California Environmental Protection Agency

Air Resources Board

Discussion of Potential Changes to ARB Test Method 435:

Determination of Asbestos Content of Serpentine Aggregate

January 24, 2008, Workshop

Operations Planning & Assessment Section
Quality Management Branch
Monitoring and Laboratory Division

Workshop Agenda

- Introduction
- Interlaboratory Study (ILS) Presentation
  - Questions & Answers
- Potential Revisions to Test Method 435 (M435)
  - Processing Procedures
  - Analytical Procedures
  - Laboratory Accreditation
- Revision Schedule/Next Workshop

M435 Revision Schedule

- January 24, 2008, Workshop (1st)
  - Rationale & identification of areas of M435 currently being examined for revision
- May/June 2008 Workshop(s)
  - More focused proposed revisions to M435
- August/September Workshop(s)
  - Proposed draft language available for comment
- February 2009 Board Hearing

ARB Asbestos Regulations

- 1986 – Identified asbestos as an airborne toxic contaminant (TAC)
- 1991 – Airborne Toxic Control Measure (ATCM) for Surfacing Applications
  - Revised 2001
- 1991 – M435: Determination of Asbestos Content of Serpentine Aggregate
- 2002 – ATCM for Construction, Grading, Quarrying, and Surface Mining Operations

➢ M435 referenced in both ATCMs

Rationale for M435 Revision

- Observations by parties regarding the variability of laboratory equipment, M435 procedures, and analytical results
- Results of ARB ILS show that certain M435 laboratory practices result in differences in the % asbestos reported.

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  - Questions & Answers
- Potential Revisions to Test Method 435 (M435)
  - Processing Procedures
  - Analytical Procedures
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Preliminary Interpretation of the Interlaboratory Study for ARB Test Method 435:

Determination of Asbestos Content of Serpentine Aggregate

ARB Test Method 435

- Airborne Toxic Control Measure (ATCM) for Surfacing Applications
  - <0.25% asbestos content limit of aggregate material
- ATCM for Construction, Grading, Quarrying, and Surface Mining Operations
  - Dust control measures required in areas with ≥0.25% asbestos
- Used by Dept. of Toxic Substances Control (DTSC) to determine asbestos content of soils at new school construction sites in CA;
- Used nationwide as a bulk method to determine asbestos content in soils.

6 Types of Regulated Asbestos in CA

Chrysotile
$Mg_3Si_2O_5(OH)_4$

Actinolite
$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$

Tremolite
$Ca_2Mg_5Si_8O_{22}(OH)_2$

Crocidolite
$Na_2(Fe,Mg)_3Fe_2Si_8O_{22}(OH)_2$

Anthophyllite
$Mg_7Si_8O_{22}(OH)_2$

Amosite
$(Fe)_2(Fe,Mg)_5Si_8O_{22}(OH)_2$

ARB Test Method 435 Issues

Observations by some parties regarding Test Method 435 (M435):

- "Guidelines in Test Method 435 allow laboratories great latitude in processing and analytical procedures;"
- "Laboratories prepare and analyze soil and rock samples in different ways;"
- "Different laboratories obtain differing results when analyzing the same samples for asbestos."

Interlaboratory Study (ILS) Objectives

- To investigate variability in preparation and analytical procedures used by laboratories applying M435;
- To determine whether these differences affect asbestos content determination.

Test Method 435 Protocol

Geologic Sample → Dry, crush to <3/8" nominal diameter, reduce to 1 pint aliquot → Pulverize majority to <75 µm diameter

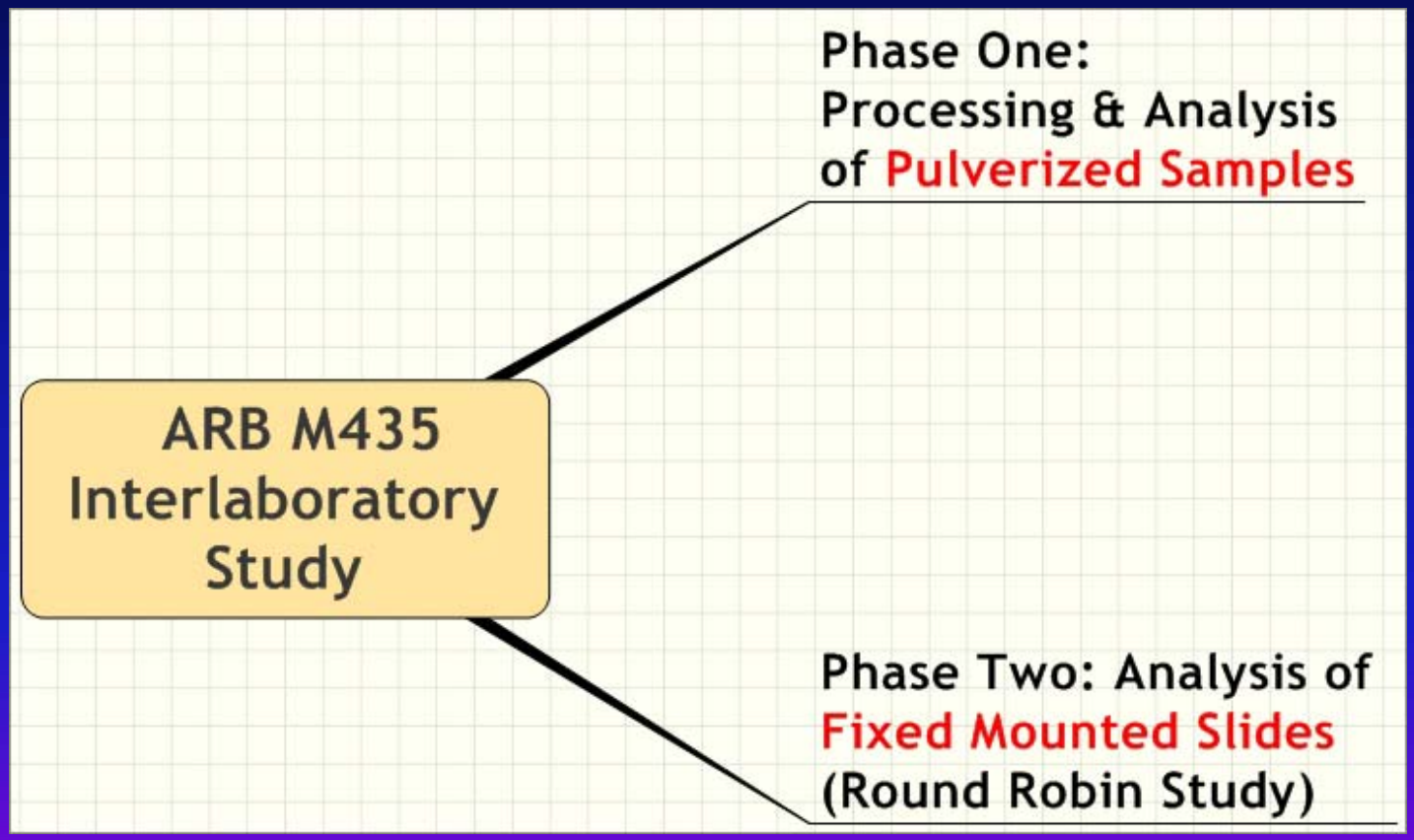
↓

Analyze using Polarized Light Microscopy (PLM)

↓

400-point count rules
Determine % Asbestos

M435 Interlaboratory Study (ILS)



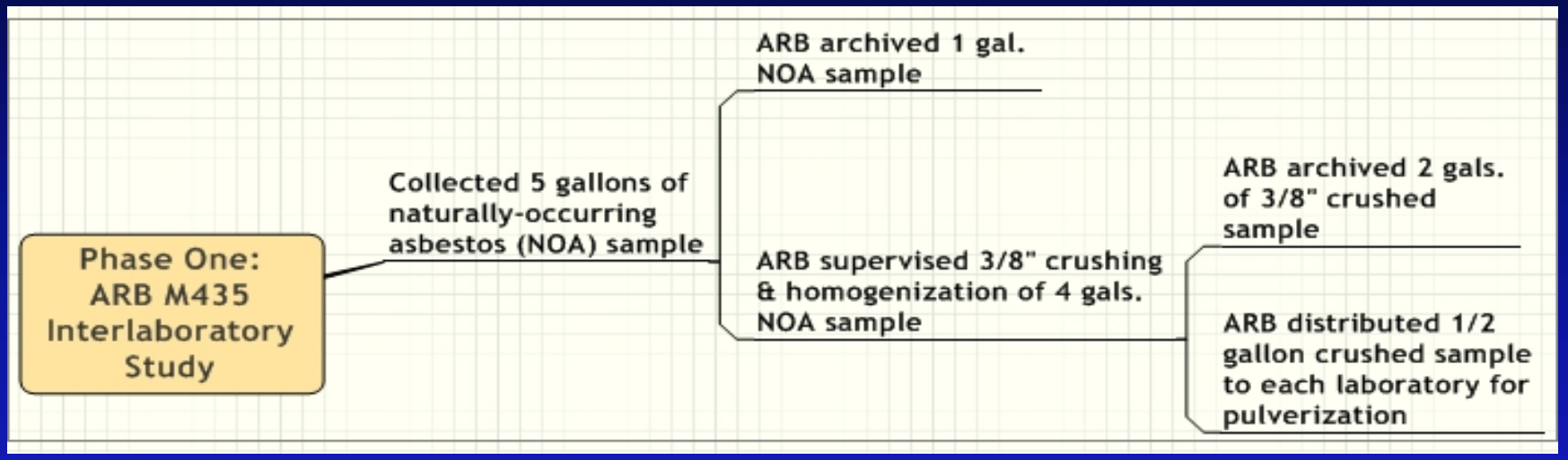
ILS Phase One

Field Sample Processing and Analysis

Phase One Objectives

- To obtain qualitative information on variability of equipment, sample processing, protocols, and fiber-counting practices among laboratories;
- To determine whether these differences result in variations of reported % asbestos.

Phase One: Crushing of Field Sample



Pulverization of Crushed Material: Four Sample Preparation Methods

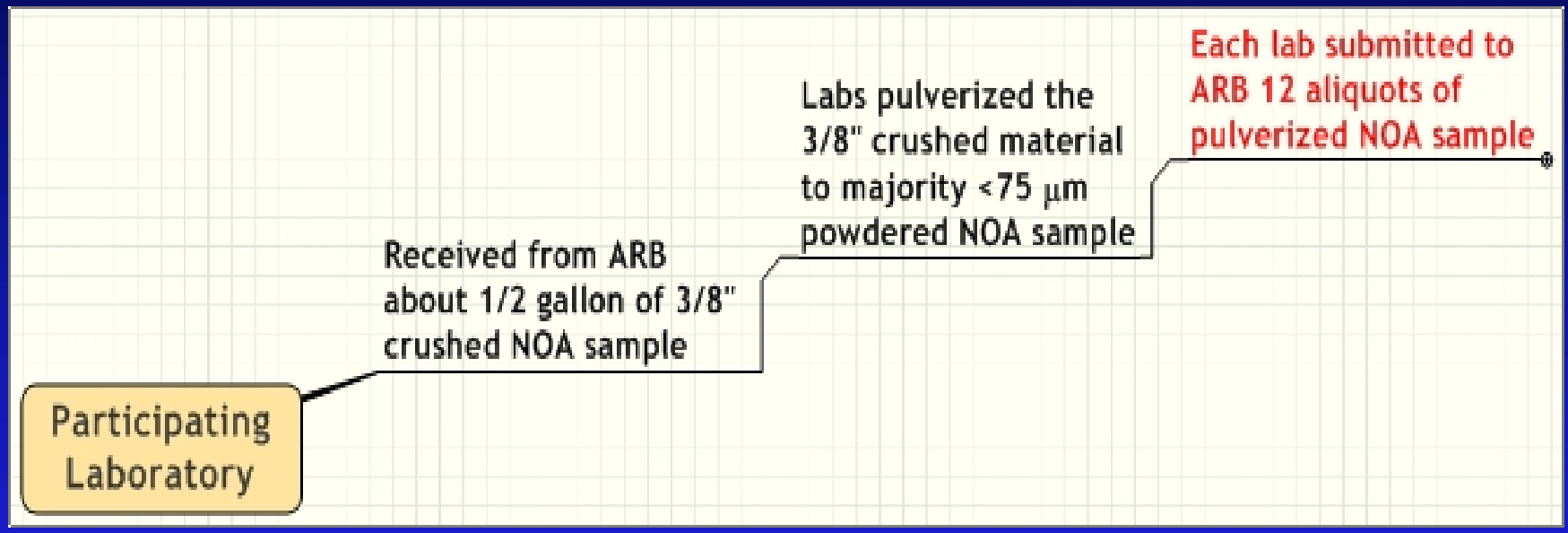
Plate Grinder (Braun Mill)

