fe compounds other than asbestos. Without treating the sample as in the Procedure, the color-forming reagent is added directly to the sample. If red color forms, Fe is present as an interference and these samples can be washed with water prior to the test to eliminate the interference. In the K² test, plaster (CaSO₄) interference is removed from the sample prior to the Mg test.

IV. SENSITIVITY, PRECISION, ACCURACY

With the glycerine treatment, the detection limit is about .2 mg of pure chrysotile per test.

From the total of 70 various field samples tested,

52 samples or 74% were correctly identified by the K² test as to whether asbestos was present or absent. Only 18 samples or 26% gave false positive reactions. The K² test was 100% accurate in identifying 22 samples that did not contain asbestos. The K² test results were verified using the electron transmission electron microscope.

V. APPARATUS

1. Teflon dish
2. Microspatula or glass rod
3. Dropping plastic pipet
4. 15 mL disposable plastic beaker
5. 25 mm size 0.8 μ m membrane filter
6. 25 mm Swinnex filter holder and gasket
7. 10 mL disposable plastic syringe

VI. REAGENTS (Reagent Grade)

1. Phosphoric acid, concentrated.
2. 10 N sodium hydroxide—Dissolve 40 g of NaOH in 100 mL of water.
3. Mg Reagent—Dissolve 1 mg of p-nitrobenzeneazo- α -naphthol in 100 mL of 2 N NaOH. Age at least a day. This reagent is stable for over a month.
4. Hydrofluoric acid—Dilute 20 mL of HF with 20 mL of water. Add 0.6 mL of concentrated HCL.
5. Fe Reagent—Dissolve 2 g of 1, 10-phenanthroline in 50 mL of ethanol. This reagent is stable for over a month.
6. Glycerine, reagent grade.
7. Double de-ionized water.

VII. PROCEDURE

A. Magnesium Test

1. If plaster is known to be absent, take a portion of sample about the size of a large pea and begin with Step 2.
 - a. Place a small portion of sample in a plastic beaker.
 - b. Add 5 drops of glycerine and mix well with a spatula.
 - c. Rinse spatula with a small amount of water into the beaker.
 - d. Filter the sample through the filtration assembly consisting of syringe attached to the Swinnex filter holder loaded with a membrane filter and a gasket.
 - e. Filter with minimum of 5 washings or about 50 mL of water.
 - f. Transfer filter to a Teflon dish.
2. Add a drop of H₃PO₄ and mix well by grinding the sample with a spatula.
3. Add 2 drops of 10 N NaOH and mix well.
4. Add 5 drops of Mg Reagent and stir briefly. Note any color change.
5. Add 5 more drops of Mg Reagent and observe the final color.
6. A blue color indicates that chrysotile may be present.

7. No color change with Mg Reagent indicates the absence of chrysotile. Even though amosite contains magnesium, this test does not produce the blue color. The iron test would produce a positive reaction indicating the presence of asbestos.

8. Check for Mg interference as in Section III if positive result is obtained.

B. Iron Test

1. Place a small portion of sample on a Teflon dish.

2. Add a drop of HF solution and mix well.

3. Add 5 drops of Fe Reagent and observe the development of red color.

4. The red color indicates that amosite or crocidolite may be present.

5. Check for iron interference as in Section III.

C. Notes

If both tests give negative responses there is a low probability that any asbestos is present. Once the presence of magnesium and/or iron is indicated, further analysis, if needed, is done in the laboratory to confirm, identify the type, and quantitate the percentage of asbestos.

VIII. REFERENCES

1. Channing L. Bete, Co., Inc. (Greenfield, Mass.): *Caution: Asbestos Dust*. Prepared in cooperation with U.S. DHEW, PHS, CDC, National Institute for Occupational Safety and Health (1973).

2. Feigl, Fritz: *Spot Tests—In Inorganic Analysis*. Translated by Ralph E. Oesper. Elsevier Publishing Company, Amsterdam (1958), pp. 225-227.

3. Kim, Walter S., James W. Carter II, and Richard E. KupeL: Quick Screening Test for Asbestos K² Test). To be published in *AIHAJ*. U.S. DHEW, PHS, CDC, NIOSH.

VOICE FROM THE FLOOR: The New York City sample, some 15% of asbestos samples, had 3% or less reported by dispersion staining. Of the samples, were any of those in that category?

MR. KUPEL: No, 3% chrysotile was the lowest we have seen that we have had confirmed. One sample did have 1% amosite which we identified.

We had two asbestos samples from New York; one contained 3% and the other 5% chrysotile. The rest of them had none. So we have not seen the very low ones. These samples were analyzed by Dr. McCrone and Mount Sinai also analyzed some of them.

VOICE FROM THE FLOOR: The concern, I think, is how this test is going to make out for that 15% of samples that are 3% or less. Thirty percent of the New York samples are 5% or less, which to me suggests that there is this grey area. Perhaps the spot test can resolve this sensitivity problem, but I think you have to be aware of the fact that there is a significant number of the samples, at least, as reported out by the PLM which are 1, 2, 3%.

What is the significance of those numbers?

MR. KUPEL: We have just received 45 more samples that we have not analyzed with the new procedure. We just developed this second step—using the glycerine to eliminate the calcium interference—since most of the samples Mr. Chiljean sent us were of the acoustical plaster type, which has a lot of calcium, and we would get a normal positive result from the plaster.

Now, we do not have any samples that have been analyzed at less than 3%, so we don't know whether it will work or not. It may, and we hope to get some of those low ones so that we can take a look at them.

VOICE FROM THE FLOOR: It may be that the formulations of the spray-on material will contain 3% or less asbestos and will test positive, not so much because of the asbestos but due to the presence of magnesium and other elements.

MR. KUPEL: Calcium is primarily the interference in these cases because the plaster is a calcium sulfate of some kind. Some persons we know will get positives with that, yes.

OSHA Analytical Experiences in Asbestos Identification and Measurement

WILLARD C. DIXON

Supervisory Chemist, Microscopy Branch, OSHA Analytical Laboratory, Salt Lake City, UT

The first thing I had better let you know is where you can reach me, in case you have questions or if you would like to have further discussions with me that you aren't able to take care of today. I am at Salt Lake City, Utah. The commercial number of my office is 801-524-5366. If you are on FTS, it would be 588-5366. The address is United States Department of Labor, OSHA, 390 Wakara Way, P.O. Box 8137, Salt Lake City, UT 84108. I will be glad to correspond or talk to you if any of you have something you would like to discuss with me.

During the analysis of asbestos samples there must be cooperation between the industrial hygienist and the laboratory. If the industrial hygienist has reason to suspect that asbestos fibers are present, then it's a good idea for him to tell the laboratory as much as he can about the type of operation that is taking place. What are they doing? Are they cutting? Are they grinding? Are they drilling? What kinds of materials are

they using? What information can be obtained from the labels to give to the analyst? If samples are to be analyzed, then it's a good idea when asking for analysis of the membranes to get a cross reference to corresponding bulk samples so one will know what kinds of fibers were found. This is quite useful sometimes in analyzing membranes.

OSHA experience with contract laboratories

I recently contacted several contract laboratories for the purpose of contracting out OSHA samples to some of them. We were temporarily getting more samples in than we could analyze ourselves. We contacted fourteen laboratories by phone. All except one were AIHA certified. The laboratories I'm talking about were those we could contract with if we decided to. We can't contract with a laboratory unless it is an independent one, so that narrows it down a little.

There were seven out of these fourteen which

claimed they were able to analyze bulk samples. The other seven only analyzed air samples. Out of the seven that analyzed bulk samples, one of the laboratories did it by electron microscopy, one by polarized light, and one by X-ray diffraction. One laboratory would do it by X-ray diffraction or optical methods. Four out of the fourteen laboratories had the capability of using polarized light. Two out of the fourteen used retardation plates and polarized light, and four used dispersion staining.

One laboratory realized that they couldn't analyze asbestos samples using dispersion staining or the other techniques such as polarized light. When we sent a request suggesting that this is the way we would like to have it done, they sent the samples back because they knew they couldn't do it that way. We didn't use one laboratory because they were charging \$670 a day for testimony. This we considered excessive. Those are some of the things we have encountered in the use of contract laboratories.

Some of the contract laboratories did make mistakes. We have a quality control program in our Salt Lake City laboratory. We repeat our analysis on one sample out of ten so we can be sure we're getting a good count on the analysis we do. In addition to cross-checking the analysis performed by private laboratories, we exchange samples with each other and cross-check to see that we are all counting the same. We are keeping statistics on that to find out what kind of standard deviation we're getting on the counts. We often get a private laboratory counter that is replicating our own counts within 8% or 10% and the difference between two counters is sometimes around 15% or 20%.

The NIOSH document (1) describing the asbestos method gives a coefficient of variation of 0.24 to .38, and they have a chart at the back of a similar publication (2) showing the statistics in terms of the total number of fibers counted.

Common mistakes in the analysis of asbestos

In checking those samples that were analyzed by contract laboratories, we found that there are certain kinds of mistakes that may occur in the analysis of asbestos. One of the most common of materials used in industry is gypsum. We find that it's possible for an analyst who is supposed to be certified to make a mistake if gypsum is present. While looking at gypsum particles, if the analyst doesn't notice that the sides are irregular, and if he doesn't check the dispersion staining, then he may think he is looking at asbestos fibers when actually what he is looking at is gypsum particles.

It's possible for an analyst to confuse something like fiberglass with crocidolite asbestos. If the analyst is not careful in the use of polarized light, he may see a fiber which doesn't show as strong birefringence as some other asbestos minerals and think he's working with fiberglass, when actually he's working with crocidolite asbestos.

Now, there are other ways to tell crocidolite asbestos. It has a natural blue absorption color

which will be observed if one has a large enough fiber bundle under the microscope. If one is using polarized light and a first order red retardation plate, one will notice that the colors reverse: where one expects to see a yellow color, one will see a blue color. In the position that one would expect to see a blue color, a yellow color will be seen. Because of the phenomenon of absorbance of natural blue crocidolite, one will get a green color instead of just a retardation yellow color. The use of some of these auxiliary methods can help to avoid mistaking crocidolite for fiberglass.

Wollastonite is used sometimes in industry as a substitute for asbestos. Occasionally one may find a situation where wollastonite and asbestos are used together, so it becomes important to be able to distinguish between the two. One of the things one will notice, if alert, is that there is a difference in the ways that wollastonite and asbestos cleave. In wollastonite, one will see a feathering between grains rather than an abrupt break as sometimes seen in asbestos fibers.

There is also an optical test that can be used to tell the difference between wollastonite and asbestos. What one can do is to put the wollastonite fibers in a position showing either yellow or blue with crossed polars and a first order red retardation plate. Then rotate that fiber around its own axis, but at an angle to the plane of polarization of the polarized light. When one does that with wollastonite, the fiber goes from yellow to blue and back to yellow again as it is rotated. If one does that with asbestos, it won't react that way.

There is a chemical method to tell the difference between wollastonite and asbestos, because the amphibole asbestos fibers, which are the ones one would most likely confuse with wollastonite, are much more resistant to acid than wollastonite. Wollastonite is easily typed by hydrochloric acid. One test is to put wollastonite into a drop of hydrochloric acid on a slide, cover with a cover slip, then heat it gently so that the hydrochloric acid boils off slowly. Upon reexamination in suitable mounting media, one will notice that the dispersion staining color is gone and that the reaction with polarized light is greatly diminished. The fiber remaining after treatment is gelatinous. Dispersion staining is another method which can be used to distinguish between wollastonite and asbestos fibers.

Occasionally we see some of the old filter membranes which show fibrous structure. This doesn't happen very much anymore. I have seen situations in which membrane structures have been counted in the membrane and considered to be asbestos by contract laboratories. If the analyst is alert, this could never happen. But if the analyst just says here is a structure that has a 3:1 aspect ratio, it has to be asbestos, then he can make a mistake.

There are some tests that can be used in the field by the hygienist to get a handle on whether asbestos might be present. If organic fibers are present, one can test them with a match and see if they burn. Use this test in a safe area to avoid an explosion or fire hazard. This same test is used

with fiberglass. Some varieties of fiberglass will melt in the flame of a match or in the flame of an alcohol lamp.

A sample came to me one time that had been analyzed elsewhere; they thought it was asbestos. I had an idea while it was still in the envelope that it was fiberglass, because I pressed it and felt it crinkle. I put it close to my ear and I could hear it crinkle. Fiberglass in an envelope will crinkle in a different way and sound different than asbestos does. My wife, who is a writer, says one should use all of the senses. The same thing can be done to differentiate fibers: use the senses of sight, feel, and hearing. All of our senses can be used.

Another difference between asbestos and fiberglass is observed when one tried to grind either material. Asbestos is a lot harder to grind than fiberglass. Fiberglass grinds easily because it is quite brittle. It even sounds different. Asbestos doesn't act the same way.

