Method for Determining Asbestos Containing Material
under Section 2 of the Air Pollution Control Ordinance

Content

1   *Introduction*

In section 2 of the Air Pollution Control Ordinance (the Ordinance), 'asbestos' includes the minerals, and substances including the minerals, amosite, crocidolite, chrysotile, fibrous actinolite, fibrous anthophyllite and fibrous tremolite while 'asbestos containing material' (ACM) means any material, substance or product which is made with or contains asbestos as determined by a method approved by the Secretary for the Environment. For the purpose of the Ordinance, the following method by gravimetric and optical microscopy techniques shall be used to determine if a substance is an ACM. It shall be effective on 4 April 2014 and supersede the method specified under Government Notice Number 2755 published in the Gazette in 1997.

2   *Application*

2.1 The method described in this document applies when it is necessary to determine if a substance is an ACM under the Ordinance.

2.2 For sample with distinctly different component layers, each layer shall be treated as a separate sample and the percentage of asbestos of each component layer shall be determined. Each material layer shall be classified as an ACM or otherwise based on the percentage of asbestos in each layer.

3   *Summary of Method*

This method gives the percentage of asbestos in a sample by two steps viz. Step 1 by visual estimation using low-power stereo binocular microscope and Step 2 by gravimetric reduction of matrix and point-counting using polarized light microscopy technique. When the percentage of asbestos cannot be determined to be more than 1% in Step 1, Step 2 is proceeded to use the gravimetric and point-counting technique. Before point-counting, the matrix of the sample is reduced or removed as much as possible by a combination of simple procedures such as organic reduction, acid dissolution and sedimentation. The weight of the sample is tracked at every stage. After matrix reduction, large asbestos bundles, if any, are removed by hand-picking and weighed. Subsamples of the final residue are then mounted on microscope slides using Cargille liquid that gives the maximum contrast and the concentration of asbestos is determined by point-counting using a polarized light microscope. The number of asbestos points and non-asbestos points are counted under the cross-hair reticle of the microscope until more than 20 asbestos points or a specified number of non-empty points have been reached.

4   *Equipment and Supplies*

4.1 HEPA-ventilated, negative pressure sample preparation work area. This can be a laminar-flow safety cabinet or a similar enclosure with all the air going through the enclosure being drawn through a HEPA filter. This should minimize cross-contamination and maintain a safe work environment.

4.2 Low-power (5-60X) stereo binocular microscope with external light source for gross examination.

4.3 Forceps, dissecting needles, probes, scalpel or razor blades, etc. for manipulating bulk sample.

4.4 Crushing equipment.

    4.4.1 Mortar and pestle.

    4.4.2 At least one of the following:

        - mini-blender (approximately 30 ml capacity),

        - liquid-nitrogen mill, or

        - Wiley mill.

4.5 Filtration apparatus for polycarbonate filters.

    4.5.1 0.4 µm pore polycarbonate filters.

    4.5.2 Petri dishes and covers.

4.6 Muffle furnace capable of sustained operation at 500°C (with proportional temperature controller).

    4.6.1 Crucibles (bottom and lid) which can withstand 500°C.

    4.6.2 High-temperature thermometer with a range to at least 500°C and with readable subdivisions of 5°C or less.

4.7 Hydrochloric acid.

    4.7.1 37% by weight, reagent grade.

    4.7.2 10% by weight, reagent grade.

4.8 Heat lamp, hot plate or drying oven.

4.9 Desiccator.

4.10 Magnetic stirrer.

4.11 Distilled water.

4.12 Reference materials.

    4.12.1 U.S. National Institute of Standards and Technology (NIST) SRM 1866:

        - chrysotile,

        - amosite (grunerite), and

        - crocidolite (riebeckite).

    4.12.2 Non-NIST asbestos bulk standards must include:

        – fibrous anthophyllite,

        – fibrous tremolite, and

        – fibrous actinolite.

    4.12.3 The U.K. Institute of Occupational Medicine can be an alternative source for these asbestos reference materials.

4.13 Microscope slides and whole (>250 sq.mm.) coverslip.

4.14 Cargille refractive index liquids:
- n_D = 1.550 high dispersion,
- n_D = 1.605 high dispersion,
- n_D = 1.630 high dispersion,
- n_D = 1.680, and
- n_D = 1.700.

4.15 Marker for identifying slides.

4.16 Polarized light microscope with:
- 10X dispersion-staining objective, and
- eyepiece of at least 8X magnification containing a fixed cross-hair.