Ball Mill

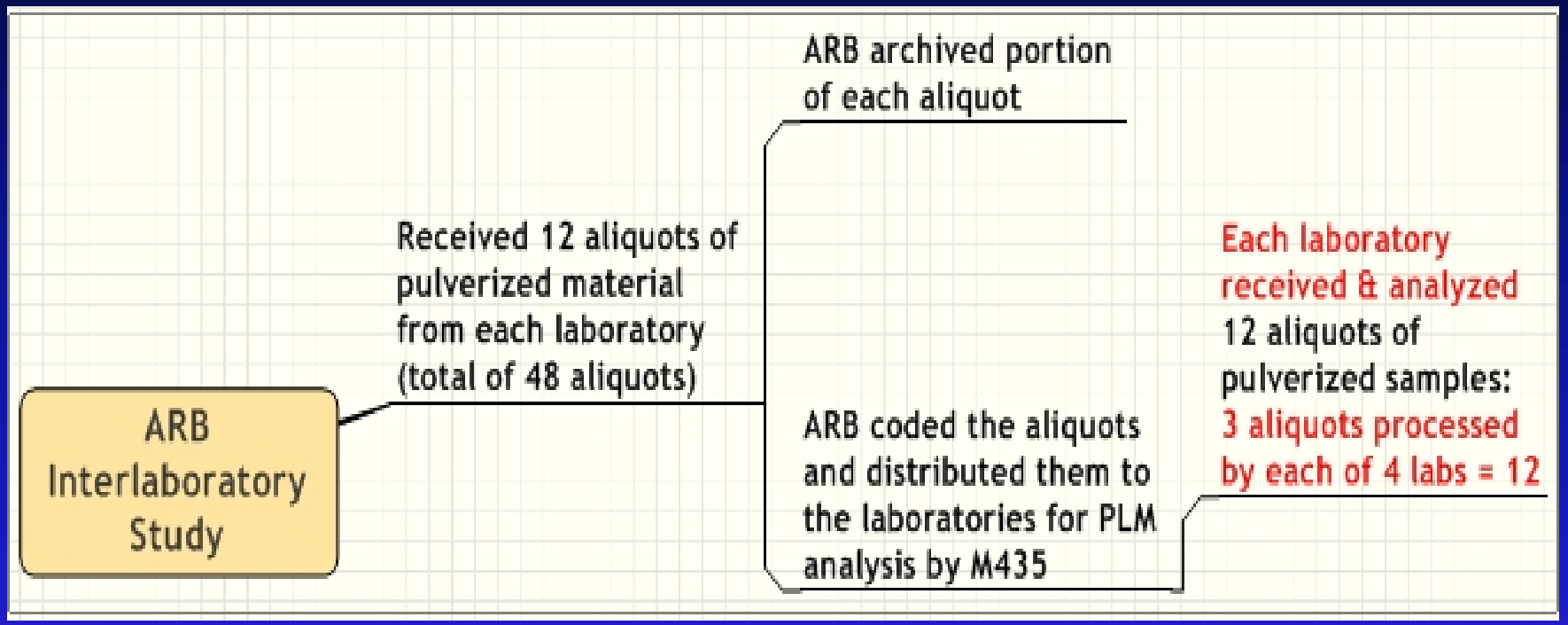
Freezer Mill

Vibrating Pulverizer
"shatter box"

Phase One: Pulverization of Crushed Material



Phase One: Analysis of Pulverized Material



Phase One: Study Design
Each Lab Pulverized 12 Samples

Prep by Lab A	xxx 1	xxx 2	xxx 3	xxx 4	xxx 5	xxx 6	xxx 7	xxx 8	xxx 9	xxx 10	xxx 11	xxx 12
Prep by Lab B	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx
Prep by Lab C	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx
Prep by Lab D	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx

Phase One: Study Design
Each Lab Analyzed 12 Samples

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab A	xxx 1	xxx 2	xxx 3	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx
Prep by Lab B	xxx 4	xxx 5	xxx 6	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx
Prep by Lab C	xxx 7	xxx 8	xxx 9	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx
Prep by Lab D	xxx 10	xxx 11	xxx 12	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx	xxx

Phase One: Coding of Results

To avoid attribution of the study results to any specific Participating Laboratory, the following tables and graphs use letter names to refer to the laboratories.

These letter names are for discussion references only, and have no continuity in the presentation.

Phase One: Analytical Results

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.0*	0.0*	0.0*	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.0*	0.0*	0.0*	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.0*	0.0*	0.25	0.0*	0.0*	0.0*	0.0*	0.0*	0.0*	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.0*	0.25	0.0*	0.75	0.50	1.00	0.00	0.00	0.00

Note: When no asbestos is detected ("0" or ND) the value "0.00" is used for this table.
When "<0.25%" or "trace" is reported, meaning the fibers observed were not under a point, "0.0*" is used for this table.

Phase One: Analytical Results

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.0*	0.0*	0.0*	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.0*	0.0*	0.0*	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.0*	0.0*	0.25	0.0*	0.0*	0.0*	0.0*	0.0*	0.0*	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.0*	0.25	0.0*	0.75	0.50	1.00	0.00	0.00	0.00

Note: When no asbestos is detected ("0" or ND) the value "0.00" is used for this table.
When "<0.25%" or "trace" is reported, meaning the fibers observed were not under a point, "0.0*" is used for this table.

- Labs A and C reported asbestos in the majority of aliquots;
- Labs B and D reported very little asbestos.

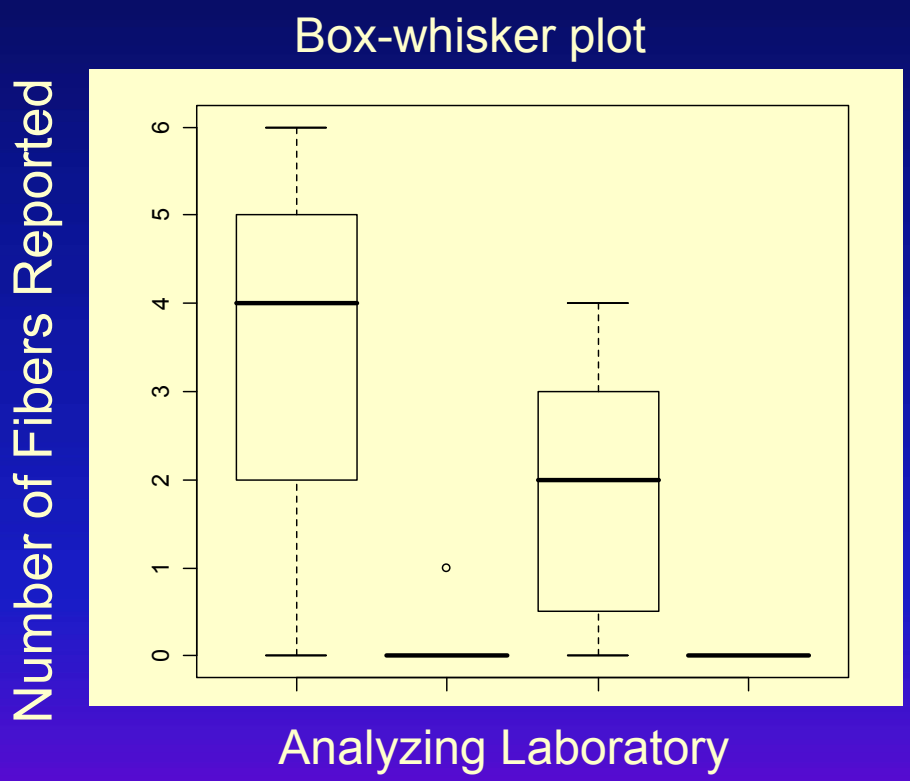
Phase One: Analytical Results

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.0*	0.0*	0.0*	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.0*	0.0*	0.0*	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.0*	0.0*	0.25	0.0*	0.0*	0.0*	0.0*	0.0*	0.0*	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.0*	0.25	0.0*	0.75	0.50	1.00	0.00	0.00	0.00

Note: When no asbestos is detected ("0" or ND) the value "0.00" is used for this table.
When "<0.25%" or "trace" is reported, meaning the fiber observed was not under a point, "0.0*" is used for this table.

- Lab D did not detect asbestos in 12 aliquots analyzed.

Phase One: Analytical Variability

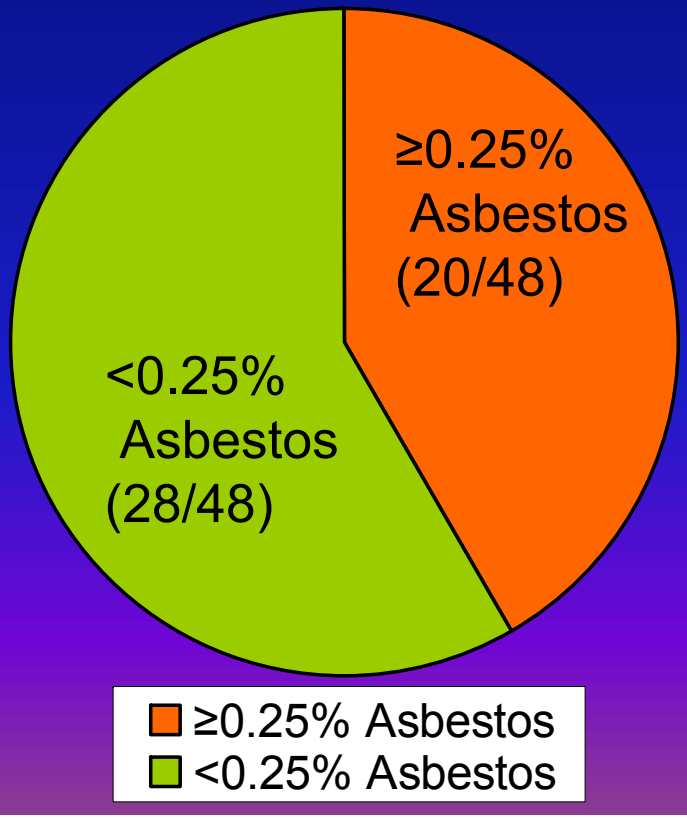


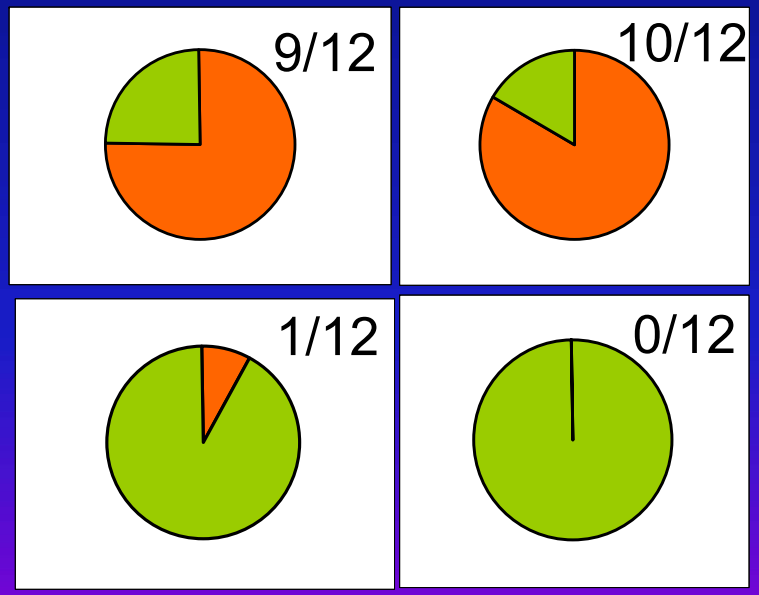
"Laboratory Analysis Effect"
Two laboratories reported statistically significantly different % asbestos content than two other laboratories.

-Boxes indicate 25th & 75th percentiles of the data.
-Thick line in the middle of box is the median.

Phase One: Analytical Imprecision

- These 48 powder aliquots are from one field sample;
- 42% of aliquot analyses (20/48) would trigger ATCM requirements.





Analytical Imprecision

Phase One: Preparation Effect

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.00	0.00	0.00	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.00	0.00	0.00	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.00	0.00	0.25	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.00	0.25	0.00	0.75	0.50	1.00	0.00	0.00	0.00

Note: When no asbestos is detected ("0" or ND) the value "0.00" is used for this table.
When "<0.25%" or "trace" is reported, meaning the fibers observed were not under a point, "0.0*" is used for this table.

