

Study Title

Water solubility of Ferro Niobium

DATA REQUIREMENT

REACH requirement EC/1907/2006

AUTHOR

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STUDY COMPLETION DATE:

2009-09-22

PERFORMING LABORATORY

CURRENTA GmbH & Co. OHG
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Federal Republic of Germany

SPONSOR

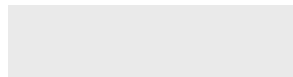
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LABORATORY PROJECT ID

Study No. 2009/0076/05



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1. **Statement of compliance with GLP (SOC) Claim**

This study was conducted in compliance with the OECD principles of Good Laboratory Practice (GLP, as revised in 1997) and with the Principles of Good Laboratory Practice according to Annex 1, German Chemical Law (Änderung des Anhangs 1 vom 8.Mai 2001).

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Dr Andreas Königer:



Date:

2009-09-22

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Dr Kreiss /
Dr Richter :



Date:

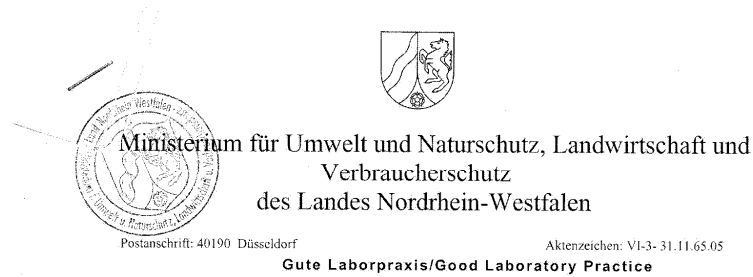
2009-10-02

2. **Archiving**

The original report, the study plan and all raw data pertaining to this study are stored in the "GLP Archiv, Services Analytik, Building Q 18, Currenta GmbH & Co. OHG, D-51368 Leverkusen". A sample of the test item is stored in "GLP-Probenlager, Services Analytik, Building DA 1, Currenta GmbH & Co. OHG, D-41538 Dormagen".

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3. GLP CERTIFICATE



Ministerium für Umwelt und Naturschutz, Landwirtschaft und
Verbraucherschutz
des Landes Nordrhein-Westfalen

Postanschrift: 40190 Düsseldorf

Aktenzeichen: VI-3-31.11.65.05

Gute Laborpraxis/Good Laboratory Practice

GLP-Bescheinigung/Statement of GLP Compliance
(gemäß/according to § 19b Abs. 1 Chemikaliengesetz)

Eine GLP-Inspektion zur Überwachung der Einhaltung der GLP-Grundsätze gemäß Chemikaliengesetz bzw. Richtlinie 88/320/EG wurde durchgeführt in: Assessment of conformity with GLP according to Chemikaliengesetz and Directive 88/320/EEC at:

Prüfeinrichtung/Test facility Prüfstandort/Test site

Bayer Industry Services GmbH & Co OHG

Prüfeinrichtung BIS-SUA-Analytiks

D-51368 Leverkusen

(unverwechselbare Bezeichnung und Adresse/Unequivocal name and address)

Prüfungen nach Kategorien

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

Kategorie 1

Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften und Gehaltsbestimmungen

Kategorie 4

Ökotoxikologische Prüfungen zur Bestimmung der Auswirkungen auf aquatische und terrestrische Organismen

Kategorie 5

Prüfungen zum Verhalten im Boden, im Wasser und in der Luft; Prüfungen zur Bioakkumulation und zur Metabolisierung

Kategorie 8

Analytische Prüfungen an biologischen Materialien

Areas of Expertise

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

category 1

physical-chemical testing

category 4

environmental toxicity studies on aquatic and terrestrial organisms

category 5

studies on behaviour in water, soil and air; bioaccumulation

category 8

analytical and clinical chemistry testing

Datum der Inspektion

(Tag, Monat, Jahr)

14. bis 16. September
und 26. bis 28. Oktober 2005

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

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

Date of Inspection

(day, month, year)

on 14 until 16 September and on 26 until 28
October 2005

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

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

Düsseldorf, den 11. Januar 2006
Im Auftrag

(Prof. Dr. David)



Dienstsiegel/official-seal

Please note: Effective January 1st, 2008, the company name Bayer Industry Services GmbH & Co. OHG was changed to CURRENTA GmbH & Co. OHG.

