

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

LAB ID:	LABORATORY NAME:
DATE:	ASSESSOR NAME:

BULK ASBESTOS AND SURFACING MATERIAL CONTAINING VERMICULITE SAMPLES BY POLARIZED-LIGHT MICROSCOPY

Method Number:
 SOP Number:
 Revision Number:
 SOP Date:

Personnel records observed (including seasonal if applicable):

Data records observed:

Method Number:
 SOP Number:
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NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
BULK ASBESTOS AND SURFACING MATERIAL CONTAINING VERMICULITE SAMPLES BY POLARIZED-LIGHT MICROSCOPY						
I. Analytical Method (G1004)						
Item 198.1 or EPA 600/M4/82/020 is for Friable Bulk Samples, and Item 198.6 is for Non-Friable Organically Bound Bulk Samples. NYS does not allow visual estimation (EPA 600/R93/116). Item 198.8 is for Surface material containing Vermiculite Bulk Samples.						
A. Necessary charts and tables are available to analyst (e.g., McCrone 1989 or Su 1994 or 2009 dispersion staining table)?						G1012
B. The lab maintains a list of non-asbestos fibers that can be confused with asbestos. (Note: This could be a poster, SOP, etc.)						G1245
a. The list includes optical properties that disqualify each fiber being identified as asbestos.						G1246
C. The lab has a textbook or reference book on mineralogy or crystallography (e.g., McCrone 1980; McCrone 1988; Deer, Howie, and Zussman 1996, Shelly 1975).						G1247
II. Polarized-Light Microscope (G1016) (Section 3 of Items 198.1 and 198.6 and Section 1.5 and 1.6 of EPA 600/M4/82/020)						
A. The PLM is equipped with the following:						
a. substage polarizer.						G1020
b. analyzer oriented perpendicular to substage polarizer.						G1024
c. eyepiece with a fixed crosshair aligned in direction of polarizer.						G1028
d. 550 nm (first-order red) retardation/compensator plate at 45° to the polarizer.						G1032
e. graduated rotating stage (360° in 1° increments).						G1036
f. focusable condenser with centerable iris diaphragm.						G1040
g. low (3.2-10X) and high, dry (30-50X) magnification objective. (Sec. 3.19.7 of Item 198.1) (Sec. 1.5.1 of EPA 600/M4/828/020 states "Objective lenses: 10X, 20X, and 40X or near equivalent.)						G1044
h. eyepiece of ≥ 8X magnification.						G1048
i. Chalkley point-count reticle? (optional)						G1049
III. Equipment and Supplies (G1052) (Section 3 of Items 198.1, 198.6, and 198.8 and Section 1.5 and 1.6 of EPA 600/M4/82/020)						
A. The lab has the following equipment/material:						
a. laminar-flow hood or negative pressure glove box with HEPA filtration.						G1056
b. low-power (10-45X) stereobinocular microscope with external source for gross examination.						G1060
c. forceps, dissecting needles, probes, scalpel or razor blades for manipulating bulk samples.						G1064
d. smooth removable substrates (glassine paper or clean glass plate) as surfaces for manipulating bulk samples.						G1068
e. homogenization equipment that includes:						
1. mortar and pestle.						G1080
2. mini-blender (approximately 30-mL capacity), liquid-nitrogen mill, or Wiley mill.						G1084
g. filtration apparatus for polycarbonate filters.						
1. 0.4-µm-pore polycarbonate filters.						G1428
2. petri dishes (50 mm diameter) and lids.						G1429