4.17 Balance with readability of 1 mg or less.

4.18 Analysis sheet with space for the following entries:
- analyst's signature,
- date of analysis,
- sample identification number,
- reference to the asbestos identification work sheet,
- type of crushing method (if any),
- matrix reduction – this should include organic reduction, acid dissolution, sedimentation or other steps used and amount of matrix removed during each step,
- weight of original sample and its residual weight after every matrix reduction procedure,
- number of asbestos points and non-asbestos points counted, and
- findings of the analysis.

5   *Asbestos Identification*

5.1   It is expected that analysts using this method are competent in the identification of asbestos by polarized light microscope. All asbestos components in the sample must be positively identified according to accredited identification procedures.

5.2   Deviation from typical properties may be observed for asbestos from atypical ores or, more frequently, for asbestos which has been altered chemically or thermally.

6   *Visual Estimation*

6.1   The sample should be of sufficient size to allow thorough examinations under a low-power stereo binocular microscope. The percentage of asbestos is determined by estimating the ratio of asbestos component to the non-asbestos component.

When the asbestos content is estimated to be more than 1%, the analysis may stop and the result be reported as

'The sample is found to be an asbestos containing material.'

6.2 When the asbestos content cannot be estimated visually or the asbestos content is estimated to be no more than 1%, proceed with the gravimetric and point-counting technique as described in the following sections.

7 *Sample Preparation*

7.1 The samples should be homogeneous and any subsampling should be at random.

7.2 Crushing.

7.2.1 The objective to crush the sample is to facilitate subsequent organic reduction and/or acid dissolution without causing unnecessary size reduction of both asbestos fibres and other ingredients. Not all samples will require crushing. Many are already homogeneous on a microscopic scale and others may simply require crushing with a mortar and pestle. Only a few would require homogenizing by mechanized methods such as by using a mini-blender, and that should be of short duration (approximately 15 seconds) to avoid significant reduction in fibre size.

7.2.2 Crushing must be performed on dry samples within the HEPA-filtered sample preparation area to prevent contamination of the overall work area.

7.2.3 Special care should be taken when crushing samples containing vermiculite or non-fibrous amphiboles: pulverization with a mill or mortar and pestle may produce asbestos-like fragments with aspect ratios greater than 3:1.

7.2.4 Care must be taken to rinse and dry equipment between sample preparations to prevent cross-contamination.

7.3 Matrix reduction.

7.3.1 Matrix removal is required whenever the matrices can be removed. For example, acid dissolution with concentrated hydrochloric acid can be used for sample containing calcite, gypsum, magnesite, brucite, bassanite, portlandite and dolomite. Diluted hydrochloric acid can be used for cementitious products. Organic reduction with muffle furnace can be used for sample containing vinyl, cellulose and other organic compound. Care must be taken when using acid or muffle furnace to minimize damage or alterations to chrysotile. The sample must be weighed before and after every matrix removal step so that the resultant asbestos percentage can be corrected to reflect its percentage in the

original material.

7.3.2 In general, the more material that can be removed and tracked gravimetrically, the fewer interferences will remain and the results should be more accurate and reliable. The steps outlined below must be followed to maximize detection and accurate quantification of asbestos. All weight determinations must be made to the nearest 1 mg or less.

7.3.3 Preliminary preparation.

(a) The sample must first be dried to constant weight inside the HEPA-ventilated, negative pressure sample preparation work area by a heat lamp, hot plate or drying oven.

(b) All extraneous materials should be removed before preparation is initiated. In the case of vinyl tiles, any mastic adhesive must be considered as a separate sample and must be removed for independent analysis.

7.3.4 Organic reduction.

(a) Take approximately 0.5-l g of sample or in the case of resinous product, shave off the same amount into a pre-weighed crucible and weigh. The weight of the subsample must be at least 200 times greater than the readability of the laboratory's analytical balance.

(b) Place in a muffle furnace at 485+/-l5°C for at least eight hours.

(c) Cool in a desiccator, reweigh and calculate percent organic loss.

7.3.5 Acid dissolution/Sedimentation.

(a) Weigh 0.5-1 g of sample. The weight of the subsample must be at least 200 times greater than the readability of the laboratory's analytical balance.

(b) Grind the sample with about 0.5-1 ml distilled water and add sufficient 10% hydrochloric acid to ensure dissolution of any carbonates present. It may be necessary to use concentrated hydrochloric acid for dissolution of dolomite or ankerite, but concentrated acid should not be used if mineral wool is present, because the products from the mineral wool are difficult to filter.

(c) Dilute to approximately 150 ml with distilled water and stir with a magnetic stirrer for about 15 minutes. Large mineral fragments and sand will settle rapidly. When stirring is stopped and avoiding the transfer of any of the large mineral fragments, decant the supernatant suspension into the filtration apparatus holding a fresh, pre-weighed 0.4 µm pore size polycarbonate filter. It is important that once wetted, the polycarbonate filter should remain wet throughout the filtration process.

