

NYSDOH Environmental Laboratory Approval Program – TEM Checklist

LAB ID:	LABORATORY NAME:
DATE:	ASSESSOR NAME:

ASBESTOS BY TRANSMISSION ELECTRON MICROSCOPY

PW: EPA 100.1 (ELAP Code 7300) and 100.2 (ELAP Code 7301)

SHW (in Non-Friable Material): Item 198.4 of Certification Manual (ELAP Code 4070)

AIR: NIOSH 7402 (ELAP Code 4592); 40 CFR 763 APX A NO. 111 (ELAP Code 4588); and YAMATE, AGARWAL GIBB (ELAP Code 4590)

Method Number:

SOP Number:

Revision Number:

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Personnel records observed (including permanent and seasonal, if applicable)

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ASBESTOS ANALYSIS BY TRANSMISSION ELECTRON MICROSCOPY	Y	N	N/A	COMMENTS	CODES
I. Transmission Electron Microscope					
J1004					
A. Is TEM voltage at least 80 keV?					J1008
B. Does TEM have a magnification range of at least 0.3 to 100 kx?					J1012
C. Is TEM resolution at least 1 nm?					J1016
D. Is TEM screen marked with a circular or 2-D calibrated scale?					J1020
E. Is TEM capable of either SAED or microbeam ED on area <0.6 μm^2 (water-only)?					J1024
F. Does TEM have a primary beam stop?					J1028
G. Does TEM have a functioning camera?					J1032
H. Are unit fibrils visible at normal analysis magnification (15-20 kx)?					J1036
I. Is tubular structure of unit chrysotile resolvable at 15-20 kx?					J1040
J. Are ED patterns readily achieved from individual chrysotile fibrils?					J1044
K. With premeasured feature, is calculated length $\pm 10\%$ of actual?					J1048
L. Do specimen translators track reliably (<25% deviation from grid bar to grid bar at 20 kx)?					J1052
M. Is smallest beam size <250 nm?					J1056
N. Does TEM have eucentric goniometer?					J1060
O. Does TEM have at least $\pm 25^\circ$ tilt?					J1064
II. Energy-Dispersive Spectrometer					
J1068					
A. Is x-ray detector located in TEM column?					J1072
B. Is there functioning software to resolve and label elemental peaks?					J1076
C. Is Al peak between 1.47 and 1.49 keV?					J1080
D. Is Cu peak between 8.03 and 8.05 keV?					J1084
E. Can Mg and Si peaks be resolved from NIST unit chrysotile fibrils?					J1088
F. Can Na in NIST crocidolite standard be resolved in spectrum?					J1092
III. Vacuum Evaporator					
J1096					
A. Is it a high-vacuum type (<0.0001 torr)?					J1100

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B. Does it use sharpened carbon rods?					J1104
D. Does it have a rotating/tilting stage?					J1112
E. Does it have a venting rate to atmosphere that is at least 1 minute in duration?					J1116
IV. Low Temperature Asher (LTA) J1120					
A. Is there documentation showing proper quarterly calibration of the ashing rate? And is it being used?					J1124
B. Is LTA using pure oxygen?					J1128
C. Does LTA have venting rate to the atmosphere that is at least 1 minute?					J1132
V. Miscellaneous Equipment/Supplies J1136 Does the lab have the following:					
A. 100-class HEPA-filtered clean room, clean bench or laminar-flow cabinet separate from bulk-sample area?					J1140
B. oven or slide warmer capable of maintaining 65-70°C temperature?					J1144
C. ultrasonic bath for cleaning apparatus?					J1148
D. fine-tipped forceps?					J1152
E. scalpel with curved blades?					J1156
F. microscope slides?					J1160
G. adhesive tape?					J1164
H. micropipet (variable 10-100 ul) with disposable tips?					J1168
I. carbon rods with 1mm x 5mm tips?					J1172
J. gold wire for evaporation?					J1176
K. petri dishes or equivalent for Jaffe wick?					J1180
L. stainless-steel mesh for Jaffe wick?					J1184
M. lens tissue?					J1188
N. copper or gold 200-mesh finder TEM grids?					J1192
O. analytical balance, minimum 0.1 mg sensitivity?					J1196
P. filter paper or equivalent for Jaffe wick?					J1200
Q. acetone or dimethylformamide or dimethylsulfoxide?					J1204

