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Project Summary

Design of a Laboratory for Particulate Analysis

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In this study the need for a state-of-the-art laboratory for particulate analysis, particularly asbestos, is reviewed. The proposed equipment and operating expenses are justified, and a conceptual framework for the laboratory is formulated. The basis for selection of optimum equipment and the results of a detailed survey of equipment manufacturers are given. The design of the laboratory, the availability of skilled personnel, and a review of analysis methodology are summarized.

It is concluded that current and past problems in particulates analysis can best be resolved by organizing the laboratory as a center of excellence. Functions of the laboratory should include methods development, training, refereeing, standard sample development and evaluation, and the analysis of difficult samples. It is recommended that an advisory panel of experts be established to review the scientific quality and effectiveness of these activities. An acknowledged expert in asbestos analysis should be the director of a staff of seven to fourteen highly qualified members. Fourteen is suggested as the optimal size of the staff.

The initial capital cost for laboratory instrumentation and specialized air-handling equipment is estimated at $764,000, excluding the cost of the building. Start-up within one year after completion of the building is believed to be achievable. Minimum staffing and an initial level of support of $308,000 per year are recommended to inaugurate the proposed laboratory. At the optimal staffing, an operating budget of $534,000 per year is anticipated with about $260,000 generated by requests for services from outside groups or agencies.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The assessment of air quality and other environmental factors for effects on health, control strategy, standards setting, and compliance demands accurate monitoring data. Establishment of relationships between health effects and environmental levels have suffered from the lack of proven monitoring methodologies for the identification and quantification of the different species of asbestos. For some time the analysis of asbestos by electron microscopy has been a source of scientific controversy. The highly elongated shape of all asbestos material has long been recognized as a determining factor in the production of adverse health effects in exposed individuals. In the past, morphological identification of asbestos by phase contrast optical microscopy was the only reliable method for monitoring the concentration of asbestos fibers in the work place.

The basic assumption in the use of this method is that any fibrous particle is asbestos. This technique is only effective in environments where asbestos is known to occur in significant quantity. Optical microscopy is not reliable for monitoring asbestos fibers less than 0.5 μm in diameter and 5 μm in length or for low concentrations. Accurate knowledge about the occurrence of such suboptical particles has become increasingly important as concern has increased about their potential health effects.

Tradition and acceptance of optical data on asbestos by the medical community prompted attempts to extend the use of optical microscopy to environmental monitoring. Electron microscopy has the desired resolution for suboptical determinations but requires a much higher level of sophistication for sample preparation, counting, and identification of asbestos. Electron microscope methods for suboptical particles have been developed, but with limited success. As a result, electron microscopy is not generally regarded as a quantitative technique for the measurement of "asbestos."

To strengthen national environmental research, EPA commissioned the design of a model particulates analysis laboratory, with an emphasis on asbestos. Specific objectives were development of specifications and recommendations for a laboratory with special emphasis on the identification and measurement of serpentine and amphibole asbestos. Specimen preparation methods; clean room standards; and equipment for optical microscopy, x-ray diffraction, electron diffraction, and computer data processing were to be evaluated and prescribed. The number, qualifications, and availability of all personnel necessary to supervise and operate the facility were to be defined.

This report addresses EPA needs for a particulates analysis laboratory and contains recommendations based on extensive experience in this area by U.S. Steel Research Laboratory. In addition to utilization of customary light and electron optical methods, the adoption of newly developed techniques for automatic measurement of particle size, shape, and composition is highly recommended. These methods have been in use in the U.S. Steel Research Laboratory for three years and have proven to be rapid, accurate, and consistent in a wide variety of samples. The report contains the following sections. The conceptual framework, rationale, justification, functions, setting, and recommended support levels for the laboratory are described in Section 1. The recommended methodology is discussed in Section 2 with emphasis on emerging techniques. In Section 3 the physical plant is described; in Section 4 the selected instruments are discussed; and in Section 5 personnel requirements are specified. Section 6 provides an estimate of initial and annual costs and the timetable required to bring the laboratory into existence.

Conclusions

Functions and Operation

The creation of a laboratory devoted to developing and maintaining excellence in the field of particulates analysis, particularly asbestos, should be of the highest priority. The establishment of this laboratory must be the responsibility of the Federal administrative agency. No other organization has the resources to acquire state-of-the-art instrumentation on a continuing basis, to assemble a well trained and experienced staff, and to commit them to the long-term objective of improving particulates analysis methods.

