

**CERTIFICATE OF ANALYSIS**

**ANALYSIS REQUESTED BY:** Mr Paul Spragg  
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**SAMPLES:** Two bulk samples of vermiculite.

**ANALYSIS REQUESTED:** Determination of the presence of asbestiform fibre content by polarised light microscopy, chemical digestion and Scanning Electron Microscopy.

**METHOD:**

1. **Sample Preparation:** Approximately 1.5 grams of each vermiculite sample was accurately weighed into a porcelain crucible, exfoliated in an oven, allowed to cool and reweighed. The vermiculite was removed by digestion in a reflux condenser with 2M H<sub>2</sub>SO<sub>4</sub>, followed by 4M NaOH. The residue was collected by filtration and reweighed. (Addison and Davies, 1990).

This process extracts vermiculite, chlorite, chrysotile and other minerals, but leaves amphiboles, feldspar, quartz, etc. effectively unaltered. The residue was divided, weighed, and sub-samples prepared as aqueous suspensions from which aliquots were deposited on to 25mm 0.2µm pore size polycarbonate filters for analysis by scanning electron microscopy.

2. **Scanning Electron Microscopy (SEM):** Segments were cut from the prepared filter, mounted on to standard SEM sample stubs and coated with gold. A standard search at 2000X magnification was carried out and all particles with morphology and elemental compositions consistent with amphibole asbestos (length >1.0 µm, aspect ratio >3:1) were counted and measured and the volume and mass concentrations were calculated. A density of 0.003 ng/µm<sup>3</sup> has been assumed for these calculations. An area of 1mm<sup>2</sup> was evaluated or 50 fibrous particles counted.

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**RESEARCH CONSULTING SERVICES**

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**METHODS (CONT):**

3. **Polarised Light Microscopy (PLM):** The undigested vermiculite and a portion of the digestion residue for each sample was mounted in appropriate refractive index liquids (1.605 – 1.640) and examined for the presence of asbestiform fibres at 125x magnification using PLM. The UK Health and Safety Executive method HSG 248 (HSE, 2005) was followed.

**RESULTS:** A summary of results is given in Table 1.

No asbestos fibres were detected in the original bulk material or digestion residues of either of the samples by PLM.

Following chemical digestion, no asbestos fibres were detected in the residue of either of the original vermiculite samples by SEM.

The detection limit for SEM analysis is approximately 0.0001% in the original vermiculite.

**COMMENTS:**

No amphibole asbestos fibres (<0.0001%) were detected in either of the samples by Scanning Electron Microscopy.

No asbestos fibres were detected in the original bulk samples or digestion residues by polarised light microscopy.

IOM Consulting cannot accept responsibility for samples, which have been incorrectly collected or despatched.

ANALYSED BY: *S Clark*

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TABLE 1. Summary of Results

| Sample ID                    | Wt. Before Heat (g) | Wt. After Digestion (g) | SEM Mass % Amphibole Asbestos Fibres |            |
|------------------------------|---------------------|-------------------------|--------------------------------------|------------|
|                              |                     |                         | In Residue                           | In Bulk    |
| Micron Crude-January 2008    | 1.12424             | 0.06614 (5.88%)         | ND<0.001*                            | ND<0.0001* |
| Superfine Crude-January 2008 | 1.25625             | 0.05753 (4.58%)         | ND<0.001*                            | ND<0.0001* |

ND - Not Detected

\* These detection limits are approximations, based on previous analysis where fibres have been detected.