I have to give you a caution in the use of these kinds of tests for distinguishing asbestos and fiberglass. Some of the old insulations will contain small amounts of asbestos, maybe 2% or 3%. One wouldn't recognize it visually without microscopy.

OSHA methods in the analysis of asbestos

When we get a bulk sample in our laboratory, we don't always start out with phase contrast microscopy. We may begin with stereo microscopy. As we look at it with a stereo microscope, we can get some visual clues as to what we are working with. We want to get as many visual clues as we can. We have stereo microscopes with which we examine our bulk samples, starting around 6x up to 50x. Sometimes if we are looking for small concentration of asbestos fibers, this is a good way to go.

I will explain what I mean by that. We can do some particle picking out of a bulk sample. If there is a bundle here and a bundle there, we will be able to pick it out and then do other kinds of tests on the bundle such as dispersion staining, microscopy or electron microscopy, or X-ray diffraction. By particle picking, it's possible to have a low concentration and still detect and analyze the fibers. If you grind it up and take out a milligram and look at that, you might miss the asbestos. But if you have gone to the trouble of particle picking, you will be able to find something that might have been missed otherwise.

I like to work with a sample before it's ground fine because if I am looking at the bundles, I get a lot more visual clues than I would if I am just looking at fine fibrils. If we grind it, we want to be careful not to grind it too much. We want to have something that gives us big enough bundles to have a good idea of what we're looking at.

If we have something which is solid, the type of material in which asbestos is solidly bound, and we want to know what's going to happen if it's cut or whether asbestos is present, one way to see the fibers is to cut that solid material with a hacksaw and save the cuttings on a piece of plastic membrane. We can look at them under our microscope and apply several optical tests such as dispersion staining.

I haven't heard much said this morning about X-ray diffraction, but it is commonly used in the analysis of asbestos. One of the methods for the cosmetics industry (3) is X-ray diffraction. This is a screening method to find out whether asbestos is present. In their procedure they look first for asbestos by X-ray diffraction; if asbestos is found by this method, they look at it with a microscope to see if fibers are present. We like to sample to see whether the fibers are present first and if so, we go after them and find out what kind of fibers they are by dispersion staining. X-ray diffraction will tell us which minerals are present, but it doesn't tell us how fibrous the minerals are.

Electron microscopy has been discussed this morning. We have an electron microscope in Salt Lake City, a Jeol Company model 100C which has the capability of transmission, scanning, and X-ray energy dispersive analysis. It is the type of microscope which has been described by the National Bureau of Standards today—they call it the analytical electron microscope. The more one does with the samples, the more expensive it is. If we can analyze a sample and have confidence in our analysis without electron microscopy, that's the way we go. The difficult samples are the ones we save for electron microscopy.

I will say a little about phase contrast. Phase contrast is a means of enhancing the contrast of a particle to make it more visible or to increase small differences in indices of refraction. With phase contrast we can see fine fibers that we might not have been able to see otherwise or at least to recognize the presence of those fibers, and it also enables us to see details of structure in some cases that would not be seen otherwise. This is helpful, but the optical microscope doesn't show morphology to the same degree that the electron microscope does. We recognize that limitation but still this is the standard method and a lot less expensive method than electron microscopy, so it is the method we use. We don't stop with phase contrast microscopy, however. Let's put it this way: if we feel certain that the types of fibers we're looking at are standard and there is no problem in recognition, then we can stop there.

As has been said, about 95% of the samples in industry will be chrysotile. Chrysotile does have characteristic morphology that the analyst can recognize. If things don't look strange, if it looks like very typical chrysotile fibers and there is no evidence of any other fibers or types of minerals, then possibly all that is needed is phase contrast microscopy. All our microscopes in Salt Lake City are equipped so that we can simultaneously use phase contrast and polarized light and retardation plates. There are a lot of microscopes in use that can't make these changes back and forth. Ours are flip-button. We can go back and forth without any loss of time in doing it.

We can analyze the air samples on membranes and use phase contrast and at the same time we can use polarized light and retardation plates. We have already mentioned that we have a big advantage when we use polarized light—we can tell whether a fiber is asbestos or whether it's amorphous. I must make the provision here that

we can distinguish only if the fiber is large enough for the eye to detect reaction of the fiber with polarized light. Likewise, with polarized light and retardation plates there will be a cutoff point where we won't see the colors very well if the fiber is too small.

However, there is this point. You can look at the fibers that are large enough to recognize, to see if there is going to be any fine stuff that may be causing some problems. Some fiberglass is manufactured in which all of the fibers are very small. But here again, we get some clues if we see a lot of very long fibers and we don't see any bundles. Then we wonder where these fibers are coming from. We know in this situation that we must do some more investigation. What we have to do is use as many visual clues as we can and tie the information together so that we can know when to be suspicious of our results.

If we do use polarized light and retardation plates, quite often plant fibers will show internal structure. We will get little cells of color adjacent to each other that will tell us we are looking at plant fibers. Occasionally a plant fiber shows a uniform color along its length as asbestos fibers do. In this kind of situation we have to look carefully at the morphology, how the fine fibers come off, are they stubby, and so on. Then if there is something about the fiber that suggests it might be borderline, we can go to dispersion staining.

Dispersion staining can be done with fibers taken from a membrane. There are a number of ways to do this. One way is to dissolve the membrane in acetone and centrifuge the fibers down. After doing this a couple times, to get rid of the material from the dissolved membrane, we put a drop of residue on a slide, let it evaporate, and then do dispersion staining on it. While working with acetone, use small quantities. You know how explosive acetone is. If you are pouring from a large container, be sure the container is grounded.

Another technique is to take the membrane, double it over, and put alcohol over it. Burn it until the membrane has burned away. I don't suggest ashing in a furnace because it will explode and then the sample is lost. Another thing one can do if the laboratory is equipped is to use a low-temperature asher. Place the membrane segment on a slide. This is convenient if one has that kind of equipment.

Dr. McCrone has mentioned the advantages of dispersion staining. I heartily agree with him as to the advantages of dispersion staining for the identification of asbestos. In his literature on the subject, he has pointed out that with three different media, one can identify asbestos fibers. In 1.67 medium we can identify amosite. In the same 1.67 medium, if we have crocidolite we will know it. In a 1.605 medium we can identify tremolite and anthrophyllite. We are also able to tell the difference between wollastonite and asbestos fibers in a 1.605 medium.

Dispersion staining is a very powerful tool. Every laboratory should know how to use it and it can save a lot of time because it's faster than any of the other techniques in use. However, I don't think dispersion staining is the way to count

asbestos fibers. I think the way to count asbestos fibers is phase contrast, with dispersion staining used as a backup for identification so that we know for sure what we are counting.

QUESTIONS AND ANSWERS

JAMES FERGUSON: How many samples do you analyze?

MR. DIXON: We analyze about 800 samples a month in our laboratory. That's quite a sample load.

VOICE FROM THE FLOOR: Are you saying that every sample that comes into the OSHA laboratory at Salt Lake City is first looked at under the microscope and if you find a fiber, you can flick over to polarized light, identify the same field that you would, say, under phase contrast, and do your counting measurements on phase?

MR. DIXON: Yes, that is correct. If, for some reason, we feel we need to use polarized light and retardation colors to check it out, particularly if it's large enough to react well, then we are equipped to use these other techniques and we can make the changes back and forth almost instantaneously.

VOICE FROM THE FLOOR: On the same field?

MR. DIXON: On the same field, yes.

VOICE FROM THE FLOOR: Does this imply that you count only those fibers that identify as asbestos?

MR. DIXON: We are making an effort, a very serious effort to try and count only asbestos. Now, it may be that sometime we will make a mistake. But, we are making a very vigorous effort to be sure that what we are counting is asbestos and, so, we use as many techniques and as much technology as we need to be sure that we are pretty accurate.

VOICE FROM THE FLOOR: You mentioned you used dispersion staining as backup when you use your phase contrast microscopy. do you take another section of the air filter and do a per cent by number of the types of asbestos on that and then apply that to the counting filter?

MR. DIXON: Yes, we do. A particle picking method is described in *The Particle Atlas* (4). It's possible to take a filter and isolate it and do dispersion staining and other tests on it. Alternatively, we can take a second wedge and analyze the fibers on it by dispersion staining to determine the percent of asbestos and the percent of non-asbestos fibers. We then apply that as a correction factor to our fiber count which may include asbestos and other types of fibers.

VOICE FROM THE FLOOR: Do you have any data that would indicate what kind of count you have in the phase contrast counts, how well you recognize the need for going on to dispersion staining?

MR. DIXON: It's hard to get a percentage breakdown. Whenever we do recognize a need, whenever there seems to be a possibility of mixed non-asbestos and asbestos fibers, we go to auxiliary techniques. I would guess that it's probably about 5%. I'm not really sure because we haven't kept data that way.

BENJAMIN LEVADIE: Did you have the opportunity to see Dr. Burney's recent article in *The Scientific Review* regarding the evaluation of fibers?

MR. DIXON: No, I don't believe I have.

MR. LEVADIE: Dr. Burney described the technique published in Europe by Dr. Baer in 1973—I think Dr. McCrone is familiar with that—in which the membrane is clarified with a solution of sycalose, enabling some formaldehyde to explore the membrane and the material you are looking at in 1.55. When using such a system, it seems to me that one would be able to do the counting and identification of chrysotile at the same time without having to buy a big \$12,000 microscope which of course the federal government can afford, but very few other laboratories can do this.

I am just wondering why nobody in OSHA or in the federal establishment has looked beyond the shores of the United States for answers to the dilemmas we have.

MR. DIXON: I would like to make a comment on that. When you do a dispersion staining, the principle of dispersion staining is to match the refractive index of the particle.

Now, the closer you come to a match, the more difficult it is to see the particle. So, you have to decide whether you want to be sure you identified every particle or whether you want to see every particle because I suspect that if you are using dispersion staining, very closely matching the index of the particles, there are going to be some of the fine particles that you do not see.

MR. LEVADIE: Not if they are colored. This is what we are talking about.

MR. DIXON: But you see, if the fibers are very fine, you won't see the color or the fibers when the index is matched.

MR. LEVADIE: I would challenge that from personal experience.

JOAN WRONSKI, National Loss Corp.: I am in a little bit of a dilemma. Are you talking about these fourteen laboratories, seven of which received samples from you?

MR. DIXON: I didn't say how many received samples from us. All I said was that of fourteen laboratories, seven offered to analyze bulk samples.

MS. WRONSKI: Then I cancel my question but I have another. If you are doing air samples for industry and you have, say, someone doing concurrent client sampling with your OSHA people, they get their own answers using the standard method of test and you are picking out not only identifying fragments, you are going to come up with two different answers on an concurrent sample.

MR. DIXON: This is true.

MS. WRONSKI: How do you explain it?

MR. DIXON: What this means is that industry, if they use only phase contrast, is going to be more restrictive and give the workers better protection in the sense that they are counting all fibers, including glass fibers, plant fibers, and other kinds of fibers, while calling that the level they are going to protect against. When we do our counts,

we have to be able to go to court and support our results; we have to be able to say to the judge, "When we count, we are counting asbestos fibers."

So we make an honest effort to do just that, and there are other laboratories that do the total fiber count.

VOICE FROM THE FLOOR: You seem to be saying, interestingly enough, that OSHA does not want to go by the law in this case because the law in Section, what is it, 1000 etcetera, doesn't give me the option of picking the particles out and counting.

MR. DIXON: *The Federal Register*, 1910.1001 (5), says that "asbestos includes chrysotile, amosite, crocidolite, tremolite, anthrophyllite, and actinolite." Section f(6) says that monitoring shall ensure that exposure to asbestos fibers is below prescribed limits, and section e(7) prescribes the membrane filter phase contrast method for counting. These sections don't say that non-asbestos fibers must be counted or controlled. They refer specifically to asbestos fibers.

I am going to refer you to the NIOSH documents (8,9) which say that in an atmosphere known to contain asbestos fibers, fibers are counted and considered to be asbestos in the absence of other information, and the entire question of how this other information is to be obtained is left open. If the hygienist goes into a plant and all they are using there is fiberglass, and if OSHA comes back with a citation for asbestos, what will happen when the judge hears that evidence? Or how about the situation in which all they are using is plant fibers?

VOICE FROM THE FLOOR: That's not what the law says. The law isn't going by the criteria document, but by what is said in 29CFR1910.

JIM FERGUSON: What he just said is on page 621 of *The Federal Register* (10) which says you use this method (11), and this method on page 28 says what Mr. Dixon just said about the absence of other information. If an atmosphere contains asbestos, fibers are assumed to be asbestos in the absence of other information; therefore, you are always on the safe side which protects the worker.

MR. DIXON: If we can find solid evidence that what we are looking at is not asbestos, we are not going to "zap" some company to put in a lot of expensive ventilation control for a problem that isn't there.

If we feel the problem is there, we are going to make them do that, but we are going to be as accurate as we can in determining the seriousness of the problem.