The primary service function of the laboratory should be that of a center of excellence. It would provide a wide variety of essential services to other laboratories or agencies concerned with the acquisition or interpretation of data about the occurrence of particulates. These functions would include acting as a national reference laboratory, performing difficult particulate analyses, acting as a quality assurance laboratory, assisting in contract monitoring, assisting in enforcement actions, providing training, and performing methods development in the area of particulate analysis.

It is presumed that the proposed facility would be located near an existing national laboratory and would be housed within its administrative structure. However, to effectively carry out the multiplicity of functions described and to attract the caliber of staff required, the laboratory should be staffed with highly qualified specialists setting their own priorities. Every effort must be made to insulate the laboratory from sudden changes in focus or priority, yet the laboratory's objectives and projects must be responsive to agency needs. The formation of an advisory panel of knowledgeable persons from government agencies, academic institutions, and industrial laboratories could provide peer review of programs, evaluate progress, and assist in setting long-term goals.

Approximately fifty percent of the operating budget should cover the cost of methods development, participation in collaborative analysis programs, enforcement assistance, technical publications, and travel. The remainder of the support could be derived from services charged to groups requesting such services.

Analytical Methodology

The methods of analysis required in the laboratory range from binocular screening, phase-contrast and polarized-light microscopy to scanning and transmission electron microscopy with capabilities for x-ray spectroscopy and electron diffraction. For suboptical particulates the scanning electron microscope (SEM) can provide particle information based on manual characterization of morphology and composition. With newly developed digital beam-control devices, automated measurement of particle size, shape, and probable identity are also possible. The scanning-transmission electron microscope (STEM) will permit conventional manual x-ray and electron diffraction analysis and with the addition of beam-scan-control accessories automated particle characterization. The SEM and STEM instruments provide similar chemical and size information about particles but on an increasingly finer scale. Correspondingly, an increased level of sophistication is required to operate and maintain the instruments and to interpret the information derived from each. The sample flow and proposed methods of analysis are shown in Figure 1.

Facilities

The general design of a building (Figure 2) to house the proposed facilities is based on the following general premises. It is to be a freestanding, one-story structure large enough to house an optimal staff and all essential requirements. The structure will include a self-contained sample receiving area, clean rooms, laboratory space, staff and administrative offices, air-handling equipment, computer room, conference room, library, and canteen. This structure must be freestanding to afford protection from the contamination that will inevitably result from the air-handling system should the facility be incorporated into or contiguous to a larger structure. Obviously, the local institutional setting

