

# MRI REPORT

POLARIZED LIGHT MICROSCOPIC (PLM) AND RELEASABILITY ANALYSIS  
OF MATERIALS USED IN SCHOOLS AND PUBLIC BUILDINGS

QUALITY ASSURANCE PROGRAM PLAN  
for the  
Office of Toxic Substances  
Office of Pesticides and Toxic Substances

EPA Prime Contract No. 68-02-3938  
Work Assignment No. 16  
MRI Project No. 7901-A(16)

For

U.S. Environmental Protection Agency  
Office of Toxic Substances  
Field Studies Branch, TS-798  
Washington, D.C. 20460

Attn: Dr. Frederick W. Kutz, Project Officer

POLARIZED LIGHT MICROSCOPIC (PLM) AND RELEASABILITY ANALYSIS  
OF MATERIALS USED IN SCHOOLS AND PUBLIC BUILDINGS

QUALITY ASSURANCE PROGRAM PLAN  
for the  
Office of Toxic Substances  
Office of Pesticides and Toxic Substances

EPA Prime Contract No. 68-02-3938  
Work Assignment No. 16  
MRI Project No. 7901-A(16)

For

U.S. Environmental Protection Agency  
Office of Toxic Substances  
Field Studies Branch, TS-798  
Washington, D.C. 20460

Attn: Dr. Frederick W. Kutz, Project Officer

## PREFACE

The following quality assurance program plan was prepared by Donna Rose of Midwest Research Institute. The plan was tailored to satisfy quality assurance needs of this bulk asbestos analytical program.

SECTION 1.0

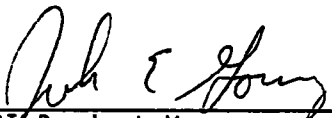
POLARIZED LIGHT MICROSCOPIC (PLM) AND RELEASABILITY ANALYSIS  
OF MATERIALS USED IN SCHOOLS AND PUBLIC BUILDINGS

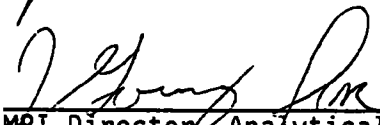
Quality Assurance Plan

EPA Contract No. 68-02-3938  
MRI Project No. 7901-A(16)

Approved for:

MIDWEST RESEARCH INSTITUTE

 7/5/84  
MRI Project Manager Date

 7/5/84  
MRI Director, Analytical Date  
Chemistry Department

for  7/5/84  
MRI Quality Assurance Manager Date

Approved for:

ENVIRONMENTAL PROTECTION AGENCY

\_\_\_\_\_  
EPA Project Officer Date

\_\_\_\_\_  
EPA Quality Assurance Officer Date

SECTION 2.0

TABLE OF CONTENTS

<u>Section</u>	<u>Heading</u>	<u>Pages</u>	<u>Revision</u>	<u>Date</u>
1.0	Title Page	1	0	7/3/84
2.0	Table of Contents	1	0	7/3/84
3.0	Project Description	1	0	7/3/84
4.0	Project Organization and Management	3	0	7/3/84
5.0	Personnel Qualifications	1	0	7/3/84
6.0	Facilities, Equipment, Consumables and Services	3	0	7/3/84
7.0	Data Generation	2	0	7/3/84
8.0	Data Processing	1	0	7/3/84
9.0	Data Quality Assessment	2	0	7/3/84
10.0	Corrective Action	2	0	7/3/84
11.0	Documentation and Reporting	2	0	7/3/84

Appendix A - Köhler Illumination

Appendix B - Analysis of Bulk Samples for Asbestos and/or Other Materials

Appendix C - Releasability Assessment Protocol

List of Plan Holders:

Midwest Research Institute

J. Going	D. Rose
C. Green	P. Constant
G. Atkinson	
C. Haile	

Environmental Protection Agency

F. R. Kutz
J. J. Breen
M. Huneycutt

## SECTION 3.0

### PROJECT DESCRIPTION

The Exposure Evaluation Division (EED) of OPTS is currently developing and implementing a multifaceted asbestos analytical program to support the asbestos-in-schools and public buildings program. The analytical program involves the development of an analytical protocol and guidance on a quality assurance program appropriate for implementation at the state and local level. The protocol and quality assurance packages are being prepared under contract, for OTS/EED through collaboration with EMSL/RTP.