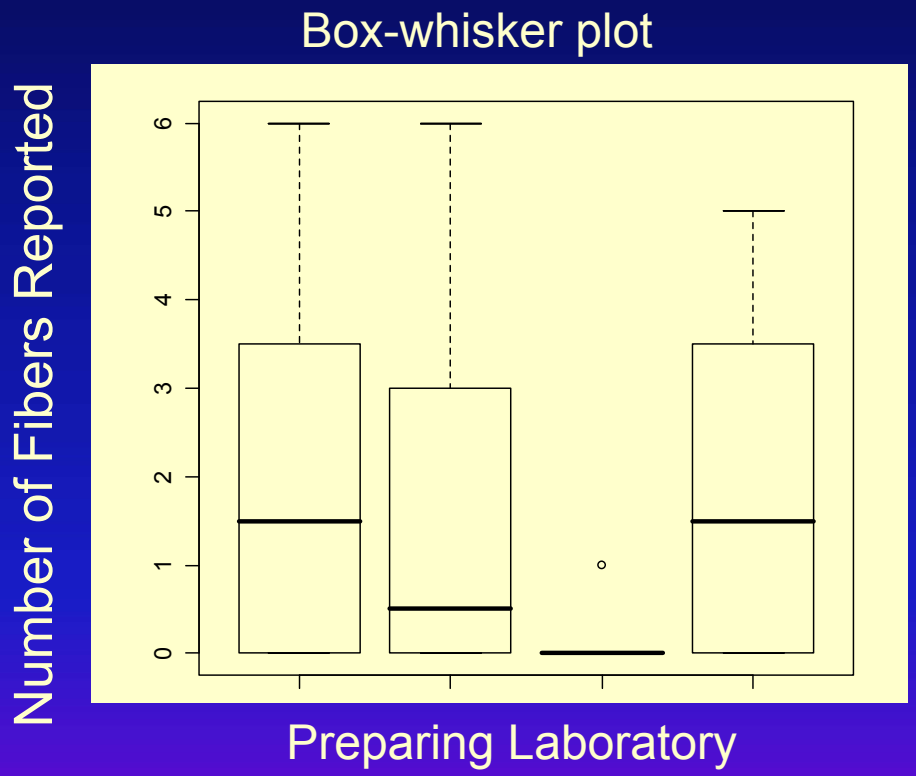
Phase One: Preparation Effect

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.0*	0.0*	0.0*	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.0*	0.0*	0.0*	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.0*	0.0*	0.25	0.0*	0.0*	0.0*	0.0*	0.0*	0.0*	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.0*	0.25	0.0*	0.75	0.50	1.00	0.00	0.00	0.00

Note: When no asbestos is detected ("0" or ND) the value "0.00" is used for this table.
When "<0.25%" or "trace" is reported, meaning fibers were observed but not under a point, "0.0*" is used for this table.

- Only one aliquot prepared by Lab G had reportable asbestos.

Phase One: Processing Variability



"Sample Prep Effect":
Asbestos samples prepared by one laboratory had statistically significantly less % asbestos content reported.

-Boxes indicate 25th & 75th percentiles of the data.
-Thick line in the middle of box is the median.

Phase One: Processing Variability

- Were there any noticeable differences among the pulverized aliquots in Phase One?
- If so, what were these differences?
- Did these differences affect the % asbestos reported?

Phase One: Evaluation of Pulverized Samples

Prepared by Lab I

Prepared by Lab K

Prepared by Lab J

Prepared by Lab L

Phase One: Processing Variability Quantitative Analysis

- Did laboratories produce pulverized aliquots with similar particle size distribution?

Phase One: Particle Size Distribution (3 aliquots from each laboratory)

- **Particle size analysis (PSA) by pipette**

  sand 50-2000µm

  silt 2-50µm

  <5µm, <10µm, <15µm, <20µm

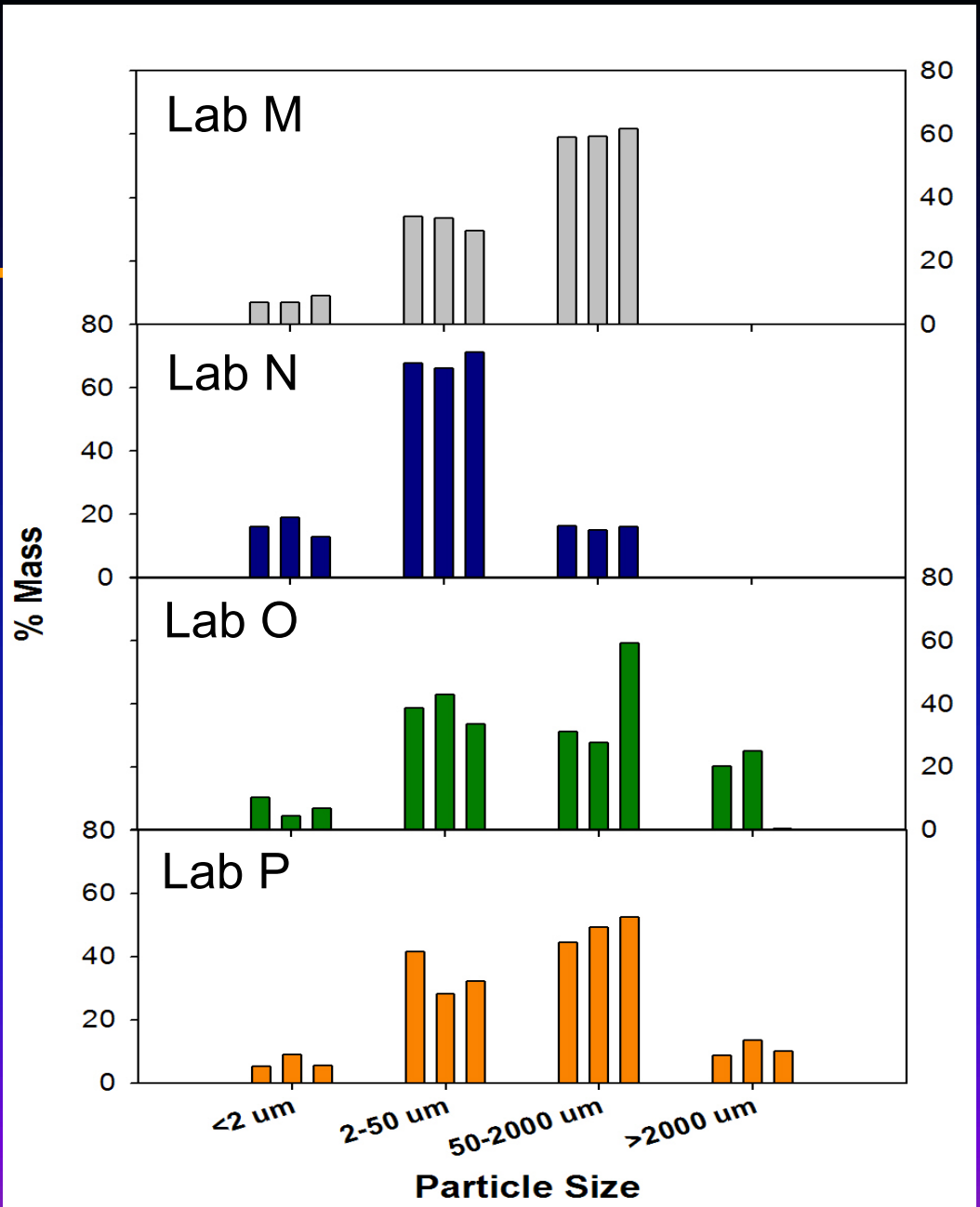
  clay <2µm

- **Dry sieving**

  50µm, 75µm (200 mesh), 100µm, 250µm, 500µm, 1000µm, 2000µm, >2000µm

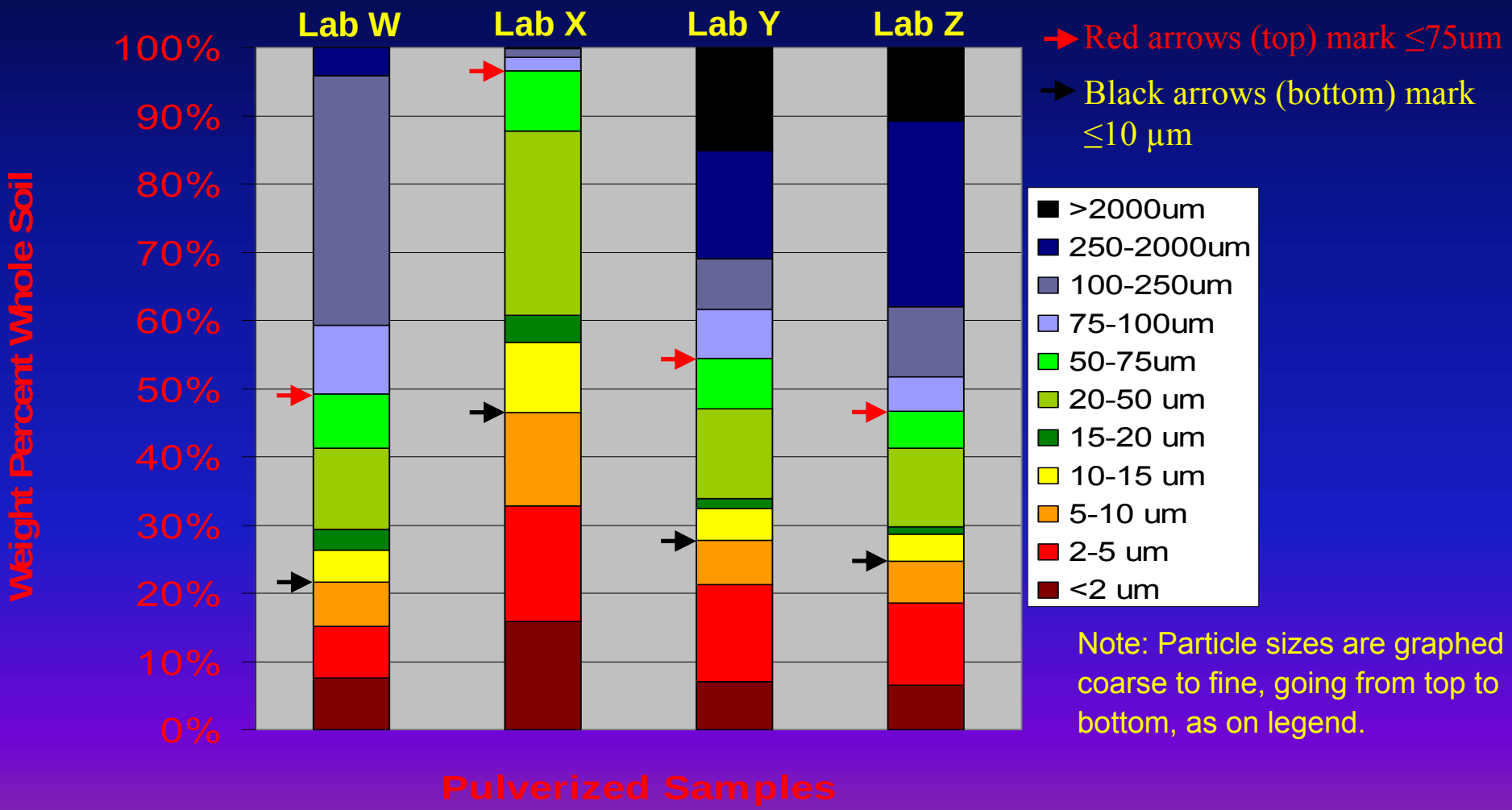
Phase One: PSA Variability

- Three aliquots from each laboratory;
- Particle size analyses (PSA) show differences among laboratories in % mass of size fractions;
- Labs O and P have particles >2000um.



