## 1 SCOPE

1.1 This method describes a general procedure for the analysis of large asbestos fibres in consumer products, as per items 16 and 18 of Part I of Schedule I of the Hazardous Products Act.

1.2 When analysed under a stereo microscope, asbestos fibres have a distinct appearance and can be isolated from the other components of a mixture. They are identified by infrared spectrophotometry.

## 2 APPLICABLE DOCUMENTS

2.1 Coates, J.P., IR analysis of toxic dusts. Analysis of collected samples of quartz and asbestos. Part 1. American Laboratory, 9 (11), November 1977. pp 105-111.

2.2 Coates, J.P., IR analysis of toxic dusts. Analysis of collected samples of asbestos. Part 2. American Laboratory, 9 (12), December 1977. pp 57-65.

2.3 Luoma, G.A., Yee, L.K. and Rowland, R. Determination of microgram amounts of asbestos in mixtures by infrared spectrometry. Anal. Chem. 54, 1982, pp 2140-2142.

2.4 Project report 99-0492 “Update of Method C-26 of the Product Safety Laboratory Test Methods Manual (Book 5).”

2.5 B. Séguin , “Revision of Method C26 - Detection and identification of various asbestos fibres in consumer products”, Health Canada, PSL, Project report no. 2002-0711, 2003.

## 3 REAGENTS AND APPARATUS

3.1 Stereo Microscope
3.2 Fine mesh sieve (Diameter: 5 cm; Mesh openings: 250 microns)
3.3 Tapered tweezers
3.4 Infrared spectrophotometer equipped with Fourier Transform acquisition system
3.5 Tetrahydrofuran, HPLC Grade
3.6 Acetone, A.C.S Certified
3.7 Potassium bromide (KBr), IR grade
3.8 Hydraulic press used for preparing KBr pellets
3.9 NIST 1866a - Asbestos standards (e.g., chrysotile, crocidolite, amosite, tremolite and anthophyllite)
3.10 KBr die kit
3.11 Polystyrene NIST standards

## 4 EXPERIMENTAL PROCEDURE

### 4.1 Sample preparation

4.1.1 Powdered samples

Sieve the sample in order to remove the fine particles. Look at the sieve and the fine residual matter under the stereo microscope. Using tapered tweezers to remove the largest agglomerates of fibres and dry them in a drying oven for one hour at 100˚C. Analyse the sample as per section 4.2 to 4.4.

4.1.2 Agglomerated samples

Dissolve a fragment of the sample in approximately 25 mL of an appropriate solvent (e.g., tetrahydrofuran) for a period of 1 hour. Filter the sample and rinse. Dry the residual matter and transfer the contents onto a fine mesh copper sieve. Rinse with the solvent and dry in a drying oven for one hour at 100°C. Under the stereo microscope, remove sample asbestos fibres using the tweezers. Deposit the fibres in a sample bottle. Analyse the sample as per section 4.2 to 4.4.

4.1.3 Semi-liquid samples

Filter the sample on a filter paper (Whatman #40). Rince with solvent (e.g. acetone) and dry in a drying oven for one hour at 100˚C. Sieve the sample as per section 4.1.1. Analyse the sample as per section 4.2 to 4.4.

4.1.4 Asbestos sheet

Remove a few fibres with tweezers and place them in a sample bottle. Analyse the sample as per section 4.2 to 4.4.

4.1.5 Asbestos textiles

Remove a few fibres using tweezers. Place them in a sample bottle and analyse the sample as per section 4.2 to 4.4.

### 4.2 Disc preparation

For better results, drying of KBr powder is best done by leaving it in a shallow dish in an oven at 120˚C for approximately 24 hours. It may then be transferred in a bottle and kept in a dessicator.

The preparation of a disc without adding any sample is prepared to check the background signal.

4.2.1 Preparation of a pellet

- Place 3 micro-spatula scoops (approximately 0.225 g) of KBr in the mortar. Smash and mix the mixture using the mortar and pestle.
- Use the KBr die kit (See Appendix) to form a disc. Before preparing the discs, make sure that all parts of the die assembly are clean and dry. Use a Kimwipe or other suitable tissue and water to clean them.
- Assemble the base and cylinder.
- Place one of the pellets polished face up into the cylinder.
- Place a quantity of powder into the cylinder, enough to cover the pellet.
- Distribute the powder as evenly as possible by lightly shaking.
- Drop the top pellet into the cylinder (polished surface down) and press down lightly with the plunger.
- Place the die under the pellet press and adjust the height by turning the black wheel at the surface of the press.
- Operate the handle until the pressure is 5 tons, wait 5 minutes.
- Release the pressure slowly to zero.
- Operate the handle until the pressure is 10 tons with the handle and wait an additional 10 minutes.
- Release the pressure slowly to zero.
- Remove the die from the press
- Invert and support the rest of the assembly on the plunger
- Remove the base slowly
- On a counter, slowly apply a pressure on the plunger until it moves up through the cylinder lifting the lower pellet and the KBr disc clear of cylinder
- Place the disc on the support for analysis