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
h. muffle furnace capable of sustained operation at 500°C.						G1096
1. crucibles (bottom and lid).						G1436
2. one of the following instruments or materials capable of calibrating a muffle furnace at 480°C: a.) high-temperature thermometer with range to at least 500°C and with subdivisions of 5°C or less, b.) melting-point solids with capability of differentiating 5°C differences between 400°C and 500°C, or c.) potentiometer capable of differentiating 5°C differences between 400°C and 500°C.						G1095
i. desiccator						G1443
j. analytical balance with sensitivity of 0.0001g.						G1445
k. concentrated hydrochloric acid (reagent grade).						G1444
l. reagent-grade dilute acetic or hydrochloric acid. (For Bulk , EPA 600/M4/82/020, Section 1.5.1)						G1104
m. surfactant such as sodium metaphosphate or aerosol OT.						G1108
n. heat lamp, slide warmer, or drying oven.						G1109
o. ultrasonic bath.						G1448
p. filtered (0.1-µm) distilled water or deionized water.						G1452
q. calibrated thermometer with range of 0 to 50°C and readability of ±1°C?						G1098
r. microscope slides (75 mm X 25 mm).						G1072
s. (whole) cover glasses (22 mm X 22 mm).						G1076
t. marker for labeling slides.						G1077
B. The lab has the following reference materials:						
a. NIST SRM 1866a (Common Commercial Asbestos – chrysotile, amosite (grunerite), crocidolite (riebeckite), and synthetic glass fiber).						G1228
b. NIST SRM 1867 (Uncommon Commercial Asbestos – anthophyllite, tremolite, and actinolite).						G1244
c. at least 10 different Vinyl Asbestos Tiles (VAT) references that have been analyzed by an ELAP-certified TEM lab.						G1456
1. at least 2 of the verified standards are negative for asbestos.						G1464
2. at least 2 of the verified standards have asbestos concentrations between 1 and 10%.						G1468
3. at least 2 of the verified standards have asbestos concentrations greater than 10%.						G1472
d. permanent mount of NIST amosite in refractive index oil with $n_d = 1.680$.						G1223
e. a complete set of RI oils ranging from $n_d = 1.49$ to 1.72 in intervals ≤ 0.005 .						G1224
f. either a solid RI calibration material (e.g., Cargille glass) or a refractometer capable of an accuracy of ± 0.004 .						G1252
Additional equipment and supplies for Surfacing Material Containing Vermiculite Bulk Sample Analysis: (G1600) (Section 3 of Item 198.8)						
C. The lab has the following equipment/material and reference materials:						
a. centrifuge, capable of 3600 rpm and accommodating at least four 15 mL centrifuge tubes.						G1088
b. glass or polypropylene, centrifuge tubes, 15 mL capacity.						G1601
c. at least 4 different SM-V specimens analyzed by outside lab.						G1602

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
d. at least 2 negative (non-ACM) standards.						G1603
e. at least 2 positive (ACM) standards.						G1604
f. magnetic stirrer (Teflon coated, 5 cm in length) and magnet.						G1605
g. Sink-Float® Standard (2.75±0.005 g/cc at 23°C).						G1606
h. water aspirator.						G1607
i. Erlenmeyer flasks (conical, 250 mL).						G1608
j. 25 mm and 47 mm diameter glass vacuum filtration assembly.						G1609
k. polycarbonate filters (0.4-0.8 um, 47 mm diameter). Note: 25 mm is allowed when there is a small amount of centrifugate.						G1610
l. porcelain or glass Buchner funnel (240 mL).						G1611
m. Whatman 40 cellulose filters (90 mm diameter).						G1612
n. 25 mm and 47 mm diameter mixed esters of cellulose filters (0.22 um porosity).						G1613
o. heavy liquid (either an aqueous solution of lithium metatungstate or sodium polytungstate).						G1614
p. reagent grade ethanol or methanol.						G1615
q. cotton applicator swabs (to remove material from upper part of centrifuge tubes).						G1616
IV. Sample Preparation – NOB Bulk Samples and Friable Sample Problem Matrices (G1112)						
A. Samples are homogenized when necessary.						G1116
a. At least 4 subsamples are prepared and mounted. (Item 198.1)						G1118
b. At least 8 subsamples are prepared and mounted. (EPA 600/M4/82/020, Section 1.7.2.4)					Option: Cite other one on page 6.	G1617
<i>Refer to the "Matrix Modification" Section (XI) of this checklist.</i>						
B. Samples are acid treated when necessary.						G1120
C. Samples are dispersed with surfactant when necessary.						G1124
D. Samples are ashed when necessary at 480°C until mass stabilizes (1-12 hours).						G1128
E. Layers in layered samples are analyzed individually.						G1130
V. Sample Amount, Storage, and Preparation – Surfacing Material Containing Vermiculite Bulk Samples (G1700)						
Gravimetric Reduction (Section 4.2.1)						
A. A minimum weight of 3 grams is used for analysis.						G1701
B. Laboratory notified its clients that a minimum of 10 grams of sample is required.						G1702
C. The laboratory keeps any unused portion of sample for a period of no less than 90 days from the date the report is transmitted to the client.						G1703
D. Samples are ashed at 485 ±5°C for at least 10 hours.						G1704
Acid Treatment (Section 4.2.2 and 4.2.3)						
E. The sub-sample is acid treated according to Section 4.2.2. and 4.2.3.						G1705
F. The floatable material in the petri dish is dried until a stable weight is achieved (< 3% difference in weight).						G1706