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R. chloroform or other PC solvent?					J1208
S. distilled, deionized or filtered water?					J1212
T. software or tables for determining hkl zone-axis measurements?					J1216
VI. Reference Standards J1220					
A. Does the lab have the following:					
A. TEM grid(s) mounted with NIST SRM 1876a or 1876b?					J1224
B. NIST-SRM-1866 chrysotile on TEM grid with hard-copy spectra (from in-house systems) and labeled SAED micrographs to accompany specific image micrographs?					J1228
C. NIST-SRM-1866 grunerite on TEM grid with hard-copy spectra (from in-house systems) and labeled zone-axis SAED micrographs to accompany specific image micrographs?					J1232
D. NIST-SRM-1866 crocidolite on TEM grid with hard-copy spectra (from in-house systems) and labeled zone-axis SAED micrographs to accompany specific image micrographs?					J1236
E. NIST-SRM-1867 anthophyllite on TEM grid with hard-copy spectra (from in-house systems) and labeled zone-axis SAED micrographs to accompany specific image micrographs?					J1240
F. NIST-SRM-1867 tremolite on TEM grid with hard-copy spectra (from in-house systems) and labeled zone-axis SAED micrographs to accompany specific image micrographs?					J1244
G. NIST-SRM-1867 actinolite on TEM grid with hard-copy spectra (from in-house systems) and labeled zone-axis SAED micrographs to accompany specific image micrographs?					J1248
H. A library containing at least four asbestos look-alikes on TEM grids and hard copies of their critical EDXA and SAED (from in-house systems)?					J1252
I. At least 6 EDXA from each of the 6 asbestos types and the determined mean, standard deviation and ranges of normalized elemental ratios to Si?					J1256
J. NIST SRM 2063?					J1260
VII. Sample Custody J1264					
A. Is there a designated sample coordinator in the laboratory?					J1268
B. Is the chain-of-custody procedure outlined in AHERA followed?					J1272
C. Are chain-of-custody records kept for all samples?					J1276
D. Have all air samples received in containers with bulk samples been rejected?					J1280
VIII. Air Sample Handling J1284					
A. Does lab have a copy of AHERA (Oct. 1987) NON-mandatory method?					J1288

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B. Are filters prescreened (at least 2%) for asbestos contamination?					J1292
C. Is there documentation showing that filter lot blanks average less than 18 s/mm ² ?					J1296
D. Is there documentation showing that filter lot blanks have less than 53 s/mm ² per single preparation?					J1300
F. Is the cassette composed of 3 pieces (cap, extension cowl or retainer ring, and base)?					J1308
G. Is either a 0.4-um polycarbonate or 0.45-um mixed cellulose ester filter on top of the cassette backing?					J1312
H. Is the filter in the cassette backed by a 5.0-um mixed cellulose ester filter?					J1316
I. For 25-mm cassettes, is collection flow rate between 1 and 10 lpm?					J1320
J. For 37-mm cassettes, is collection flow rate between 3 and 30 lpm?					J1324
K. Are sample volumes above the minimum volumes given in Table I of Section III (FR 52 (21):41876)?					J1328
L. For abatement projects, are at least 13 samples submitted?					J1332
1. Are at least 5 samples collected per abatement area?					J1336
2. Are at least 5 ambient air samples per abatement area?					J1340
3. Are at least 2 field blanks included per abatement set?					J1344
4. Is at least 1 sealed blank included per abatement set?					J1348
IX. Water Sample Handling J1352					
A. Does lab have a 500-ml graduated cylinder?					J1356
B. Does lab have a copy of Chatfield (1991)?					J1360
C. Does lab have appropriate vertical-walled filtration apparatus?					J1364
D. Does lab have 0.1-um polycarbonate filters or 0.1-0.22-um MCE filters?					J1368
E. Does lab have 0.45-um mixed-cellulose ester filters for backing 0.1-um filters?					J1372
F. Is final filtration of water samples through a 0.1-um polycarbonate filter or 0.1-0.22-um MCE filters?					J1376
G. Are samples kept cool (4°C) and in the dark?					J1380
H. Are samples filtered within 48 hours of collection?					J1384
I. Have fresh water blanks been prepared with each batch of water samples to check for contamination?					J1388
J. Do lab blanks have less than 3 long fibers counted in the 20 grid squares or less than 1% of sample concentration?					J1392