In the interim period, however, there are serious sampling and analysis problems which need to be addressed in support of the asbestos-in-schools and public buildings program.

The present absence of a standardized analytical protocol has resulted in conflicting analyses of bulk samples with complex matrices. The OTS task manager will identify and provide MRI with samples of these analytically troublesome materials and copies, as available, of the conflicting analyses. MRI will analyze these samples using PLM with or without dispersion staining (DS) with an eye towards resolving the analytical discrepancies. When requested by the EPA work assignment manager, MRI will provide detailed comments and guidance on what elements or components of the material matrix may be the source of the discrepancies.

## SECTION 4.0

### PROJECT ORGANIZATION AND MANAGEMENT

#### 4.1 Organization

The project organization is presented in Figure 4.1.

4.1.1 Department Management: Dr. John Going will represent department management. He will:

- Assure that all necessary resources are available.
- Assure that the Quality Assurance Monitor (QAM) is fully informed and involved in the project.
- Assure that all personnel are informed of project QA policy.
- Review all communication from QA regarding the project.
- Assure that any problems, deviations, etc., reported by QA receive immediate corrective action.

4.1.2 QA Management: Carol Green, Quality Assurance Manager, will be the QAM for this project and will:

- Help prepare the project QA plan.
- Assure that all MRI QA policies and procedures are available and understood.
- Conduct a systems audit of the asbestos analysis.
- Assure management that the facilities, equipment, personnel, methods, records, and controls are in conformance with program objectives and requirements.
- Inform the work assignment leader and department management representative of any problems in the project and request corrective actions by way of reports to management.

4.1.3 Work Assignment Leader: Donna Rose will be the work assignment leader. She will:

- Help prepare the project QA plan.