VOICE FROM THE FLOOR: One comment. The inter-laboratory and the environmental variability between two very close samples taken concurrently is so great that the asbestos contained in both of these, there is little difference in the overall condition, in other words, a prudent employer will look at, in order to determine upper confidence levels in order to determine whether or not there is a possibility of exceeding any legal standard—and those are extraordinarily broad—there are very great differences between samples and between laboratories, so it really doesn't make

much difference if the employer is prudent and wishes to avoid a citation.

References

1. Taylor, David G.: *NIOSH Manual of Analytical Methods*. 239:1-21 (1977). NIOSH, Cincinnati, OH.
2. Leidell, N.A., S.G. Bayer, R.D. Zumwalde, and K.A. Busch: "USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibers." 79-127: 81 (1979). NIOSH.
3. Cosmetic, Toiletry, and Fragrance Association: "Method J-4." 1133 15th St., N.W., Washington, DC 20005, (202) 331-1770.
4. McCrone, W.C. and J.G. Delly: *The Particle Atlas*. Ann Arbor Science Publishers, Inc., Ann Arbor, MI, pp. 222-257.
5. *Code of Federal Regulations*. 29 Labor, Parts 1900-1919, revised July 1, 1977; 29 CFR 1910.1001, sections (a) 1,2, and (b) 2.
6. Reference 5, section f.
7. Reference 5, section 3.
8. Reference 2, paragraph 3, p. 2.
9. Reference 3, paragraph 3, p. 28.
10. Reference 5, section e.
11. Reference 1 and Reference 2.

Problems in Assessing Asbestos Concentrations Under Realistic Conditions

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The time has come for some kind of link between OSHA, MESA, and EPA. These agencies are obviously interested in asbestos in ambient air and in the workplace. As far as proposed or existing standards are concerned, there is about a thousand-fold difference between the workplace standard and any proposed ambient standard.

EPA Round Robin

The Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. EPA (Research Triangle Park, NC 27711) has conducted the *first* comprehensive "Comparison of Ambient Asbestos Levels Determined by Various Laboratories." The most informative results were published in September 1977.

The EMSL/RTP was asked by Region III to provide technical support to the effort being made by Montgomery County, Maryland, the State of Maryland, and EPA (Region III) to assess the problem of airborne chrysotile asbestos in the vicinity of the Rockville Crushed Stone Quarry. One of the efforts proposed was a comparison of laboratory results obtained from the analysis of samples taken from air which would include replicate samples—samples taken at *one* site and at *one* time by identical samplers. The study was to provide data for an assessment of the State of the Art in analysis of air samples for asbestos by participating laboratories.

Sampling methods

Real samples suffer from inhomogeneity. This results from the collection of a large chrysotile fiber which can contain as many as a hundred thousand fibrils along with homogeneously distributed fibrils of chrysotile. In addition, real samples contain extraneous matter.

Sampling was performed using a modified membrane particulate sampler. Flowrates were reduced to 2.5 cfm. A 0.45 micron porosity 102 mm diameter membrane filter HAWP 10250 was used for the collecting medium. Samplers were operated on a 24-hour basis from midnight to midnight for a period of ten days. In all, samples were obtained in triplicate on three separate days—one set of triplicates had high, one set

intermediate, and one set low particulate loading. Samples were removed from samplers using forceps and placed in a sealed glassine envelope.

Upon return from the field, the nine filter samples were placed in an equilibrating rack in a temperature-humidity-controlled room for 48 hours. After equilibration, each filter was weighed and replaced in the envelope in which it came from the field. All filters and an unexposed (blank) filter were packaged carefully and sent to the Northrup Corporation in Hawthorne, CA, where they were sectioned under clean room conditions. All segments were placed in an individual glassine envelope appropriately numbered. Ten envelopes containing one segment from each of the ten filters were placed in a petri dish and sent to these participating laboratories:

- University of California (Dr. Jack Murchio)
- McCrone (Mr. Ian Stewart)
- Mount Sinai (Srs. Langer and Rohl)
- EPA Duluth (Dr. Phil Cook)
- ITTRI (Dr. John Stockham)
- Bureau of Mines (Dr. William J. Campbell)
- Battelle (Mr. Carl Melton)

The letter of transmittal requested the laboratory to measure, if practical, and report the following for each type of asbestos (May 5, 1977):

- mass
- fiber count (define fiber)
- fiber size distribution

in terms of the total quantity on the segment examined. The laboratories were told the approximate mass of particulate matter per sample for each level. Each laboratory used its own routine procedure. All data were converted to the total quantity of interest on an entire filter.

Results

The reported fiber data (not fibrils) are shown in Table I. Chrysotile mass information is presented in two tables. Some laboratories calculated a mass by assuming a density of chrysotile. The volume of the fibers was estimated from fiber length and diameter and multiplied by density to find the mass of the material, as shown in Table II. Alternatively, other laboratories ground all chrysotile present to fibrils (by trituration or

ultrasonication), determined a fibril count, and obtained an estimate for the mass from a calibration curve based on measurements of fibril counts obtained from quantities of chrysotile. These data are presented in Table III. Data presented in Tables I, II, and III are summarized in Tables IV and V together with the appropriate standard deviations. Summary information is also presented in Table VI where, for each of the classes of procedures identified, the geometric means of the three levels are listed followed by —the within-laboratory geometric standard deviation —the between-laboratory geometric standard deviation —the expected 95% tolerance limits for the within-laboratory and the between-laboratory variability.

The within laboratory variability (standard deviation) is an estimate of the ability of the average laboratory in this group of participants to reproduce a single result of its own for each of the three levels of samples supplied. The between-laboratory variability (standard deviation) estimates the variability of results between the participating laboratories for the sample. These standard deviations may seem curiously small with respect to the mean values cited. However, this is due to the fact that they are geometric standard deviation.

CONCLUSIONS

* —“The limits of variability are so wide in every situation for the data of this study that the probability of accurate judgement based on this data is small.”

—“Because of the extremely broad dispersions it would be more fruitful to engage in method evaluation and development than to perform further statistical analysis of data.”

—“The most important inference to be gained from this study is the need for a standard sample of asbestos for inter- and intra-lab comparisons and analytical development work.”

There is indeed not much to be added to EPA's current assessment of ambient asbestos concentration measurements. However, it should now be abundantly clear that any previous (and current) measurements of asbestos concentrations (either by mass or by fiber size distribution) must inherently have *uncertainty* factors similar (probably greater) to those reported above from carefully controlled experiments (not routine samples). Hence, any epidemiological study might correctly assess the *medical* parameter but its relationship to the ambient *absolute* level of asbestos (chrysotile) is uncertain.

For example, Figure 1 prominently displays 30 ng/m³ as proposed standard. In light of the quoted EPA experiment, the 95% tolerance limits (see Table VI) for this concentration range are of

the order of

- * 624 ng/m³ and 1104 ng/m³ on volume/density basis (within labs) (between labs)
- * 121 ng/m³ and 88 ng/m³ on weight/count basis.

If one were to “roll back” this experimental uncertainty to the appropriate emission level (as was done with the 30 ng/m³ proposed asbestos standard), it would yield values of

- * 500 g/day and 880 g/day for the volume/density ambient mass determination and
- * 100 g/day and 70 g/day for the weight/count based ambient mass determination.

Obviously these emission levels calculated on the basis of a 95% confidence level for ambient concentrations of around 30 ng/m³ are only of academic interest.

The main conclusion to be drawn at this time is that implementation of an air-quality standard and consequently an emission standard for asbestos should be delayed until more reliable information becomes available.

Limits of detection

The deficiencies and serious problems associated with accurate asbestos mass/fiber determinations in ambient air are well recognized and acknowledged by the scientific community, although the severity of the situation was not so clearly documented before the EPA “Round Robin” experiment of 1977. Rather than elaborating on the seemingly endless list of reviewed publications on asbestos-related measurement problems, a short summary from a recent EPA report of 1978 on limits of detection might be appropriate. Under the supervision of Dr. Jack Wagman, Director of EPA's Emissions Measurement and Characterization Division, Sumudra et al prepared a manual (1) describing a provisional optimum electron microscope procedure for measuring the concentration of asbestos in air samples. Electron microscopy is currently the principal technique used to identify and characterize asbestos fibers in ambient air samples.

—“The several laboratories that perform such analyses generally have reasonable internal self-consistency. However, interlaboratory comparisons have shown that the results obtained by the separate laboratories are often widely different.”

—“The minimum detection limits using a high-volume air sampler are shown in Table VII. “The main features of the method include depositing an air sample on a polycarbonate membrane filter, examining an E.M. grid specimen in a transmission electron microscope, and verifying fiber identity by Selected Area Electron Diffraction.”

Extrapolating this minimum detection limit linearly to sample duration of 30 days and volume of air sampled on the order of 3000 m³ would yield a hypothetical lower detection limit for fibrils of 7500 m⁻³. The prolonged exposure time undoubtedly has a detrimental effect on the overall accuracy. Also note that the minimum detection limit is solely based on counting

statistics and does not include any systematic or random differences occurring during sampling, preparation, transfer, etc. As a result, those numbers are reliable only within a factor of 10 at best.

Recommendations

It is somewhat sobering to realize that millions of dollars ride on the results that are being derived from such measurements. Standards must be set on the basis of reproducible, reliable measurements based on accepted procedures and methods. Any other approach will unnecessarily

drain the cooperative spirit between the industrial, governmental, regulatory, and scientific sectors and, in the long run, discredit the environmental cause.

Reference

1. Sumudra, A.V., C.F. Harwood, and J.D. Stockham: *Electron Microscope Measurement of Airborne Asbestos Concentrations*. ITT Research Institute, Chicago, IL 60616. EPA-600/2-77-178. Revised June 1978.

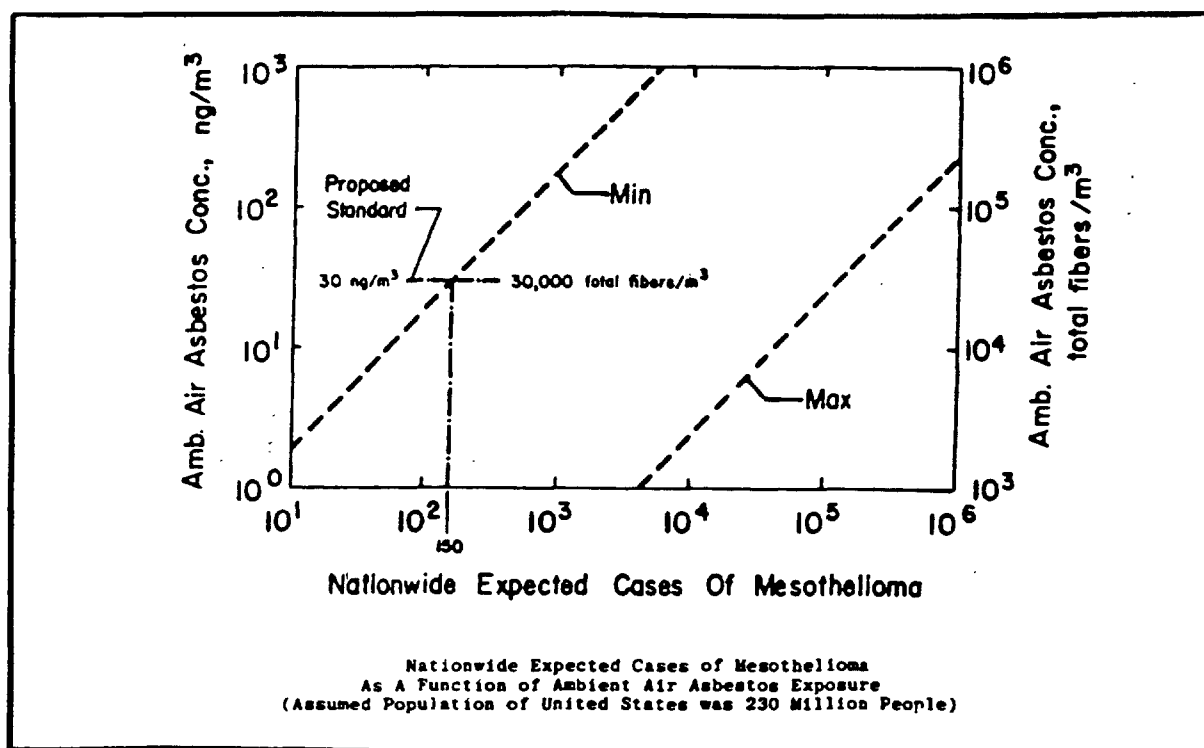


Figure 1—Nationwide expected cases of mesothelioma as a function of ambient air asbestos

exposure (assumed population of United States was 230 million people).

