



Report to Client

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

For

**Mr M Darling,  
Palabora Europe Limited  
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Guildford  
Surrey  
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**CONTRACT NO: 609-00473  
DATE OF ISSUE: 20.09.04**

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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, Limpopo Province, 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. 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 6<sup>th</sup> July 2004. All samples were collected by Steve Klek of IOM Consulting. At the North Killingholme site, there are normally five 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 and Large PP & V). At the time of this exercise there were five grades in stock and the approximate stocks at the time of sampling are summarised in Table 1.

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 grade 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.

In addition to the samples collected by IOM Consulting, two samples of Micron Grade PP&V vermiculite were supplied by Palabora Europe for crystalline silica analysis. The samples identified as "North Killingholme, Silo 912 10/01/2001" and "MV Marylaki, Rotterdam 04/12/2003", were used to compare past levels of crystalline silica with current levels.

**Table 1 Details of Crude Vermiculite Stocks at North Killingholme on 6<sup>th</sup> July 2004**

Vermiculite Grade	Silo	Approx. Stock on 06.07.04 (tonnes)
Micron (PP & V)	924	1130
Superfine (PP & V)	932	1748
Fine (PP & V)	906	844
Medium (PP & V)	915	286
Large (PP & V)	915	335

### **3. METHODS OF ANALYSIS**

#### **3.1 Asbestos**

The main objective was to identify any hazardous asbestos fibres present in the samples of vermiculite collected by IOM Consulting. 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 magnifications, 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 the 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 July 2004 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 2004 Micron PP&V, 2004 Fine PP&V, 2001 Micron PP&V and 2003 Rotterdam Micron PP&V samples was carried out to achieve a lower detection limit. Detailed method descriptions are shown in the certificates 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 of the July 2004 bulk materials did not detect any crystalline silica in any of the samples of vermiculite.
- 4.3. For the samples subjected to chemical digestion prior to analysis by XRD, no cristobalite was detected in any of the samples and quartz was detected in all four samples as follows: Micron PP&V (2004) 0.12%, Fine PP&V (2004) 0.07%, Micron PP&V (2001) 0.05% and Rotterdam Micron PP&V (2003) 0.04% quartz.

- 4.3 A summary of results is given in Table 2 below. Certificates of Analysis are attached (Appendix 1).

**Table 2a Summary of Results 2004 Sampling Exercise**

<b>Silo Number</b>	<b>Description</b>	<b>Asbestos</b>	<b>Crystalline Silica</b>
915	Large (PP & V)	None Detected	None Detected
915	Medium (PP & V)	None Detected	None Detected
906	Fine (PP & V)	None Detected	None Detected
932	Superfine (PP & V)	None Detected	None Detected
924	Micron (PP & V)	None Detected	None Detected

**Table 2b Quartz Comparison Exercise**

<b>Sample Description</b>	<b>Cristobalite %</b>	<b>Quartz %</b>
2004 Killingholme Micron (PP & V)	None Detected	0.12
2004 Killingholme Fine (PP & V)	None Detected	0.07
2001 Killingholme Micron (PP & V)	None Detected	0.05
2003 Rotterdam Micron (PP & V)	None Detected	0.04

## 5. CONCLUSIONS

- 5.1 The vermiculite materials tested comply with the proposed 0.1% European packaging and labelling of carcinogen (asbestos) 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 five bulk 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.
- 5.3 The comparison exercise for quartz indicated that small amounts of crystalline quartz are present in current stocks of Micron and Fine Grade PP&V vermiculite, albeit at low levels (close to the detection limit for the method). Comparison with historical samples of Micron Grade PP&V vermiculite shows that current levels have slightly higher levels of quartz than those of the 2001 and 2003 samples.

**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:** 002280-1

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 14.07.04.

**Date of Issue:** 29.07.04

**Date of Analysis:** 20.07.04

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
R8202	SILO 915	<i>CRUDE VERMICULITE SAMPLE:</i>  LARGE GRADE PP+V	NONE DETECTED

**No. of Samples:** ONE

**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:** 002280-2

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 14.07.04.

**Date of Issue:** 29.07.04

**Date of Analysis:** 20.07.04

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
R8203	SILO 915	<i>CRUDE VERMICULITE SAMPLE:</i> MEDIUM GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

**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:** 002280-3

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 14.07.04.

**Date of Issue:** 29.07.04

**Date of Analysis:** 20.07.04

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
R8204	SILO 906	<i>CRUDE VERMICULITE SAMPLE:</i> FINE GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

*Sheet 1 of 1*

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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:** 002280-4

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 14.07.04.

**Date of Issue:** 29.07.04

**Date of Analysis:** 20.07.04

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
R8205	SILO 932	<i>CRUDE VERMICULITE SAMPLE:</i> SUPERFINE GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

**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  
Guildford, Surrey GU2 8XG

**Contract No:** 002280-5

**Requested By:** Mike Darling

**Project No:** 610

**Date Samples Received:** 14.07.04.

**Date of Issue:** 29.07.04

**Date of Analysis:** 20.07.04

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
R8206	SILO 924	<i>CRUDE VERMICULITE SAMPLE:</i> MICRON GRADE PP+V	NONE DETECTED
<b>No. of Samples: ONE</b>			

**Analysed by:**



T Sodergren  
Scientific Technician

**Authorised by:**



S Clark  
Senior Scientific Technician

*Sheet 1 of 1*

## CERTIFICATE OF ANALYSIS

**ANALYSIS REQUESTED BY:** Mr M Darling  
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**CONTRACT NO:** 02280-6  
**PROJECT NO:** 610  
**DATE OF ISSUE:** 29.07.04

**ANALYSIS REQUESTED:** Crystalline silica content of five bulk vermiculite samples, with further more detailed analysis of the Micron (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 (Micron 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).