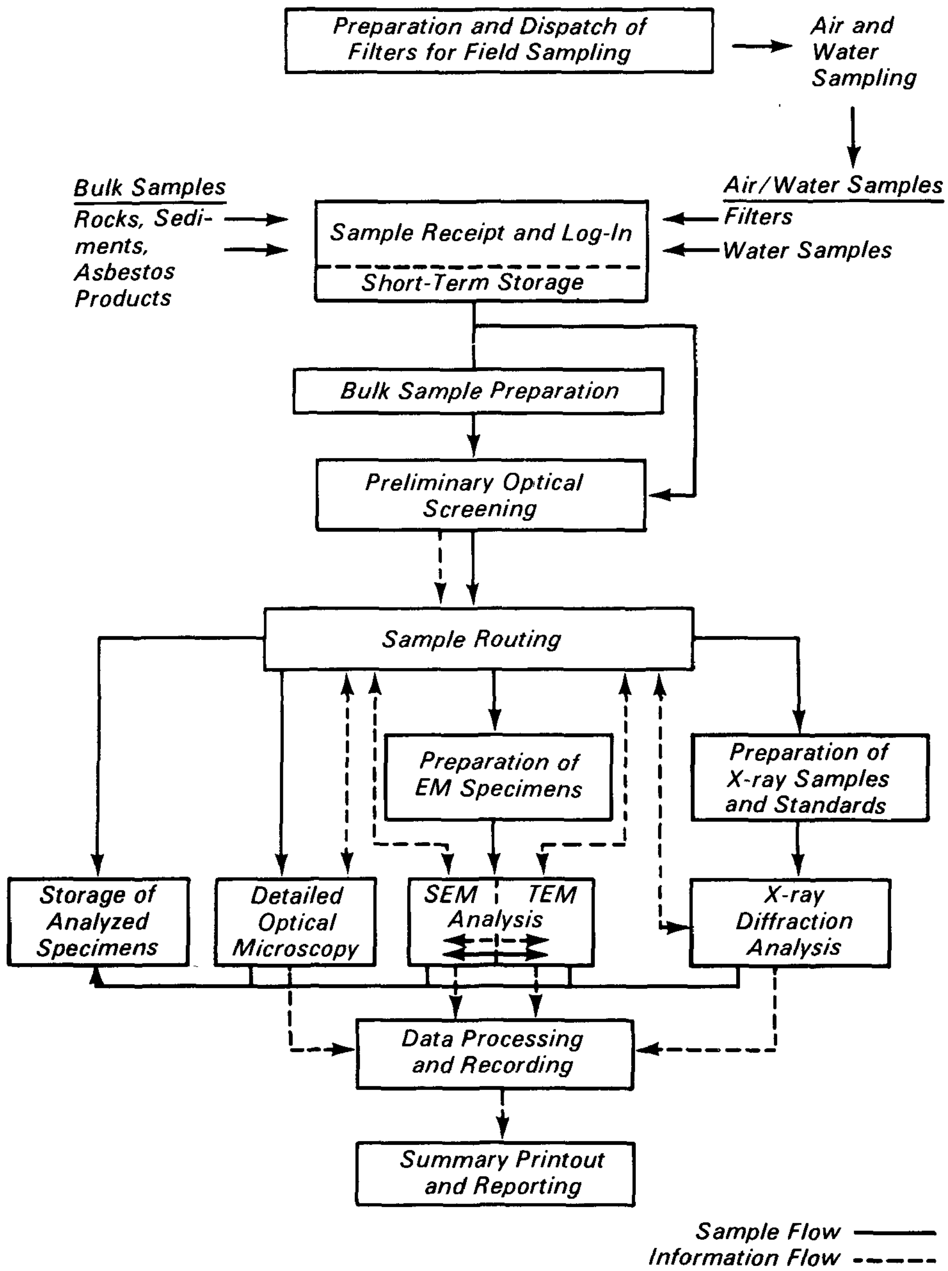


Figure 1. *Sample flow sheet.*

will affect some of these choices. The nominal dimensions of the proposed building are 56 feet by 104 feet for a total area of about 5600 square feet. Approximately one-fourth of the space is devoted to clean rooms, one-fourth to laboratories, one-fourth to offices, and one-fourth to utilities.

The philosophy underlying the design of the building is that the clean areas, laboratory areas, and office areas should be spacially separated. The air-handling equipment for the building can then be installed so that pressure differentials are maintained between the clean areas, the laboratories, and the office space. This will permit smoking and other activities in the office space without affecting the laboratory and clean areas.