(d) Add more distilled water to the original container, stir rapidly and decant the supernatant suspension into the filtration apparatus, again avoiding the transfer of the large mineral fragments. Repeat this procedure a second time.

(e) Transfer the filter and residue carefully to a clean, pre-weighed petri dish and allow the filter to dry to stable weight by using a heat lamp, hot plate or drying oven.

(f) Transfer any large mineral fragments remaining in the original container after decanting, to a petri dish, and allow to dry. The drying process can be accelerated by washing in ethanol and decanting. Examine this material under a binocular microscope to confirm that there are no large bundles of asbestos among the mineral fragments. Any large bundles of asbestos should be hand-picked from the sediment and from the residue on the filter, and placed into pre-weighed containers. If the weight of the large asbestos bundles is more than 1% of the original weight of the subsample, the analysis may be stopped and the sample reported as ACM. In any case, the weight of the large asbestos bundles should be accounted for in the calculation of asbestos concentration from the gravimetric data.

(g) Weigh the filter and residue in the petri dish and calculate the percent weight loss. If the residual weight and the weight of any large hand-picked asbestos bundles are no more than 1% of the original weight of the subsample, another two subsamples shall be prepared to receive the same treatment to determine their percent weight loss. When all the subsamples give a 'no more than 1%' residual weight, the analysis may be terminated and the sample reported as non-ACM.

7.4 Slide preparation.

7.4.1 After crushing and matrix reduction, subsamples must be mounted on clearly labelled microscope slides under separate coverslip. The number of subsamples required for analysis depends on the degree of matrix reduction (see column C of Table 1) and in any case, at least two subsamples shall be analyzed.

7.4.2 For each subsample, a small drop of Cargille liquid is placed on the slide. The refractive index of the Cargille should be chosen to give the maximum contrast e.g. 1.550 would be suitable for chrysotile.

7.4.3 A small sample from the homogenized material is removed using the edge of a scalpel blade. This sample must be taken at random, ie.

without preference to fibrous (or non-fibrous) material and without the aid of a stereo binocular microscope.

7.4.4 The small sample should be removed from a different spot of the homogenized material for each individual slide.

7.4.5 The sample is transferred to the mounting medium on the slide and dispersed evenly throughout the drop. One way to do it is by rubbing gently the preparation against another slide placed at 90° in a rapid shearing motion. Any 'spilled' mounting medium can be moved back to the middle of the slide using the edge of the second slide, and the process repeated several times. This usually produces a very uniform dispersion of particles.

7.4.6 A coverslip is placed on the preparation and more medium is added at the coverslip edge as necessary.

7.4.7 If the coverslip is raised obliquely because of large grains, the sample may require further sedimentation to remove the grains or crushing or milling to reduce grain sizes.

8 *Sample Quantification*

8.1 Point-counting criteria.

8.1.1 A point is the intersection of two mutually perpendicular lines ie. the cross-hair in the eyepiece reticle.

8.1.2 A non-empty point is the visual superimposition of a point over any material in the slide preparation. A non-empty point must be categorized as asbestos or as non-asbestos material. Empty points are those points which lie over areas containing no materials. Ideally, slide preparations should contain approximately 50% non-empty points.

8.1.3 Moving to new fields of view must be done at random, with the analyst looking away temporarily while moving the slide. The slide must never be deliberately moved to preferred fields of view under the reticle.

8.1.4 If the point lies over an area where particles are heavily clumped, the analyst should move the slide to a new field to avoid attempting to count multiple layers under a point. The maximum number of occasions that an analyst may reject a field because of the presence of a clump is 10, exceeding that the slide in question shall be discarded and replaced with a new one.

8.1.5 For the occasional superimposition of two particles under a point, the analyst should count both particles as separate points.

8.2   Counting rules.

8.2.1   Point-counting must be done on a polarized light microscope, usually with the slide between crossed polars and with a first-order red compensator inserted in the 45° port above the slide.

8.2.2   A standard objective without dispersion staining is preferred for point-counting. It gives a sharper image and allows closing down of sub-stage diaphragm to enhance contrast.

8.2.3   In some situations where extremely fine asbestos fibres are present, it may be preferable to analyze the sample between slightly uncrossed polars without the compensator. Other situations may warrant point-counting in a dispersion-staining mode.

8.2.4   All point-counting must be done at 100 X magnification although it will be advantageous at times to switch to higher magnification(s) for enhanced visualization of identification criteria.

8.2.5   The total non-empty points to be counted vary with the degree of matrix reduction of sample and they are given in column B of Table 1. The points shall distribute evenly among the slides but no more than 250 non-empty points shall be counted for each slide.