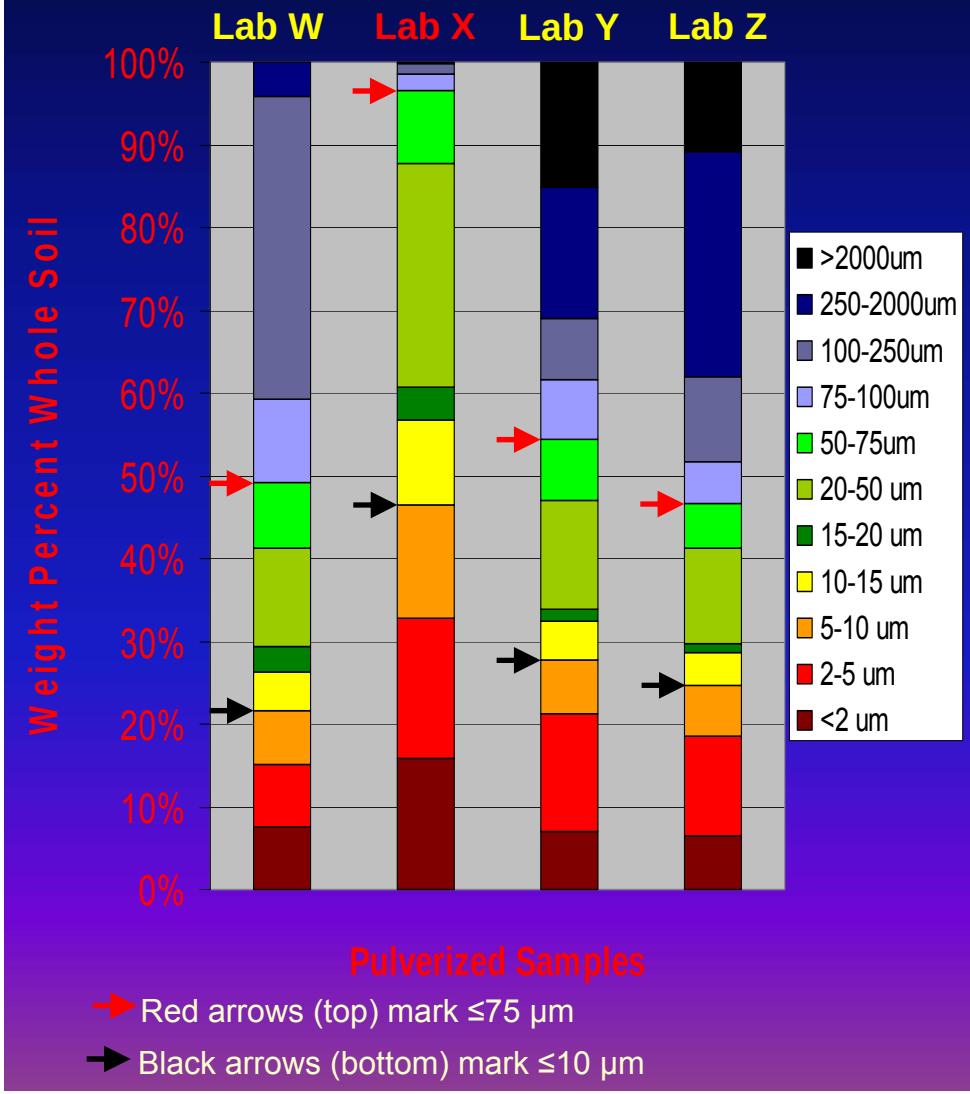
Phase One: Particle Size Distribution

(averages of 3 pulverized samples from each laboratory)



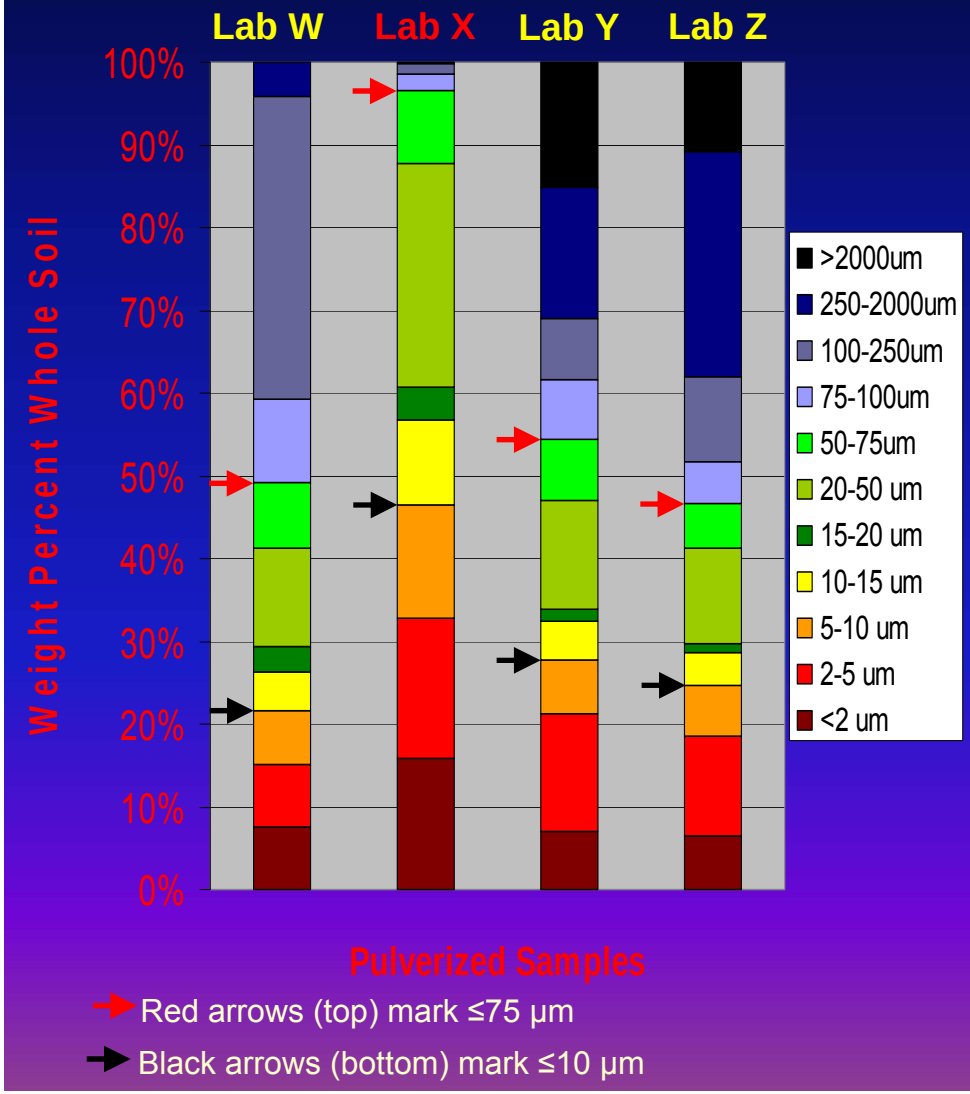
Note: Particle sizes are graphed coarse to fine, going from top to bottom, as on legend.

Phase One: Processing Variability



-Particle size distribution of samples prepared by Laboratory X were much finer.

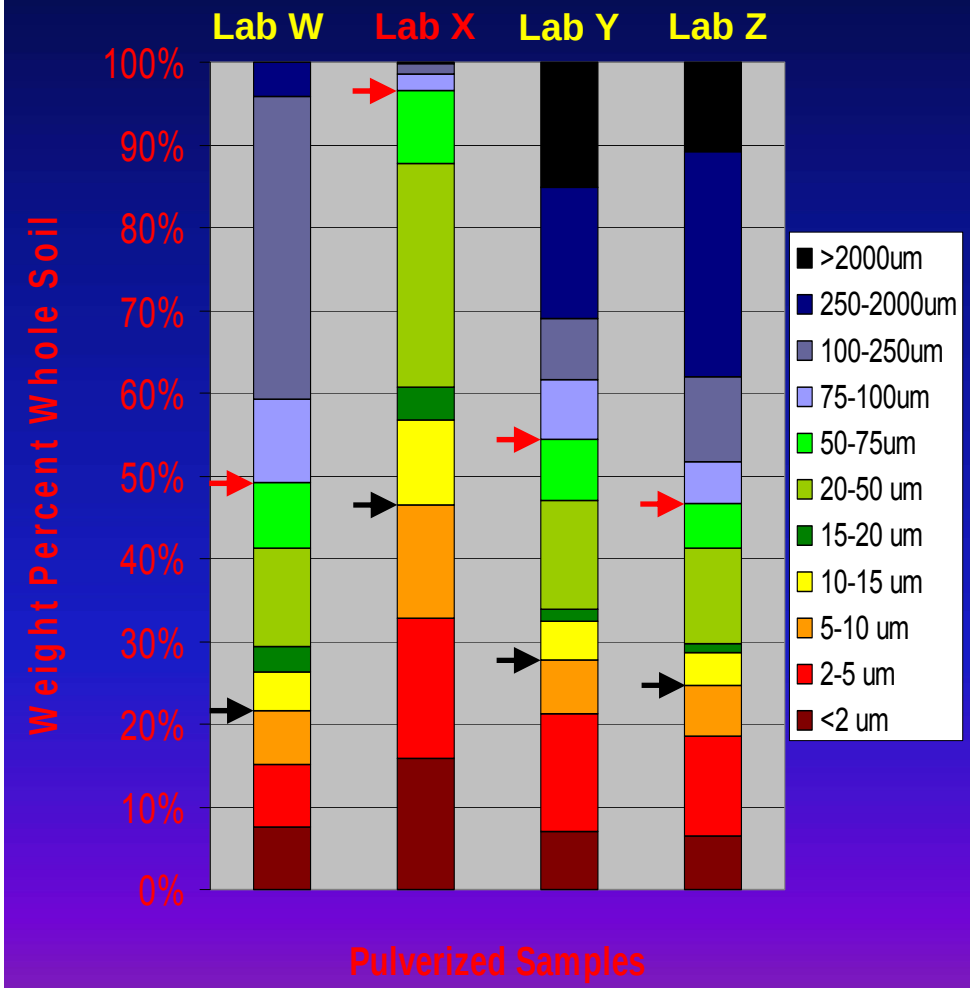
Phase One: Processing Variability



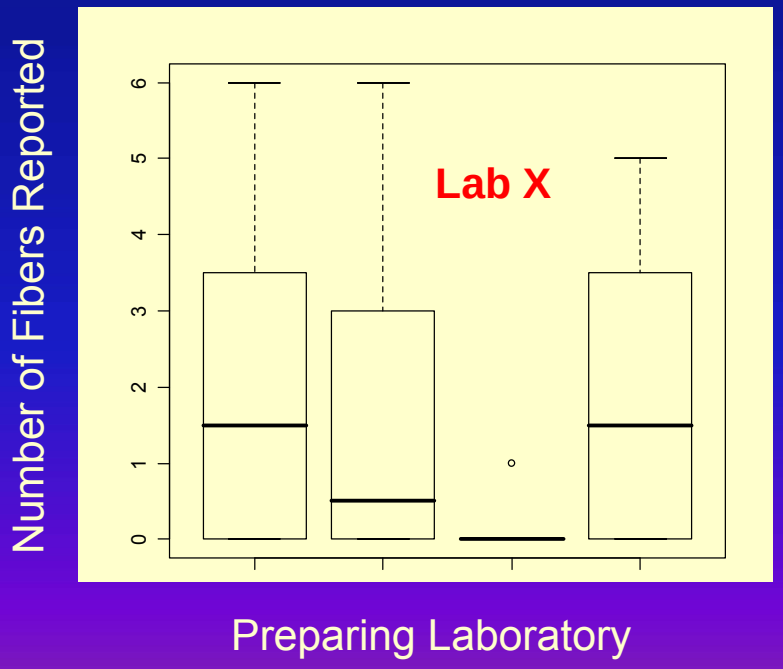
-Smaller particles may be difficult to visualize at 100x with PLM;

-Point counting at 100x magnification is required by M435.

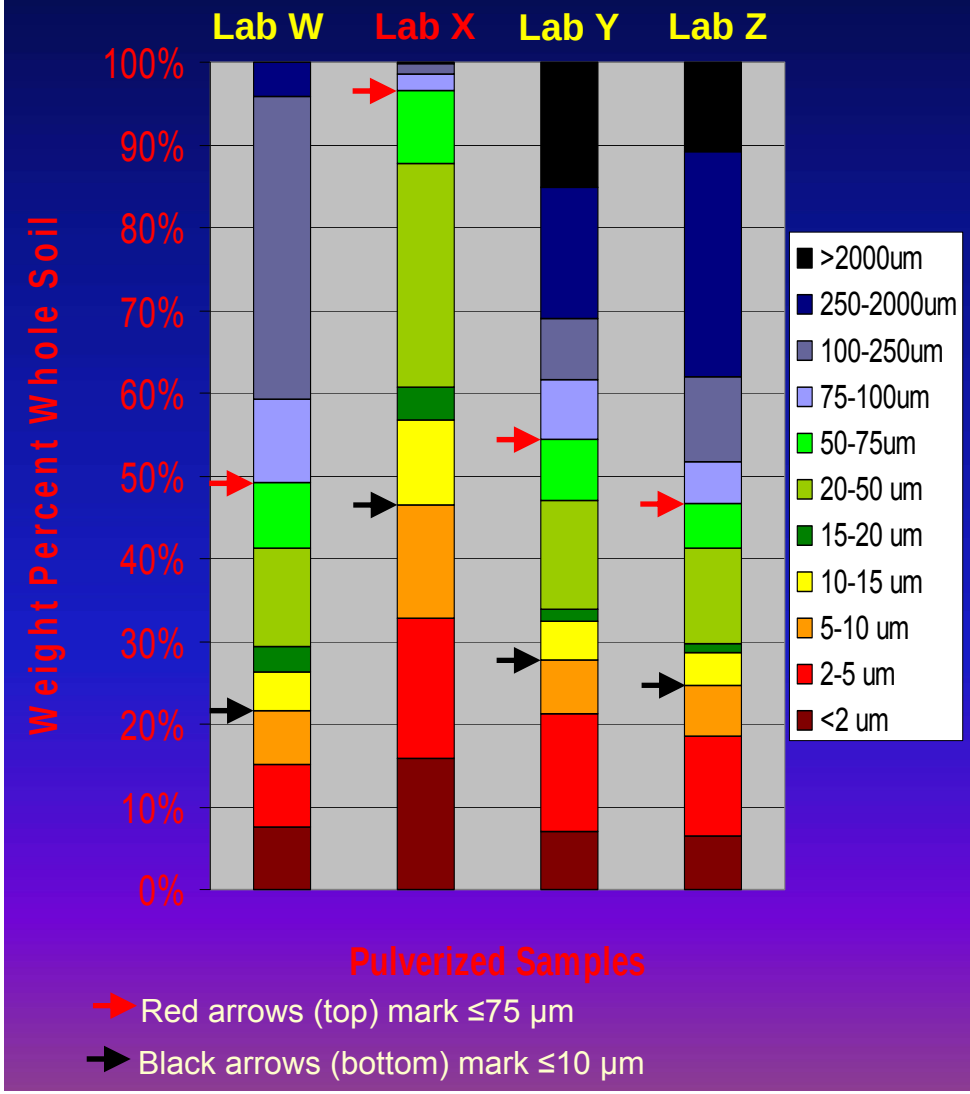
Phase One: Processing Variability



-Almost no asbestos was reported from aliquots prepared by Laboratory X;



Phase One: Processing Variability



-Asbestos, due to its needle-like shape, may be more susceptible to pulverization;

-Length to width ratio of asbestos fibers may be reduced to <3:1, and asbestos definition will not be satisfied.