Instrumentation

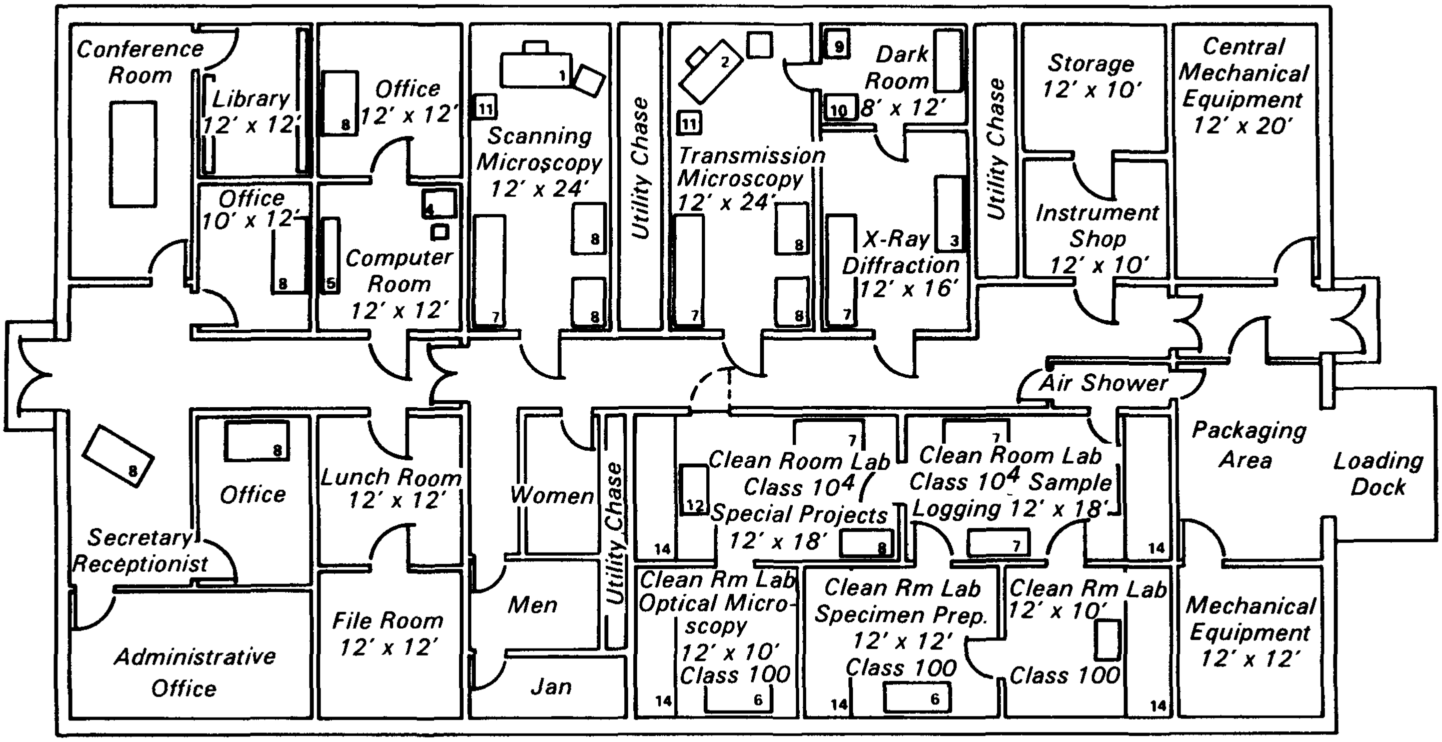
To keep abreast of and contribute to advances in particulate analysis methodology, to analyze a wide variety of air and water samples, and to corroborate data obtained by others, the laboratory must be equipped with top quality light and electron microscopes and an x-ray diffractometer. A multiterminal computer system is also required to monitor the status of current samples and to retrieve data needed for comparison in reporting programs. The criteria for selection of particular instrumentation include specifications, performance, actual compatibility with expected future developments, availability of replacement parts, service, and relative cost. Recommended major equipment for the laboratory includes a Siemens D-500 x-ray diffraction system; an AMR SEM; a Hitachi HU-6002 STEM, A Tracor Northern TN-4000 Energy Dispersive spectrometer; and an Ohio Scientific Challenger data base manager system. Specifications of major instruments, costs, and lists of other required equipment are given in the report.

Personnel

The challenging role to be assumed by the proposed laboratory demands that it be staffed by scientists and technicians of the highest caliber. Anything less will negate the point of establishing a new facility dedicated to excellence in particulate analysis. The key individual is the senior scientist selected to inaugurate and direct the facility. Considerable emphasis should be placed on obtaining an experienced person with outstanding credentials. The specific educational background of this person is not as important as established reputation and experience in particulates research. There are very few persons with the necessary qualifications. To attract one of these persons will require adequate initial and ongoing funding for developing and maintaining expertise of the highest caliber. If the laboratory is established as recommended in this report, the excellent opportunity presented should be sufficient to attract one of the best individuals in the country.

The scanning electron microscopists required should have expertise in computer-based data collection and manipulation systems. Knowledge of mineralogy and of x-ray microanalysis is essential to this position. Candidates with these skills are available.

The optimum staff would comprise trained specialists for each type of instrumentation installed in the laboratory with sufficient support personnel to



Equipment Legend:

Floor Plan 1

1. Scanning Electron Microscope
2. Scanning Transmission Microscopy
3. Automatic X-Ray Diffractometer
4. Computer Terminal
5. Disk Storage
6. Optical Microscope
7. Work Benches
8. Desks
9. "Logetronics" Printer
10. Enlarger
11. Image Analysis Equipment
12. Evaporator
13. Class 100 Fume Hoods
14. Circulating Fan & pre-Filter Enclosure (Typ. Five Places)

Figure 2. *Plan of proposed laboratory.*

run all the instrumentation at full capacity. The minimum staff is that required to maintain the instrumentation, stay abreast of developments, and perform a very limited number of special analyses. The optimum staff would include two electron microscopists, one petrographer, one senior scientist, eight technicians, a secretary, and a computer operator. The minimum staff would include one electron microscopist, one petrographer, four technicians, and a secretary.