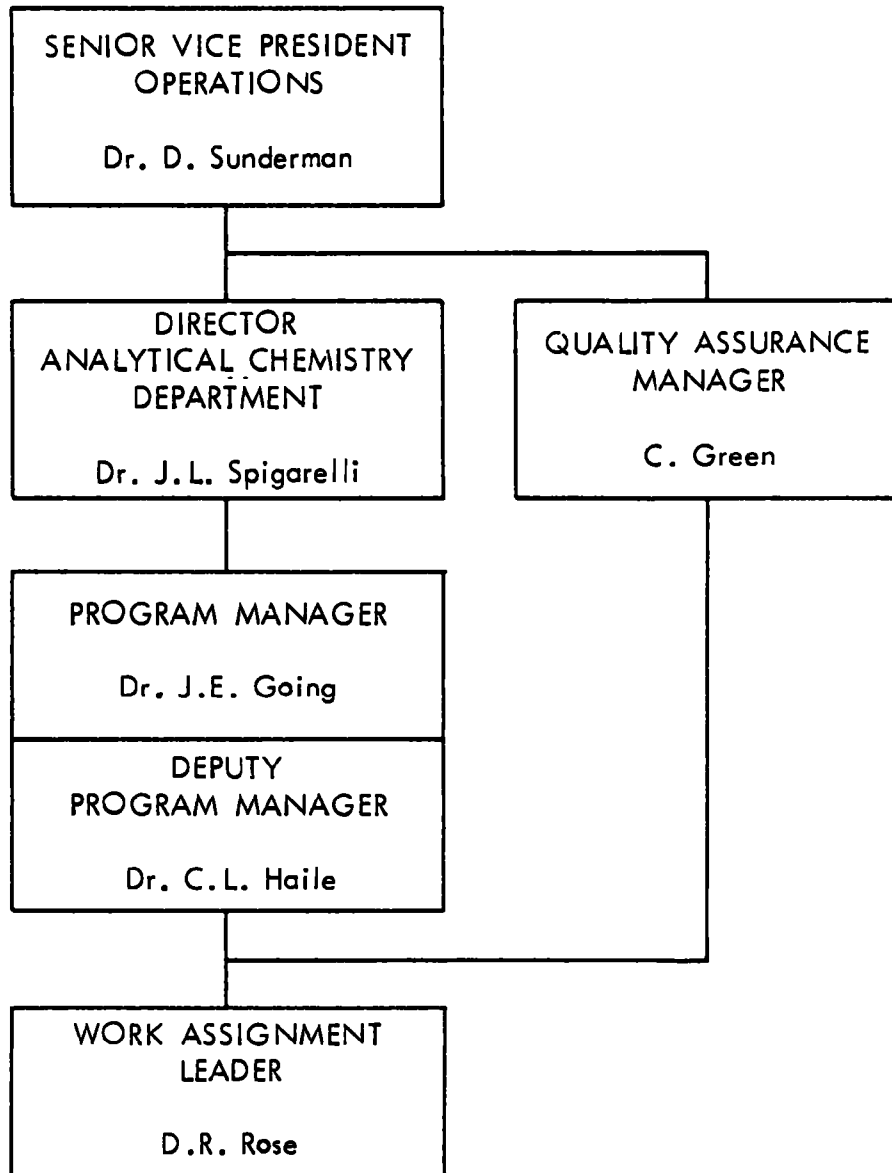


Figure 4.1 - Project Organization



- Be responsible for training all staff where required.
- Enforce instrument calibration and maintenance procedures.
- Be responsible for sample custody and traceability.
- Maintain document control of lab data, notes, records, and other hard copy information.
- Examine notebooks at appropriate intervals and verify authenticity by initialing at bottom of the appropriate page.
- Take corrective action for any problems and communicate in writing to the QAM and department management.

4.1.4 Physical/Chemical Analysis Staff: Donna Rose will be responsible for asbestos analysis. She will be assisted by Sam Ferro.

4.1.5 Consultant: Gaylord Atkinson will be available for consultation.

## SECTION 5.0

### PERSONNEL QUALIFICATIONS

Donna Rose has considerable experience in sampling and analysis of asbestos-containing materials. She is project leader of asbestos analysis projects and has served as subtask leader on EPA asbestos analysis work assignments.

Sam Ferro has completed an audiovisual course in asbestos identification by PLM and has experience in PLM analysis of asbestos-containing materials.

Gaylord Atkinson manages a program specializing in microanalytical chemistry, including microscopy. He has been task manager of several EPA asbestos-related tasks.

All analysts have successfully participated in the EPA "Asbestos Bulk Sample Analysis Quality Assurance Program" administered by RTI.

## SECTION 6.0

### FACILITIES, EQUIPMENT, CONSUMABLES AND SERVICES

#### 6.1 Facilities and Equipment

6.1.1 Microscopy Laboratory: The microscopy laboratory is adequately designed and equipped for asbestos analysis. Sample preparation and microscopic analyses are performed in a fume hood. The following microscopes and accessories are available:

- Unitron MPS Polarizing Light Microscope\*
- Unitron MPS-3 Polarizing Light Microscope\*
- Bausch and Lomb Stereo Zoom Microscope
- Olympus Stereo Zoom Microscope
- Zeiss Phase Contrast Microscope and Illuminator
- Various Illuminators

\* This microscope is equipped with McCrone dispersion staining objectives and "first-order-red" compensators.

6.1.2 Infrared Spectroscopy Laboratory: The infrared spectroscopy laboratory is equipped with:

- Perkin-Elmer Model 283 spectrophotometer with communications accessory and Model 3500 intelligence terminal.
- Wilks Model 8B multiple internal reflection spectrophotometer, a modified Perkin-Elmer Model 237, used for both reflection and transmission.
- Perkin-Elmer Infracord 137B IR unit.
- Various ancillary equipment, including KBr press and special cells with CsCl, AgCl, and quartz windows.