Phase One: Processing Variability Quantitative Analysis

- Did laboratories produce pulverized aliquots with similar particle size distribution?

  No, different methods of pulverization produced different particle size distributions.

- Very fine particle size distribution appears to significantly decrease the % asbestos reported.

ILS Phase Two

Analysis of Fixed Mounted Slides

Phase Two Objectives

- To observe variability between laboratories in asbestos sample analysis, while minimizing sample processing effects;

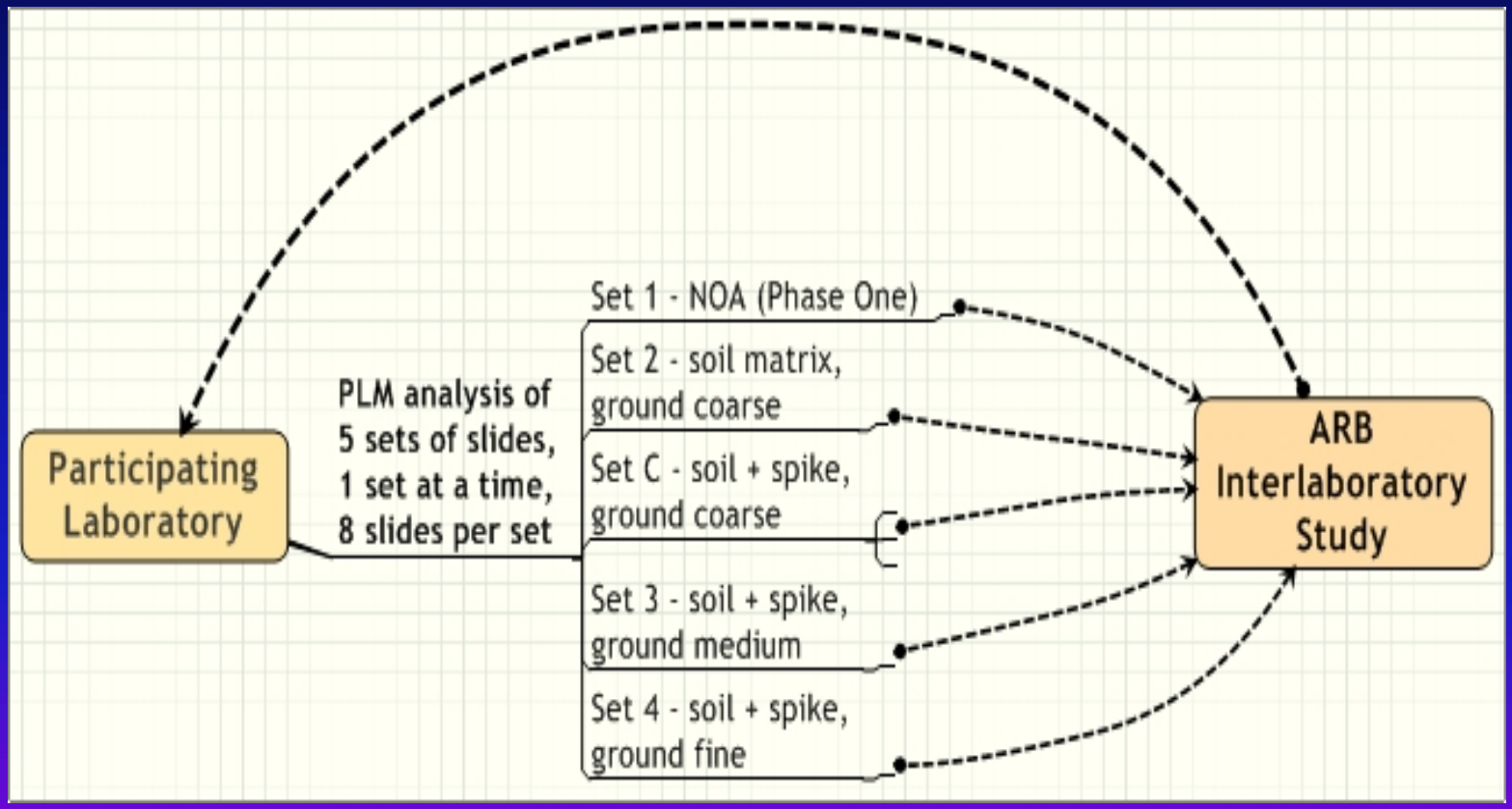
- To observe the effect of counting rules on number of asbestos fibers reported;

- To observe the effect of sample particle size distribution on number of asbestos fibers reported;

- To observe variability among laboratories quantifying naturally-occurring asbestos (NOA) versus NIST* standard reference asbestos.

***NIST – National Institute of Standards and Technology**

Phase Two: Round Robin Study

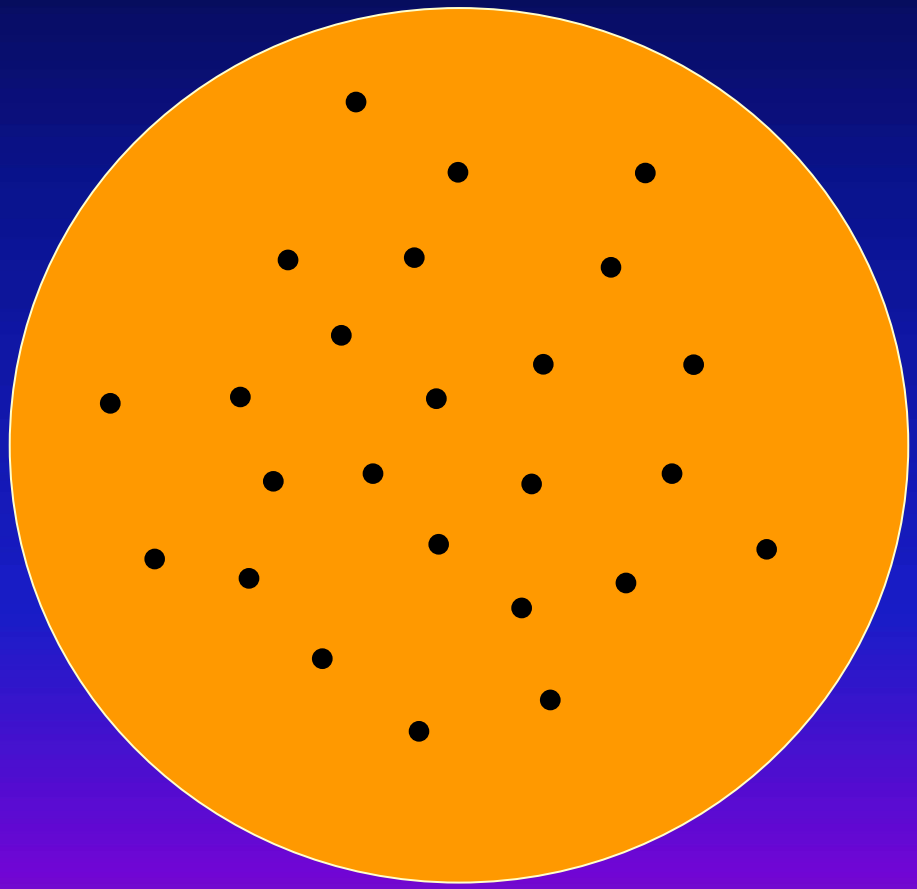


Phase Two: 5 Sets of Fixed Slides + Ground Samples

	Description	Objectives
Set 1	NOA sample, previously analyzed in Phase 1	NOA vs. NIST* reference asbestos
Set 2	Soil matrix, no spike, ground coarse	Assess asbestos content of soil matrix
Set C	Soil matrix + 0.5 wt% NIST* tremolite, ground coarse 400-point ct,1000-point ct, field-of-view ct	Compare asbestos content using 3 counting methods
Set 3	Soil matrix + 0.5 wt% NIST tremolite, ground medium	Assess effect of particle size distribution on asbestos count
Set 4	Soil matrix + 0.5 wt% NIST tremolite, ground fine	

* NIST – National Institute of Standards and Technology

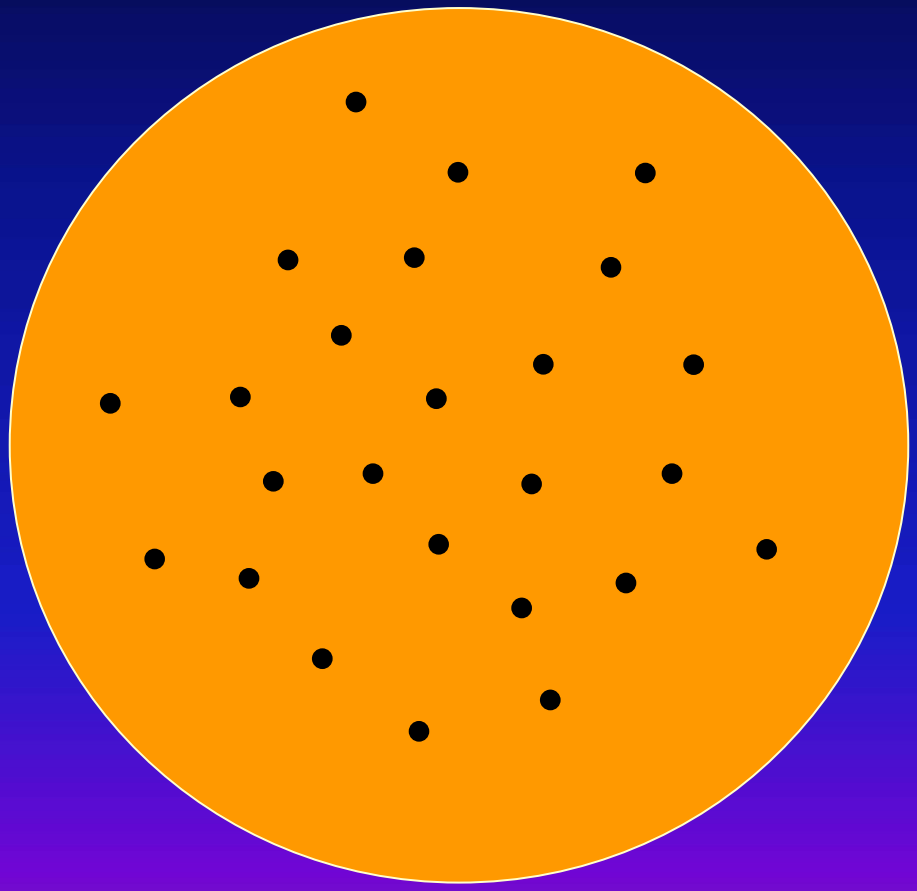
Phase Two: 400-point Count Method



(dot size exaggerated)

- 25-point Chalkley array was used;
- 50 non-empty points counted in each of 8 slides;
- 1/400 = 0.25% sensitivity;
- Only asbestos fibers under a point can be reported;
- “Trace” is reported when asbestos fibers are seen, but not under a point.