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X. TEM Analysis Sheet J1396					
A. Is there a space for sample identification?					J1400
B. Is there a space for analyst's name?					J1404
C. Is there a space for date of analysis?					J1408
D. Is there a space for recording correct grid opening area?					J1412
E. Is there a column for SAED confirmation of identity?					J1416
F. Is there a column for X-ray peaks identified?					J1420
G. Is there a column for number and identity of grid squares analyzed?					J1424
H. Are identity of specific grids (grid box locations) and each analyzed grid square (finder grids) recorded?					J1428
I. Is there a space for volume filtered?					J1432
J. Is there a space for filter's effective area?					J1436
K. Is the space for the grid opening area to be filled in?					J1440
L. Is there a space for the number of grid openings analyzed?					J1444
XI. Analysis of AHERA Samples J1448					
A. Are analyses performed at an actual screen magnification of at least 15,000X?					J1452
B. Is each analysis split between two different grid preparations?					J1456
C. Are SAED patterns verified for at least the first 4 asbestos fibers per sample?					J1460
D. Is there a record of each SAED pattern being checked by a qualified individual?					J1464
E. If any asbestos was detected in a set of 5 samples, was at least one SAED pattern from each type of asbestos recorded on film?					J1468
F. Is there verification of qualified individual maintaining at least 80% accuracy in SAED ID?					J1472
G. Are EDXA taken for at least the first 4 amphiboles?					J1476
H. Are counts continued until sensitivity is at least 0.005 s/cm³?					J1480
I. Were fiber lengths separated for > 5 um and < 5 um?					J1484
XII. Analysis of Water Samples J1488					
A. Was analysis on short fibers continued to the point that sensitivity was at or below 10 MFL?					J1492

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B. Was analysis for short fibers performed at a screen magnification of at least 15,000X?					J1496
C. Was analysis for long fibers continued to the point that sensitivity was at or below 0.2 MFL?					J1500
D. Was analysis for long fibers performed at a screen magnification of at least 10,000X?					J1504
E. Were fiber lengths separated for > 10 µm and < 10 µm?					J1508
F. Was analysis for long fibers performed on at least 4 grid openings?					J1512
G. Was analysis split among at least 3 different grid preparations?					J1516
H. Have at least one micrograph, one SAED pattern, and one EDXA spectrum been recorded for each type of asbestos identified in each sample?					J1520
XIII. Data Handling J1524					
A. Are hand-calculations orderly and double-checked and initialed by a second analyst?					J1528
B. Is there hard-copy documentation of in-house-developed computer programs?					J1532
C. Is there flexibility in entering grid square area?					J1536
D. Is there flexibility in entering effective filtration area?					J1540
E. Is there flexibility in entering volume filtered?					J1544
XIV. Reports to Clients J1548					
A. Are structure concentrations per mm ² and per cm ³ reported (air only)?					J1552
B. Is the analytical sensitivity for method reported?					J1556
C. Is the total number of asbestos structures reported?					J1560
D. Is the total area analyzed reported?					J1564
E. Is the air or water volume sampled reported?					J1568
F. Is the average grid opening size reported?					J1572
G. Is the number of grid openings analyzed reported?					J1576
H. Is micrograph number sent to client for asbestos-containing AHERA samples?					J1580
I. Is a copy of analytical count sheet(s), with the type(s) of asbestos, sent with report?					J1584
J. Is the signature of the lab director included on the report?					J1588
K. Does report include MFL for both total and long-fiber analyses?					J1592
L. Is there a disclaimer about structures/cm ³ if air samples were collected by non-lab personnel?					J1596