6.1.3 Inspection and maintenance: MRI's instrument maintenance program consists of both scheduled (or preventive maintenance) and nonscheduled maintenance procedures. Records of maintenance performed on the instruments are maintained in the respective instrument logbooks. In addition, any instrument repair not performed by the laboratory personnel is handled by the Instrument Services Department, which also adheres to a recordkeeping program.

Routine maintenance of microscopes consists of cleaning lenses when dirty with lens tissue. Nonroutine maintenance and/or repair is provided by MRI's Instrument Services Department who will contact an authorized repairman for service.

#### 6.1.4 Calibration procedures

6.1.4.1 Microscopes: Koehler illumination is achieved in the polarizing light microscopy in a manner similar to that described by McCrone<sup>2</sup> (see Appendix A), and checked by the analyst at the start of each analysis day.

6.1.4.2 Infrared: The IR spectrum of a polystyrene film is run by Chemical Management personnel of the Bio-analytical Chemistry Section once a week. The pattern of peaks and the position of eight of the more prominent absorbance maxima compared to standard values is recorded. If significant discrepancies are observed, the Instrument Services Department is contacted.

#### 6.1.5 Standard and reference materials

6.1.5.1 Asbestos and other standards: Bulk standards containing known amounts of asbestos are available for assisting in the visual estimation of volume percentages of asbestos. Various asbestos mine dusts of known identity as well as other known minerals and fibers are also available.

##### 6.1.5.2 Printed reference materials:

1. U.S. Environmental Protection Agency. Asbestos; friable asbestos-containing materials in schools; identification and notification. Final Rule. 40 CFR Part 763, Federal Register, Vol. 47, No. 103, May 27, 1982.
2. McCrone, W. C., and J. G. Delly, The Particle Atlas, 2nd Ed., Volumes I-IV (1974), Volumes V-VI (1978), Ann Arbor Science Publishers.
3. McCrone, W. C., et al., Polarized Light Microscopy, (1979), Ann Arbor Science Publishers.
4. McCrone, W. C., The Asbestos Particle Atlas, (1980), Ann Arbor Science Publishers.

5. Rajhans, G. S., and J. R. Sullivan, Asbestos Sampling and Analysis, (1981), Ann Arbor Science Publishers.
6. McCrone, W. C., Identification of Asbestos: An Audiovisual Training Program for Microscopists, (1983), Brian Howard and Associates.
7. Zeller, M. V., and M. P. Juszli, Reference Spectra of Minerals (1975), Perkin-Elmer.

## 6.2 Consumables and Supplies

Cargille or equivalent refractive index liquids, as specified in McCrone's analytical scheme, will be utilized. Other materials used will include precleaned standard microscope slides and cover glasses, and infrared grade KBr.

## SECTION 7.0

### DATA GENERATION

This section presents a detailed outline of the analytical approach to bulk samples suspected of containing asbestos.

#### 7.1 Sample Collection

Samples will be collected and coded by EPA.

#### 7.2 Sample Analysis

Samples are analyzed according to the protocol given in Appendix B (see also Ref. No. 1, Section 6.1.5.2. For the microscopic analyses, MRI uses a stereo zoom microscopes capable of 7X to 40X magnification equipped with an external illuminator for oblique illumination, and a polarizing microscope (100X magnification) equipped with an external illuminator and dispersion staining objective.

Each bulk sample is examined as a whole through the stereo microscope for layering, homogeneity, and the presence of fibrous material. Identification of macrosize nonfibrous components is usually possible at this point.

Subsamples of the bulk sample selected using the stereo microscope are mounted onto a clean microscope slide in the appropriate index of refraction liquids for examination through the polarizing microscope.

The polarized light microscopy procedure consists of observing the characteristics of the subsample components with transmitted polarized light, crossed polars, slightly uncrossed polars, crossed polars plus the first-order red compensator, and the central stop dispersion staining objective. The observations obtained using the various techniques are used to identify the fibrous and some of the nonfibrous components on the basis of morphology, sign of elongation, and refractive index/dispersion staining colors.

