

Final Report

Determination of the Particle Size Distribution of Ferro-niobium < 0.4 mm

Guidelines

CIPAC MT 187

Study Director

Kai Birnschein, Dipl. Chem. (FH)

Date

08 September 2014

Testing Facility

Eurofins Agrosience Services
EcoChem GmbH
Eutinger Str. 24
D-75223 Niefern-Öschelbronn
Germany

Sponsor

CBMM Europe BV
WTC H-Tower
Zuidplein 96
1077 XV Amsterdam
The Netherlands

Study Identification Code

Test item: Ferro-niobium < 0.4 mm
Study code: S14-02218
Trial/Lab Phase code: S14-02218-L1_PCPD

Statement of Confidentiality

This report contains confidential and proprietary information of CBMM Europe BV which must not be disclosed to anyone except the employees of this company or to persons authorised by law or judicial judgement without the expressed and written approval of CBMM Europe BV.

Statement of Compliance with the Principles of Good Laboratory Practice

The study described in this report was conducted in compliance with the most recent edition of:

- The Principles of Good Laboratory Practice (GLP), Chemical Act, attachment 1, Germany (July 11, 2008)
- The OECD Principles of Good Laboratory Practice

The German requirements are based on the OECD Principles of Good Laboratory Practice which are accepted by regulatory authorities throughout the European Community, the United States of America (FDA and EPA) and Japan (MHW, MAFF and METI) on the basis of intergovernmental agreements.

Head of testing facility
(Dr. Martin Feyerabend/ Dr. Susanne Timmermann)

09 Sep 14 S. Timmermann
Date / Signature

Study director
(Kai Birnschein, Dipl. Chem. (FH))

08 Sep 2014 K. Birnschein
Date / Signature

Statement of Quality Assurance Unit

Study code: S14-02218

Study title: Determination of the Particle Size Distribution of Ferro-niobium < 0.4 mm

This study has been audited by the relevant Quality Assurance Unit(s) in accordance with the OECD principles of Good Laboratory Practice and respective national regulations. Dates of inspection and reporting are listed in this section, or in the phase reports supplied by the test site(s). Documents were audited as draft versions. Facilities and/or processes and systems are monitored as part of a regular program.

	Date of audit	Date of report to Principal Investigator	Date of report to Study Director ¹⁾	Date of report to Management ²⁾
Study plan	14 Apr 2014	-	14 Apr 2014	14 Apr 2014
Experimental phase³⁾	20 Mar 2014	-	20 Mar 2014	20 Mar 2014
Final report	15 Aug 2014	-	15 Aug 2014	15 Aug 2014

¹⁾ including Lead QA and test facility management if audit reported to Principal Investigator

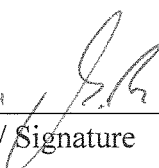
²⁾ test site management if audit reported to Principal Investigator, otherwise test facility management

³⁾ process based audit; recipients of audit reports may be different to those in this study

- not applicable

According to the inspections detailed above, and the QA Statements provided by the test sites it can be confirmed that the methods, procedures, and observations described in this final report are a full and accurate account of the raw data.

Quality assurance
(Marie Risch)

09 Sep 2016 
Date / Signature

Contents

	Page
Title Page	1
Statement of Confidentiality	2
Statement of Compliance with the Principles of Good Laboratory Practice	2
Statement of Quality Assurance Unit.....	3
Contents.....	4
List of Tables.....	4
List of Figures	4
1 Summary	5
2 Time Schedule	6
3 Study Objective.....	6
4 Material	6
4.1 Test Item.....	6
4.2 Apparatus.....	7
4.3 Method.....	7
4.4 Definition and Units	7
5 Method and Performance of the Test.....	7
6 Deviations to the Study Plan.....	8
7 Results of Particle Size Distribution.....	8
8 Archiving	10
9 References.....	10
10 Distribution	10
10.1 Study Plan.....	10
10.2 Final Report.....	10
10.3 Raw Data	10
11 Appendix.....	11
A 1 Certificates	11