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XV. Analyst's Sample Preparation Performance					
J1600					
A. Did analyst maintain clean-room conditions during filter preparation?					J1604
B. Was a lab blank prepared alongside audit filter?					J1608
C. Was etching performed according to latest calibration?					J1612
D. Did low-temperature asher take at least one minute to return to room pressure?					J1616
E. Did evaporator vacuum go below 0.0001 Torr?					J1620
F. Did analyst use tilt/rotate mode during carbon film evaporation?					J1624
G. Did carbon film integrity meet AHERA criteria for acceptable preparation?					J1632
1. Did audit-filter preparation have most grid openings (>50%) covered by carbon film replica?					J1636
2. Did audit-filter preparation have complete dissolution (>90%) of filter medium?					J1640
3. Did audit-filter preparation have less than 50% of carbon film overlapped or folded?					J1644
4. Did audit-filter preparation have at least 20 useable, non-adjacent grid openings?					J1648
5. Was carbon film sufficiently thin for analysis?					J1652
H. Did analyst follow acceptable procedures during filter dissolution?					J1656
I. Were particles on audit filters reliably transferred to carbon film?					J1660
J. Was etching satisfactory?					J1664
XVI. Analyst's Sample Analysis Performance					
J1668					
A. Did analyst attempt SAED on the asbestos fiber before using EDXA?					J1672
B. Did analyst measure audit diffraction spacing(s) to within $\pm 5\%$ of true value?					J1676
C. Did analyst predict layer-line orientation from fiber axis orientation?					J1680
D. Did analyst accurately draw chrysotile diffraction pattern and label 5 different reflections from memory?					J1684
E. Did analyst correctly identify actinolite audit sample?					J1688
F. Did analyst correctly identify tremolite audit sample?					J1689
G. Did analyst correctly identify fibrous talc audit sample?					J1690
H. Did analyst disqualify sepiolite?					J1692
I. Did analyst disqualify potassium richterite?					J1696

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J. Did analyst disqualify hornblende in actinolite audit sample?					J1700
K. Did analyst correctly differentiate crocidolite from amosite?					J1704
L. Did analyst disqualify palygorskite?					J1708
M. Did analyst adjust goniometer eucentricity before attempting SAED?					J1712
N. Did analyst scan grid with linear single-translator movements?					J1716
O. Did analyst achieve zone-axis diffraction pattern(s) with amphiboles?					J1720
P. Did analyst measure and match amphibole zone-axis SAED pattern to correct published values?					J1724
Q. Did analyst achieve identical results in blind re-analysis of one of their archived samples?					J1726
R. Did analyst have a clean surface for handling TEM grid before being placed in specimen holder?					J1727
XVII. On-Site Audit Sample Results J1728					
A. Were MFL results for short fibers within $\pm 10\%$?					J1732
B. Were MFL results for long fibers within $\pm 10\%$?					J1736
C. Were asbestos types(s) correctly identified?					J1740
D. Were structures/mm ² within $\pm 10\%$?					J1744
E. Were structures/cm ³ within $\pm 10\%$?					J1748
XVIII. Quality Control J1752					
A. Are there records that at least 2% of all analyses (same grid squares) have been re-analyzed blindly by the same analyst and all discrepancies have been resolved?					J1756
B. Has precision for each analyst been determined from same grid-square recounts?					J1760
C. Are there records that at least 4% of all analyses (same grid squares) have been re-analyzed blindly by a second analyst and all discrepancies have been resolved?					J1764
D. Are control charts monitoring intra- and inter-analyst variability kept up to date?					J1768
E. Are there records that samples (same grid squares) have been re-analyzed by an independent outside lab at least twice per year and all discrepancies have been resolved?					J1772
F. Are there records that verified analyses have been performed at the rate of at least 1% of total analyses by all analysts?					J1776
G. Is the average concentration used for verified analyses between 1000 and 5000 fibers/mm ² ?					J1780
H. Are cumulative records kept for each analyst documenting false positives and negatives?					J1784
I. Are true/false positive/negatives for each analyst within AHERA guidelines?					J1788