Implementation Plan and Cost

A two-year schedule for the completion of this project is projected. When the laboratory is approved, the distinguished scientist who will eventually direct the laboratory should be brought into the project along with the architect. This scientist and the architect should work closely together in designing the building in detail using the conceptual design as a model. There will be adequate time available for the scientist selected to make the necessary visits to other laboratories and to become fully versed in the latest methodologies, instrumentation, and federal regulations on particulates.

After the completion of the architects' work, bids for construction may be requested. Four months after approval of the laboratory the construction contract for the building may be awarded. At this time the scientist in charge should order long-lead-time equipment such as electron optical instruments, x-ray diffractometers, and clean-room modules. Interviewing of the skeletal staff for the laboratory (two senior technicians and one more scientist) should begin eight months after approval of the laboratory. Short-lead-time equipment such as optical microscopes and evaporators should be ordered at this point in the schedule.

With the completion of the building and clean rooms after ten months, the equipment installation and testing period should begin. Calibration and trial runs on the equipment using well-characterized particulate samples should be carried out during this period. After twelve months the laboratory should be able to handle some real-world specimens at a limited rate, with full capabilities taking an additional twelve months to develop. The number of staff to be added with time will be determined by the sample demand.

The initial cost of equipment, excluding the building, will be $764,000. The annual laboratory operating cost including salaries, supplies, and equipment maintenance for minimal staffing will be $308,000. For optimum staffing this figure is $534,000.

Recommendations

The recommended conceptual design of the particulate analysis laboratory, if fully implemented, will achieve the objectives established by the EPA in commissioning this project. The broad purpose is to rectify the lack of reliable information about the exposure to asbestos and other particulates in non-occupational settings and their health effects. The current situation is not the result of a lack of effort on the part of EPA or other groups. To a large extent, it is a direct result of the ambiguity in the definition of asbestos and the intrinsic complexity and interdisciplinary nature of the analysis. Unfortunately, many of the projects sponsored to develop rigorous, robust methods for the analysis of asbestos and other particles have had limited success, and studies using recommended methods have been found wanting. Clearly a new approach is needed. Thus, establishment of a center of excellence for particulates analysis within EPA is timely and well conceived.

Specific recommendations stemming from this project are listed below:

1. Establishment of a federal laboratory specializing in the analysis of particulates should be of the highest priority.
2. The laboratory should be equipped with the best available instruments and staffed with highly qualified and experienced personnel.
3. It should function as a center of excellence qualified to monitor and evaluate relevant efforts in any laboratory.
4. An advisory panel should be established to aid in selecting programs, setting priorities, and evaluating progress, but otherwise the laboratory should be autonomous.
5. Peer review of the quality of the research program should be conducted under the auspices of the advisory panel.
6. A substantial part of the program should be devoted to methods development and to providing a definitive analysis for critical samples; a minimum portion should be devoted to routine analysis.
7. After start-up, funding should be provided to maintain state-of-the-art instrumentation as improvements become available.
8. A contract should be arranged with a qualified laboratory to develop the necessary hardware and software to measure and interpret electron diffraction patterns from particulates.
9. Initial research programs should be aimed at the development of suitable standards and reference samples, such as synthetic mixtures of asbestos and other materials, for general use.
10. The laboratory should participate actively in round-robin studies sponsored by ASTM or other groups to establish the degree of reproducibility that can be achieved and should initiate such studies if necessary.
11. The laboratory should screen and evaluate existing sample preparation techniques and develop new methods particularly suitable for automatic image analysis in both the scanning electron microscope and scanning transmission electron microscope.
12. The laboratory should develop automatic image analysis methods, including more sophisticated computer software.

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***R. J. Thompson** is the EPA Project Officer (see below).*
The complete report, entitled "Design of a Laboratory for Particulate Analysis," (Order No. PB 81-191 132; Cost: $9.50, subject to change) will be available only from:
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