**Note:** It is advisable to handle the discs with tweezers, NEVER touch disc with bare hands.

4.2.2 Preparation of a standard disc

- Mix the standard with some KBr in an approximate proportion of 1 part standard: 3 parts KBr (1:3) and follow the instructions of section 4.2

4.2.3 Preparation of a sample disc

- Mix the sample with some KBr in a approximate proportion of 1 part sample: 3 parts KBr (1:3) and follow the instructions of section 4.2

### 4.3 FT-IR Instrument set-up parameters

Select the following experiment "C26 - Detection of asbestos" on the FT-IR which included all parameters below.

- Number of scans: 32
- Resolution: 4
- Spectral Range: 4000 to 400
- Source: IR
- Detector: DTGS KBr
- Beamsplitter: KBr
- Collect a new background each 60 minutes

**Note:** If you obtain a spectra with a percent transmittance lower than 60%, you should remake your disc with less mixture of sample and KBr by following the step 4.2 and 4.3. When the percent transmittance is low, it means that your disc is too opaque.

### 4.4 Analysis of sample

Analyse the disc using the FT-IR. Be sure to make all controls of the instruments before every analysis and complete the log book.

## 5 REPORTING

5.1 Obtain an infrared spectrum of the sample. Refer to Table 1 for identification, and compare the spectrum obtained with the spectrum of an appropriate standard.

Example of a spectra: NIST 1866a (Chrysotile)

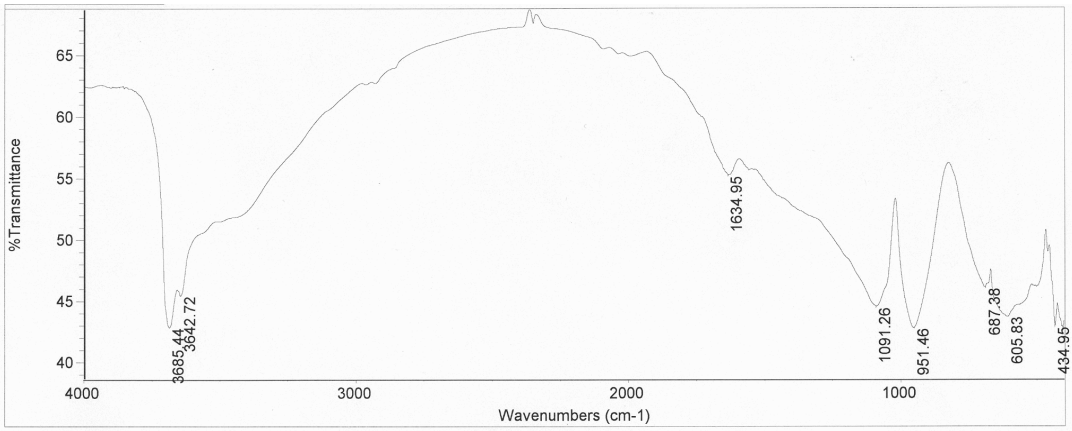
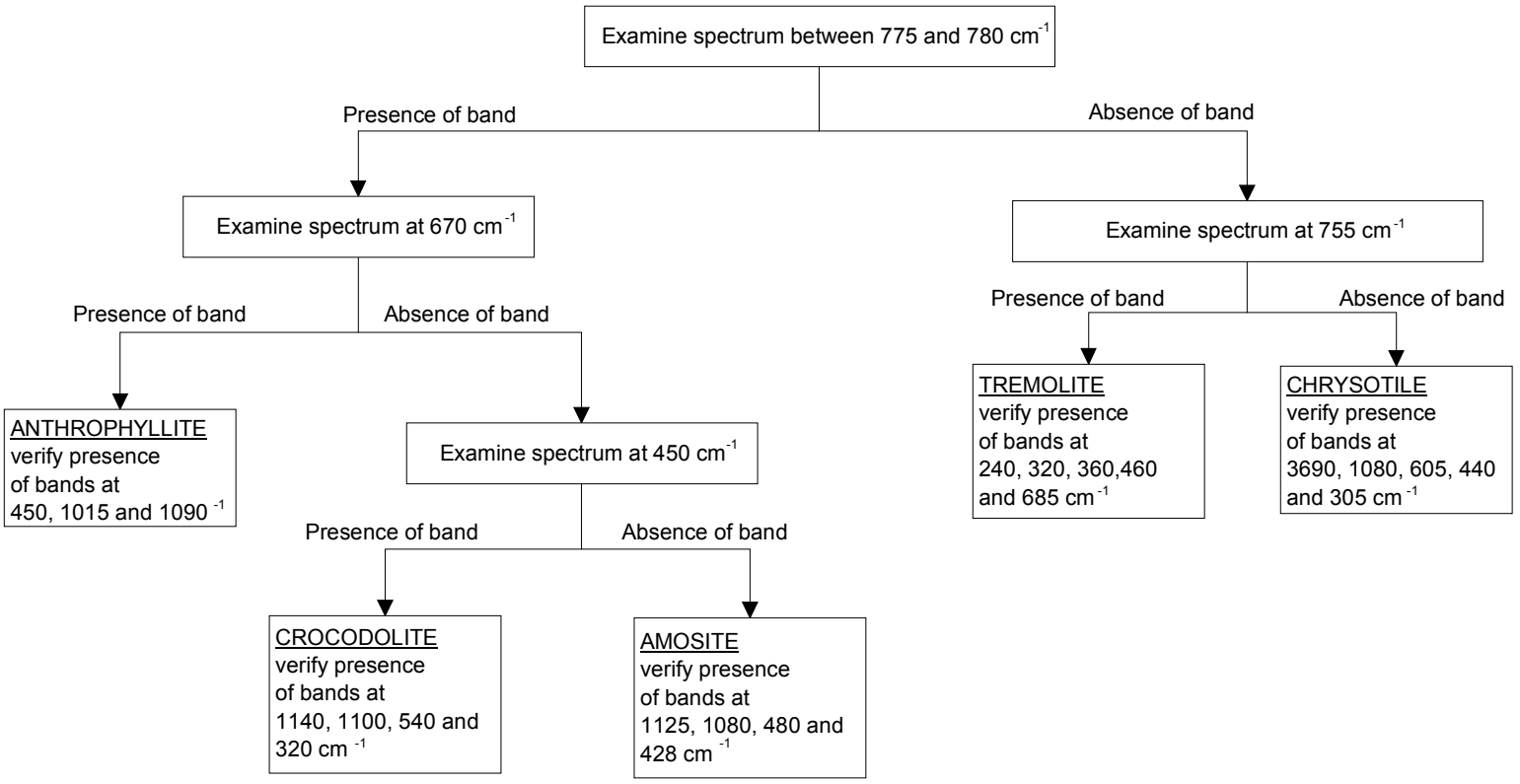