Phase Two: 1000-point Count, Field-of-View Count (FOVC)

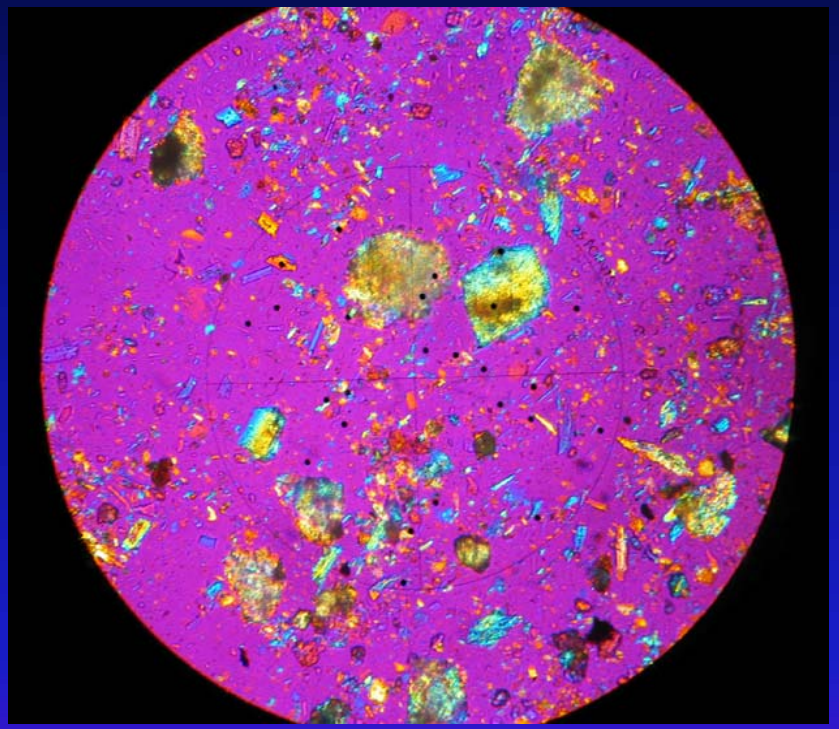


(dot size exaggerated)

- 25-point Chalkley array was used;
- 125 non-empty points counted in each of 8 slides;
- 1/1000 = 0.10% sensitivity;
- Only asbestos fibers under a point can be reported;
- FOVC counted all fibers visible in view while doing 1000-point count.

Phase Two: Set C
Effect of Counting Method

- Set C : ground coarse + 0.5 wt% NIST tremolite;
- 400-pt count: 50 pts/slide;
- 1000-pt count: 125 pts/slide;
- Field-of-view count: all fibers in view;
- How many fibers are detected but not counted?
- Increased sensitivity of results for better comparison between laboratories.

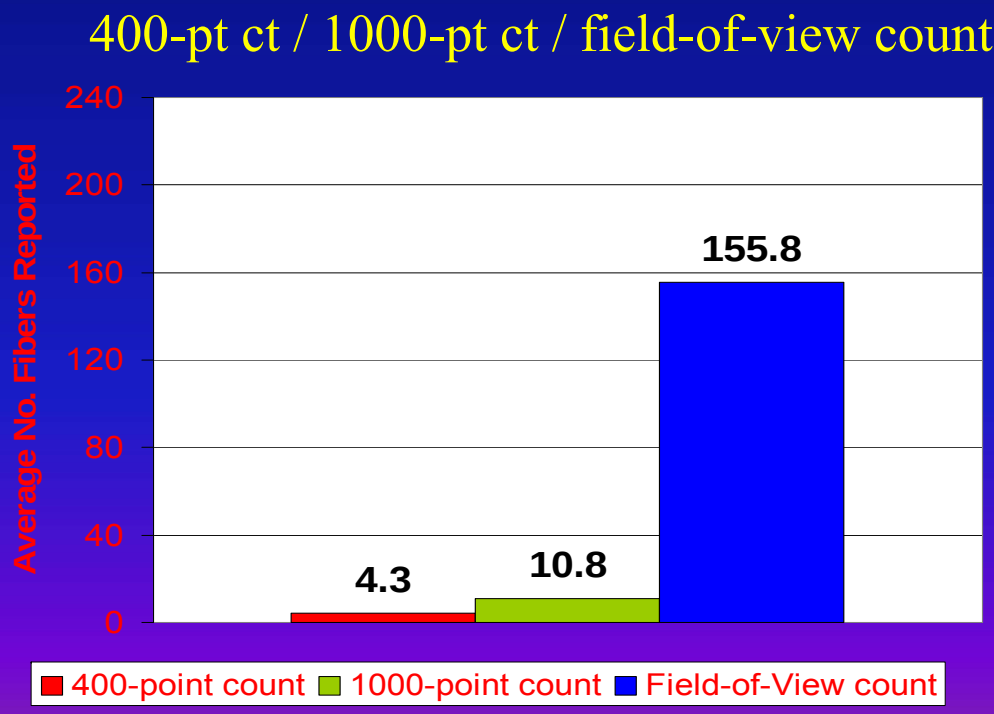


Phase Two: Effect of Counting Method Set C: 0.5 wt% NIST Tremolite, Coarse

		Lab W	Lab X	Lab Y	Lab Z	Ave. No. of Fibers
Set C 400-point count	# of fibers	6	0	4	7	4.3
Set C 1000-pt count	# of fibers	8	0	9	26	10.8
Set C Field-of-View count	# of fibers	53	0	205	365	155.8

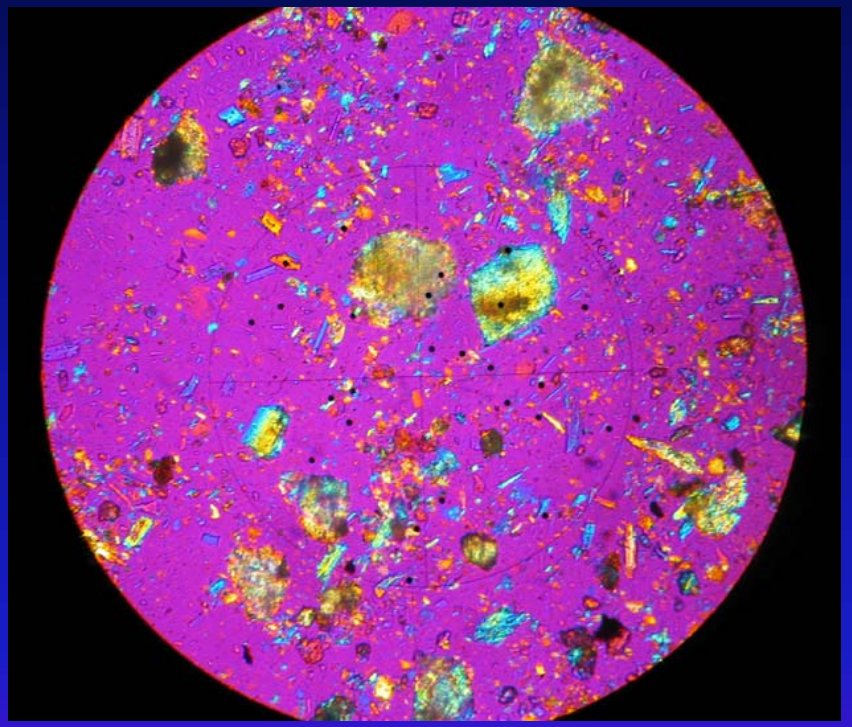
Phase Two: Effect of Counting Method on Average Number of Fibers Reported

- Many asbestos fibers observed in the field of view were not reported, due to the counting rules.

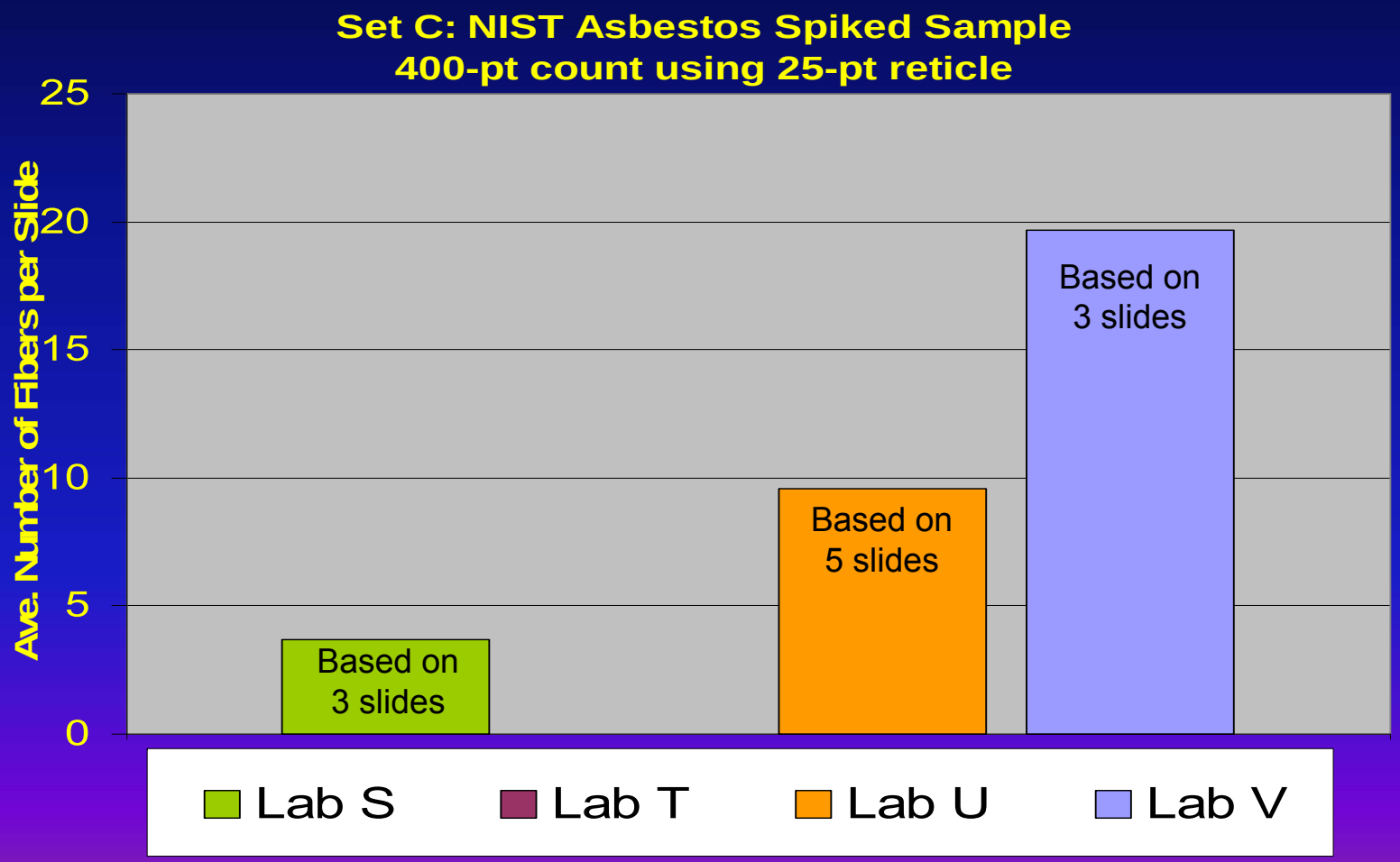


Phase Two: Set C
Effect of Counting Method

- Set C : ground coarse + 0.5 wt% NIST tremolite
- In a 400-point count: How many fibers were seen in the field of view when no fibers were reported on a slide?



Average Number of Fibers in FOV when No Fibers Fell on a Point



Phase Two: Effect of Particle Size

- Compared the number of asbestos fibers reported in a 400-point count analysis of:

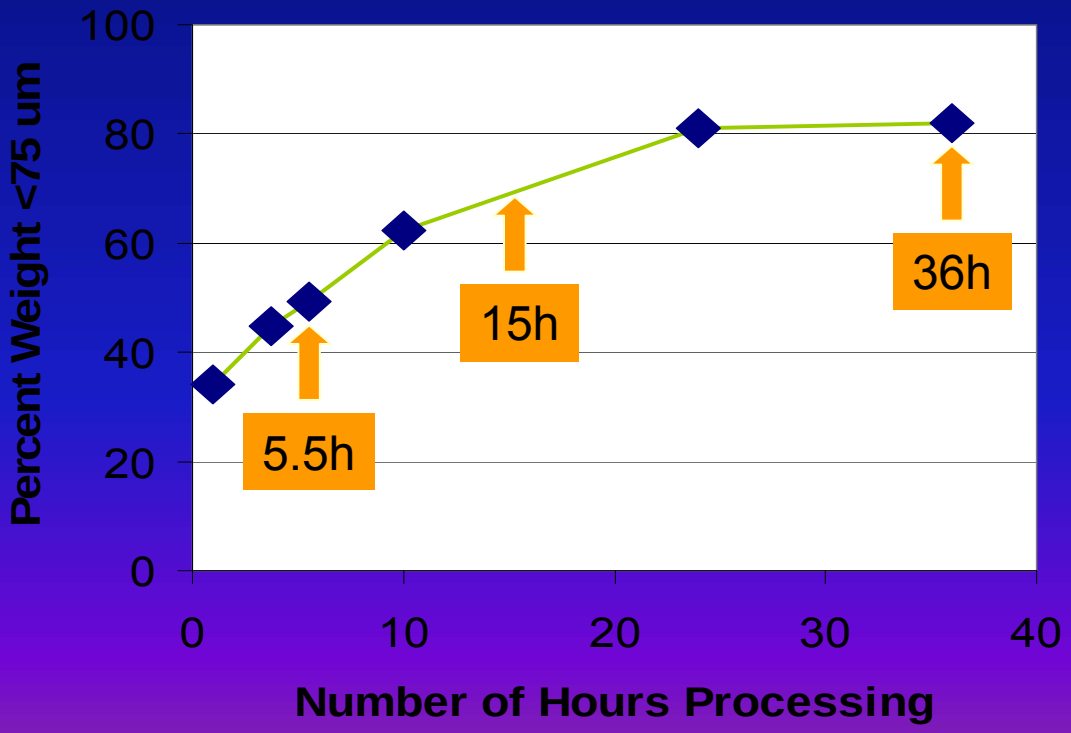
  Set C – ground coarse with 0.5 wt% NIST tremolite;

  Set 3 – ground medium with 0.5 wt% NIST tremolite;

  Set 4 – ground fine with 0.5 wt% NIST tremolite.

