



Report to Client

## **Sampling and Analysis of Crude Vermiculite Samples For Possible Asbestiform Fibre and Quartz Content**

For

**Mr M Darling,  
Palabora Europe Limited  
1A Guildford Business Park  
Guildford  
Surrey  
GU2 8XG**

**CONTRACT NO: 00943-1  
PROJECT NO: 609-00324(610)  
DATE OF ISSUE: 30.12.03**

**Report Prepared by: Stephen R Klek, BSc(Hons), CEng, MIMMM, MIOSH, MIOA.  
Senior consultant**

A handwritten signature in blue ink, appearing to read 'SR Klek', is positioned below the name of the report preparer.

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Office manager**

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## **1. INTRODUCTION**

Palabora Europe Limited is the European distributors of vermiculite produced by the Palabora Mining Company from open cast mines at Phalaborwa, Transvaal, South Africa.

In order to address concern on Health and Safety issues in the world-wide market for vermiculite, Palabora Europe have previously commissioned IOM Consulting to undertake a detailed analysis of vermiculite from the main PP & V ore body at Phalaborwa and one grade from the adjacent VODT deposit. This original study produced favourable results and has been followed up by routine screening of stocks.

The main aims were to collect representative samples from each grade of vermiculite as available in the European market, split them and analyse them at the IOM Consulting's Edinburgh headquarters for asbestiform mineral and crystalline silica.

## **2. PROCEDURE**

Mr Steve Klek of IOM Consulting and Mr Mike Darling of Palabora Europe Limited visited the Palabora Europe Ltd vermiculite bulk store at North Killingholme Humberside, on 2nd December 2003. At the North Killingholme site, there are normally six different grades of vermiculite stored inside large silos of approximately 27 metres diameter and 9 metres high (micron PP & V, superfine PP & V, fine PP & V, medium PP & V, large PP & V and superfine VODT). At the time of this exercise there were only five types in stock, there being no Superfine VODT.

Samples were collected from each grade as representatively as possible, by clearing away loose dust from the top of the material and digging into the stock as far as possible, avoiding any material falling in and re-mixing. A number of sub samples per site were extracted and mixed from each stockpile. Each sample was split on site with one sample being retained by Palabora Europe and the rest by IOM Consulting.

## **3. METHODS OF ANALYSIS**

### **3.1 Asbestos**

The main objective was to identify any hazardous asbestos fibres present in the vermiculite. This was achieved as follows:

- i) Initial examination by stereo-binocular microscopy and polarised light microscopy using methods described in HSE document MDHS 77 (HSE, 1994a).

A portion of each sample was examined for the presence of fibrous asbestos minerals at X8 – X40 magnification, using stereo-binocular microscopy. Any fibres detected, were mounted in appropriate refractive liquid and identified at 125X magnification using Polarised Light Microscopy (PLM) and dispersion staining microscopy.

This part of this analysis was carried out under IOM's external quality accreditation, awarded by the United Kingdom Accreditation Service (UKAS).

- ii) Quantitative assessment of amphibole asbestos by electron microscopy and X-ray diffractometry would normally then be carried out for samples in which asbestos fibres were detected in (i) above. In this instance, this was not done as there were no fibres identified.

### 3.2 Crystalline silica

A portion of each sample was ground up and analysed using X-ray diffraction techniques using modified versions of MDHS 51/2 (HSE 1988) and MDHS 76 (HSE 1994). Additional preparation of the Superfine PP&V sample was done to achieve a lower detection limit. Detailed method description is shown in the certificate of analysis in Appendix 1.

## 4 RESULTS

- 4.1 Polarised Light Microscopy examinations did not detect any amphibole or chrysotile asbestos fibres present in any of the samples of vermiculite. As there were no amphibole or chrysotile asbestos fibres detected in any of the samples of vermiculite collected, quantitative analysis of the vermiculite materials was not undertaken.
- 4.2 X-ray diffractometry did not detect any crystalline silica in any of the samples of vermiculite.
- 4.3 A summary of results is given in Table 1. Certificates of Analysis are attached (Appendix 1).