Sample #	Fibers x 10 ⁴ /m ³						
	Univ. of California	McCrone	Mt. Sinai	EPA Duluth	ITTRI	Bureau of Mines	Battelle
1	BDL*	BDL	1350	14.5	BDL	216	3.3
2	BDL	BDL	1530	BDL	BDL	125	19.8
3	BDL	BDL	1260	26.4	0.38	17	103
11	.768	.0636	3330	13.5	2.53	273	618
12	1.27	2.62	2430	148	7.26	660	1710
13	.211	3.03	2340	103	4.21	336	428
21	.521	4.17	5310	168	-----	2820	1370
22	.158	3.33	9450	33.8	15.9	1670	5480
23	.768	6.91	10200	27.0	----	918	3070

* Below detectable level
**No value reported

State of Maryland reported fibril counts--not included here

TABLE I

LAM 001233

Mass (Volume/density basis) ng/m ³				
Sample #	McCrone	EPA Duluth	Bureau of Mines	Battelle
1	BDL*	2.24	65	0.31
2	BDL	BDL	30	4.3
3	BDL	14.2	---**	7.6
11	BDL	1.31	43	56
12	137	87.5	83	207
13	2.1	12.0	28	162

TABLE 2

Mass (Weight/count calibration basis) ng/m ³			
Sample	Mt. Sinai	Battelle	State of Maryland
1	44	2.1	BDL*
2	128	9.9	0.017
3	55	51	0.007
11	371	281	0.35
12	336	734	1.02
13	206	216	0.55
21	807	686	0.40
22	1,225	2,803	0.20
23	1,305	1,535	0.56

*Below Detectable Level

TABLE 3

Asbestos Fiber Count; Fibers x 10 ⁴ /m ³												
Sample Set	Suspected Level	Laboratory										*Overall Avg. & Std. Deviation X σ
		A	B	C	D	E ¹	F	G	H			
	Low	BDL ²	BDL ³	1400 200	20 11 ⁴	0.6 0.3 ⁵	0.4 ⁶	120 120	40 60	316 602		
11,12,13	Medium	0.7 0.5	1.9 1.7	2700 600	90 80	32 19	4.7 2.8	420 220	900 800	588 999		
21,22,23	High	0.5 0.3	4.8 2.1	8000 3000	80 80	19 11	16 ⁶	1800 1100	3300 2400	1886 2960		

¹ Reported as fibrils and not fibers.
² BDL--Below Detectable Limit; unknown value.
³ BDL--Below Detectable Limit; 870 fibers/cm².
⁴ Does not include one sample reported as not statistically significant (less than 9000 fibers/cm²).
⁵ Does not include one sample reported as BDL (value unknown).
⁶ All three samples analyzed; two BDL (value unknown).

The overall averages and standard deviations were obtained by applying the statistical technique of R.B. Dean and W.J. Dixon, Analytical Chemistry, 23 636 (1951). Data from Laboratory E not included.

A -- University of California
 B -- McCrone
 C -- Mt. Sinai
 D -- EPA Duluth
 E¹ -- State of Maryland
 F -- ITTRI
 G -- Bureau of Mines
 H -- Battelle

TABLE 4

Mass (ng/m ³)											
Sample Set	Suspected Level	Laboratory									*Overall Avg. & Std. Deviation X σ
		A ¹	B	C	D	E	F ¹	G	H ²	H ²	
1,2,3	Low	--	--	75±49	8±11 ³	0.012±0.009 ⁴	--	48±31 ⁵	21±27	4±4	26±30
11,12,13	Medium	--	46±81	304±97	34±51	0.64±0.40	--	51±33	410±306	142±89	141±151
21,22,23	High	--	44±77	1110±292	13±17	0.39±0.21	--	173±127	1615±1249	477±266	490±497

¹ No mass estimate furnished.
² Laboratory H estimated mass by two different techniques
³ Does not include one sample reported as not statistically significant (less than 9000 fibers/cm²).
⁴ Does not include one sample reported as BDL (value unknown).
⁵ Based on two samples only.

The overall averages and standard deviations were obtained by applying the statistical technique of R.B. Dean and W.J. Dixon, Analytical Chemistry, 23 636 (1951).

A¹ -- University of California
 B -- McCrone
 C -- Mt. Sinai
 D -- EPA Duluth
 E -- State of Maryland
 F¹ -- ITTRI
 G -- Bureau of Mines
 H² -- Battelle (weight/count)
 H² -- Battelle (volume/density)

TABLE 5

(Deleting One Laboratory's Mass Assessment)*

Sample Sets	Geometric Mean Fibers x 10 ⁴ /m ³	Avg. Geometric Standard Deviation		95% Tolerance Limits	
		Within Labs Single values	Between Labs Single values	Within Labs	Between Labs
Low	27.2	2.4	27.8	157.4 4.7	21,000 .04
Medium				213.7 6.4	28,600 .05
High				533.7 16.0	71,400 0.1
	Mass (Volume/density basis) ng/m ³			Mass (Volume/density basis) ng/m ³	
Low	7.5	4.4	5.9	147 0.4	259 0.2
Medium	31.8			624 1.6	1,104 0.9
High	37.7			738 1.9	1,306 1.1
	Mass (Volume/count basis) ng/m ³			Mass (Volume/count basis) ng/m ³	
Low	25.3	2.2	1.9	121 5.3	88 7.3
Medium	257			1,231 54	895 74
High	1,250			5,985 261	4,353 359

*The mass data from one laboratory were not from the same data population and thus deleted

TABLE 6

Minimum Detection Limits Using High-Volume Air Sample

	Sample Duration	Volume of Air Sampled m ³	Field of View Method (1 fiber in 100 fields) fiber/m ³
Point Source	½ hr	21	1,070,000
Near Source	2 hr	84	270,000
Distant Source	8 hr	336	67,000

$$\text{Detection limit} = \frac{1 \text{ Fiber}}{100 \text{ Fields}} \cdot \frac{406 \text{ cm}^2}{0.18 \times 10^{-6} \text{ cm}^2/\text{field}} \cdot \frac{1}{\text{Volume of m}^3}$$

Table taken from Sumudra et al: *Electron Microscope Measurement of Airborne Asbestos Concentrations*. (1)

TABLE 7

Moderator's Opening Remarks

BENJAMIN LEVADIE

Retired Director of the Vermont Division of Occupational Health Laboratory, Barre, VT

First of all, I'd like to say something about the last speaker's remarks. I thought that was a splendid presentation. In a nutshell, what he did was to tell us, "Look, we have many problems in the analytical methodology, we have a good deal of confusion there which needs to be cleared." What he didn't say, but what he certainly alluded to, was that there is a similar confusion in the epidemiologic aspects of this problem—we are being told to look for "asbestos", and in the colloquial, "there ain't no asbestos."

There is chrysotile. There is amosite. There is crocidolite. You name it, but there is *no* such thing as "asbestos" itself. "Asbestos" is a generic term which has confused our problems to a very serious degree.

The setting of the OSHA standard around that generic term has added more confusion. What are we doing now? We are hearing that the permissible level of the number of "asbestos-form" fibers in an environment will be reduced to whatever number that is going to be, and that the reduction is *not* addressing itself to the one salient fact in the United States: 95% of all the material used in this country, which we call "asbestos", is actually chrysotile. I will say, at the risk of being slapped by an epidemiologist here, that the indictment of chrysotile as a carcinogen is not yet in. The latest work coming out of Canada raises a grave question with regards to *any* carcinogenicity for chrysotile, whereas the question of carcinogenicity for amosite and crocidolite is clear.

We are told: "We know chrysotile is a substance which will cause asbestosis or pneumoconioses related to 'asbestos.'" But we are not willing to say, "Yes, it causes mesothelioma; yes, we know it causes cancer." Not even the McDonalds up in Montreal are ready to do that yet, if ever.

The Moderator is supposed to keep the meeting within bounds, to restrain discussion. That is Webster's definition of a moderator. Although I hope we shall keep within bounds of our subject to a degree, I hope as well that we shall not confine ourselves only to the measurement of airborne asbestos but will also include questions respecting the identification of asbestos-forms, and I invite all, all of you, *not* to restrain discussion. Please! There has been too much restraint and I am afraid too much patience with the blunderings which have guarded the technology of determining asbestos-forms. As I see it, the ultimate purpose of this symposium is to arrive or to try to arrive at unequivocal conclusions, conclusions which are long overdue.

To begin with, we need an honest definition of what we mean by "asbestos." Perhaps we can get that on the record. I think there has been some reference to that here already. There is so much shading, fuzzing, and slithering of this definition in both the analytical and the epidemiological

fields that one becomes tempted to suspect the confusion is deliberate.

There are geologists who like to divide the asbestos-forms of minerals into two groups of fibers: chrysotile and amphiboles. As I pointed out earlier, we have clear evidence that the amphiboles are dangerous, extremely dangerous, but the story on chrysotile is not yet in. Let us remember, too, that these fire-resistant fibrous materials have been in use for at least 2000 years, and that their morphologies and compositions have been studied for about a hundred years. It might be thought that by this time, methods for their identification and quantification would be commonplace and relatively non-controversial.

We know after what we have heard here during the last few hours that they are not noncontroversial. The determination of asbestos-form fiber content in the air is at present a muddled and confusing technology. At the root of the problem, I believe, is traditionalism. You have heard a good deal of that here today.

Traditionally, the methods for sampling and analysis of airborne dusts called for impingement in a liquid concentration. When size-selective sampling by impaction became available, the various techniques built around that concept offered a new route. During the course of the last decade, industrial hygiene has been provided with a cheap and relatively simple sampling system—the cyclone, which I would call a kissing cousin of the impactor. Regrettably, none of these devices have become readily available for the sampling of fibers, and so we continue to be saddled with simple filtration devices for fiber-sampling and the problems associated with them.

But this is not all. Tradition has it that when one wants to quantitate a dust, one uses a microscope. In the evaluation of other dusts, tradition has been aborted. I no longer count dust particles to get an idea of the quartz content of the air in the granite sheds in Vermont.

The major justification for counting fibers is cost. It's supposed to be cheaper with a microscope. By point of view, the most attractive and meaningful equipment for the evaluation of fibrous dust content in the air is the electron microscope; I'm using the term in a generic sense. You can take your choice as to what form it takes or how you employ it to obtain morphological information and also to count fibers, as we heard this morning. However, we are told it is too costly an approach. The inference to be drawn from the NIOSH recommendations to count fibers is this: it is more useful for the controlling of an occupational hazard to work from relatively meaningless information which is cheaply obtained than from meaningful information which is costly.

Yesterday I talked to Dave Trayer about this aspect. He has a problem in his system and I

asked him in so many words whether it would be worth his while to spend \$150,000 or so to find out what was needed, to have the instrumentation to find out what was *truly needed*, before he spent \$100 million to rectify or try to rectify conditions in his system. I think you know the answer. This is where the game is played. We do talk about money like that \$100 million, not \$50,000 or \$40,000 for proper instrumentation to get the facts. So let us remember this: with respect to asbestos-fiber control, it is important that we *know* the kind of asbestos involved.

This point you brought out beautifully this morning and it applies just as well to the establishment of threshold limit values. I am frightened by the present trends towards the establishment of a new threshold limit value—one value—for all asbestos-form fibers.

Optical microscopy, no matter how sophisticated tell us what kinds of fibers are being counted. We can make a very good educated guess as Dr. McCrone did, I'm sure, but there is always an element of uncertainty, for optical microscopy cannot quantitate those fibers which are below the size (diameter) which it is within the power of the optical microscope to distinguish, but which are long enough to have a profound physiological effect.

Furthermore, I haven't seen anything in the epidemiological literature which will make me feel comfortable about the fibers which are less than three-tenths of a micrometer in diameter and more than five micrometers in length. They make up a large and profound proportion of the fibers present environmentally and in the workplace, but they cannot be seen by optical microscopy.

I would say this in defense of the legally sanctified phase contrast microscopy fiber count: it has been said that when it is applied to the environment for which it is intended, in mines and factories where *only* asbestos-form fibers are being processed, then it is satisfactory. Even so, it is not satisfactory to the extent that it does *not* evaluate the invisible fibers which are physiologically active—those below three-tenths of a micron in size. Beyond that, given the extremely low permissible fiber counts and the generally low concentrations of these fibers found in most environments now, what assurances are there that all the fibers being counted by this method, even when looking at a factory where "only asbestos" is being used, are obviously derived from asbestos-forms? This is a question we need to answer.

There is one other area I want to touch on. In 1970 Dr. McCrone, following a well-beaten scientific path, redefined an excellent method for the quantitative analysis of chrysotile. He published on it and I'm grateful that he elaborated on it again today.

But, unfortunately, in 1977 Dr. McCrone also described the usefulness of polarized light microscopy. I say "unfortunately" not because I regret it—it's a good method—but because there was a statement in the paper which, when read by the unwitting, appeared to condemn all other methods so far as their ability to identify asbestos-

form fibers was concerned. I think he knows what I am talking about.

There are a lot of the unwitting reading scientific papers. After the present concern with respect to asbestos-form fibers in school insulation became fully exploded (and one may trust the unwitting who write stories for the media to do this sort of thing), the Environmental Protection Agency was obliged to respond to public furor. It came forward with an *implicit recommendation* that polarized light microscopy would be the method for consulting laboratories to use in identifying asbestos. I don't need to make any more comments on this area.