Phase Two: Evaluation of Grinding Time on <75 µm Fraction by Dry Sieve

- Grinding times of 5.5 hours, 15 hours, and 36 hours were used for sample preparation.



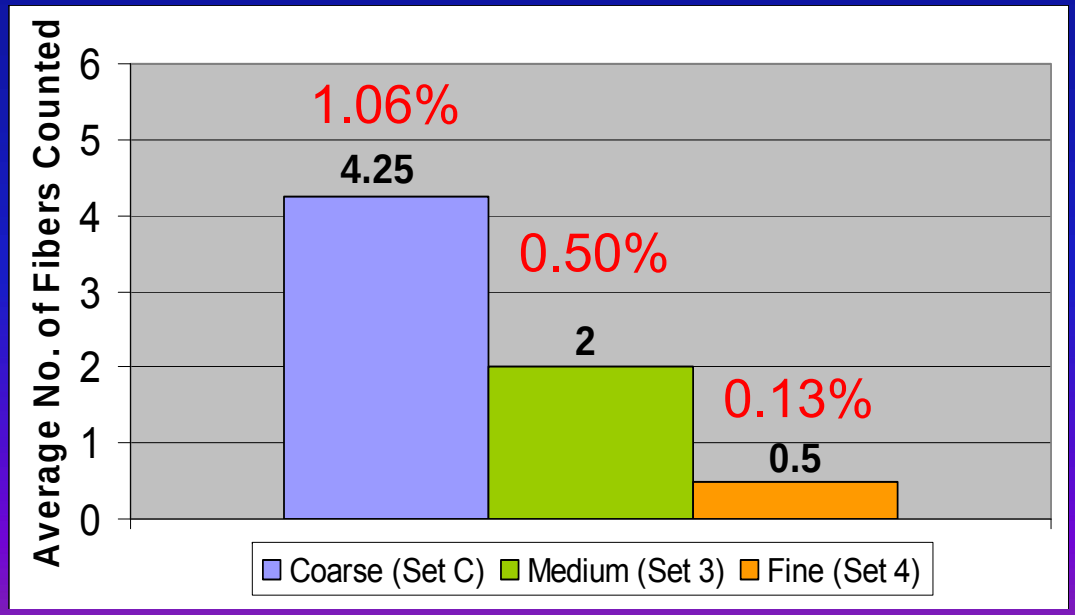
Phase Two: Effects of Particle Size Results and Interpretation

		Lab I	Lab J	Lab K	Lab L	Observations
Set C Coarse Spiked	# fibers	4	7	6	0	Asbestos fibers reported by three laboratories in coarse sample.
Set 3 Medium Spiked	# fibers	1	7	0*	0*	Asbestos fibers reported by two laboratories in medium sample.
Set 4 **Fine** Spiked	# fibers	0*	2	0	0	Asbestos fibers reported by one laboratory in fine sample.
		0* - indicates “trace” or “<0.25%” was reported (asbestos seen but not under point).				

Phase Two: Effects of Particle Size Average Number of Fibers Counted

- Average number of asbestos fibers reported from spiked samples decreased with decreasing sample particle size. Percent asbestos in red.

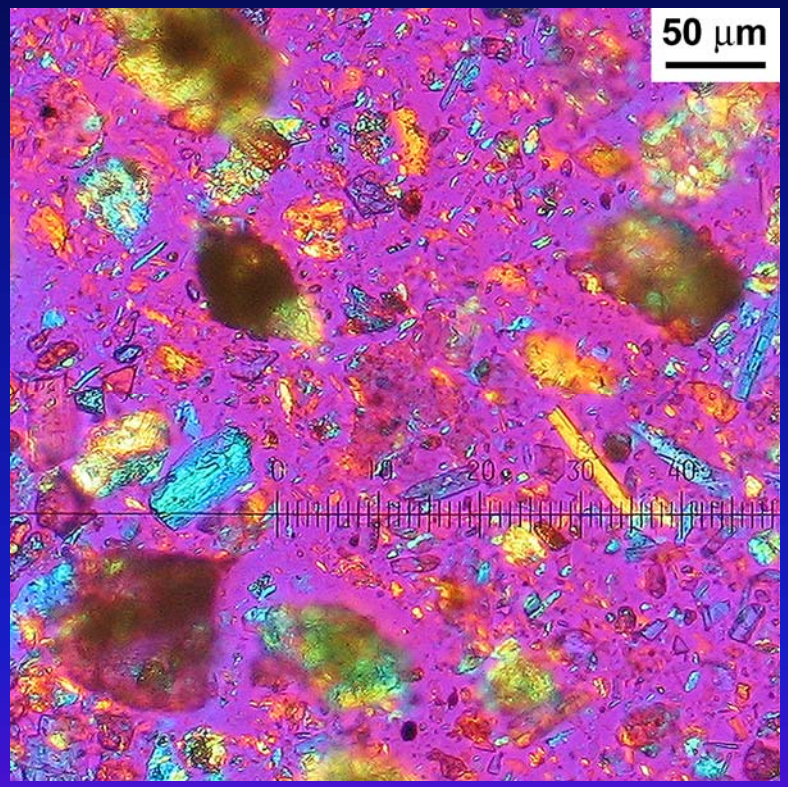
•ATCM requirements not applicable for fine sample (orange).



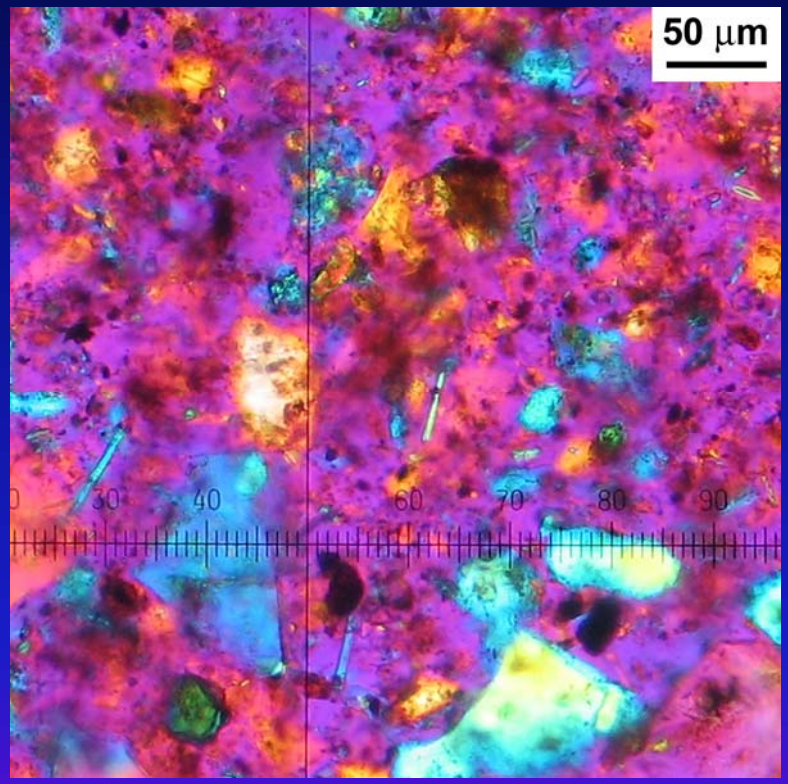
Phase Two:
NOA and NIST Asbestos

- Set 1 – NOA sample from Phase One;
- Set C, Set 3, Set 4 – spiked with NIST tremolite asbestos;
- 400-point count analysis.

Phase Two: NOA and NIST



Set 1 - NOA



Set C - NIST Asbestos

Phase Two: NOA vs. NIST Number of Fibers in 400-pt Count

	Lab M	Lab N	Lab O	Lab P
Set 1 NOA	0	0*	35	0*
Set 2 Soil Matrix	0	0	5	0
Set C – 0.5 wt% spike NIST tremolite	0	4	7	6

0* - indicates fibers were seen but not under a point

- NOA reported by one laboratory;
  - -Another lab reported NOA in Phase One from the same set of 12 aliquots;
- NIST asbestos reported by three laboratories.

Phase One & Phase Two: 400-point Count, Fiber Totals

		Lab Q	Lab R	Lab S	Lab T
Phase One 400-pt count NOA	Sum of all fibers, 12 aliquots	1	41	24	0
Phase Two 400-pt count NIST tremolite + NOA	Sum of all fibers, Sets C, 3, 4 + Set 1, 2	6	61	5	0
	Totals	**7**	**102**	**29**	**0**

Some Conclusions from ILS

- Laboratories use different processing equipment and protocols;
  - Result in varying particle size distribution of samples;
- Finer particle size distribution is one factor resulting in lower % asbestos reported;
  - Observed in Phase One:
    - Preparation Effect
  - Observed in Phase Two:
    - Set C (coarse) vs. Set 4 (fine)

Some Conclusions from ILS

- Sampling using 400-point count analysis lowers reportable number of asbestos fibers by 1 to 2 orders of magnitude when compared to the field-of-view count;

Some Conclusions from ILS

- Fiber identification criteria are not uniform among laboratories;
  - Phase One: Laboratory Analysis Effect
  - Phase Two: Laboratories reported a wide range of number of asbestos fibers
  - One laboratory did not detect NOA nor report NIST standard reference asbestos.

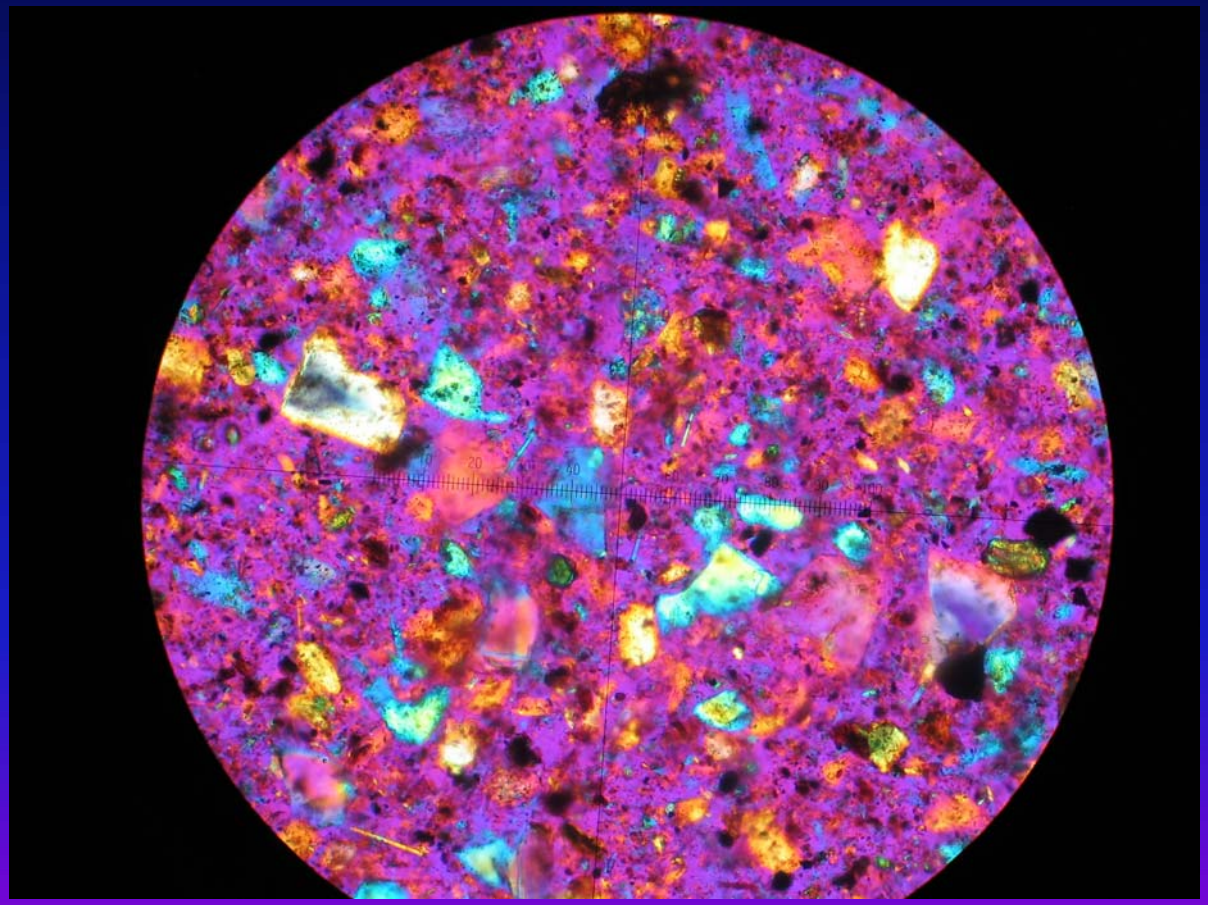
ARB Future Activities

- To work with stakeholders in identifying variables that can reduce laboratory processing and analytical variability when applying Test Method 435.
- To revise Test Method 435 accordingly.