List of Tables

Table 1: Particle Size Distribution of Ferro-niobium < 0.4 mm	8
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List of Figures

Figure 1: Example graph of the Particle Size distribution of Ferro-niobium < 0.4 mm (measurement 1)...	9
Figure 2: Certificate of analysis of Ferro-niobium < 0.4 mm, page 1/2	11
Figure 3: Certificate of analysis of Ferro-niobium < 0.4 mm, page 2/2	12
Figure 4: Certificate of test facility	13

1 Summary

The particle size distribution of Ferro-niobium < 0.4 mm was determined according to CIPAC MT 187.

The optical observation of the test item is depicted in the following table.

Determined particle size range:

L ₁₀ D(v,0.1): 123.8 µm
L ₅₀ D(v,0.5): 346.5 µm
L ₉₀ D(v,0.9): 695.2 µm

The mass median aerodynamic diameter (MMAD) was calculated to be 991 µm.

2 Time Schedule

Study initiation date:	18 June 2014
Start of the experimental phase:	03 July 2014
End of the experimental phase:	31 July 2014
Draft report:	04 August 2014
Study completion date:	08 September 2014

3 Study Objective

The aim of this study was the determination of the particle size distribution of the test item according to CIPAC MT 187.

4 Material

4.1 Test Item

Test Item			
Name	Ferro-niobium < 0.4 mm	Batch number	01.030675
Product	Ferroniobium 111-11		
EAS Test item code	2014-002137	Appearance / colour	solid / sliver, grey metallic
CAS number of Niobium (Nb)	7440-03-1	Purity nominal (FeNb)	95.0 % w/w
CAS number of Ferrum (Fe)	7439-89-6	Purity analysed (FeNb)	95.1 % w/w
Density	8.1997 g/cm ³	Risk symbol(s)	not available
Date of analysis report	13 Feb 2014	Expiry date	04 Apr 2015 *
		Storage conditions	ambient (+5 to +30 °C), dark, dry

* assumed as one year after receipt

All specifications given on the certificate of analysis, provided by the sponsor/supplier, are essential for correct identification of the test item for use under GLP. They have not been verified by the test facility and no claim of GLP compliance will be made for these data, except where this is explicitly claimed on the certificate of analysis.

4.2 Apparatus

- Master Sizer 2000 and autoaccessoires, Malvern, Germany

4.3 Method

The particle size distribution was determined using the laser diffraction method with dry dispersion.

4.4 Definition and Units

The median particle size $L_{50} D (v, 0.5)$, also called MMD (mass median diameter), of solids or liquids is the particle size defining the value at which 50 % of particle volume or particle mass is present with a lower particle diameter.

The particle size $L_{10} D (v, 0.1)$ of solids or liquids is the particle size defining the value at which 10 % of particle volume or particle mass is present with a lower particle diameter.

The particle size $L_{90} D (v, 0.9)$ of solids or liquids is the particle size defining the value at which 90 % of particle volume or particle mass is present with a lower particle diameter.

The particle size of a particle is given in μm .

The MMAD (mass median aerodynamic diameter) can be calculated using the given density of the test item ($\rho = 8.1997 \text{ g/cm}^3$) with the following equation (see references):

$$\text{MMAD} = \text{MMD} * \rho^{0.5}$$

5 Method and Performance of the Test

Before determination of the particle size distribution the laser was adjusted by measuring the background of the measuring cell and the Mastersizer 2000 was checked according to the SOP of the testing facility.

To determine the particle size distribution of Ferro-niobium < 0.4 mm the test item was directly given into the measuring cell which was filled with ethanol.