Quantitation of the asbestos is achieved by stereo microscopic observation of the entire bulk sample through the stereo microscope and PLM examination of the subsamples. The volume percentages of the various components are estimated in relationship to the whole sample.

A releasability assessment is made according to the protocol in Appendix C.

7.3 Internal Quality Control Checks

Every tenth sample received will be analyzed as a blind duplicate by a second analyst. Results will be recorded in a laboratory notebook reserved exclusively for internal quality control analyses. Results of both analyses will be reported to the EPA task manager.

7.4 Performance and Systems Audits

The MRI QA Manager will perform a systems audit of the asbestos analysis and report the findings to the task leader.

## SECTION 8.0

### DATA PROCESSING

#### 8.1 Collection

Data collection will consist of recording in a bound laboratory notebook the gross and microscopic observations for all samples.

#### 8.2 Data Validation

Procedures for validation of data will include screening laboratory notebooks for completeness of sample information and analytical observations, and comparing duplicate analysis results. This will be done when work is completed on each group of samples analyzed if the number of samples received in a group is less than 10, or at the end of each work day if the number of samples in a group is more than 10.



## SECTION 9.0

### DATA QUALITY ASSESSMENT

#### 9.1 Precision

Precision will be determined from the results of duplicate analyses. Neither of the duplicate estimated percentages should vary from the average of the two by more than  $\pm 50\%$ . Duplicate identifications of species of asbestiform materials (e.g., chrysotile, amosite) shall agree.

#### 9.2 Accuracy

Accuracy will not be determined, because the percentages are visually estimated.

#### 9.3 Completeness

No data losses are anticipated.

#### 9.4 Comparability

Other laboratories routinely use PLM analysis for asbestos; therefore, our results are comparable.

#### 9.5 Standards

Various UICC asbestos mine dusts and miscellaneous matrices containing known amounts of asbestos are available for reference. Their use, when required, will be recorded in the project laboratory notebook.

#### 9.6 Traceability of Samples

Traceability methods will be utilized as described below.

A unique MRI number will be affixed to each sample. The following information will be entered in an MRI logbook.

- MRI number (as above).
- Sample description.

- Source (if available).
- Date of receipt.
- MRI logbook number and page where analytical data are entered.
- Sample disposition (storage box number, returned to EPA, forwarded to subcontractor, etc.).

#### 9.7 Traceability of Data

Data will be documented from recording of sample receipt through reporting results to the EPA work assignment manager.

#### 9.8 Representativeness

Samples will be collected by EPA and shipped to MRI. Samples received are assumed to be representative of the environment from which they were taken. Each sample received will be examined as a whole.

## SECTION 10.0

### CORRECTIVE ACTION

The work assignment leader has primary responsibility for taking corrective action; if she is unavailable, the department management and/or the QAM shall be contacted for instructions. Some of the types of problems and corrective actions to be taken are listed below.

#### 10.1 Systems Audits

If problems are detected by the QAM during any audit:

- The QAM shall immediately notify the lab person responsible and the work assignment leader of the problem(s) and any action(s) taken.
- The work assignment leader and the responsible field/lab person shall correct the problem, then notify the QAM.
- The QAM shall then prepare and send a problem/action-taken memo to the department management.
- The work assignment leader, department management, and the QAM shall then collectively decide on the appropriate action.
- The work assignment leader shall then implement the corrective action.
- The work assignment leader shall prepare and send a documentation memo to department management and the QAM.

#### 10.2 Loss of Data

The work assignment leader shall investigate the problem, then perform one or more of the following actions:

- If the problem is limited in scope, the problem/action-taken is documented in the MRI notebook; the work assignment leader then prepares and sends a problem/action-taken memo to the QAM and department management.
- If a large quantity of data is affected, the problem/action-taken is documented in the MRI notebook; the work assignment leader then prepares and sends a problem/action-taken memo to the QAM, department management, and the EPA work assignment manager.

### 10.3 Significant QA Problems

In general, the work assignment leader shall identify technical problems.