Acknowledgment

- Asbestos TEM Laboratories, Inc.
- EMSL Analytical, Inc.
- Forensic Analytical Laboratories, Inc.
- R J Lee Group, Inc.
- NOA Laboratory Working Group, SAGE of El Dorado County
- Southard Laboratory, Dept. of LAWR, University of California - Davis

End



Workshop Agenda

- Introduction
- Interlaboratory Study (ILS) Presentation
  - Questions & Answers
- Potential Revisions to Test Method 435 (M435)
  - Processing Procedures
  - Analytical Procedures
  - Laboratory Accreditation
- Revision Schedule/Next Workshop

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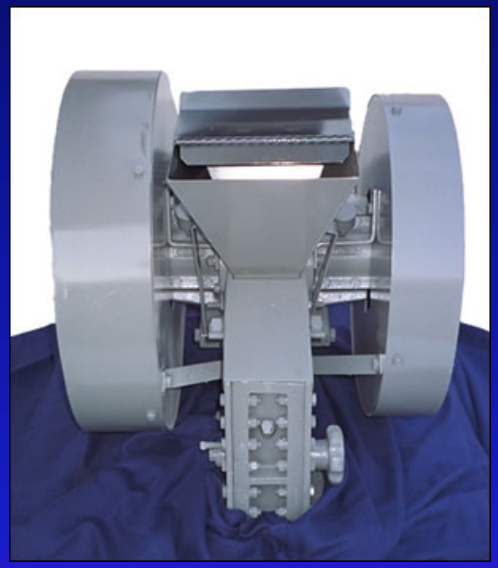
Potential Revisions to Processing of Samples

Crushing Equipment



Bico Braun Crusher

or

Hammer

or ?

- ENTIRE composite sample needs to be reduced to <3/8” & riffle split

Potential Revisions to Processing of Samples

Pulverization Equipment

Braun Mill (plate grinder)

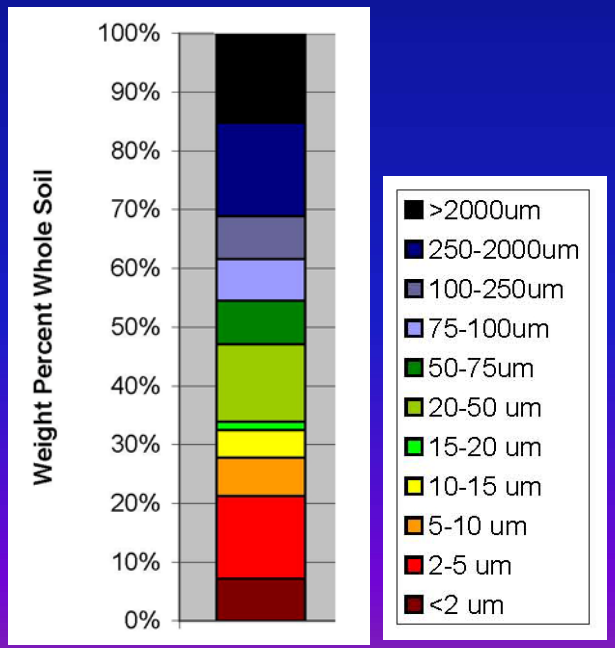
- Need to be able to calibrate to desired particle size distribution
- No large chunks leftover

Potential Revisions to Processing of Samples

Particle size distribution of pulverized samples

- Majority less than 200 Tyler mesh (75 µm) material (M435)
  - Not specific enough
  - Ideal particle size distribution?
- Continue to calibrate at 75 µm?

Sieves



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Potential Revisions to Analytical Procedures

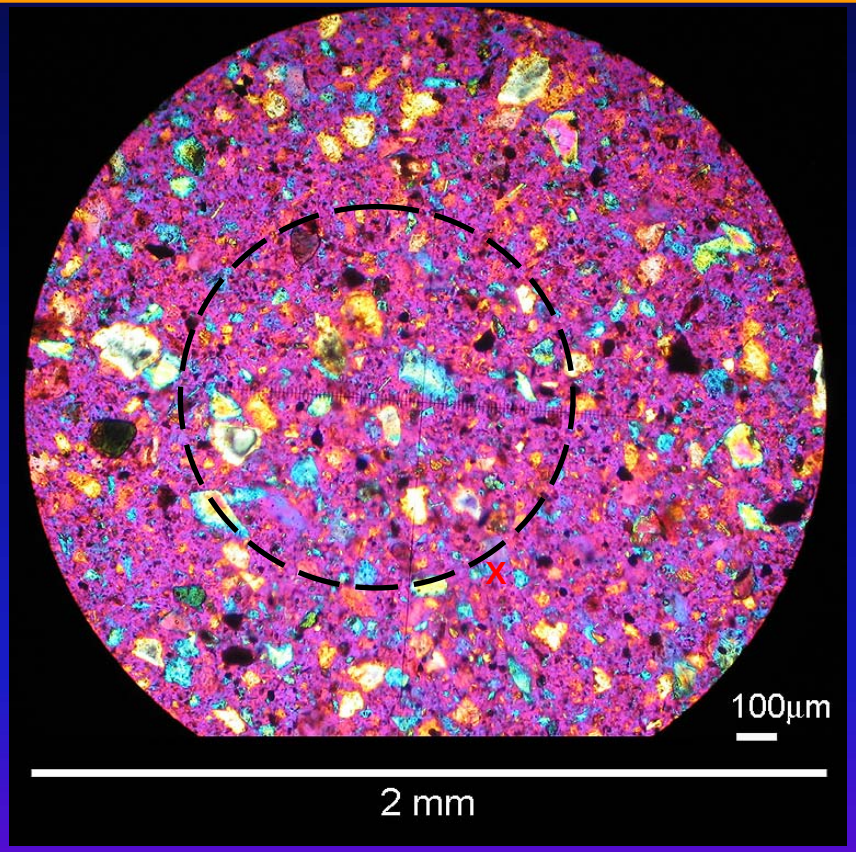
Magnification

- Specify magnification during identification
  - M435 currently silent
- Increase magnification during point count
  - morphology more easily determined
  - reticle dot becomes "smaller"

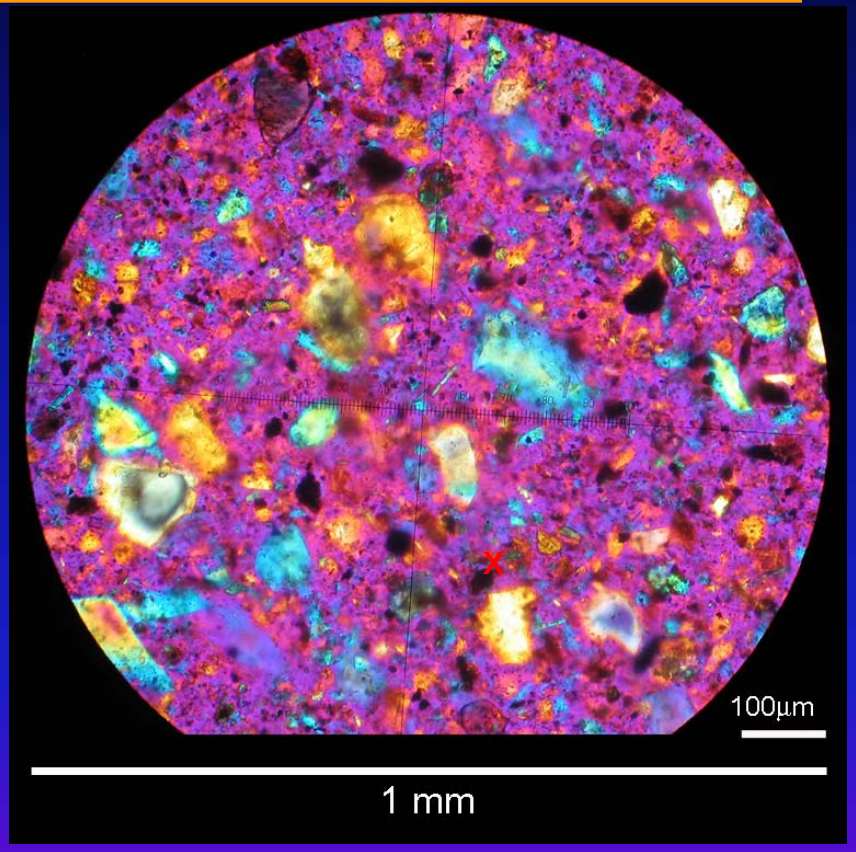
Increase in Point Count

- Would lower detection limit below ATCM's "trigger" level
- Would keep spatial representativeness intact with increase in magnification

Potential Revisions to Analytical Procedures



100x

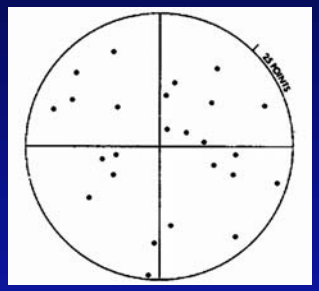


200x

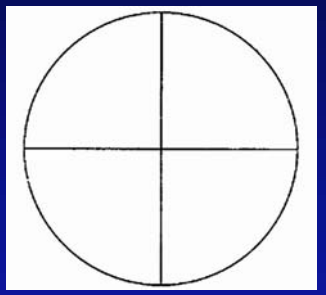
- Submission of photomicrographs with lab reports?

Potential Revisions to Analytical Procedures

- Reticles
  - Unbiased sampling?

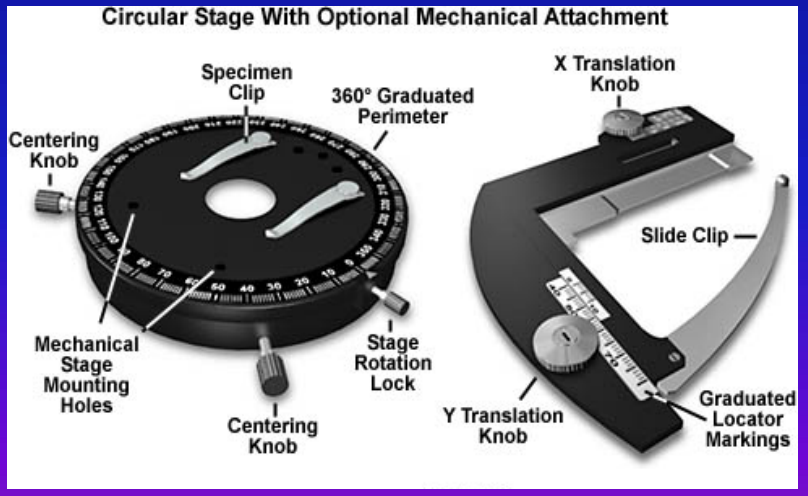


25-pt Chalkley array,
100-pt Chalkley array



Cross-hair

- Mechanical Stage
  - Predetermined movement of cross-hair reticle



Potential Revisions to Analytical Procedures

Fiber Identification / Definition

- Labs have different interpretation of what should be identified as asbestos
- M435 criteria need to be more explicit
- ARB will work with OEHHA to ensure revised definition reflects health information

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Laboratory Accreditation Promotes

- Measurement accuracy
- Accepted quality control and good laboratory practices
- Quality assessment through proficiency testing
- Corrective action for nonconformities

➢ No current accreditation requirement to perform M435 analysis

Need for M435 Accreditation

- ILS showed significant variability in M435 results among labs accredited for PLM analysis of bulk asbestos products;
- Current accreditation programs are not specific to naturally-occurring asbestos (NOA) samples
  - NVLAP/AIHA accreditation for PLM use an EPA method for the analysis of asbestos in building materials (EPA600/R-93/116);

Laboratory Accreditation Organizations

- NVLAP - National Voluntary Laboratory Accreditation Program
- NELAP/ELAP - National Environmental Laboratory Accreditation Program / California ELAP within the California Department of Public Health
- AIHA - American Industrial Hygiene Association

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M435 Revision Schedule

- January 24, 2008, Workshop (1st)
  - Rationale & identification of areas of M435 currently being examined for revision
- May/June 2008 Workshop(s)
  - More focused proposed revisions to M435
- August/September Workshop(s)
  - Proposed draft language available for comment
- February 2009 Board Hearing

Workshop Two

- Time frame: May/June 2008;
- Possible venues?

California Environmental Protection Agency

Air Resources Board

Potential Changes to Method 435

January 24, 2008, Workshop

Thank you for your participation.
For questions and comments, please contact:

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