8.2.6   Point-counting can be stopped if more than 20 asbestos points are counted before the total non-empty points have been reached.

8.2.7   To account for statistical fluctuation in the number of asbestos points detected and in the homogeneity of subsample, the counts shall be compared against the one-sided, lower 95% confidence limit of the Poisson distribution. The total non-empty points given in Table 1 are meant to ensure that 95% of repeat measurements would correctly classify the sample. It is necessary to observe more than 20 asbestos points in the total non-empty points specified to demonstrate by point-counting the concentration of asbestos in the sample is more than 1%.

8.3   Analytical records. Detailed records must be kept for all phases of analysis. An analysis sheet that includes all the data required in Section 4.18 must be filled out completely, signed and dated by the analyst.

9   *Test Report*

Report to client must include at least the following:

9.1   A unique report identification number.

9.2   Client. Identify name and address.

9.3   Method. Detailed description of the steps taken for examination of the

sample(s).

9.4   Sample identity.   The identification number assigned by the laboratory must be clearly cross-referenced to field identification number and location provided by the sample collector.

9.5   Analytical results.   Each sample or layer quantified should be reported giving the following information: the percentage reduction of matrix, percentage of asbestos removed by hand-picking, the number of asbestos points and non-empty points counted, and the percentage of asbestos by point-counting together with the measurement uncertainty for the particular matrix.

10   *Interpretation of Results*

10.1   When the weight of hand-picked asbestos bundles in the subsample is more than 1% under the circumstances described in Section 7.3.5(f), report

'The sample is found to be an asbestos containing material.'

10.2   When the residual weights of all three subsamples are not more than 1% under the circumstances described in Section 7.3.5(g), report

'The sample is not classified as an asbestos containing material.'

10.3   If more than 20 asbestos points are counted in the total non-empty points specified in Section 8.2.5, report

'The sample is found to be an asbestos containing material.'

10.4   When no more than 20 asbestos points are counted in the total non-empty points specified in Section 8.2.5, the result should be reported as follows:

(a)   When no asbestos bundles have been hand-picked as described in paragraph 7.3.5(f), report

'The sample is not classified as an asbestos containing material.'

(b)   When large asbestos bundles of weight less than 1% of the original subsample have been hand-picked as described in paragraph 7.3.5(f), the concentration of asbestos shall be calculated in the following manner:

% asbestos =   % asbestos (hand-picked) +
                        % asbestos (point-counting)

where % asbestos (point-counting) =

$$\frac{\text{Number of asbestos points x } (1 - \% \text{ reduction})}{\text{Total non-empty points counted}}$$

(i)   If % asbestos is more than 1%, report

'The sample is found to be an asbestos containing material.'.

'The sample is not classified as an asbestos containing material.'.

Table 1

A	B	C	A	B	C	A	B	C	A	B	C
0	1325	6	25	994	4	50	663	3	75	331	2
1	1312	6	26	981	4	51	649	3	76	318	2
2	1299	6	27	967	4	52	636	3	77	305	2
3	1285	6	28	954	4	53	623	3	78	292	2
4	1272	6	29	941	4	54	610	3	79	278	2
5	1259	6	30	927	4	55	596	3	80	265	2
6	1246	5	31	914	4	56	583	3	81	252	2
7	1232	5	32	901	4	57	570	3	82	239	2
8	1219	5	33	888	4	58	557	3	83	225	2
9	1206	5	34	875	4	59	543	3	84	212	2
10	1193	5	35	861	4	60	530	3	85	199	2
11	1179	5	36	848	4	61	517	3	86	186	2
12	1166	5	37	835	4	62	504	3	87	172	2
13	1153	5	38	822	4	63	490	2	88	159	2
14	1140	5	39	808	4	64	477	2	89	146	2
15	1126	5	40	795	4	65	464	2	90	133	2
16	1113	5	41	782	4	66	451	2	91	119	2
17	1100	5	42	769	4	67	437	2	92	106	2
18	1087	5	43	755	4	68	424	2	93	93	2
19	1073	5	44	742	3	69	411	2	94	79	2
20	1060	5	45	729	3	70	398	2	95	66	2
21	1047	5	46	716	3	71	384	2	96	53	2
22	1034	5	47	702	3	72	371	2	97	40	2
23	1020	5	48	689	3	73	358	2	98	26	2
24	1007	5	49	676	3	74	345	2	99	20	2

Notes

Column A: Percentage reduction in weight of subsample given by

$$\frac{OW - FW}{OW} \times 100\%$$

Where   OW - original weight of subsample

FW - final weight of subsample

Column B: Number of total non-empty points to be counted.   The points shall be distributed evenly among the slides.

Column C*:* Number of slides required to be analyzed.

KS WONG *Secretary for the Environment*

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