Table 1: Determination of asbestos fibres by infrared analysis.



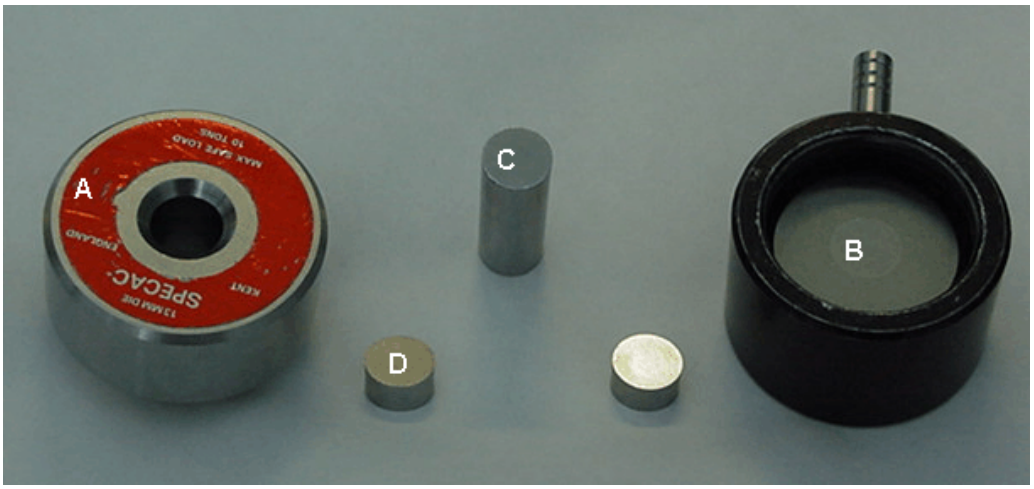

## 6 QUALITY CONTROL PROCEDURE

6.1 The normal and correct operation of the spectrometer shall be verified according to the following guideline:

6.1.1 Run the system validation of the instrument using the polystyrene NIST standards 1.5 mil and 3.0 mil. If the results pass all the validation tests, the system validation report shall be included in the log book. Also, all the different section of the log book should be complete. If the spectrometer failed the validation tests, the instrument shall be checked or repaired and the validation tests shall be ran again to meet the requirements prescribed by the manufacturer.

**APPENDIX**

Figure 1: 13 mm KBr dies parts (Max Safe Load of 10 tons)




A: Cylinder
B: Base
C: Plunger
D: Two Pellets with a polished face

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