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4. Quality Assurance Statement

This report was audited by the Quality Assurance Unit Currenta, Services Analytik, Quality Management at Currenta GmbH & Co. OHG and this statement confirms that the final report reflects the raw data. The dates of Quality Assurance inspections and audits are given below.

| Audits | Dates of QAU Inspections | Dates of Reports |
|----------------------------------|--------------------------|------------------|
| Study plan inspection | 2009-09-02 | 2009-09-02 |
| Inspection of experimental phase | 2009-04-20* | 2009-04-20* |
| Report inspection | 2009-09-30 | 2009-09-30 |

* Process based inspection

FOR THE HEAD OF QUALITY ASSURANCE

Ms D.I. Senic /
~~Dr Doerzbach-Lange /~~
~~Dr Neupert-~~

A. Senic

Date:

2009-09-30

5. Study Time Table

| | |
|------------------------------|------------|
| Study initiation date: | 2009-09-02 |
| Study completion date: | 2009-09-22 |
| Start of Experimental Tests: | 2009-09-07 |
| End of Experimental Tests: | 2009-09-17 |

6. Summary

Report: Dr Andreas Königer: Water solubility of Ferro niobium; Currenta, report no.: 2009/0076/05

Guidelines: The test was performed according to OECD Guidelines for Testing of Chemicals, Section 1 – Physical-Chemical Properties OECD TG 105 (1995)

Deviation from Guidelines: No

GLP: Yes (certified laboratory)

Time of experimental tests: 2009-09-07 to 2009-09-17

Materials and Determinations: With Ferro niobium, batch no.: AD/4204, content: 65.9 % Nb and 29.4 % Fe, the water solubility was calculated by determination of niobium and iron.

Due to the expected low solubility or insolubility of the test item in water no preliminary test was performed. Three tests with each approx. 1 g of the sample in 1 l deionized water (Millipore water) and a blank test were performed. The contents of niobium and iron were determined by ICP-MS.

Results:

The very small parts of the very finely grounded material seem to build a suspension which is stable over a few days and very difficult to separate from the solution. Therefore the separation step is of high importance and done twice.

After filtration and centrifugation the following results were obtained:

| Blank values | Determination 1 | Determination 2 | Mean value |
|-----------------|-----------------|-----------------|------------|
| Niobium content | < 0.5 µg/l | < 0.5 µg/l | < 0.5 µg/l |
| Iron content | Not determined* | < 10 µg/l | < 10 µg/l |

* Determination invalidated due to a carry-over.

| Water solubility at 20 °C (after filtration and centrifugation) | Flask 1 | Flask 2 | Flask 3 | Mean value |
|--|-----------------------------|-----------------------------|-----------------------------|------------------------------------|
| Niobium content (Determination 1 Determination 2) | <0.5 µg/l < 0.5 µg/l | < 0.5 µg/l < 0.5 µg/l | 0.85 µg/l 0.82 µg/l | |
| Niobium content (mean value) | < 0.5 µg/l | < 0.5 µg/l | 0.84 µg/l | 0.8 µg/l (1 µg/l Alloy) |
| Iron content (Determination 1 Determination 2) | Not determined* <10 µg/l | Not determined* <10 µg/l | Not determined* <10 µg/l | |
| Iron content (mean value) | <10 µg/l | <10 µg/l | <10 µg/l | <10 µg/l |

* Determination invalidated due to a carry-over.

The limit of quantification for the ubiquitous element iron is set to 10 µg/l in this determination due to the blank test.

Due to the uncertainty of the iron determination in such low concentrations, the water solubility is only determined on the basis of niobium.

The water solubility of the alloy Ferro niobium is approx. 1 µg/l.

7. **Methods and Documents**

The determination of the water solubility was based on the following guidelines:

Currenta-internal SOP 00190 Version 2, covering OECD TG 105 (1995).

Currenta-internal SOP 00178 Version 2 for the determination of the pH-value.

Currenta-internal method 2011-0366401-92D: Determination of niobium and iron by ICP-MS technique.

8. **Sample description**

| | | | |
|-------------------------------|----------------------|------------------|---------------------|
| Product name: | Ferro Niobium | Chemical name: | Ferro niobium alloy |
| Empirical formula: | FeNb | Molecular mass: | -- g/mol |
| CAS-No: | -- | Batch No.: | AD/4204 |
| Content: | 65.9 % Nb, 29.4 % Fe | Expiry date: | 2010-01-21 |
| Date of arrival at test site: | 2009-07-21 | Sample no./year: | 1000/2009 |

9. **Test Methods**

9.1 The determination of the water solubility by the flask method.

9.1.1 Preparation of the test solutions

SOP: 00190 Version 2 (water solubility)
This procedure corresponds to test method OECD 105 for the determination of the water solubility.

Supervisor: Dr Königer

Procedure:

Due to the expected low solubility or insolubility of the test item in water no preliminary test was performed. Three tests with each approx. 1 g of the sample in 1 l deionized water (Millipore water) and a blank test were performed. The content of niobium and iron was determined by ICP-MS.