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
(Using Erlenmeyer flask, stirring rod, 2 M HCl, 0.1 um filtered water, and magnet. Sample is stirred for 15 minutes. Removing any floatable material. Repeating rinses at least 3 times.)						
Filtration (Section 4.3)						
G. The remaining materials (liquids and solids) is collected during acid treatment filtered according to Section 4.3.						G1707
(Using glass filtration apparatus, 0.4-0.8 um polycarbonate filter, and 0.1 um filtered water. Repeating rinse at least 2 times.)						
H. The filtered material in the petri dish is dried until a stable weight is achieved (< 3% difference in weight).						G1708
VI. PLM examination for Chrysotile (Section 6), Determination of Amphibole Asbestos Using Heavy Liquid Centrifugation (7), PLM examination for Amphibole Asbestos (8), and Calculations (9) – Surfacing Material Containing Vermiculite Bulk Samples						
Section 6 (G1800)						
For the examination of chrysotile, ...						
A. Eight slides are prepared from the residue using high dispersion liquid of RI 1.630 or 1.680.						G1801
B. Each slide is scanned using crossed polars with a 550 nm compensator plate to determine if structures morphologically consistent with chrysotile are present.						G1802
C. If no structures are detected, zero chrysotile points and 50 occupied points are assigned to each slide.						G1803
D. If structures are detected, at least 1 additional slide using RI 1.550 oil is prepared and at least 4 structures are positively identified as per Section 5.						G1804
For the quantiation of chrysotile, ...						
E. The lab uses the 400 point count method with 50 non-empty points per slide.						G1805
F. The lab uses the original 8 slides (in RI 1.630 or 1.680 oil).						G1806
(Chalkley point-count reticle is not allowed.)						
Section 7 (G1825)						
For the determination of amphibole asbestos concentration using heavy liquid centrifugation, ...						
A. The residue is properly dried and weighed. (i.e., If the residue has been exposed to room air for > 1 hour, the residue must be placed back in the oven or desiccator for a minimum of 1 hour before weighing.)						G1826
B. The residue is properly divided and transferred between 2 centrifuge tubes.						G1827
C. The heavy liquid is calibrated as per Appendix D.						G1828
E. The residue is properly dispersed throughout the heavy liquid. (i.e. using a glass rod, adding at least 10 mL of heavy liquid to bring the liquid level up to 2 cm from the top of each tube)						G1829
F. The tubes are properly centrifuged. (i.e. Centrifugation times vary depending on the dimensions and rotation speed of the particular centrifuge. Refer to Appendix E.)						G1830
G. The heavy fraction is properly washed. (i.e. washing 5 times with 2 mL reagent water)						G1831
H. The centrifugate is properly washed to remove the heavy liquid. (i.e. using 5 mL 0.1 um water; one wash)						G1832
Section 8 (G1850)						
For the examination and quantitation of amphibole, ...						
A. Eight slides are prepared from the residue using high dispersion liquid of RI 1.630.						G1851

