Report to Client

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

For

Mr M Darling,
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CONTRACT NO: 609-00741
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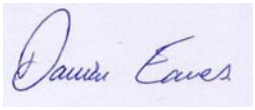

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1. INTRODUCTION

The Palabora Mining Company produces crude vermiculite from open cast mines at Phalaborwa, Limpopo Province, South Africa. The crude vermiculite is transported by rail from the mine to the port of Richards Bay on the Indian Ocean coastline of KwaZulu-Natal in South Africa. From this port it is shipped in bulk to a variety of destinations. Palabora Europe Limited stores Palabora vermiculite in both Rotterdam in the Netherlands and North Killinghome in the U.K.

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 Damien Eaves of IOM Consulting and Mr Mike Darling of Palabora Europe Limited visited the Palabora Europe Ltd vermiculite bulk store at North Killingholme Humberside, on 30th November 2004. All samples were collected by Damien Eaves 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.

Table 1 Details of Crude Vermiculite Stocks at North Killingholme on 30th November 2004

Vermiculite Grade	Silo	Approx. Stock on 30th November 2004 (tonnes)
Micron (PP & V)	924	1450
Superfine (PP & V)	932	1455
Fine (PP & V)	906	344
Medium (PP & V)	915	205
Large (PP & V)	915	711

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 November 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 Medium PP&V sample 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 November 2004 bulk materials detected no concentrations of crystalline silica present in the samples of vermiculite.

4.3. For the sample subjected to chemical digestion prior to analysis by XRD, concentrations recorded were below the limit of detection for the methodology adopted.

4.3 A summary of results is given in Tables 2a, 2b and 2c below. Certificates of Analysis are attached (Appendix 1).

Table 2a Summary of Results November 2004 Sampling Exercise

Silo Number	Description	Asbestos	Crystalline Silica
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 Crystalline Silica Results

Silo Number	Sample Description	Cristobalite %	Quartz %
915	Large (PP & V)	None Detected (<0.3)	None Detected (<0.3)
915	Medium (PP & V)	None Detected (<0.3)	None Detected (<0.3)
906	Fine (PP & V)	None Detected (<0.3)	None Detected (<0.3)
932	Superfine (PP & V)	None Detected (<0.3)	None Detected (<0.3)
924	Micron (PP & V)	None Detected (<0.3)	None Detected (<0.3)

Table 2c Crystalline silica content of Medium grade PP&V, after chemical digestion.

Sample	Original weight (g)	Weight after digestion (g)	Percentage Cristobalite	Percentage Quartz
Medium PP&V	1.55760	0.11163	None detected <0.03	< 0.1

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 (<0.3%) detected in the five bulk samples and the result obtained from the Medium grade vermiculite after digestion <0.1% quartz, there should not be any health hazard, specifically related to crystalline silica, when the materials are used in a controlled environment.

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

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CERTIFICATE OF ANALYSIS
FIBRE IDENTIFICATION IN BULK MATERIAL

Client Details: Palabora Mining Company, 1A Guildford Business Park
Guildford, Surrey GU2 8XG

Contract No: 03686-1a

Requested By: D Eaves – IOM Consulting

OH Contract Code: 609-00741

Date Samples Received: 07.12.04

Project No: 610

Date of Analysis: 08.12.04

Date of Issue: 05.01.05

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
R14705	00473/1	***CRUDE VERMICULITE SAMPLE:*** SILO 932 SUPERFINE PP&V	NONE DETECTED
No. of Samples: ONE			

Authorised by: Steve Clark

S Clark
Mineralogy Section Manager

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CERTIFICATE OF ANALYSIS
FIBRE IDENTIFICATION IN BULK MATERIAL

Client Details: Palabora Mining Company, 1A Guildford Business Park
Guildford, Surrey GU2 8XG

