Study Title

Water solubility of Diniobium pentaoxide

DATA REQUIREMENT

REACH requirement EC/1907/2006

AUTHOR

Dr Andreas Königer

STUDY COMPLETION DATE:

2009-09-01

PERFORMING LABORATORY

CURRENTA GmbH & Co. OHG
Services Analytik
D-51368 Leverkusen
Federal Republic of Germany

SPONSOR

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

STUDY MONITOR

Mr. Jorge Davo
CBMM Companhia Brasileira de Metalurgia e Mineracao
Córrego da Mata s/n, 38183-903 Araxá – MG
Brasil

LABORATORY PROJECT ID

Study No. 2009/0074/01

This page is intentionally left blank for the purpose of submitting administrative information that is required by regulations promulgated by various countries.

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 Anhanges 1 vom 8.Mai 2001).

STUDY DIRECTOR
CURRENTA GmbH & Co. OHG
Services Analytik
Building Q 18
D-51368 Leverkusen
Federal Republic of Germany

Dr Andreas Königer: _____

Date: 2009-09-01

FOR THE HEAD OF TEST FACILITY
CURRENTA GmbH & Co. OHG
Services Analytik
Building Q 18
D-51368 Leverkusen
Federal Republic of Germany

Prof. Dr Caspers /
~~Dr Kreiss /~~
~~Dr Richter :~~ _____

Date: 2009-09-02

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".

This page is intentionally left blank for the purpose of submitting administrative information that is required by regulations promulgated by various countries.

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-Analytics
D-51368 Leverkusen

(unverwechselbare Bezeichnung und Adresse/Unequivocal name and address)

Prüfungen nach Kategorien (gemäß ChemVwV-GLP Nr. 5.3/OECD guidance)	Areas of Expertise (according ChemVwV-GLP Nr. 5.3/OECD guidance)
Kategorie 1	category 1
Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften und Gehaltsbestimmungen	physical-chemical testing
Kategorie 4	category 4
Ökotoxikologische Prüfungen zur Bestimmung der Auswirkungen auf aquatische und terrestrische Organismen	environmental toxicity studies on aquatic and terrestrial organisms
Kategorie 5	category 5
Prüfungen zum Verhalten im Boden, im Wasser und in der Luft; Prüfungen zur Bioakkumulation und zur Metabolisierung	studies on behaviour in water, soil and air; bioaccumulation
Kategorie 8	category 8
Analytische Prüfungen an biologischen Materialien	analytical and clinical chemistry testing

Datum der Inspektion (Tag.Monat.Jahr)	Date of Inspection (day.month.year)
14. bis 16. September und 26. bis 28. Oktober 2005	on 14 until 16 September and on 26 until 28 October 2005

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

The above mentioned test facility 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 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 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.

Table of contents

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	2009-07-23	2009-07-23
Inspection of experimental phase	2009-04-20*	2009-04-20*
Report inspection	2009-09-02	2009-09-02

* Process based inspection

FOR THE HEAD OF QUALITY ASSURANCE

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

Date: 2009-09-02

5. Study Time Table

Study initiation date:	2009-07-23
Study completion date:	2009-09-01
Start of Experimental Tests:	2009-07-27
End of Experimental Tests:	2009-08-14

6. Summary

Report: Dr Andreas Königer: Water solubility of Diniobium pentaoxide; Currenta, report no.: 2009/0074/01

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: 2009-07-31 - 2009-08-14

Materials and Determinations: With Diniobium pentaoxide, batch no.: AD/4199, purity 99.2 %, the water solubility was calculated by determination of niobium.
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 was determined by ICP-MS.

Results:

Diniobium pentaoxide powder seems to build a dispersed or colloidal system in water. The separation of the solution (dissolved diniobium pentaoxide) from the colloidal and/or dispersed solid material is an important step during the test process. Therefore the separation was 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**
Diniobium pentaoxide content	< 0.5 µg/l	< 0.5 µg/l	**< 0.5 µg/l**
pH-value	6.62	--	**6.6**

Water solubility at 20 °C (after filtration and centrufigation)	**Flask 1**	**Flask 2**	**Flask 3**	**Mean value**
Niobium content (Determination 1 Determination 2)	23.1 µg/l 23.1 µg/l	7.9 µg/l 7.9 µg/l	30.6 µg/l 30.3 µg/l	
Niobium content (mean value)	23.1 µg/l	7.9 µg/l	30.4 µgl	**20 µg/l**
Diniobium pentaoxide content	33.0 µg/l	11.3 µg/l	43.6 µg/l	**30 µg/l**

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

the solution. Due to the fact that the water solubility calculated in the second determination is very low no third phase separation was performed.

The water solubility is therefore estimated to be lower than 30 µg/l.

7. Methods and Documents

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

Currenta-internal SOP 00190 Version 1, 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 by ICP-MS technique.

8. Sample description

Product name:	Diniobium pentaoxide	Chemical name:	Diniobium pentaoxide
Empirical formula:	Nb_2O_5	Molecular mass:	265.8 g/mol
CAS-No:	1313-96-8	Batch No.:	AD/4199
Content:	99.2 %	Expiry date:	2010-01-21
Arrival at test site:	2009-07-21	Sample no./year:	998/2009

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 1 (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 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 thermostatted water bath. Following this procedure, the temperature is reduced to 20 °C and the bottles were kept in the thermostatted water bath for another 24h.

After phase separation by filtering through a membrane filter (0.45 µm) directly into the measuring tubes the concentration of niobium was determined by ICP-MS (standard laboratory procedure).

Due to the low reproducibility of the results of this standard laboratory procedure (see table 1), the filtrates were investigated again and found to be slightly turbid.

They therefore 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 but still of low reproducibility (see table 2).

Due to the fact that the water solubility calculated in the second determination is very low no third phase separation was performed. Nevertheless, it can not be excluded, that a small amount of colloidal distributed test item is still present in the solution. The water solubility is therefore expected to be lower than the results obtained.

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]	1.01	1.00	0.99
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 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 limit of quantification (LOQ) for this determination is 0.5 µg/l.

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**
Diniobium pentaoxide content	< 0.5 µg/l	< 0.5 µg/l	**< 0.5 µg/l**
pH-value	6.62	--	**6.6**

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)	296.0 µg/l 297.3 µg/l	214.8 µg/l 195.0 µg/l	414.7 µg/l 409.8 µg/l	
Niobium content (mean value)	296.7 µg/l	204.9 µg/l	412.2 µgl	**300 µg/l**
Diniobium pentaoxide content	424,4 µg/l	293.1 µg/l	589.8 µg/l	**440 µg/l**
pH-value	5.89	5.94	6.01	**6.0**

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

Water solubility at 20 °C (after filtration and centrufigation)	**Flask 1**	**Flask 2**	**Flask 3**	**Mean value**
Niobium content (Determination 1 Determination 2)	23.1 µg/l 23.1 µg/l	7.9 µg/l 7.9 µg/l	30.6 µg/l 30.3 µg/l	
Niobium content (mean value)	23.1 µg/l	7.9 µg/l	30.4 µgl	**20 µg/l**
Diniobium pentaoxide content	33.0 µg/l	11.3 µg/l	43.6 µg/l	**30 µg/l**

The results show a low reproducibility. Although the solution was visibly clear after the second separation, it can not be excluded that a small amount of colloidal distributed test item is still present in the solution. Due to the fact that the water solubility calculated in the second determination is very low no third phase separation was performed.

The water solubility is therefore estimated to be lower than 30 µg/l.