I hope that during this discussion we will be able to come forward with some clear and affirmative guidelines concerning the appropriate methods to use for the identification of different forms of asbestos fibers and perhaps even bulk materials. I hope, too, that our discussion will be free and energetic. That's what we need. Now, folks, if this doesn't turn into a donneybrook, I'm going to be disappointed.

Panel Discussion

RICHARD LEE: I would like to start off by suggesting that we propose a proper physical definition of asbestos, one which is in term of the aspect ratio and size to be measured and one which is consistent with mineralogical observations or some other such measurement—namely, that we throw out the aspect ratio which gives everybody fits. I would welcome comments from any of you.

DOUG HOLLAND: I think we are getting into dangerous territory in that such a definition must necessarily include some consideration of the health effects and I don't believe that this is the right gathering for such consideration. I certainly don't feel qualified to make such a definition myself and that's what we are all after in the end. We are after exploring the health standards and preventing any potential health effects that might exist.

MR. LEE: I would suggest that there is no consideration, there has been no consideration of health effects involved in the adoption of the 3:1 definition, so I don't think that's appropriate.

But secondly, I would say as a hygienist which most of us are, that we know there are morphological criteria—there are shape aspect criteria which any one of us sitting on a microscope use to say, well, that's a 3:1 fiber and that's an asbestos fiber. There are some gray areas, as I think everyone will agree, but I think you can make a very good distinction. You might want to make it 15:1, you might want to make it 10:1, you might want to make it 20:1.

The yellow booklet characterizes it as 100:1, something that is physically meaningful rather than getting down to the point of asking, did you see a 3:1 particle? Or it has something else that you don't count as asbestos and I do or vice versa. Neither of us are ever going to get anywhere.

MODERATOR LEVADIE: I would like to add that in July 1976 Ed Baier told me, on the lawn of

Johnson College in Vermont, that the NIOSH definition of an asbestos fiber was the Washington Monument. We can take it from there.

PETER BELL: In that same vein, I believe that the term amosite should be stricken from the literature. For asbestos mines in South Africa some people believe it's grunerite. I just believe that the term amosite should be stricken from the literature and we should deal with a distinct mineral name such as antigorite, crocidolite, chrysotile, whatever. It's an ambiguous term.

MODERATOR LEVADIE: Do you want to address anyone specifically on the panel to pick up this question?

MR. BELL: Anyone can comment.

WILLARD DIXON: We know that amosite stands for an asbestos mine in South Africa. We are talking about a commercial product. This is a designation used commonly in commerce. I personally see nothing wrong with calling it by the name which is used in commerce.

We recognize that it's a member of the Commingtonite series, grunerite series. But a rose by any other name would smell as sweet. Asbestos fiber by any other name is just as toxic, so whatever we call it, as long as we are speaking in the same language, that's okay with me. If everybody agrees to call it amosite, which they seem to have done already, then at least we know what we're talking about.

STEVE BAYER: I would like to make a comment. Back in the sixties I was out taking a lot of asbestos samples, as probably you have too. We were told you count fibers and with all the trash in the air, the air is not pure amosite, the air is not pure chrysotile, there are all kinds of trash in the air, so you do the best you can and you count the fibers. If you see a bag labeled "Amosite" you pretty much know what it is, so you go back and count and do the best you can with all the trash floating around that ends up on the filters.

I think your comments are justified. I think they should be more accurate, but there are so many ways you are kicking around terminology which, I agree, sometimes in terms of private count may not make that much difference. Who is to say that in some cases it is just asbestos fibers, anyway who is to say that it is not the 3 to 1 ratio particulate causing the problem. I will be the first to admit that the count statistics have probably improved because we would be getting rid of some particles, but that 3 to 1 aspect ratio is recognized almost on a worldwide basis. Perhaps they are physiologically insignificant. I think you can kick that around for a long time too.

MODERATOR LEVADIE: We are dealing with traditionalism, aren't we, folks?

JOHN DAVIS: At the risk of being out of order at a symposium to discuss asbestos identification, let me suggest, on the basis of some of the research reports I've seen, that it may not be necessary to identify asbestos.

I am referring to a report on fiber size distributions in the NBS symposium two years ago. Those gentlemen delineated a fiber diameter

and a fiber length which were carcinogenically active. They asked themselves if their proposed Fiberglass standard did specify a fiber diameter and a fiber length. Perhaps what will happen in the future, as we do more research in this area, is that there should really be a standard for airborne fiber exposure which would delineate that fiber diameter and the fiber length, and that all we have to address ourselves to is the method which will give us an accurate count of those fibers that are physiologically active.

MODERATOR LEVADIE: Does the panel want to take that up?

MR. DIXON: It sounds like it's working now. I agree that we should be regulating fibers in terms of the length and the dimensions of the fibers so that we are giving workers protection against those fibers that are physiologically active.

Now, I can't voice that as an official representative of OSHA because I am not the one who has the authority to set policy in that regard, but, as a private person who has some knowledge of asbestos, I will agree with that remark.

And I say further that we see situations in the laboratories where we know the workers are not being protected under the present standard. What I am talking about, for example, is when wollastonite is substituted for asbestos, because asbestos is regulated and wollastonite is not. What do we have to show that wollastonite fibers are any safer than asbestos fibers?

Another example is the use of stock that is not regulated, fiber stalk asbestos. You go to the asbestos standard and it lists six asbestos minerals. It doesn't mention stalk. We have to go to court, and we have samples with stalk fibers and samples with fibers which are similar in composition. We are not counting those. Here are workers being exposed to all these fibers that are not controlled under the present standard because it is not set up to protect workers in terms of fiber content rather than just asbestos content.

DR. ERIC STEEL: You mentioned that wollastonite and asbestos have the same morphologies—is that true?

MR. DIXON: In my talk earlier, I pointed out the differences between wollastonite and asbestos. It is possible to distinguish between asbestos and wollastonite.

DR. STEEL: Do they have the same dimension?

MR. DIXON: Well, as far as fiber size distribution is concerned, you won't find wollastonite fibers as long and thin as asbestos.

MODERATOR LEVADIE: I am going to have the last word on that. Would the gentleman over on the other side like to comment?

SLIM THOMPSON: I jump around here. I want to comment in reference to the 3:1, 5:1 government definition of a fiber. Nobody ever saw wollastonite fiber, at any rate of the dimensions that this gentleman was talking about at the NBS—who I believe was Stanton—and these dimensions were a quarter of a micron and down; longer than an eighth is where they started.

This is a 3:1 aspect ratio, which has been stated to be accepted worldwide. But my experience has

been that in other countries the majority laugh at us. Three to one came primarily from the Asbestos Research Council in London.

I served on a committee with Steve Holmes that developed this criteria, as a counting technique in a chrysotile atmosphere below which there was no way it could be a fiber. Steve says that if he had made it 10:1—which he now wishes he had—he would have had no difference in his counts whatsoever. You guys across the ocean seem to think that we set anything less than 3:1 as not a fiber, then all of a sudden you say that everything lower than 3:1 is a fiber—that's not what we had in mind at all.

I think this is a very important criteria. I don't think NIOSH would indicate that these three to ones are ours. If they are, they still do not become a fiber, they still do not become asbestos.

MODERATOR LEVADIE: I will come to you next.

VOICE FROM THE FLOOR: I would like to make a comment. I hope you can hear me. I am not a hygienist, but Steve Bayer said something a minute ago which strikes me as perhaps speaking to the heart of this problem and that's this: he said who is to prove or who is to say that fibers of this length or that length don't cause trouble.

I would like to submit that that's the same kind of medical criteria which the government now is proposing as standard. That is the same as Dr. Mohnen talked about.

I would say to you, Steve, that to write standards on the basis of a negative causing the people, forcing people to prove a negative is so unscientific it isn't even worthy of discussion.

I am not aware of one bit of legitimate scientific evidence that states that fibers less than 5 microns cause damage because of the physiologic action of the lung.

Now, if you don't get it into your lung, it can't very well make you sick. And, I object to having people tell me that we are going to have to prove that that's harmless. That's just plain not scientific.

If we would limit ourselves to known fiber lengths that do stay in the lung, we have all we can handle, but whether we measure that fiber length, we would be doing something—

MODERATOR LEVADIE: You made an excellent point. You stopped me because I was going to address it that way.

DR. VOELKER MOHNEN: Two comments. The larger fiber bundle that one can look at under a microscope, the more you approach the 3:1, the more you can mix it up with the smaller, and I mean a bundle that is a hundred microns in length. It's big and it looks like a bundle, so 3:1 is better classified as that, but this we have measured for the Washington area for this round robin experiment or whatever we call it. The measured then was about .2 micron in diameter, .3 micron diameter, about 2 to 3 micron length, so, the second point, to the medical expert I have been exposed a little bit to the discussion of epidemiology.

While they all agree verbatim that the fiber size

distribution ought to be the most important criteria for determining health effects, I have yet to find an epidemiologist who really does this.

These data are not available, so, on the one hand you cannot agree and say this is the most important information and then say it isn't, but it doesn't matter. We will go ahead anyway. I have similar problems as you have. No resolution to it.

MR. LEE: I would suggest that the major difficulty in terms of the use of electron microscopy for counting has been this traditionally.

The first guy who stuck a sample of asbestos or rock forming mineral, crushed rock forming mineral in the microscope and saw fibers shorter than 5 microns should have started to ask, now that I can improve my information about the size distribution, why don't I improve my information about the aspect ratio distribution as well? Instead, he stuck with the traditional definition of 3:1.

The second thing he should have done is said, damn it, if I pick up a handful of asbestos and a handful of non-asbestos rock and can tell the difference, why can't I under the microscope? That is a serious scientific question we should all be addressing ourselves to.

MR. DIXON: I want to comment, first of all, on this 3:1 aspect ratio. It's true we don't have information about what is the absolutely safe level or what is the ratio that we can tolerate, because when you get to safety, the harder it is to measure whether it is safe or not.

MR. BAYER: I am in agreement with you in the back, who made the comment. The only reason I pointed that out, I am not implying a 3:1 is dangerous. I don't know if it is or it isn't. We have spent like probably 25 or 30 minutes arguing epidemiology, what is doing what to whom, you know, which ones are doing it and this and that.

I came to this conference as a microscopist with a whole different concept of why we were here. I thought we were going to talk about microscope techniques. We are talking about epidemiology.

MODERATOR LEVADIE: I think you will have to recognize that one cannot separate microscopy from epidemiology, and this is a very serious flaw in the thinking of too many people who try to rationalize this whole matter in a simplistic way so that we can do a fiber count—this is safe and this is not safe.

I raised this issue earlier today. Chrysotile at the present time according to the medical profession is considered to be at least as toxic as amphiboles and amtraphite and this is fact. This is the way it is now. 95% of all of the asbestos used in this country is chrysotile.

The new threshold limit values which are being considered are being considered on the basis of the fact that asbestos causes mesothelioma cancer. We can't run away from that. All right.

Therefore, the people here who have raised the question of the validity of the counting technique and the identification technique vis-a-vis epidemiology are on very sound ground. We cannot Mickey Mouse this any longer. That's

what we have been doing, Mickey Mousing it.

So, it can be a simple thing to go ahead and sit down and do a count using the phase contrast system and get a number. This doesn't go. It shouldn't have gone on as long as it has.

VOICE FROM THE FLOOR: What I would like to do, Ben, is maybe take this up on another level.

MR. LEVADIE: Will you identify yourself?

VOICE FROM THE FLOOR: John Prohobka. I think what has been happening here is we have been lowering to a point where there are no answers, no solutions to a point.

I am going to use the word "asbestos" for this simple reason: we talk about a law and it says asbestos, this concentration in the air, this length, this aspect ratio. Now, that's the law we have to work with.

The second point is that asbestos might have different kinds of physiological effects. No one here is going to stick out his neck and say this is the clinical significance because of legal, et cetera, matters and so many court matters that he would be going dizzy.

I think the separation that I would like to bring up comes at this point. If we take that aspect ratio, increase it, if you take your length and increase it, the question I have to pose to all of you people who discussed very fine techniques and methods is this: if you change that law, are your people in this country who are going to be taking those samples, going to take them accurately, and what type of method are you going to use so that this gentleman, the other gentleman over there does the sampling techniques. You cannot say it's reproducible, it is accurate. That's the question I have to pose to you now.

Do you honestly think that you have the methods, even if you changed the law, to say that you can do specific sampling throughout this entire country?

MODERATOR LEVADIE: Do any of you gentlemen want to address this question?

MR. DIXON: That's a tough question. We feel that the analytical work that we are doing in our own laboratory is very consistent based on our own quality control data, that we are getting reproducible results internally.

I suspect that if we cross check against a private laboratory that we are going to be in situations where a private laboratory does things differently than we do.

As I have mentioned already, they may not be able to make a distinction between asbestos fibers or non asbestos fibers or they may have a microscope that has an objective which has a lower resolution than ours does.