Contract No: 03686-1b

Requested By: D Eaves – IOM Consulting

OH Contract Code: 609-00741

Date Samples Received: 07.12.04

Project No: 610

Date of Analysis: 08.12.04

Date of Issue: 05.01.05

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
R14706	00473/2	***CRUDE VERMICULITE SAMPLE:*** SILO 924 MICRON GRADE PP&V	NONE DETECTED
No. of Samples: ONE			

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CERTIFICATE OF ANALYSIS
FIBRE IDENTIFICATION IN BULK MATERIAL

Client Details: Palabora Mining Company, 1A Guildford Business Park
Guildford, Surrey GU2 8XG

Contract No: 03686-1c

Requested By: D Eaves – IOM Consulting

OH Contract Code: 609-00741

Date Samples Received: 07.12.04

Project No: 610

Date of Analysis: 08.12.04

Date of Issue: 05.01.05

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
R14707	00473/3	***CRUDE VERMICULITE SAMPLE:*** SILO 915 LARGE GRADE PP&V	NONE DETECTED
No. of Samples: ONE			

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Sheet 1 of 1

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CERTIFICATE OF ANALYSIS
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Client Details: Palabora Mining Company, 1A Guildford Business Park
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Contract No: 03686-1d

Requested By: D Eaves – IOM Consulting

OH Contract Code: 609-00741

Date Samples Received: 07.12.04

Project No: 610

Date of Analysis: 08.12.04

Date of Issue: 05.01.05

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
R14708	00473/4	***CRUDE VERMICULITE SAMPLE:*** SILO 915 MEDIUM GRADE	NONE DETECTED
No. of Samples: ONE			

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CERTIFICATE OF ANALYSIS
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Contract No: 03686-1e

Requested By: D Eaves – IOM Consulting

OH Contract Code: 609-00741

Date Samples Received: 07.12.04

Project No: 610

Date of Analysis: 08.12.04

Date of Issue: 05.01.05

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
R14709	00473/5	***CRUDE VERMICULITE SAMPLE:*** SILO 906 FINE GRADE	NONE DETECTED
No. of Samples: ONE			

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CERTIFICATE OF ANALYSIS

ANALYSIS REQUESTED BY:

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CONTRACT NO: 03686-2

PROJECT NO: 610

DATE OF ISSUE: 05.01.05

ANALYSIS REQUESTED: Crystalline silica content of five bulk vermiculite samples, with further more detailed analysis of the Medium grade (PP&V) sample (see 03686-3).

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).

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PROJECT NO: 610
DATE OF ISSUE: 05.01.05

RESULTS: Crystalline silica in bulk vermiculite.

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

COMMENTS:

No crystalline silica was detected in any of the bulk samples.

ANALYSED BY: A. Hamer **AUTHORISED BY:** Steve Clark

A Hamer
Scientific Technician

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.

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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:

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CONTRACT NO: 03686-3

PROJECT NO: 610

DATE OF ISSUE: 05.01.05

ANALYSIS REQUESTED: Detailed analysis of one vermiculite sample for crystalline silica content – PMC Medium 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_2SO_4, 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).

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PROJECT NO: 610
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RESULTS:

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

Sample	Original Weight (g)	Weight After Digestion (g)	Percentage Cristobalite	Percentage Quartz
Medium PP&V	1.55760	0.11163 (7.2%)	ND<0.03*	<0.1[1]

ND - Not Detected

* - Detection Limit. Our current detection limit for crystalline silica on filter by XRD is 0.02 mg. 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.

[1] As a result of mineral interference the result for this sample was based upon the absence of a secondary quartz peak (detection limit 0.07mg).

COMMENTS:

Due to the presence of mineral interference it was not possible to assess the level of quartz that may be present in this sample by the detection of a primary quartz peak. The absence of a secondary quartz peak enables us to say that any quartz that may be present is <0.1%.

ANALYSED BY: Steve Clark

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AUTHORISED BY: Craig Lewis

C Lewis
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