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
B. The whole area of each slide is scanned at a 100x magnification as per Section 5.						G1852
C. If no amphibole asbestos is observed, 50 occupied points and zero asbestos points are assigned to each slide.						G1853
D. If amphibole asbestos is observed, scanning is discontinued and 400 point count started.						G1854
E. The lab calculates the concentration of amphibole asbestos in the centrifugate and in the original sample as per Appendix B and Section 9.						G1855
Section 9 (G1875)						
For the calculation of chrysotile, the lab follows the calculations in Appendix B and Section 9.						G1876
For the calculation of amphibole, the lab follows the calculations in Appendix B and section 9.						G1877
For the calculation of total asbestos content, the lab follows the calculations in Appendix B and Section 9.						G1878
VII. Analytical Records (G1132)						
A. The analysis sheet includes/indicates the following:						
a. analyst's signature or initials and date of analysis.						G1136
b. sample ID number.						G1140
c. gross description of material including color, homogeneity and texture.						G1144
d. disqualifying optical property for each non-asbestos fiber identified.						G1177
e. matrix reduction. (See Section X, too.)						G1148
f. at least four subsamples are prepared and mounted. (Item 198.1)						G1180
g. at least eight subsamples are prepared and mounted. (EPA 600/M4/82/020)					Option: Cite other one on page 4.	G1617
h. use of EPA 600/M4/82/020 and/or ELAP Item 198.1 point count methods.						G1184
i. tally of points for each type of asbestos.						G1188
j. original quantitation results that are based on point counting.						G1192
B. The analysis sheet for SM-V captures the following (in addition to items a.-j. noted in Section VII, A, above):						
a. confirmation of vermiculite by stereo binocular microscope.						G1193
b. matrix reduction (mass of original sub-sample, mass after ashing, mass after acidification, mass after acid float, mass of centrifugate).						G1148
C. For each type of asbestos type identified, the following is recorded:						
a. morphology.						G1156
b. birefringence.						G1160
c. angle of extinction.						G1164
d. sign of elongation.						G1168
e. RI (to the nearest 0.004) for fiber length (parallel).						G1172
f. RI (to the nearest 0.004) for fiber width (perpendicular). (Note: Fiber width and fiber length should be different.)						G1176
VIII. Calibration Records (G1248)						
A. There are records of the following:						

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
a. semi-annually or next use, whichever is less frequent , calibration of refractive index oils to within 0.004, <u>with a temperature accuracy of 2°C using a refractometer or RI glass beads.</u>	Volume 1, Module 3 1.7.1.3.2					G1256
b. calibration of RI oils to within 0.004 when a new container is opened.						G1257
c. daily or next use alignment of PLM.						G1260
d. monthly determinations of dispersion-staining or Becke-line colors from the lab's permanent amosite mount.						G1264
e. semi-annual measurements of HEPA-ventilated enclosure(s) demonstrating a face velocity of at least 75 fpm.						G1268
f. quarterly calibrations of muffle furnace in the range 450-480°C.						G1270
g. room temperature being checked daily or next use .						G1271
IX. Quality Control and Personnel Records (G1376)						
A. All QC analyses have been performed and evaluated before final reports are submitted to clients.						G1301
B. QC samples are submitted blindly to the original analyst so that the analyst is unaware that the sample will be reanalyzed. Note: A random number generator could be used for 1-person labs.						G1303
C. For intra-analyst precision, the original analyst reanalyzes at least 2% of blind QC samples. Note: This is the same as at least one (1) out of fifty (50) samples.	Volume 1, Module 3 1.7.3.1.3a					G1305
a. For single analyst labs, at least 1 out of every 10 blind QC samples is reanalyzed.	Volume 1, Module 3 1.7.3.1.3a					G1304
D. For inter-analyst precision, a different analyst reanalyzes at least 6.7% of blind QC samples given to the original analyst. Note: This is the same as at least one (1) out of fifteen (15) samples.	Volume 1, Module 3 1.7.3.1.3b					G1307
E. QC re-analyses include complete and independent reparation and analysis of the sample.						G1309
F. R-bar charts are showing intra- and inter-analyst precision kept up-to-date for each analyst.						G1310
a. Records of each analyst's replicate and duplicate analyses are kept.						G1388
b. Corrective is action taken when R values are > 1 or < -1 for inter-analyst QC. Note: Acceptable range = 1 to -1						G1311
c. Corrective action is taken when absolute R values are > 1 for intra-analyst QC.						G1313
G. At least 1% (1 out of 100) of samples analyzed are a standard or reference sample that has been routinely resubmitted.	Volume 1, Module 3 1.7.4.3a					G1312
a. For friable materials, at least 50% of the QC reference samples submitted contains between 1 and 10% asbestos.						G1315
H. X-bar charts showing analyst's accuracy are kept up-to-date.						G1314
a. The records from reference standard analyses are kept for each analyst.						G1392
I. Re-analysis of inter-laboratory QC samples are performed at least quarterly or at a rate of 1 sample per 500 routine samples (whichever is less). Note: Inter-Laboratory Precision. The laboratory shall participate in round robin testing with at least one (1) other laboratory. Samples shall be sent to	Volume 1, Module 3 1.7.3.1.3c				All labs can meet this requirement provided they are routinely participating in NYS DOH PT studies or AIHA PT studies.	G1317