You may find those kinds of differences in making comparisons between laboratories, but at the present time all samples are coming into one single laboratory in which all samples are being cross checked against each other so that we are getting a uniform count in our own laboratory. So, maybe we are kind of high. Maybe we are kind of low, but the point is that when we have cross checked with the other laboratories, we are getting excellent counts and, at least, we can say that our

counts are consistent.

MODERATOR LEVADIE: So, what you seem to be saying then is that we do have or we have at least the insight for potentially accurate methods for doing the analysis. This is what you are saying?

MR. DIXON: Our deviation is about an error of plus or minus anywhere from 10% up to plus or minus 20%.

JOHN ROSKI: I am a laboratory manager for accredited laboratories, the same as you are in Salt Lake City. We belong to the round robin program.

For that program, a test under the PAT program for asbestos, you can do whatever method you want, whether it's electron microscope or whatever—they never tell you what method of analysis to use.

They won't and shouldn't publish these data on asbestos anywhere, because they are not using a consistent method. Everyone can do whatever they want so you can never compare different types of testing methods in any kind of meaningful way. But it does tell the participating labs in the case of asbestos that, on the average, in 124 laboratories that need these samples that are prepared by a contract in the same way and as deeply as they can do that on any given sample depending on the concentration—you can go from plus or minus 35, from plus or minus 120% in 120 labs. That's the methodology.

I can check in my lab of 10 who analyze asbestos, we can check within 15% of the slides. The OSHA people come in and we could easily differ by 50% on the same slide. Internally, we have good precision, but going from this lab to that lab to this lab, then using different methods, you are going to see what you saw in the EPA study. There is no consistent method for asbestos identification in this country.

MODERATOR LEVADIE: Let me make a comment here apropos of this. Presumably, all the samples in the PAT program are being counted in the same way. I believe that's the idea, isn't it? Aren't they all being counted the same way?

MR. BAYER: I am not involved in the PAT program.

MODERATOR LEVADIE: Let me give you some statistics on this. Before I came here on Friday, I did a little bit of quick and dirty arithmetic with a calculator.

Looking at PAT, 32 to 52, inclusive, there is one sample that was, I think, eliminated by the program, which left a total of 80 samples. The standard deviation of the percent, standard deviation per count was 33%. The mean standard deviation of the percent standard deviation of the count—remember I am talking now about the error—was 68.4%. And the percent standard deviation of the above, 48%.

I don't think I have to make any comment on those numbers.

MR. DIXON: I agree with Joe about the potential for differences between laboratories that are counting, and yet we do see a great similarity between counts at the same laboratory.

What this suggests is that one possibility for overcoming this type of problem is regulating your standards of having an exchange between laboratories so that they can take back a uniform technique to each laboratory.

Now, this is being organized. It is going to be up to a higher level authority. I see a way where a problem can be solved.

MR. PRONOBKA: What I see is no answer to my first question, to be honest with you, so, let's squeeze it a little bit tighter.

Let me ask you and I would appreciate an answer from every one of you, what method or methods do you feel should be standardized in each and every single lab that all the techniques should be working?

MODERATOR LEVADIE: Do you want to make you comments now before the panel addresses this question?

VOICE FROM THE FLOOR: I would like to talk about something that John was talking about. I am from General Motors and we are in the PAT program, also, and when we talk about being able to compare within our laboratory, we can't even do that.

I have very well trained people and there are seven of them all counting asbestos and we can't come up within our own group with a comparison.

These people have been counting asbestos for the last, anywhere from seven to four or five years, that's how much training they have had and they count asbestos frequently, but they do not count well together.

VOICE FROM THE FLOOR: There is one other thing. You talked about having good agreement when you counted with laboratories outside which you said were good laboratories. How do you establish what a good laboratory is?

MODERATOR LEVADIE: I will let him answer that in due course. Do you want to address that question?

IAN STEWART: I think, first of all, I have to say that I am thoroughly in agreement with Dr. Trayer, that we are really battling things around in the air until we know what the epidemiologist can tell us.

As an analyst you can tell me what you need to measure and we will find a way to measure it. Against the situation it does rather look like there is a growing body of opinion saying that high power phase contrast was right—until you read the next paragraph, which says it looks like you are going to be pushed to electron microscopy.

If we are, then I think we ought to become aware of it now. The EPA has already looked into this and come up with provisional procedures both for air and water. They have established what these procedures can do. We seem to be getting smarter all the time. As more people get into it, the data is, becoming more numerous and, fortunately, the errors are beginning to narrow down a little bit.

The other thing we have to note, however, is that we are going to be pushed towards limits where we will be looking for needles in haystacks. One of the problems is that there are an awful lot of haystacks and there are an awful lot of high aspect ratios in haystacks.

What happens if we don't get a clear fiber—if we have a fiber where we can get neither the electron diffraction information nor the information on chemistry? I think we have to realize that we are still going to be fighting a lot in the dark. Any number we can come up with in those situations is almost certainly a minimum number, but I think we do have to start looking a lot more into epidemiology.

This question of amosite was "defrocked," if you like, way back about 1948. That's no longer regarded as a mineral. We should be talking about something of this sort.

We have a regulation which states quite categorically that asbestos is chrysotile. Asbestos is one of the amphiboles. It doesn't specify that they have to be fibrous. Then it goes on to say that it regulates asbestos fibers. I do think we have problems in the terminologies. From the analyst's point of view, we have to look for better ways. Our problem is going to be perhaps a little bit simpler.

But I don't think we can come up with a magic way to dissolve all the problems associated with this without becoming cognizant of the fact that this has to be within the government's jurisdiction and has to be based on biological significance.

DR. STEELE: I agree with that. It becomes a question, more so, on the amphiboles, what you term them, than in the mineralogies. An article by Leake, B.E., "Nomenclature of Amphiboles," *Am. Min.*, 63, 1978, pp. 1023-1052, described the new accepted classification of the amphiboles.

That's a good reference if anybody wants to use it for correct mineral terminology, they can look in those references.

As to the analytical technique, I would again agree with Ian in that it depends on what you are looking for. If you are looking for really large particles, the electron microscope is going to have a problem. If you are looking for very thin particles, then the electron microscope is the way to go.

For large bundles, I have a tendency to have less preference for the electron microscope because it's the less definite way of identifying particles.

The light microscope has some definite advantages in identifying samples if you are looking for very long and very thick fibers.

DR. WALTER McCRONE: I think I would just say that it's usually the analyst rather than the method that you have trouble with. The methods, in most cases, could be made to work; you could come out with the right standard of deviation of error and so on.

For most of the methods, in order to do that, you have to be sure that the people have adequate training and that you have adequate standards and known samples, but I think your problem is really with methods. I think if anybody tells us what has to be analyzed and to what extent and how far down and so forth, we have the methods to do it.

I have to defend myself. There are a number of things I have written in my life that I wish I hadn't, but that one he cited is not one of them.

MODERATOR LEVADIE: It's a good paper. I didn't criticize the paper.

DR. McCRONE: I think you are referring to my saying that the light microscope, optical microscope based on that are the only sure ways that I know of to identify—

MODERATOR LEVADIE: The statement was with direct reference to polarized light. I have it here, but I think you really meant what you said and the way that you said it.

DR. McCRONE: I will say what I think I meant. I don't think when this comes up again that everybody uses the methods in which they have confidence and we have confidence in the light microscopes and, naturally it's better than PBM or anything else I can think of.

You have more confidence in that, therefore, staining doesn't look very good to you. It's understandable. But, if you try to, let's say with my condition, I would say that the optical microscope data as determined by the dispersion staining is the only single method that I know of that will certainly identify any asbestos minerals or any other mineral. For that matter, therefore, combination methods, X-ray diffraction plus elemental analysis, but X-ray diffraction alone will not do it. PEATEPM alone will not do it. Anything else I can think of will just not do it.

DR. MOHNEN: The question was what method would the panelists prefer. I think at this time I would not prefer any method.

I would say it would be extremely dangerous if we were to settle down for one or more methods. If we haven't received the proper questions yet—and the proper questions are what you want—somebody, obviously it is the epidemiologist, decides the stuff is dangerous. He has to tell us what he has to know so that we can properly identify the technique.

Now in the meantime, since we probably have to wait another twenty years for that to come true, to me it's just out of the question why no one has sent around known standards. NBS is beginning to look at that with the Asbestos Institute. How do you make an asbestos sample a standard sample so that we know how many milligrams are in that sample?

We know it's only certain that the deviations there are can be used by the individual who deviates as a means of identifying the property he has. If you tell me that I am 20% off in a small part of the sizes, then I can go back to the instrument, back to the technique, and say why, where it becomes a research question.

We have not identified your problem, why your operators are different from Dr. McCrone's—but it's people, obviously techniques and machines. These problems can be resolved to some degree on a research basis if we have a standard sample, a standardization that can be sent around. That's the first step that has to be developed by your group.

Now you will have a chance. Once that is discussed and we all agree on the proper standard, that can be sent around. Once you have asbestos, I believe it can then be done in a way that uses a

hundred-milligram sample. These are the size distributions. Next you try to verify it by identification and so identify the weaknesses in your technique and the weaknesses in your people all the way down.

Since we won't have it for another ten years, we are left with what the individuals prefer at this time.

Roger Cheng is the manager of our laboratory. He tells me that he will come up then and only then with a method if he has identified under SEM with EDXA at the same time with the dispersion staining procedure Dr. McCrone taught him some years ago—if it's small samples, TEM and SEAM, so that he knows he didn't miss a particle.

In this event, you talk about ten-day research for just one sample. Not too many people have that time. We are employees of the State of New York, which lets us do that.

MODERATOR LEVADIE: I might add that OSHA gives us only ten days.

MR. DIXON: First of all, I have also recognized the need for counting asbestos fibers for quality control of calibration and methodology. I have recommended to the Washington office that the proposal of the NBS to develop standard asbestos fibers is a good proposal and that there should be something done in this area. I believe EPA is also working with the Bureau of Standards on the method of developing standard asbestos fibers.

A second question has come up. Somebody remarked that they are unable to get good cross checks with personnel in their own laboratory.

When new people who are being trained first start counting, their cross checks are not going to be as good as they are when they have more experience. But we sit down and take turns looking through the microscope at the same field, to count the fibers that we see in that field. When we find some difference in our counts, we draw diagrams showing what the fibers look like and discuss why this fiber is counted and that one is not. Is it glass? Is it in the same bundle? What is the problem causing one person to count it and another not to count it? By practical experience and discussion, we can get together for some kind of consensus counting the same fields together, and we will get uniform counts. This can be done between laboratories as well as between individuals in a laboratory, and we will get a consensus as to how we are going to count.

Now, as to methods, it is my feeling that every laboratory can use optical microscopy. It isn't expensive, and it doesn't take that much training. Every laboratory should be able to use the methodology of phase contrast. They should understand the use of polarized light. They should understand the use of cross polars, the use of retardation plates. They should know how to use dispersion staining.

Then, if they have something beyond their capability, they should know when they need to send this sample out to another laboratory that has an electron microscope, if they don't have one themselves. They should know when the sample is of such a nature that it needs more testing.

DR. STEEL: The National Bureau of Standards is working on a program with the EPA to find where the errors are in their electron microscope provisional method.

This includes the making of TEM grids with fibers on and eventually the making of filters with a predictable distribution of fibers on them.

This may not be possible, but that's what they are working on at the moment. Again, it will be defined statistically. It won't be that you have so many fibers on the filter or you have so many fibers in a field of view that have to be defined statistically.

IAN STEWART: Just a brief comment. EPA is offering guidelines and also has a series of test samples out in connection with upcoming contracts. There is a lot of work going on in developing standards for the development of microscopy.

MR. BAYER: I guess all I can say is that since about 1971 one of my major responsibilities has been to do everything I can to make sure that everybody counts the same way. Whether it's right, whether it's wrong, I think that's important. We have a standard which right now specifies a certain method, and there are loopholes here and there in that method.

I will agree with it just the same as the rest of you, but then again, I can't train 28 people this week to count asbestos this way and then say, well, let's see, I think we ought to do it that way, and then next week plug that in, train them in a slightly different way and keep passing it on. A lot of people have been in my particular course and I have tried to maintain the integrity of procedure, whatever that is, over a series of years.

There are a few things I would like to see improved that I think would help a lot. I wholeheartedly agree with the aspect ratio problem. I think a 5:1 ratio would improve the counting statistics considerably and still probably protect a lot of people's health.

I know, at least one particular microscope that I have, considering of course that all the different types of asbestos fibers we are involved with are bi-refrigent under polarized light.

With this particular microscope I can get a total fiber count for that particular field, then, without moving the field, I can remove the phase contrast condenser, plug in a polarizing light condenser, flip on an analyzer in one position of the polars, count how many bi-refrigent fibers I see.

I can rotate the polarizer and the analyzer both 45 degrees to account for some fibers that may be an extension and continue it again and get an idea of all those fibers I counted in phase contrast.