- The work assignment leader prepares and sends a problem memo to the QAM and department management; if the problems are significant, the action is determined collectively.
- The action taken is documented in the MRI notebook.

## SECTION 11.0

### DOCUMENTATION AND REPORTING

#### 11.1 Documentation

All entries made in logbooks, laboratory notebooks and other task documents shall be in ink. Errors shall be corrected by drawing a line through the error and entering the correct information. The correction will then be initialled, dated, and a written explanation for the correction given. All additions to existing data will be dated and initialled at the time they are entered.

#### 11.2 Document Control

11.2.1 Sample traceability: All samples submitted by EPA shall be logged in by assigning each sample a unique MRI number and recording that number and other pertinent information in the task sample logbook. Entries (book number and page number) in the analytical task notebook and date(s) of reports shall be cross-referenced in the task sample logbook.

Chain-of-custody procedures will be instituted only on request of the EPA work assignment manager.

11.2.2 Data archiving and storage: Institute QA SOP's will be followed for task sample logbooks, laboratory notebooks and other pertinent documents.

#### 11.3 Quality Assurance Reports to Management

The QAM, in cooperation with the work assignment leader, shall identify critical phases of the project which will be subject to inspection. The inspection will include a review of:

- Results of performance and systems audits.
- Equipment maintenance and calibration records.
- Data entry.
- Data errors, deletions, and corrections.
- Records and other information.

- Document control.
- Assessment of data accuracy, precision, and completeness.

The results of inspections and audits will be reported by the QAM to MRI management; summaries of the audits will be reported to the EPA project officer.

#### 11.4 Report Design

Reports will consist of telephoned results to the EPA work assignment manager, followed by a confirming letter report through normal channels. The analysis schedule is unknown, but telephone contact followed by letter report will be the norm for each sample shipment received on this task.

Reports will identify each sample by the MRI log number, as well as any information on the sample label as-received. Analysis results given will include, but will not be limited to:

- Type and percent by volume of asbestos.
- Type and percent by volume of non-asbestos fibrous materials.
- Type and percent of other identified components.
- An assessment of releasability.
- Other information as requested by the EPA Task Manager.

## APPENDIX A

### KOHLER ILLUMINATION

"To arrange the microscope and illuminator for Köhler illumination, it is well to proceed methodically through the following steps:

"a. Remove the diffusers and filters from the lamp.

"b. Tilt the lamp until the beam is centered on the microscope mirror. Open the lamp diaphragm (also called field diaphragm, field iris or radiant field stop).

"c. By moving the lamp condenser, focus a sharp image of the filament on the plane of the microscope substage iris. The filament image should be large enough to fill, even though unevenly, the microscope substage condenser opening. If it does not, move the lamp away from the microscope to enlarge the filament image and refocus.

"d. Place a specimen on the microscope stage and focus sharply with a 16 mm (10X) objective. Open the substage diaphragm completely. If the light is too bright, temporarily place a neutral density filter in the lamp.

"e. Close field iris somewhat and adjust the mirror to center it in the field of view.

"f. Move the specimen so that a clear area is under observation. Place the Bertrand lens in the optical path, or remove the ocular and insert an auxiliary telescope (sold as a phase contrast accessory) in its place, or remove the ocular and observe the back focal plane of the objective directly. Now observe the lamp filament through the microscope.

"g. If the filament does not appear to be centered, swing the lamp housing in a horizontal arc centered at the field diaphragm. The purpose is to maintain the field diaphragm on the lamp in its centered position. If a vertical movement of the filament is required, loosen the bulb base and slide it up and down. If the base is fixed, tilt the lamp housing in a vertical arc around the field diaphragm (again endeavoring to keep the lamp diaphragm centered). If you have mastered this, you have accomplished the most difficult step. (Better microscope lamps simplify this step with adjustments to move the bulb independently of the lamp housing.)

"h. Put the specimen in place, replace the ocular and the desired objective and refocus.

"i. Open or close the field diaphragm until it falls just outside the field.

"j. Observe the preparation and adjust the contrast by opening or closing the substage iris. It must be as wide open as possible.