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J. Are lab blanks analyzed at the rate of 5% of sample load?					J1792
K. Is there an orderly system for storing grids?					J1796
L. Is there an orderly system for storing negatives?					J1800
XIX. Calibrations J1804					
A. Are there records of monthly phosphor screen magnification calibrations?					J1808
B. Are phosphor screen measurements (mm) for 0.5 and 5 um recorded monthly (AHERA)?					J1812
C. Are phosphor screen measurements (mm) for 0.5 and 10 µm recorded monthly (water)?					J1816
D. Are there records of monthly micrograph negative magnification calibrations?					J1820
E. Are phosphor screen calibrations for 5.3A spacings recorded monthly?					J1824
F. Are there records of monthly micrograph negative camera constant calibrations?					J1828
G. Are there records of quarterly spot-diameter calibrations demonstrating <250 nm?					J1832
H. Are there records of quarterly beam-dose calibrations demonstrating >90% reliability?					J1836
I. Are there records of daily verification of Cu peak between 8.03 and 8.05 keV and Al peak between 1.47 and 1.49 keV?					J1840
J. Are there records of semi-annual verification of resolution <175 keV at the Mn K (alpha) peak?					J1844
K. Are there records of semi-annual determinations of k factors relative to Si?					J1848
L. Is Mg/Fe sensitivity factor 1.5 or less?					J1852
M. Are there hard-copy spectra of quarterly determinations of resolvable Na peaks from NIST crocidolite?					J1856
N. Are there hard-copy spectra of quarterly demonstrations of resolvable Mg and Si peaks from NIST chrysotile unit fibrils?					J1860
O. Does the lab have calibration records for the batches of TEM grids received from vendors or measurements on each analysis sheet for individual square measurements?					J1864
XX. Non-Friable Organically Bound Samples J1896					
A. Does the lab have 0.4-µm-pore polycarbonate filters?					J1900
B. Does the lab have filtration apparatus for polycarbonate filters?					J1904
C. Does the lab have crucibles for muffle oven that can withstand 500°C?					J1908
D. Does the lab have a high-temperature thermometer (at least 500°C with 5°C divisions) or equivalent melting-point standards or potentiometer?					J1912
E. Does the lab have concentrated HCl?					J1916

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F. Does the lab have an ultrasonic bath?					J1920
G. Does the lab have filtered (0.1-µm) distilled water?					J1924
H. Do NOB analysis sheets show calculation of percent matrix loss during muffle furnace ashing?					J1928
I. Do NOB analysis sheets show calculation of percent matrix loss during acid digestion?					J1932
J. Are percentages of matrix loss used in calculating final asbestos percentage?					J1940
K. Does the lab have a muffle oven capable of reaching 480°C?					J1944
L. Have all QC re-analyses involved complete re-preparation of the sample?					J1945
M. Are there records that at least 2% of all analyses have been re-analyzed blindly by the same analyst and have all discrepancies been resolved?					J1946
1. Are r values plotted according to Section 8.2.1 of Item 198.4?					J1947
N. Are there records that at least 6.7% of all analyses have been re-analyzed blindly by a different analyst and have all discrepancies been resolved?					J1949
1. Are r values plotted according to Section 8.2.2 of Item 198.4?					J1950
O. Have blanks been submitted at the rate of at least 5% to monitor contamination?					J1951
P. Does the laboratory have records demonstrating monthly calibration of the muffle oven at 480°C?					J1948
XXI. Evaluation of Archived TEM Grid J1952					
A. Is filter surface correctly etched?					J1956
B. Did structure count during re-analysis match the originally reported structure count?					J1960
C. Did the grid preparation meet the following criteria:					
1. Carbon film covered >50% of grid openings?					J1968
2. More than 90% of filter is dissolved?					J1972
3. Less than 50% of the carbon film is folded or overlapped?					J1976
D. Did at least 20 grid openings meet the following criteria:					
a. No overlap or folded carbon film?					J1984
b. Less than 5% holes or tears?					J1988
c. Less than 5% opaque material?					J1992
d. Less than 10% particulate loading?					J1996
XXII. NIOSH 7402 J2000					

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A. Is the filter not etched?					J2001
B. Did all structures counted have a diameter >0.25 µm and length >5 µm?					J2002
C. For light loading (<5 f/GSO), were 40 grid openings counted?					J2003
D. For moderate loading (5 to 25 f/GSO), were 40 grid openings or 100 fibers counted?					J2004
E. For heavy loading (> 25 f/GSO), were 100 fibers and a minimum of 6 grid openings counted?					J2005
F. Was the analysis split among 3 different grid preparations?					J2006
G. Were at least 2 field blanks analyzed?					J2007
H. Were the following EDX spectra taken:					
a. For chrysotile for the first 5 fibers and one out of 10 thereafter?					J2008
b. For amphibole for the first 10 fibers and one out of 10 thereafter?					J2009
c. For ambiguous structures for every fiber?					J2010
I. Was a diffraction pattern taken for a minimum of 10% of the fibers and at least 3 asbestos fibers?					J2011
J. Does the final report include:					
a. The fraction of optically visible asbestos fiber?					J2012
b. The asbestos fiber count as applied to the original PCM results?					J2013
c. The type of asbestos identified?					J2014