I now have a pretty good idea of the bi-refrigent fibers. That would mean they are mineral fibers of some sort and I would be closer to answering the question of how many of those fibers are asbestos.

Granted, there are lots of other bi-refrigent fibers, but that would be at least two, possibly three times slower than counting the fibers, maybe a little better, I don't know.

Just like I say, I take the stand—I don't always agree with everything, but—I take the stand that

right now we have something to work with. That's what I am working with. That's what I am sticking with until somebody comes out, OSHA changes their standard, declares a new technique, I will train people how to do it.

JAMES FERGUSON: I shouldn't have any comment, because it's been a long time since I've done microscopic analysis regularly. However, when you ask what I would recommend as a technique, I would say it depends on what you want to know.

We have talked today about at least three different problems: the identification of asbestos or something similar from bulk samples such as ceiling insulation; the identification of asbestos collected on membrane filters in the workplace and other places; and the identification of fibers collected in the outside environment, which I suppose includes water samples and food samples by various techniques, membrane filters, etc.

You can't really answer it with one technique, that's the problem. In the first place, to look at ceiling tile, I think we ought to use Dick Kupel's method. Any time you get a positive and you really want to be certain if you have asbestos, that's going to bring you to one of the other methods that worked for the other two. I think the first stage has to be optical microscopy as a screening technique to tell you whether or not you have to take the next step, such as TEM or other electron microscopy techniques—so, depending on how quickly you have to get the answer.

You must remember that recent statistics, which are pretty much the same over the past seven, almost nine years, since the passage of the Occupational Safety and Health Act, tell us that 14,000 workers are dying in the workplace every year from traumatic injuries. They don't have any idea how many are dying right now and will die within the next 20-30 years as a result of chronic occupationally related diseases.

All of the test techniques are good. The phase contrast technique can tell you better than the dispersion staining technique whether or not a fiber is present. It can't tell you as well what it is, but then as it gets smaller and smaller, remember that anything that relies on visible light for resolution can't be much smaller than the lower wavelength of visible light, about a fourth of a micrometer.

So when you get down to those limitations, you are pretty well stuck with the method. I say to the person who has a problem with agreement within counters, that you have a problem in the uniform training of those counters. That's what the standard doesn't speak to. It doesn't say that anyone who is going to count asbestos had better have a week's course at Dr. McCrone's or a four-day course at NIOSH, but some kind of basic training so that we all begin at more or less the same point.

VOICE FROM THE FLOOR: They have all been very well-trained. This is what I am trying to point out. Even with their good training, they are not counting well together because of the method, because there is operator judgment involved, and

that leads to a difference in counts, I don't care how well they are trained.

MR. FERGUSON: Well, I don't agree with you. I know that operators vary considerably. I have worked with a number of different training organizations over the years and used similar techniques for making pollen counts. For example, if people have all been trained the same way and they are not given the results before the next one does the count, they are more likely to get closer. I cite the example of NIOSH.

Steve Bayer can show some very interesting statistics that show reproducibility within the same class and from one class to another. Here is one person, though, giving one course repeatedly. Even though it's to different people, he changes very little and he can show good reproducibility.

I don't think the reproducibility is nearly as bad a problem as it has been made out to be. I think the problems lie elsewhere.

I would like to make one point in defense of the PAT technique. NIOSH started the proficiency and analytical testing round robin procedure in which samples were made up of many things and sent to a number of laboratories. One of them was asbestos. In the beginning all of those people used the same technique. In fact, if you look at the light yellow booklet that was given you on the NIOSH membrane filter technique, there is some of that history described there.

Everyone then did it the same way using phase contrast microscopy. You could compare it from laboratory to laboratory and there were some generally reproducible statistics of about 80%. Now that may not be as great as you might want, but it's not all that bad. I can't really say where it went wrong. Bill Kelley might be able to shed a little light on that because he worked with the PAT program in its early days. It is now administered by AIHA.

If they have not been specific as to the technique that should be used, obviously there will be variation. I don't know where we went wrong in not continuing to specify the procedure that is in the *Federal Register*. Maybe it isn't so good, but again I'll go back to past experience in my air pollution days. If you heard a pollen count on the radio, it was really a sample that was put out yesterday, brought in this morning, and really more or less yesterday's count.

The sampling procedure is a very simple one called the Durham sampler. There were fifty years of data using the Durham sampling technique and it could be used for a variety of conditions. It wasn't a highly efficient sampler. You could take quantitative samplers, put them out, and get lots more material. The information gained from that technique was very crude, but simple to do and very inexpensive. There was a great deal of knowledge gained and I think the phase contrast technique backed up by dispersion staining and finally backed up by electron microscopy is going to give you all the information you ever need.

MODERATOR LEVADIE: Bill, would you like to respond to Jim's query?

MR. KELLEY: No, I'm afraid I was out of that

program before the change occurred, if the countdown is historically correct.

MR. BAYER: I would like to make a few more points. My experience in asbestos training has, over the years, permitted a unique overview of fiber-counting personnel and microscopy in general.

Many think the proper use of the microscope can be achieved through little more than reading the instructions. Most problems I encounter in dealing with microscopists are due to their tremendous inadequacy in training. Many of those who sell microscopes cannot tell the buyer how to properly set up the microscope. Sometimes the buyer must incorporate instructions from several different books in order to assemble and align all the components.

I have found it difficult to acquire the training I need in microscopy. I have taken a couple of very short classes through McCrone Associates and they were quite useful. When microscopy is one's profession or, more often, when it is used as a tool in one's profession, the proper application of the microscope to solving a problem will be a tough row to hoe without specific training.

Once a certain degree of experience and training has been accumulated, there are still stumbling blocks. Often I receive inquiries asking, "Can asbestos be positively identified by using dispersion staining?" Within certain optical limitations, the answer is yes. Off they go from there to buy a dispersion-staining microscope. Of the seven or so brand-name microscopes, I can think of only one which can be used straight from the factory as a dark-field dispersion-staining microscope. Granted, McCrone Associates sells a dispersion-staining objective which fits all microscopes. It does not function properly on all of NIOSH's microscopes used in the asbestos training course. Why? Dispersion staining requires a special optical system (of which the major component is McCrone's objective) and provisions for aligning almost every optical component. Most people who count asbestos fibers are using biological-laboratory microscopes which, with rare exceptions, are not suitable.

Using dispersion staining requires purchasing and assembling an optical system not usually available commercially as a package; attendance in a detailed training course on the principles of dispersion staining theory; and considerable experience using the procedure before one feels comfortable with it. Walter McCrone supplies two out of three of those requirements, and anyone attempting dispersion staining should acquire the training before buying microscopy equipment.

My last comment is in regard to electron microscopy. There are two objections often raised relative to the membrane filter method. First is that the counting reproducibility between operators is poor. Second is that individual fibers cannot be conclusively identified. In many cases (but not in all) both kinds of electron microscopes can be used—and sometimes it may take both SEM and TEM and their analytical attachments to identify as asbestos a single fiber. In most cases, though, identification can be made.

But do you ever hear electron microscopists brag about the wonderful data agreement between various microscopes and operators? Of course not, simply because dust counting variability can arise from a number of causes common to all microscopy. Examples include: variations in dust deposition on the sample medium; differences in resolution between instruments; differences in measurement criteria between operators; the facts that no two individual instruments or operators looking at the same sample will see the same fields and that microscope operators looking at the same sample under different magnifications will count different fibers; and so forth. Basically, every problem associated with counting variability on optical microscopes will be encountered on electron microscopes.

DR. McCrone: I just want to comment that Steve is right about the difficulties with dispersion staining. They aren't as insurmountable as he makes them seem.

Perhaps the objective comes with standard RMS. It's supposed to fit in with the microscope. There is a problem if you try to fit it on. The microscope doesn't have a centerable condenser, but this is no great problem. One, they don't do that much. I am sure that they would be glad to fit the whole thing together and send it to you. Really, we don't have that many problems.

MODERATOR LEVADIE: As the last member of the panel, I would like to comment with respect to this in Vermont.

I was saying to one of the participants here earlier that I did my first asbestos fiber count in 1951. I don't know whether that makes me an expert. We sampled in alcohol. We used the impinger technique. We had a plant where they were actually working with asbestos fibers making globes and various asbestos equipment in Northfield, Vermont, using chrysotile.

They brought me a sample, they said, well, tell us what's in it. And, I looked at it and I said, we have got fibers in it.

We will let you know how many particles there are per cubic foot and how many fibers there are and try, please, to give us an idea of the size and the length and so on of the fibers.

I looked at this study a few weeks ago and was amazed to the extent that one could go with a common microscope to get an idea of what was present in the air and at that time I think, looking back at it now, I felt pretty pleased.

When Germaine Crossman came out with his techniques with respect to dispersion staining, we then purchased in 1955, Steve, a Zeiss microscope which is equipped with a rotating stage. It was a phase contrast microscope and we went back to this mill, to this factory where they were using asbestos and, after all, now, I have something a little more sophisticated, and with the appropriate dispersion oil, I took advantage of the first shift and found if I used the 1.56 refractive index oil I could very nicely delineate and identify the chrysotile.

We did counts and we found that we came out for the defense operations in the plant with

comparable information.

Now, with regards to the determination of asbestos, chrysotile asbestos, I want to stay with that in respect to the school study.

We are not running into insurmountable problems there. Again, the same technique, we are backing it up with infra red spectroscopy. That gives a peak at about 3750 centimeters at a peak of about 10 millimeters in height; double it.

We have found that the only problem we have had in this sort of thing is the presence of organic matter. After all, a chemist is supposed to have a certain amount of inertia, laziness and some imagination.

We fire off at 450 degrees C. We burn off the organic matter overnight. We put this droplet on the slide, let it sit there overnight. The next morning there is no more organic matter, so, we don't have that interference.

This works, also, very well when we take a slide and look at it with the phase contrast microscope and use dispersion oil.

I would like to say that one of the most confusing things in this whole program that was organized with respect to the asbestos in schools was the sampling of the sample at the source and the sampling of the sample in the laboratory.

Somebody was talking about picking off a piece of sample from the flock and looking at it. I don't like to do that because it doesn't tell me what I want to know in terms of quantity.

What we do—I am going to pass this on to you—I am not going to publish this. We have an old-fashioned mechanically operated mortar and the 10% suspension in distilled water of the sample, we like to work with from 10 to 15 grams of sample and take the thing and grind it for 10 minutes. You get a very nice mush. I don't care whether there is Fiberglass in it or not.

When we have Fiberglas under the microscope, it might be a piece of Fiberglas 10 micrometers in length, it looks like a monster.

I take a droplet of the material and put it on the slides, I use five slides, so to prepare five slides, fire them overnight. The next day we look at them using the oils.

And, I tried this—I know it isn't fair—I thought, well, if we can do this, I will know whether we can quantitate it. I find that the microscope is much, much more sensitive than the IR method.

I can identify a fiber in 10,000 if I want to look for it and it works very well. The distribution is right and the depression of statistics with respect to representative sample has been eliminated by the mixing.

So, in listening to the conversations and discussions here with respect to it, I find it difficult to appreciate that problem.

What I don't like is the official N.I.O.S.H. count, because in Vermont we have a problem of not knowing what is in the sample and when you go to sampling in a garage where people are working on brake shoes, some of the fibers may be asbestos. They have the correct 3 to 1 ratio. Some of those fibers may be something else.

When we tell the man who is involved in the operation to go through a considerable expense because he is in violation and also fine him so he has to pay on the basis of a count which, I know that if it were challenged in court would not stand up, I feel very uncomfortable and I believe that this is where tradition comes in.

People like yourself, Steve, are reluctant to back off and this is unfortunate. I don't want to take the Hyman approach because you are simply doing what you have been asked to do at N.I.O.S.H.

I think we need to back off because now that we are counting two and three and four fibers per centimeter, if there is one, if there are two non asbestos fibers in the count, we punish somebody economically because of something that isn't there.

Now, I agree that is working in terms of protecting the worker. There is something that OSHA has to start understanding and that is this, that we can protect the worker out of his job and there is a threat here which we must recognize if we are going to be chancey and inaccurate in our work, where do we hurt him more.

This is why I say that the OSHA part makes me uncomfortable. I would like to feel more reliance on what I am doing.

MR. PROHOBKA: To continue along your lines, first, I would like to just jump a little bit into the epidemiology of this whole symposium.

I think what we are doing is we don't have good understanding of what we mean by that. We are working backwards. If you want to study what is the effect of a CO₂ in a vat you have to have methods by which you can measure.

You don't walk up to a rat and say because of the cherry red blood, et cetera, what do you think is the concentration which is affecting you? In the same way you don't walk up to a man who is dying and say, what do you think is the aspect ratio here that is killing you right now?

We have to develop good methods and means for measuring these things so we can detect them in the atmosphere and then relate that to the physiological effects on the person.