**Table 1. Summary of Results**

<b>IOM Sample No.</b>	<b>Description</b>	<b>Asbestos</b>	<b>Crystalline silica</b>
R763	Medium (PP & V)	None Detected	None Detected *
R764	Micron (PP & V)	None Detected	None Detected *
R765	Fine (PP & V)	None Detected	None Detected *
R766	Large (PP & V)	None Detected	None Detected *
R767	Superfine (PP & V)	None Detected	None Detected *

\*The detection limit for quartz and cristobalite by this method is around 0.3%. This figure is based upon three times the standard deviation of the measurement of a blank sample run on a quantification programme. Previously, the detection limit was based upon the lowest amount, which could be detected on a qualitative programme, which was 0.1%. Although this has not changed, we are now required by the United Kingdom Accreditation Service (UKAS) to base our detection limit on the quantitative rather than the qualitative part of the analysis.

## 5. CONCLUSIONS

- 5.1 The vermiculite materials tested comply with the proposed 0.1% European packaging and labelling of carcinogen requirements (HSE, 1994b) and the 0.1% trigger value for asbestos required for labelling of hazardous materials in the US.

The levels are also lower than the 0.001% for asbestos in loose aggregates proposed by Addison *et al* (1988) based on work at the IOM using asbestos and soil mixtures. Therefore, it is concluded that these materials or products containing them should not present a significant asbestos-related health hazard when used in controlled occupational environment.

- 5.2. Similarly with there being no crystalline silica detected in the samples and in consideration of the detection limits of the analysis, there should not be any health hazard, specifically related to crystalline silica, when the materials are used in a controlled environment.

Considering an airborne total inhalable dust concentration of 10 mg/m<sup>3</sup>, based on the higher limit of detection quoted in the certificate of analysis of <0.3%, the concentration of crystalline silica would be <0.03 mg/m<sup>3</sup>; based on the lower limit of detection achieved for the Superfine PP&V sample, the airborne crystalline silica concentration would be 0.003 mg/m<sup>3</sup>. This is in comparison with the current Maximum Exposure Limit of 0.3 mg/m<sup>3</sup> (8 hour TWA) given in HSE document EH40/2002.

## **REFERENCES:**

Addison J, Davies LST. (1990). Analysis of amphibole asbestos in chrysotile and other minerals. *Ann.Occ.Hyg* ; 34: 159-175.

Health and Safety Executive (1987). Quartz in Respirable Airborne Dusts. MDHS 37. HMSO London.

Health and Safety Executive (1998). Occupational Exposure Limits 1998. EH40/98. HSE Books, Sudbury.

Chung (1974). Quantitative Interpretation of X-ray Diffraction patterns of Mixtures. III Simultaneous Determination of a set of Reference Intensities. Sherwin-Williams Research Centre, Chicago, Illinois.

Health and Safety Executive (1988). "Quartz in respirable airborne dusts". Laboratory method using x-ray diffraction (direct method). Methods for the Determination of Hazardous Substances No. 51/2. HMSO, London.

Health and Safety Executive (1994). "Cristobalite in respirable airborne dusts". Laboratory method using X-ray diffraction (direct method). Methods for the Determination of Hazardous Substances No. 76. HMSO, London.

## **Appendix 1**

### **Certificates of Analysis**

## CERTIFICATE OF ANALYSIS FIBRE IDENTIFICATION IN BULK MATERIAL

**Client Details:** Palabora Europe Ltd., 1A Guildford Business Park  
Guildford, Surrey GU2 8XG

**Contract No:** 00943-1

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 5.12.03.

**Date of Issue:** 17.12.03

**Date of Analysis:** 16.12.03

The samples detailed below have been analysed qualitatively for asbestos by polarised light and dispersion staining as described by the Health and Safety Executive in MDHS 77. The results are given below:

IOM No.	Clients Sample No.	Sample Details	Asbestos Type(s) Present
R763	SILO 915	<i>CRUDE VERMICULITE SAMPLE:</i>  MEDIUM GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

The Institute of Occupational Medicine accepts responsibility only for results obtained from samples as received. No responsibility is accepted for errors, which may have arisen during sampling or transportation of samples by external clients.