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
this other laboratory at least four (4) times per year. These samples shall be samples previously analyzed as QC samples. Results of these analyses shall be assessed in accordance with QC requirements. <u>The QC requirements shall address misclassifications (false positives, false negatives) and misidentification of asbestos types.</u>						
J. All misclassifications (false positives and false negatives) and misidentifications of asbestos types associated with inter-laboratory reanalyses have been resolved.						G1320
K. At least one non-ACM blank is prepared daily or with every 50 samples analyzed, whichever is less. (Item 198.1, Section 8.3.2)						G1325
a. Or, a blank check is made after every 20 uses of each piece of homogenization equipment. (Item 198.1, Section 8.3.2)						G1323
L. At least one non-ACM non-friable material is prepared and analyzed with every 20 samples analyzed. (Item 198.6, Section 8.3.2)						G1327
M. Monthly summaries reveal an error rate of less than 1% on the classification of samples.						G1326
N. All analysts are able to correctly identify the six regulated asbestos types. (Note: The six types are chrysotile, amosite (grunerite), crocidolite (riebeckite), anthophyllite, actinolite, and tremolite)						G1395
O. Records are kept for each analyst outlining resolutions of any QC deficiencies.						G1396
P. At least 10% of SM-V analyses are re-analyzed.						G1397
X. Results and Reports (G1196)						
A. The final results include the type and percentage of each asbestos type.						G1200
B. The final results include the type and percentage of each non-asbestos fiber type.						G1204
C. The percentage of asbestos detected is rounded off to two significant digits .						G1230
D. A recent client report on an ACM includes the following:						
a. color of the bulk sample.						G1208
b. type and percentage of each asbestos type.						G1212
c. type and percentage of each non-asbestos fiber type.						G1216
E. Results from layered samples are reported as separate layers. (Note: Labs can do composites, but lab needs to record results for original layers.)						G1217
F. The samples are stored in a secure area for at least 60 days after result reporting.						G1218
G. Samples determined by the laboratory to be NOB and analyzed by Item 198.4 and/or 198.6 are clearly noted as NOB material on reports.						G1219
H. Final results for SM-V include all of the following: % chrysotile and amphibole detected, total % asbestos, % organic fraction and water, % floats, % residue, and % centrifugate.						G1231
XI. Matrix Modification for NOB Bulk Samples and Friable Sample Problem Matrices (G1424)						
A. Analysis sheets show calculation of percent matrix loss during muffle furnace ashing.						G1476
B. Analysis sheets show calculation of percent matrix loss during acid digestion.						G1478
C. Reduced samples are cooled in a dessicator prior to weighing.						G1488
D. Percentages of matrix loss are used in calculating final asbestos percentage.						G1480