"k. Observe the back focal plane of the objective, preferably with the Bertrand lens or the auxiliary telescope, and note the position of the substage iris. If it is not open at least two thirds of the diameter of the back focal plane, the preparation has too little inherent contrast or you are a bad judge of good illumination. It is instructive to vary the opening of the substage iris and observe the image and the objective back focal plane critically during this manipulation.

"l. If the illumination is too strong, insert an appropriate neutral density filter between the illuminator and the condenser. Do not use the condenser iris or the lamp field diaphragm to control illumination intensity."

Source: McCrone, W. C., et al., Polarized Light Microscopy, Ann Arbor, Michigan, 1979.



## APPENDIX B

### ANALYSIS OF BULK SAMPLES FOR ASBESTOS AND/OR OTHER MATERIALS

CAUTION: Perform all asbestos analyses in laboratory fume hoods.

#### General and Stereomicroscopic Observations

- Empty the entire bulk sample onto clean weighing paper. Describe general appearance in laboratory notebook (e.g., floor tile, acoustical tile, etc.).
- Examine the sample under the stereomicroscope. Use tweezers and probes to expose all materials to view. Write stereomicroscopic observations in laboratory notebook.

#### Polarized Light Microscope (PLM) Observations

All fibrous components in a bulk sample must be identified, although nonasbestos fibers may be designated by class only (e.g., synthetics).

- Set up the PLM for Köhler illumination (see Appendix A) in a manner similar to that described by McCrone (McCrone, W. C., et al., Polarized Light Microscopy, Ann Arbor Science, Ann Arbor, Michigan 1979). Use only the steps appropriate for the specific PLM and associated illuminator used (e.g., all PLM's do not have a Bertrand lens, all illuminators are not equipped with diffusers).

- Using tweezers and the stereomicroscope if needed, place representative examples of each type of material present onto a microscope slide and mount them in one or more drops of 1.550 HD and other appropriate index of refraction liquids for PLM analysis. Separate fibers in the liquid, if necessary, then add coverslip.

- Place prepared slide on PLM stage and focus on sample.

- Identify asbestiform minerals and other bulk sample components using prescribed polarized light microscope techniques. These techniques include but are not limited to:

Crossed polars (+): Insert upper analyzer and cross the polars. Rotate stage and observe angle of extinction of anisotropic minerals. Observe morphology of fibers.

Slightly uncrossed polars (X): (Optional: may help in determining glass).

Crossed polars plus first order red plate (+R1): Insert upper analyzer and cross the polars. Insert first order red plate. Rotate stage and observe sign of elongation.

Dispersion staining central stop (CS) or annular stop (AS): Uncross polars and remove upper analyzer. Insert Bertrand lens and rotate central or annular stop into place. Remove Bertrand lens and refocus microscope. Rotate the stage and determine dispersion staining colors of sample materials. Enter in lab book.

Transmitted light ( ): (Used when crocidolite is suspected). Insert Bertrand lens and remove dispersion staining stop from field of view. Remove upper analyzer. Remove Bertrand lens. Rotate stage and determine pleochroism of suspect fiber(s). Note all PLM observations in laboratory notebook.

### Determination of Carbonate vs. Noncarbonate Binders

Using tweezers, transfer small representative portions of the bulk sample to black spot plate depression.

While observing this subsample under the stereomicroscope, add hydrochloric acid (approximately 3N) dropwise.

Estimate carbonate (soluble) versus noncarbonate (insoluble) binders on the basis of amount of material soluble with  $\text{CO}_2$  evolution. Enter estimate in lab book.

### Estimation of Sample Components

Using PLM results (specific materials identification) combined with carbonate/noncarbonate binder determination and whole-sample observation under the stereomicroscope, estimate volume percentages of all materials and enter in lab book. Standards containing known percentages of asbestos are available for reference.

### Disposition of Analyzed Samples

Return bulk sample to its original primary container and reseal.

Damp-wipe the outside of the container and remove sample from hood.