Approx. 1 g of the test item were weight each into stoppered glass bottles and agitated on a magnetic stirrer for 24h, 48h and 72h, respectively, at 30 °C in a thermostated water bath. Following this procedure, the temperature is reduced to 20 °C and the bottles were kept in the thermostated water bath for another 24h.

After phase separation by centrifugation (9000 RPM for 20 min) the solution was pipetted into the measuring tubes and the concentration of niobium and iron was determined by ICP-MS.

Due to the low reproducibility of the results of this first determination (see table 1), the solutions were filtered again through a smaller membrane filter (0.1 µm) and centrifuged (9000 RPM for 20 min). The second filtrates were visibly clear solutions. The solutions were analyzed again. The results are much lower (see table 2).

A blank test with water was also performed.

Agitating time for the test at 20 °C: Flask 1 : 72 hours at 30 °C and 24 hours at 20 °C
 Flask 2 : 48 hours at 30 °C and 24 hours at 20 °C
 Flask 3 : 24 hours at 30 °C and 24 hours at 20 °C

Test concentrations

| | Flask 1 | Flask 2 | Flask 3 |
|----------------------|----------------|----------------|----------------|
| Initial weight [g] | 1.03 | 1.03 | 1.00 |
| Volume of water [ml] | 1000 | 1000 | 1000 |

9.1.2 Analysis of the test solutions

9.1.2.1 Test: pH measurement

SOP: SOP 00178 Version 2

Supervisor: Dr Königer

Description of the method: After phase separation of the test mixtures the determination of the pH-value was performed with a pH-meter with single-rod glass electrode after previous calibration.

9.1.2.2 Test: Content of niobium and iron by ICP-MS

Method no.: 2011-0366401-92D

Supervisor: Dr Schweer

Procedure: ICP-MS

The aqueous solutions were acidified with hydrofluoric acid and determined by ICP-MS using rhodium as internal standard. The limits of quantification (LOQ) for this determination are:

Niobium: 0.5 µg/l

Iron: 10 µg/l due to the blank test

9.1.3 Results

| Blank values | Determination 1 | Determination 2 | Mean value |
|-----------------|-----------------|-----------------|------------|
| Niobium content | < 0.5 µg/l | < 0.5 µg/l | < 0.5 µg/l |
| Iron content | < 10 µg/l | < 10 µg/l | < 10 µg/l |

Table 1: Water solubility at 20 °C (first determination)

| Water solubility at 20 °C (Standard laboratory procedure) | Flask 1 | Flask 2 | Flask 3 | Mean value |
|--|-----------------------|-----------------------|--------------------------|--|
| Niobium content (Determination 1 Determination 2) | 26.6 µg/l 8.4 µg/l | 19.8 µg/l 4.4 µg/l | 182.0 µg/l 143.7 µg/l | |
| Niobium content (mean value) | 17.5 µg/l | 12.1 µg/l | 162.8 µg/l | 64 µg/l (97 µg/l Alloy) |
| Iron content (Determination 1 Determination 2) | 18 µg/l < 10 µg/l | 14 µg/l < 10 µg/l | 44 µg/l 34 µg/l | |
| Iron content (mean value) | < 10 µg/l | < 10 µg/l | 39 µg/l | Not calculated because of low reproducibility |
| pH-value | 6.2 | 6.3 | 6.0 | 6.2 |

Table 2: Water solubility at 20 °C (after centrifugation)

| Water solubility at 20 °C (after filtration and centrifugation) | Flask 1 | Flask 2 | Flask 3 | Mean value |
|--|-----------------------------|-----------------------------|-----------------------------|------------------------------------|
| Niobium content (Determination 1 Determination 2) | <0.5 µg/l < 0.5 µg/l | < 0.5 µg/l < 0.5 µg/l | 0.85 µg/l 0.82 µg/l | |
| Niobium content (mean value) | < 0.5 µg/l | < 0.5 µg/l | 0.84 µg/l | 0.8 µg/l (1 µg/l Alloy) |
| Iron content (Determination 1 Determination 2) | Not determined* <10 µg/l | Not determined* <10 µg/l | Not determined* <10 µg/l | |
| Iron content (mean value) | <10 µg/l | <10 µg/l | <10 µg/l | <10 µg/l |

* Determination invalidated due to a carry-over.

The results show a low reproducibility in the first determination. Therefore a second separation step was performed.

Although the solution was visibly clear after the second separation, it can not be excluded that a small amount of distributed test item is still present in the solution.

The limit of quantification for the ubiquitous element iron is set to 10 µg/l in this determination due to the blank test.

Due to the uncertainty of the iron determination in such low concentrations, the water solubility is only determined on the basis of niobium.

The water solubility of the alloy Ferro niobium is approx. 1 µg/l.