I think we should be working in that direction. In terms of what the panel brought up here I feel that, number one, we seem to need a standard, number two, and I regret that the gentleman in the blue sweater didn't make a comment, we have to have a method by which, because of the multitude of samples coming into the lab, we can rapidly distinguish as to which needs the analysis.

MODERATOR LEVADIE: This is on bulk? you are talking about bulk samples now?

MR. PROHOBKA: Right.

MODERATOR LEVADIE: I wanted to make that point for the record.

PROHOBKA: Number 3, in any lab it seems that we need a method by which we could identify length, aspect ratios and a method to further take that to the exact type.

I think the fourth point that we have got to bring up here is that we need a good training program.

If we have these four factors incorporated into

every single lab, I get the feeling we can start looking at accurate and consistent results. I think everybody will agree to that.

MODERATOR LEVADIE: I have got a fifth factor, to make the legislature of the state of Vermont aware of the fact that in order for me to do the kind of work that I need to and want to do, it should vote money so that I can buy appropriate equipment.

MR. PROHOBKA: Excellent! I am glad you brought this up because this takes us to the final point coming from industry.

I will throw this out in a very direct question because that's the way things are put to us and it goes like this: do you agree that all of these five factors are important? My question to you is now you tell me when, give me a date by which you will have all of these incorporated in your lab that we can get moving.

MODERATOR LEVADIE: There is a statement here—20 years. I think that that statement is probably made in humor. Somebody over here said that Dixon has it now. But, Dixon has a very wonderful microscope that was purchased for him. What did it cost? Is \$25,000 a good number?

MR. DIXON: We do have one that is actually a Universal which is around 12,000 with maybe another eight or ten.

MODERATOR LEVADIE: There you go, \$20,000. You have it now, but your equipment costs about \$20,000.

When I want to buy it, it will cost 25, 26.

MR. DIXON: It doesn't have to be that high.

MODERATOR LEVADIE: I do quite well with a \$3,000 one.

MR. PROHOBKA: Is it necessary to have that?

DR. MOHNEN: It goes back to the question of every time somebody comes up with a new environmental hazard, we all stick our tail between our legs and run.

This goes even one step further. We all know that asbestos and all the rest of it is a hazard. We have hundreds of other things that run and run continuously.

I was involved with this Rockville crushed stone business. It was my very first involvement. It's a very strange event to me seeing the newspapers going overboard, seeing government agencies going overboard. At one time there were people from about 15 federal and local agencies walking around taking different samples.

I presume something else had to be dropped during that time so they could find the time to do this.

You are well aware of the problem I am pointing at. It's this old question of who decides at some point why we have to invest and this is the figure, about \$180,000 in getting this equipment, these four instruments. I don't know why I came up with 40,000 for a microscope because I want the automatic analyzer, then about 80,000 for the ISI, another hundred thousand dollars for the TV.

I agree that I should have this for research purposes, but then people should listen not only to me, but to research groups like Dr. McCrone's and

others. These are the groups that then have to come up in three years with the answers.

VOICE FROM THE FLOOR: Speaking of information, Mr. Ferguson has brought up a point there. I have done some checking and acted as a consultant of some school systems around the country.

DICK KUPEL: There is one comment I would like to make. It seems that EPA says that if there is any asbestos in the schools, it has to be removed. It doesn't make any difference whether it's 3%, 5%, 90%, or 100%.

I think this spot test we have developed will give you that information and this can eliminate an awful lot of further analyses.

I think this test ought to be tried first. With all due respect to Ben, his samples take over a day to analyze and give, very simply, only asbestos. He is also going to give you percentage of asbestos and I don't believe you need that. I think you need to know, is there asbestos or isn't there? If there is no asbestos, forget the whole thing and you don't need to worry about this little kid getting his fingers on that cassette. You don't need to take those samples.

In a lot of places you go into they know already that asbestos is there because the contractor said, yes, we put asbestos there. But then there are those few contractors who say absolutely not, we did not put asbestos there, and those are the guys you have to check out.

MODERATOR LEVADIE: How much is a few?

MR. KUPEL: Probably about 80%. No, all kidding aside, I think you need to know whether asbestos is there before you waste your time in taking those air samples, and going back to the lab and counting them and finding, no, there is no asbestos there, no fibers there at all, so, personally, I am recommending that you try this test first. We don't say that it's going to work 100% of the time, but we hope it will.

As I said this morning, we found no false negatives. Again, we have heard that there are some from New York that have something like .3% asbestos in them, and if EPA allows a 1% mix of asbestos, why are we worrying about .3%?

MR. STEWART: That was from zero to 3%, not .3%.

MR. KUPEL: We see 3% and we may be—

JOE BREEN: What was found in the state of New York was that 5% of the samples were 1%; 2% of the samples were 2%, 9% were 3%.

Sixteen point five per cent of the samples that had asbestos in them had 3% or less. Thirty per cent had 5% or less, so we are not talking about a trivial problem in terms of the number of samples that may be a problem.

We are not making any distinction. We are just talking about the absence or presence.

I think that the concern that we have in terms of the optical method and focusing on the use of polarized light microscopy is the historical fact that the samples have been sent out to people who

apparently have been, as best we can determine, certified in the PAT program, who have reported back positive for asbestos.

What they have done is a fiber count and reported it back positive for asbestos. The schools have then properly or improperly—hindsight is always 20/20—ripped the asbestos out.

Subsequent to this, very expensive action has found that the material was of either cellulose or Fiberglas. So, it's a real dollar question.

We feel that it is really important that you identify the fiber as being asbestos or not.

VOICE FROM THE FLOOR: What method did you use on the less than 3% to identify that?

MODERATOR LEVADIE: The gentleman wants to know what method was used on the less than 3% asbestos in the samples.

MR. BREEN: The state of New York had the samples analyzed by polarized light microscopy with dispersion staining, I believe. This is not my data. This is data that I have obtained from the state of New York.

I think that that's fairly important with the question of identification.

The question relative to the percentages is a difficult one that the administrator is going to have to face on the local level in terms of what action he is going to take if he gets a positive back and it's got one, two or three per cent, whether you make any distinction between that and one that has 90%.

I think he has to factor it into an assessment scheme which will consider what his options are, in terms of the actions of whether he is going to encapsulate the surface, whether he is going to hang a spanner and put a ceiling in (enclosure techniques) or, whether the fact that you have the asbestos plus the accessibility factors of whether the children can get at it, whether it is a vandalized area might tend to consider going the full nine yards and removing the material.

All of those options are available to him and all of them may well be appropriate in a full asbestos program in responding to the presence of asbestos.

While I have the mike, perhaps I can indicate to you that out of 476 samples that the state of New York ran, 50% were negative for the presence of asbestos.

As to the positives, of approximately 250 positives, 43 were amosite; so we are not talking about 5% but 17% occurrence. One was crocidolite and the rest were chrysotile.

I don't know whether you can see it, but this is the frequency distribution for all of the asbestos samples and you can see that it is a skewed histogram. The first bar on your left is for samples from 1 to 10 or 11%.

Over a hundred samples out of the 250 are in that category and those are obviously going to be the ones that are difficult for the administrator to deal with in terms of an action statement.

The chrysotile distribution is somewhat similar. There are an awful lot of them that are high as well.

I don't have the amosite figures here with me which would just indicate that there are a greater

number of them which are in the 10 to 20% range.

It is skewed a little to the higher side than the general scheme. The other point I would indicate is that we have looked at the analysis in terms of trying to break out the types of materials and there are general categories to which one can assign the various types of asbestos, in this particular sample, for whatever reason. I think the important point is that these materials were sampled. There are an awful lot of acoustical samples, things which I don't think most of us would consider to be friable; however, they were sampled and included in this set.

Of cementitious materials some 70% were negative for asbestos which is to say on the other hand that 30% are positive.

More importantly, or, in relief to that, is the fact that in the other categories which are the more friable materials, the three categories reflected 79%, 81% and 76% as positive for asbestos.

The question that remains and begs to be asked is what about the accuracy of the analysis. I would just indicate to you that it is a bona fide problem and we are talking about on the basis of, yes, no, that the limited data that we have available to use as given by other people suggests that split sample analysis is a problem on the basis of yes, no.

What the problems are in terms of the identification and the percent, I think, is something that one is going to have to deal with down the line. It may be fairly moot as to whether one is going to get upset once you have identified the fact that the asbestos is there and the real concern is that among split samples between 2 laboratories that are prestigious, if you will, some 26% showed differences on the basis of yes, no.

I think you are really going to have to think about that in terms of what you might consider to do in-house yourselves to make sure that the output of your laboratories is credible.

VOICE FROM THE FLOOR: Mike Davis, industrial hygienist with Argonne National Laboratories. I just wanted to address the practical significance of getting the sample in the first place.

You had a comment before about whether there was a representative sample from road dust.

It seems to me that we mentioned the epidemiology being tied in with the determination of the analytical method. The analytical method then puts certain constraints on the sampling method as well, and in Dr. Mohnen's one table he talked about an air sample volume of 21 cubic meters taken within a half hour.

Most of the operations involving asbestos that I get into are under the two-hour category and I haven't seen the portable battery powered sampling pump that could pull 21 cubic meters in a half hour.

MODERATOR LEVADIE: I would like to call the conference to an end within another five minutes. Just a moment, in case you have the floor, is there anybody else who would like to speak before I call on two who have already spoken.

I would like to give the others here an opportunity to speak before you do if they want to because time is running out.

MR. BREEN: What I would just like to indicate is that we are well aware of bugaboos. We obviously don't have a standard procedure for using polarized light microscopy—quote unquote, whatever modifications thereof—but what we are trying to do in terms of generating some reference materials—again quote unquote, these are not NBS sanctioned reference materials—is the following:

We have provided Bill Campbell of the Bureau of Mines with a set of real world insulation samples, both positive and negative, for asbestos on the different types of asbestos, we hope. He is going to have them examined by various Bureau of Mines Laboratories (I think six). In an attempt to characterize them, these may then serve as materials which would be made available to laboratories who might be interested in checking themselves.

We are also trying to identify laboratories who claim that they can do polarized light microscopy and will make available to them sets of these "reference materials."

MODERATOR LEVADIE: All right, now, Mr. Dixon.

MR. DIXON: We have been talking about the exposure to asbestos in the schools. I just wanted to point out there may be another institution where there is exposure to asbestos that nobody has thought about and that's the prisons of the United States. Some of these prisons may have asbestos covering their pipes and I have learned by talking to some of these prisoners that one of their favorite places for hiding weapons is in those pipes.

This means that they have got to rip out the asbestos, if that's what the stuff is, so they can get their weapons in.

If they have a shakedown, that means that the guards will be exposed if they have asbestos to rip out to find where the guns are hidden.

MR. FERGUSON: Serves them right. (Applause) Not the guards, that is.

I think the man deserves an answer even though the question may not have been all that relevant to the way he said it. There is no personal sampling pump, obviously, that pulls 21 cubic meters in a half-hour, but they weren't doing any personal sampling anyway. The question then becomes, if it's there, who is being affected and by how much and so on. That's another story.

I would like to remind all of you, though, that perhaps we did not achieve the objective we came here for today. Our ideals might have been very lofty from the start, but scientists in general forget quite often that they are also voters and have voices which are very often heard—and that's why we find ourselves doing the things today that we have done for years and years. If we see an area that needs change, instead of sitting in rooms talking about it we need to get the word to legislators and enforcement agencies such as OSHA.

That's what I hoped we would talk about here today. I really think we did point out that there is now a technique which can tell you quickly whether asbestos—or at least a solid containing magnesium and iron—is present or not.

Then, if you are disposed to go further, you have dispersion staining techniques for identification. If you want to go further yet, as you may have to in some cases, you have the electron microscopy techniques.

We have several techniques for identifying

fibers. We ourselves need to work out the bugs, and there must be ways. After all, we have developed these techniques through our own ingenuity. We must be able to solve the difficulties inherent in them.

Closing Remarks

JAMES S. FERGUSON

**Deputy Director of the Division of Training and Manpower Development, NIOSH,
Cincinnati, Ohio**

I certainly hope it was worth your time and your efforts to come here. I know that our speakers put forth considerable effort and I want to thank each and every one of them now: Dave Trayer, chairman of ACGIH; Dr. McCrone, who has helped immeasurably by his being here; Ian Stewart, Steve Bayer, Dr. Mohnen, Roger Cheng, Dr. Steel, Dick Kupel, Willard Dixon, and Ben Levadie. All of the speakers tried to tell you as much as we could about our feelings in the matter of asbestos identification, what our experiences have been, and a lot of our own opinions.

If you can in any way bring about change, I am sure it will be welcomed by all of the people from

whom you have heard today, who say that we realize there are problems. But we are really all trying to do the best job possible with the information at hand.

Last but not least, I want to thank Lee Saltgaver who just walked into the back of the room. We are here today at OSHA on purpose. They probably have the largest task in continuing to protect 93 million workers in this country, and I felt they would be especially interested in hosting a conference to deal with the problem which concerns OSHA as much as it does all of us here.

So, thank all of you for coming, and I hope we will see you again.