If asbestos was found, place "cancer hazard" or other type of carcinogen sticker on outside of primary container.

Place analyzed sample in designated secondary container to await storage or other disposition as directed by project/task leader.

## Handling Discrepancies

If blind analysis by a second MRI analyst or by outside QA contractor produces serious discrepancy, the sample will be compared with standards containing known percentages of asbestos. These standards are available for reference, should they be needed by the analyst to compare with any sample being analyzed.

## Infrared Spectroscopy (IR)

An occasional sample may require IR to confirm identity of one or more components. (CAUTION: Perform all grinding and mixing in a laboratory fume hood.)

### A. KBr Pellet

#### 1. If the material appears pure:

a. Take approximately 1 to 2 mg and grind to a fine powder using a small mortar and pestle.

b. Add approximately 150 mg KBr to the ground sample and mix thoroughly.

c. Transfer sample mixed with KBr to a mini press plugged at one end with the appropriate bolt.

d. Secure the second bolt and compress the KBr using bench-top wrench and t-handle socket wrench.

e. Remove both bolts and examine pellet--it should be fairly clear. If not, re-press.

f. Consult instrument SOP for specific IR instrument used.

g. Run spectrum.

h. If spectrum is too weak, add more sample and make another pellet. Or, use computer (consult instrument manual) to enhance the spectrum. If spectrum is too strong, add KBr and make another pellet.

i. Compare sample spectrum with known mineral spectra for identification.

#### 2. If the material is impure:

a. Remove adequate amount (1 to 2 mg of the fiber type of interest from the bulk sample).

- b. Scrape any adhering binder from the fibers.
- c. Grind the cleaned sample to a fine powder using a mortar and pestle.
- d. Proceed from Step 1(b) above.

#### B. Cast Film

High concentration of organic-soluble material may be cast as a film on silver chloride or sodium chloride discs and spectra taken of the cast film to be compared with reference spectra. Other procedures also may be applicable.

#### C. Detection and Quantification Limits

The limits of detection and quantification of asbestos in a bulk sample are a function of the presence of materials that tend to coat or otherwise obscure the fiber. In the absence of interferences, detection and quantification limits are simply related to the quantity of sample examined and are well below 1%. Estimated quantification values are relatively accurate under these conditions.

When obscuring materials (e.g., gypsum, calcium carbonate, cement) are present, the limits can be greatly increased and may approach or exceed 1%. Special treatments to visualize the asbestos may then be required, such as treatments to dissolve the obscuring materials.

#### MRI Internal QC

Ten percent 10% of the bulk samples will be reanalyzed by a second MRI analyst. Every 10th sample analyzed by the primary analyst will be re-coded and analyzed blind by a second person. Results will be compared after the second analysis is complete. Comparison of results may be on a sample by sample basis, or after several of the internal QC samples have been analyzed.

## APPENDIX C

### RELEASABILITY ASSESSMENT PROTOCOL\*

The releasability rating is a subjective determination requiring the good judgment of an experienced analyst. The releasability rating is assigned after completing the following steps:

1. Determine the identity and volume percent of sample components by the usual microscopic means.

2. Examine the sample under a stereomicroscope at approximately 10X magnification. Note the size and freedom of the fibers.

3. Probe the sample with needles and note the brittleness, toughness, or resilience of the matrix.

4. Rate the releasability on a scale of 0 to 9. A low rating is assigned samples with low potential for release of asbestos, a high rating for samples with high release potential. The rating is assigned following the consideration and judgment of the following factors:

- Assign a high rating for a large number of free asbestos fibers.
- Assign a high rating for a brittle or fragile matrix that can be readily broken or abraded.
- Assign a low rating for a resilient or tough matrix (such as a resin-bonded glass wool or resin-bonded vermiculite).

---

\* Midwest Research Institute. 1983. Releasability of asbestos containing materials as an indicator of airborne asbestos exposure. Draft Final Report. MRI Project 4901-A(55). Washington, DC: Office of Pesticides and Toxic Substances, U.S. Environmental Protection Agency. Contract 68-01-5915.