After adjusting the laser, measuring the background in ethanol, an obscuration of about 6-10 % was adjusted by feeding the ethanol filled sample unit directly with the test item. The sample was stirred with 3500 rpm and 100 % ultrasound in the sample unit. Subsequently the diluted suspensions were pumped through the optical system and were measured promptly. The particle size distribution was determined four times and the average result was given.

6 Deviations to the Study Plan

The study was performed according to the study plan dated 18 June 2014 with the following deviation.

Deviation: Ethanol was used as dispersing agent instead of water.

Reason: Sample did not allocate homogeneous using water as dispersing agent. Parts of the sample locate on the surface of water. A representative sampling was not possible.

Impact on study: Measurement using Ethanol as dispersing agent will be reported in the final report

This report reflects the conduct of this study.

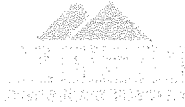
7 Results of Particle Size Distribution

Table 1 shows the results of the test. Figure 1 shows a plot of the particle size distribution.

Table 1: Particle Size Distribution of Ferro-niobium < 0.4 mm

Measurement	1 [µm]	2 [µm]	3 [µm]	4 [µm]	Average [µm]
L ₁₀	136.359	136.641	99.208	127.879	123.8
L ₅₀ (Median particle size)	348.337	350.424	318.304	368.961	346.5
L ₉₀	691.471	690.224	677.186	721.846	695.2

The mass median aerodynamic diameter (MMAD) was calculated to be 991 µm.



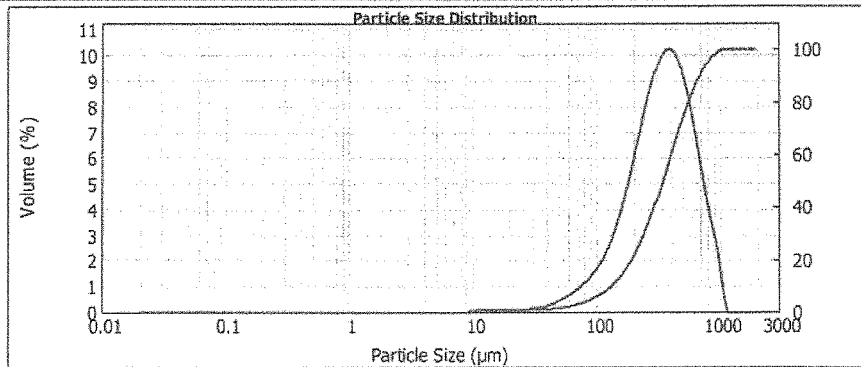
Result Analysis Report

Sample Name: S14-02218
 Sample Source & type: 2014-02137 = FeNb 0.4mm
 Sample bulk lot ref: 01.030675
 SOP Name:
 Measured by: Chrom
 Result Source: Measurement
 Measured: Donnerstag, 17. Juli 2014 17:38:45
 Analysed: Donnerstag, 17. Juli 2014 17:38:46

Particle Name: Fraunhofer
 Particle RI: 0.000
 Dispersant Name: Ethanol
 Accessory Name: Hydro 2000S (A)
 Absorption: 0
 Dispersant RI: 1.360
 Analysis model: General purpose
 Size range: 0.020 to 2000.000 um
 Weighted Residual: 0.434 %
 Sensitivity: Normal
 Obscuration: 6.07 %
 Result Emulation: Off

Concentration: 0.2119 %Vol
 Specific Surface Area: 0.0252 m²/g
 Span: 1.594
 Surface Weighted Mean D[3,2]: 238.159 um
 Uniformity: 0.486
 Vol. Weighted Mean D[4,3]: 385.043 um
 Result units: Volume