**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

*Sheet 1 of 1*

## CERTIFICATE OF ANALYSIS FIBRE IDENTIFICATION IN BULK MATERIAL

**Client Details:** Palabora Europe Ltd., 1A Guildford Business Park  
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**Contract No:** 00943-2

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 5.12.03

**Date of Issue:** 17.12.03

**Date of Analysis:** 16.12.03

The samples detailed below have been analysed qualitatively for asbestos by polarised light and dispersion staining as described by the Health and Safety Executive in MDHS 77. The results are given below:

IOM No.	Clients Sample No.	Sample Details	Asbestos Type(s) Present
R764	SILO 924	<i>CRUDE VERMICULITE SAMPLE:</i> MICRON GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

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**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

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## CERTIFICATE OF ANALYSIS FIBRE IDENTIFICATION IN BULK MATERIAL

**Client Details:** Palabora Europe Ltd., 1A Guildford Business Park  
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**Contract No:** 00943-3

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 5.12.03

**Date of Issue:** 17.12.03

**Date of Analysis:** 16.12.03

The samples detailed below have been analysed qualitatively for asbestos by polarised light and dispersion staining as described by the Health and Safety Executive in MDHS 77. The results are given below:

IOM No.	Clients Sample No.	Sample Details	Asbestos Type(s) Present
R765	SILO 906	<i>CRUDE VERMICULITE SAMPLE:</i> FINE GRADE PP+V	NONE DETECTED

**No. of Samples:** ONE

The Institute of Occupational Medicine accepts responsibility only for results obtained from samples as received. No responsibility is accepted for errors, which may have arisen during sampling or transportation of samples by external clients.

**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

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## CERTIFICATE OF ANALYSIS FIBRE IDENTIFICATION IN BULK MATERIAL

**Client Details:** Palabora Europe Ltd., 1A Guildford Business Park  
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**Contract No:** 00943-4

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 5.12.03

**Date of Issue:** 17.12.03

**Date of Analysis:** 16.12.03

The samples detailed below have been analysed qualitatively for asbestos by polarised light and dispersion staining as described by the Health and Safety Executive in MDHS 77. The results are given below:

IOM No.	Clients Sample No.	Sample Details	Asbestos Type(s) Present
R766	SILO 915	<i>CRUDE VERMICULITE SAMPLE:</i> LARGE GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

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**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



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## CERTIFICATE OF ANALYSIS FIBRE IDENTIFICATION IN BULK MATERIAL

**Client Details:** Palabora Europe Ltd., 1A Guildford Business Park  
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**Contract No:** 00943-5

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 5.12.03

**Date of Issue:** 17.12.03

**Date of Analysis:** 16.12.03

The samples detailed below have been analysed qualitatively for asbestos by polarised light and dispersion staining as described by the Health and Safety Executive in MDHS 77. The results are given below:

IOM No.	Clients Sample No.	Sample Details	Asbestos Type(s) Present
R767	SILO 932	<i>CRUDE VERMICULITE SAMPLE:</i> SUPERFINE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

The Institute of Occupational Medicine accepts responsibility only for results obtained from samples as received. No responsibility is accepted for errors, which may have arisen during sampling or transportation of samples by external clients.

**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

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## CERTIFICATE OF ANALYSIS

**ANALYSIS REQUESTED BY:** Mr M Darling  
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Guildford  
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**CONTRACT NO:** 00943-6  
**PROJECT NO:** 610  
**DATE OF ISSUE:** 16.12.03

**ANALYSIS REQUESTED:** Crystalline silica content of five bulk vermiculite samples, with further more detailed analysis of the Superfine (PP&V) sample.

**SAMPLES:** Five bulk samples of Palabora vermiculite (see Table 1)

### METHOD:

Portions of the samples were ground up to a uniform size then scanned qualitatively using routine X-ray diffraction techniques. The resultant diffraction patterns were then matched with those of standard minerals (quartz, cristobalite etc.) to determine the presence of crystalline silica in the samples. The method followed was as described by Chung (1974).