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

<b>Vermiculite Grade</b>	<b>% Cristobalite</b>	<b>% Quartz</b>
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 Micron PP&V, after chemical digestion.**

<b>Sample</b>	<b>Original Weight (g)</b>	<b>Weight After Digestion (g)</b>	<b>Percentage Cristobalite</b>	<b>Percentage Quartz</b>
Micron PP&V	1.45166	0.08754 (6.0%)	ND<0.02*	0.12 <sup>1</sup>

ND - Not Detected

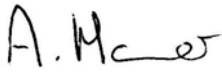
\* - Detection Limit. Our current detection limit for crystalline silica on filter by XRD is 0.02mg. The detection limits quoted for the sample above are based upon 0.02 mg expressed as a percentage of the weight of dust on the sample filter. The detection limits therefore vary as a function of the weight of dust on the filter and weights of the original samples and digestion residues.

<sup>1</sup> As a result of mineral interference the result for this sample was based upon visual comparison of calibration data with sample data.

**CONTRACT NO:** 02280-6  
**PROJECT NO:** 610  
**DATE OF ISSUE:** 29.07.04

**COMMENTS:**

1. No crystalline silica was detected in any of the bulk samples.
2. Quartz was detected in the sample of Micron PP&V (0.12%) prepared by chemical digestion.
3. The digestion of Micron PP&V and subsequent analysis by XRD is not covered by UKAS accreditation.
4. Opinions and interpretations are outside the scope of our UKAS accreditation.

**ANALYSED BY:**   
**A Hamer**  
Scientific Technician

**AUTHORISED BY:**   
**S Clark**  
Senior Scientific Technician

## **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.

## CERTIFICATE OF ANALYSIS

**ANALYSIS REQUESTED BY:** Mr M Darling  
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GU2 8XG

**CONTRACT NO:** 02437  
**PROJECT NO:** 610  
**DATE OF ISSUE:** 13.08.04

**ANALYSIS REQUESTED:** Detailed analysis of one vermiculite sample for crystalline silica content – Fine Grade (PP&V).

**SAMPLES:** One bulk sample of Palabora vermiculite

### **METHOD:**

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 0.2µm 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).



CONTRACT NO: 02437  
PROJECT NO: 610  
DATE OF ISSUE: 13.08.04

## RESULTS

Crystalline silica content of Fine grade PP&V, after chemical digestion.

Sample	Original Weight (g)	Weight After Digestion (g)	Percentage Cristobalite	Percentage Quartz
Fine PP&V	1.45739	0.11436 (7.8%)	ND<0.02*	0.07 <sup>1</sup>

ND - Not Detected

\* - Detection Limit. Our current detection limit for crystalline silica on filter by XRD is 0.02mg. The detection limits quoted for the sample above are based upon 0.02 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.

<sup>1</sup> As a result of mineral interference the result for this sample was based upon visual comparison of calibration data with sample data.

## COMMENTS

Quartz was detected in the sample of Fine grade PP&V (0.07%) prepared by chemical digestion.

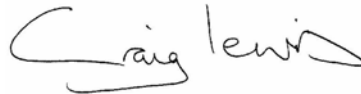
ANALYSED BY:



**S Clark**

Senior Scientific Technician

AUTHORISED BY:



**C Lewis**

Chemist

## CERTIFICATE OF ANALYSIS

**ANALYSIS REQUESTED BY:** Mr M Darling  
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**CONTRACT NO:** 02518  
**PROJECT NO:** 610  
**DATE OF ISSUE:** 27.08.04

**ANALYSIS REQUESTED:** Detailed analysis of two vermiculite samples for crystalline silica content – Micron Grade (PP&V) from North Killingholme, silo 912 – 10/01/2001 and Micron Grade PP&V from MV Marylaki, Rotterdam – 04/12/2003.

**SAMPLES:** Two bulk samples of Palabora vermiculite

### **METHOD:**

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 0.2µm 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).

**RESULTS**

**Crystalline silica content of Micron grade PP&V, after chemical digestion.**

Sample	Original Weight (g)	Weight After Digestion (g)	Percentage Cristobalite	Percentage Quartz
North Killingholme Silo 912 10/01/2001	1.47461	0.06806 (4.6%)	ND<0.03*	0.05 <sup>1</sup>
MV Marylaki Rotterdam 04/12/2003	1.52721	0.07322 (4.8%)	ND<0.03*	0.04 <sup>1</sup>


ND - Not Detected

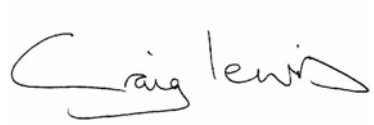
\* - Detection Limit. Our current detection limit for crystalline silica on filter by XRD is 0.02mg. The detection limits quoted for the sample above are based upon 0.02 mg expressed as a percentage of the weight of dust on the sample filter. The detection limits therefore vary as a function of the weight of dust on the filter and weights of the original samples and digestion residues.

<sup>1</sup> As a result of mineral interference the result for this sample was based upon visual comparison of calibration data with sample data.

**COMMENTS**

Quartz was detected in the samples of Micron grade PP&V; North Killingholme 2001 (0.05%) and Rotterdam 2003 (0.04%) prepared by chemical digestion.

ANALYSED BY:   
**S Clark**  
Senior Scientific Technician

AUTHORISED BY:   
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Chemist