d(0.1): 136.359 um d(0.5): 348.337 um d(0.9): 691.471 um



S14-02218, Donnerstag, 17. Juli 2014 17:38:45

Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %
0.010	0.00	0.105	0.00	1.090	0.00	11.482	0.07	120.226	2.33
0.011	0.00	0.120	0.00	1.259	0.00	13.183	0.08	136.038	3.00
0.013	0.00	0.138	0.00	1.445	0.00	15.130	0.09	158.489	3.90
0.015	0.00	0.158	0.00	1.650	0.00	17.378	0.09	181.970	4.59
0.017	0.00	0.182	0.00	1.905	0.00	19.963	0.09	208.930	6.11
0.020	0.00	0.209	0.00	2.198	0.00	22.809	0.08	239.883	8.28
0.023	0.00	0.240	0.00	2.512	0.00	26.303	0.10	275.423	7.27
0.026	0.00	0.275	0.00	2.854	0.00	30.250	0.10	316.228	8.28
0.030	0.00	0.316	0.00	3.311	0.00	34.674	0.13	363.070	9.98
0.035	0.00	0.363	0.00	3.802	0.00	39.851	0.16	416.869	9.23
0.040	0.00	0.417	0.00	4.355	0.00	45.709	0.23	479.650	8.96
0.045	0.00	0.479	0.00	5.012	0.00	52.481	0.36	549.041	6.19
0.052	0.00	0.560	0.00	5.794	0.00	60.298	0.60	630.957	7.00
0.060	0.00	0.651	0.00	6.607	0.00	69.180	0.95	724.436	5.58
0.069	0.00	0.724	0.00	7.566	0.00	79.433	1.11	831.764	4.12
0.079	0.00	0.832	0.00	8.710	0.00	91.201	1.41	954.963	2.81
0.091	0.00	0.965	0.00	10.000	0.00	104.713	1.81	1095.478	1.35
0.105	0.00	1.096	0.00	11.482	0.00	120.226	0.00	1258.925	0.00

Operator notes: Probe in EtOH; 100 % US; 3500 Stirr Pump; nach reinigung
 Neueinwaage2 nach Zeilenreinigung für reproduzierbarkeitscheck
 hohe Einwaage um inhomogenität auszugleichen

Figure 1: Example graph of the Particle Size distribution of Ferro-niobium < 0.4 mm (measurement 1)

8 Archiving

For the periods demanded by the principles of GLP the following documents and materials will be archived:

- Study plan, raw data, comments of the sponsor on the draft report and the final report.
- All documentation generated by the Quality Assurance Unit
- A sample of the test item.

All documents and materials will be stored in the archives of Eurofins Agrosience Services EcoChem GmbH. The premises for storing the documents and materials are settled according to the principles of Good Laboratory Practice in the organisation of the testing facility.

9 References

W. DOBRAT AND A. MARTIJN: Collaborative International Pesticides Analytical Council Limited, 2003, CIPAC Handbook Volume K: Analysis of Technical and Formulated Pesticides

J. SWARBRICK, J. C. BOYLAN: J. Encyclopedia of Pharmaceutical Technology, Vol. 20, p. 98ff, Marcel Dekker, 10. Auflage (2001)

10 Distribution

10.1 Study Plan

Original: Testing facility (1 x)

Copy: Sponsor (1 x) (paper or pdf file possible)

10.2 Final Report

Original: Sponsor (1 x)

Testing facility (1 x)

10.3 Raw Data

Original: Testing facility

11 Appendix

A 1 Certificates



Companhia Brasileira de Metalurgia e Mineração
 Córrego da Mata S/N - Araxá - MG
 38.183-970 - Brazil
 Tel: +55 (34) 3669-3000- Fax: +55 (34) 3669-3300



Analysis Report

Date: 13 Feb 2014
 Report Number: 7280
 Version: 1
 Product: FERRONIOBIUM 111-11
 Country of Origin: Brazil
 REF. #: 4500229508
 Packaging: Boxes Carton on pallets
 Lot: 01.030675
 Lot Quantity (kg): 52,191.00
 Labware Number: 764707
 Sampling Plan: 5237
 Customer: CBMM NORTH AMERICA, INC.
 Address: 1000 OLD POND ROAD
 City: BRIDGEVILLE, PENNSYLVANIA-15017-0217
 Country: USA