One of the samples (Superfine PP&V) was subjected to additional sample preparation, in order to achieve a lower detection limit. For this sample, approximately 1.5 grams of the vermiculite was accurately weighed into a porcelain crucible, exfoliated in an oven, allowed to cool and re-weighed. The vermiculite was removed by digestion in a re-flux condenser with 2M H<sub>2</sub>SO<sub>4</sub>, followed by 4M NaOH. The residue was collected by filtration and re-weighed (Addison and Davies, 1990). This process extracts vermiculite, chlorite, chrysotile and other minerals, but leaves amphiboles, feldspar, quartz, etc. effectively unaltered. Aqueous suspensions were prepared from which aliquots were deposited on to 25mm 0.4µm pore size polycarbonate filters for analysis by X-ray diffractometry for crystalline silica content. Samples were analysed using modified versions of MDHS 51/2 (HSE 1988) and MDHS 76 (HSE 1994).

*Page 1 of 3*

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**CONTRACT NO:** 00943-6

**RESULTS: Crystalline silica in bulk vermiculite.**

Vermiculite Grade	% Cristobalite	% Quartz
Superfine (PP&V)	ND<0.3*	ND<0.3*
Fine (PP&V)	ND<0.3*	ND<0.3*
Micron (PP&V)	ND<0.3*	ND<0.3*
Medium (PP&V)	ND<0.3*	ND<0.3*
Large (PP&V)	ND<0.3*	ND<0.3*

\*The detection limit for quartz and cristobalite by this method is around 0.3%. This figure is based upon three times the standard deviation of the measurement of a blank sample run on a quantification programme. Previously, the detection limit was based upon the lowest amount, which could be detected on a qualitative programme, which was 0.1%. Although this has not changed, we are now required by the United Kingdom Accreditation Service (UKAS) to base our detection limit on the quantitative rather than the qualitative part of the analysis.

**Crystalline silica content of the Superfine PP&V, after chemical digestion.**

Sample	Original Weight (g)	Weight After Digestion (g)	Percentage Cristobalite	Percentage Quartz
Superfine PP&V	1.50608	0.11634 (7.7%)	ND<0.03*	ND<0.03*

ND - Not Detected

\* - Detection Limit. The detection limit for crystalline silica on filter by XRD is 0.01 mg. The detection limits quoted for the sample above is based upon 0.01 mg expressed as a percentage of the weight of dust on the sample filter. The detection limits therefore vary as a function of the weight of dust on the filter and weights of the original samples and digestion residues.

**COMMENTS:**

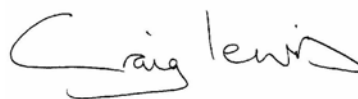
No crystalline silica was detected in any of the bulk samples or the sample prepared by chemical digestion.

ANALYSED BY:



**S Clark**  
Senior Scientific Technician

AUTHORISED BY:



**C Lewis**  
Chemist

**CONTRACT NO:** 00943-6

**PROJECT NO:** 610

**DATE OF ISSUE:** 16.12.03

**REFERENCES:**

Addison J, Davies LST. (1990). Analysis of amphibole asbestos in chrysotile and other minerals. *Ann.Occ.hyg.*; 34: 159-175.

Health and Safety Executive (1987). Quartz in Respirable Airborne Dusts. MDHS 37. HMSO London.

Health and Safety Executive (1998). Occupational Exposure Limits 1998. EH40/98. HSE Books, Sudbury.

Chung (1974). Quantitative Interpretation of X-ray Diffraction patterns of Mixtures. III Simultaneous Determination of a set of Reference Intensities. Sherwin-Williams Research Centre, Chicago, Illinois.

Health and Safety Executive (1988). "Quartz in respirable airborne dusts". Laboratory method using x-ray diffraction (direct method). Methods for the Determination of Hazardous Substances No. 51/2. HMSO, London.

Health and Safety Executive (1994). "Cristobalite in respirable airborne dusts". Laboratory method using X-ray diffraction (direct method). Methods for the Determination of Hazardous Substances No. 76. HMSO, London.