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

	NELAC Reference	Y	N	NA	Comment	Code
E. Inconclusive (≤1%) NOB asbestos results by PLM are reported with the ELAP required disclaimer. (See disclaimer in Item 198.6, Section 6.3.2.2.)						G1484
F. The gravimetric reduction method is used to generate the asbestos result.						G1487
XII. ELAP On-Site Audit Materials and Samples (G1328)						
ELAP Audit Samples – Please use in-house samples from lab (i.e. past ELAP or AIHA PTs).						
A. Proper dispersion-staining colors or Becke lines were visible with the ELAP amosite slide.						G1332
B. Complete extinction was observed when the ELAP amosite mount was viewed with crossed polars.						G1336
C. The analyst correctly described the morphology of the fibers.						G1340
D. The color of the sample was recorded on the analysis sheet.						G1344
E. Subsamples were taken at random and without preference to fibers.						G1348
F. a. At least 4 subsamples were prepared and mounted using whole coverslips. (Item 198.1)						G1352
b. At least 8 subsamples were prepared and mounted using whole coverslips. (EPA 600/M4/82/020)						G1374
G. The analyst was able to accurately determine if the fiber's refractive index was lower or higher than the initial mounting medium.						G1356
H. The analyst was finally able to determine the refractive index of the fiber length and width to within 0.004 of the known refractive index.						G1360
I. Sign of elongation was correct.						G1364
J. Extinction angle was correct.						G1368
K. a. The Item 198.1 stratified point counting was done correctly.						G1372
b. EPA 600/M4/82/020 point counting was done correctly.						G1375
Note: The analyst must use a uniform scan pattern when analysis is performed with a multi-point eye piece. (Item 198.1, Section 5.2.2)						
L. Analyst accurately identified fibrous components.						G1373

Additional Observations/Notes:

Other Useful Information:

Non-Friable Organically Bound materials (NOB): vinyl asbestos tile (VAT), resilient floor tiles, mastic, asphalt shingles, paint chips, caulking, glazing, etc.

R-bar (Item 198.1 and 198.6, Sec. 8.2.2) - **Inter-Analyst**

$$R = \frac{(A - B)}{\left(\frac{A + B}{2}\right)}$$

R-bar (Item 198.1 and 198.6, Sec. 8.2.1) – **Intra-Analyst**

$$R = \left| \frac{(A - B)}{\frac{A + B}{2}} \right|$$

NYSDOH Environmental Laboratory Approval Program – PLM Checklist

Where A = result from the analyst being checked and B = result from other analyst for same sample.

Multiple analysts: Inter-analyst, at least 1 per 15 samples and Intra-analyst, at least 1 per 50 samples

Single analyst: Intra-analyst, at least 1 per 11 samples

X-bar (Item 198.1 and 198.6, Sec. 8.2.3)

Accuracy of each analyst shall be monitored by determining percent recovery.

$$\% \text{ Recovery} = \left(\frac{A}{W} \right) \bullet 100$$

Where A = analytical result read by analyst and W = formulated weight for reference standard slide

Disclaimer (from Item 198.6, Sec. 6.3.2.2)

"Polarized-light microscopy is not consistently reliable in detecting asbestos in floor coverings and similar non friable organically bound materials. Quantitative transmission electron microscopy is currently the only method that can be used to determine if this material can be considered or treated as non-asbestos containing."

Table 1 (from Item 198.1, Table I and Item 198.6, Table I)

Asbestos Type	Color and Morphology	Refractive Index		Sign of Elongation	Extinction Angle
		⊥			
Chrysotile	White to pale green; v. flexible w/ "kinks"; Wavy w/ "knuckles"	1.493-1.559	1.517-1.567	+	; undulose
Amosite	Tan; mod. flexible, but straight bundles; easily splayed ends	1.657-1.686	1.696-1.729	+	;
Crocidolite	Dark blue; flexible; some "kinks"; splayed ends; strongly pleochroic	1.654-1.701	1.668-1.717	-	
Anthophyllite	White to light tan; stiff; ends splayed to blunt	1.596-1.652	1.615-1.722	+	
Tremolite	White to light tan; stiff; large bundles; may have splayed ends	1.599-1.628	1.625-1.655	+	; v. thin fibers or cleavage fragments (≤ 15°)
Actinolite	White to green; stiff; large bundles; may have splayed ends; often pleochroic	1.600-1.668	1.625-1.688	+	; v. thin fibers or cleavage fragments (≤ 20°)

% Asbestos NOB Calculation

$$\% \text{ Asbestos} = \left(\frac{PAM}{OM} \right) \bullet AP$$

Where PAM is mass of residue after furnace and acid treatment (in mg), OM is mass of original subsample (in mg), and AP is mean percentage of asbestos (versus inorganic residue) in final slide preparations.