Result:

Element	Result	Uncertainty	K	Analysis Date	QL	Method
Nb (%)	65.1	0.2	1.96	11 Feb 2014	NA	ITL-AICP-04

Notes:

- 1 - Analysis Methodology:
ITL-AICP-04 (Analysis by ICP-OES)
- 2 - Reported results refer to the entire lot.
- 3 - Sampling is performed according to the IT-DEBE-02 (FeNb sampling) method .
- 4 - Uncertainty in the certified value is calculated in accordance to GUIDE EURACHEM/CITAC 2nd Ed. $U=k.U_c$ where k is the coverage factor for a 95% coverage probability and U_c is obtained from the standard Uncertainty. U is the expanded uncertainty which includes the contribution of the volume, weight, calibration curve and other contributions of the measuring system.
- 5 - NBR ISO/IEC 17025 accreditation valid until 05 Jul 2017.
- 6 - % Nb testing refers to the Nb content in the FeNb product.
- 7 - Accreditation Seal: CRL 0502. Accreditation applies only to services provided by this laboratory displayed in the site: <http://www.inmetro.gov.br/>
- 8 - This certificate may only be reproduced in its entirety.
- 9 - The unit "%" is used to represent "g/100g".

Report Number: 7280 V.1
 Lot: 01.030675

Page: 1/1

Approved by: LEANDRO OLIVEIRA LIMA
 Title: CHEMIST
 Professional Registration: CRO 02101207 2nd Region

This product was manufactured in a plant whose management system is certified accordance with the requirements defined in ISO 9001, ISO 14001, OHSAS 18001 and ISO 27001.

4.4.2014 Kca

Figure 2: Certificate of analysis of Ferro-niobium < 0.4 mm, page1/2


Companhia Brasileira de Metalurgia e Mineração

Córrego da Mata S/N - Araxá - MG

38.183-970 - Brazil

Tel: +55 (34) 3669-3000- Fax: +55 (34) 3669-3300

Analysis Report

Date: 13 Feb 2014
 Report Number: 7280 A
 Version: 1
 Lot: 01.030675

Element	Result	Analysis Date	QL	Method
Si (%)	1.6	10 Feb 2014	0.45	ITL-AFRX-03
Al (%)	1.1	10 Feb 2014	0.28	ITL-AFRX-03
P (%)	0.1	10 Feb 2014	0.039	ITL-AFRX-03
Mn (%)	0.4	10 Feb 2014	0.059	ITL-AFRX-03
Pb (%)	0.1	10 Feb 2014	0.033	ITL-AFRX-03
Ti (%)	0.54	10 Feb 2014	0.10	ITL-AFRX-03
Cu (%)	0.1	10 Feb 2014	0.013	ITL-AFRX-03
Fe (%)	30.0	11 Feb 2014	NA	ITL-AICP-04
C (%)	0.1	10 Feb 2014	0.003	ITL-ACS-02
Ta (%)	0.1	10 Feb 2014	0.055	ITL-AFRX-03
Sn (%)	0.1	10 Feb 2014	0.044	ITL-AFRX-03
S (%)	<0.1	10 Feb 2014	0.002	ITL-ACS-02
N (%)	0.1	10 Feb 2014	0.0010	ITL-AHNO-02

Size Distribution		
Screen (mm)	% Accumulated Retained	Method
12,5	0	IT-DELB-07
1	98	IT-DELB-07

Legend:

-- = Not Analyzed

NA = Not Applicable

QL = Quantification Limit

Notes:

1 - Analysis Methodology:

IT-DELB-07 (Sieving analysis)

ITL-ACS-02 (LECO CS Determinator)

ITL-AFRX-03 (Analysis by XRF)

ITL-AHNO-02 (LECO ONH Determinator)

ITL-AICP-04 (Analysis by ICP-OES)

2 - Reported results refer to the entire lot.

3 - Sampling is performed according to the IT-DEBE-02 (FeNb sampling) method.

4 - This certificate may only be reproduced in its entirety.



Report Number: 7280 A V.1
 Lot: 01.030675

Page: 1/1

Approved by: LEANDRO OLIVEIRA LIMA

Title: CHEMIST

Professional Registration: CRQ 02101207 2nd Region

This product was manufactured in a plant whose management system is certified accordance with the requirements defined in ISO 9001, ISO 14001, OHSAS 18001 and ISO 27001.

Figure 3: Certificate of analysis of Ferro-niobium < 0.4 mm, page 2/2


Baden-Württemberg

LANDESANSTALT FÜR UMWELT, MESSUNGEN UND NATURSCHUTZ BADEN-WÜRTTEMBERG

Gute Laborpraxis / Good Laboratory Practice

GLP-Bescheinigung / Statement of GLP Compliance

(gemäß / according to § 19 b Chemikaliengesetz)

Eine GLP-Inspektion zur Überwachung der Einhaltung der GLP-Grundsätze gemäß Chemikaliengesetz bzw. Richtlinie 2004/9/EG wurde durchgeführt in: Assessment of conformity with GLP according to Chemikaliengesetz and Directive 2004/9/EC at:

 Prüfeinrichtung / Test facility

 Prüfstandort / Test site

Eurofins Agrosience Services EcoChem GmbH
Eutinger Straße 24
75223 Niefen-Öschelbronn

(Unverwechselbare Bezeichnung und Adresse / Unequivocal name and address)

Prüfungen nach Kategorien / Areas of Expertise

(gemäß / according ChemVwV-GLP Nr. 5.3 / OECD guidance)

1 Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften	Physical-chemical testing
4 Ökotoxikologische Prüfungen zur Bestimmung der Auswirkungen auf aquatische und terrestrische Organismen	Environmental toxicity studies on aquatic and terrestrial organisms
5 Prüfungen zum Verhalten im Boden, im Wasser und in der Luft; Prüfungen zur Bioakkumulation und zur Metabolisierung	Studies on behavior in water, soil and air; bioaccumulation
6 Prüfungen zur Bestimmung von Rückständen	Residue studies
7 Prüfungen zur Bestimmung der Auswirkungen auf Mesokosmen und natürliche Ökosysteme	Studies on effects on mesocosms and natural ecosystems
8 Analytische Prüfungen an biologischen Materialien	Analytical and clinical chemistry testing

Datum der Inspektion / Date of Inspection

(Tag, Monat, Jahr / day, month, year)

10.10.2013

Die/Der genannte Prüfeinrichtung/Prüfstandort befindet sich im nationalen GLP-Überwachungsverfahren und wird regelmäßig auf Einhaltung der GLP-Grundsätze überwacht.

The above mentioned test facility/test site is included in the national GLP Compliance Programme and is inspected on a regular basis.

Auf der Grundlage des Inspektionsberichtes wird hiermit bestätigt, dass in dieser Prüfeinrichtung/Prüfstandort die oben genannten Prüfungen unter Einhaltung der GLP-Grundsätze durchgeführt werden können.

Based on the inspection report it can be confirmed, that this test facility/test site is able to conduct the aforementioned studies in compliance with the Principles of GLP.

Unterschrift, Datum / Signature, Date


Dr. Volker Giraud

Leiter der Abteilung Technischer Umweltschutz



Karlsruhe, 08.01.2014

(Name und Funktion der verantwortlichen Person / Name and function of responsible person)

 LUBW Landesanstalt für Umwelt, Messungen und Naturschutz Baden-Württemberg
 Postfach 10 01 63, 76231 Karlsruhe

(Name und Adresse der GLP-Überwachungsbehörde / Name and address of GLP Monitoring Authority)

Figure 4: Certificate of test facility