

Final Report

**Stability, Homogeneity and Accompanying Analysis of
Diniobium pentaoxide
in Water**

DATA REQUIREMENT

REACH requirement EC/1907/2006

AUTHOR

Dr Andreas Königer

STUDY COMPLETION DATE:

2009-12-17

PERFORMING LABORATORY

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

SPONSOR

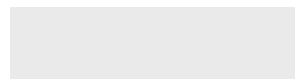
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LABORATORY PROJECT ID

Study No. 2009/0074/05



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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 Königler:



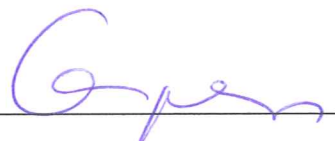
Date:

2009-12-17

FOR THE HEAD OF TEST FACILITY

CURRENTA GmbH & Co. OHG
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Federal Republic of Germany

Prof. Dr Caspers /
Dr Kreiss /
Dr Richter :



Date:

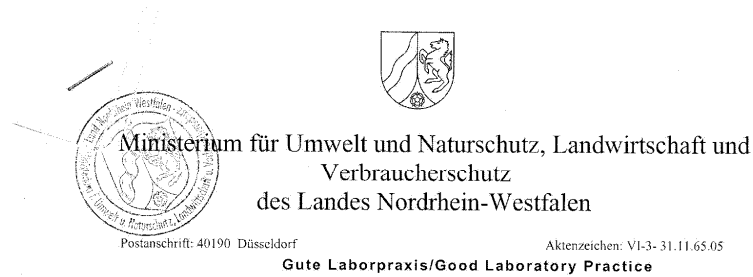
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2. **Archiving**

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". No samples of the formulations were stored.

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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-Analytics

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)



Dienstsigel/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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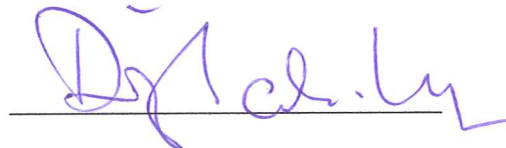
4. Quality Assurance Statement

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	2009-09-08	2009-09-08
Inspection of experimental phase	2009-10-02	2009-10-05
Inspection of experimental phase	2009-11-17	2009-11-19
Report inspection	2010-01-06	2010-01-06

FOR THE HEAD OF QUALITY ASSURANCE

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



Date:

2010-01-06

5. Study Time Table

Study initiation date:	2009-09-08
Study completion date:	2009-12-17
Start of Experimental Tests:	2009-10-02
End of Experimental Tests:	2009-11-26

6. Summary

Report: Dr Andreas König: Stability, homogeneity and accompanying analysis of Diniobium pentaoxide in water; Currenta, report no.: 2009/0074/05

Guidelines: CURRENTA-internal method 2011-0366401-92D

Deviation from Guidelines: No

GLP: Yes (certified laboratory)

Time of experimental tests: 2009-10-02 to 2009-11-26

Materials and Determinations:

All samples of the test item in water were prepared and labelled at BSL Bioservice Scientific Laboratories GmbH, BSL study no.: 092790

Results:

For detailed results see tables in paragraph 9.

7. **Methods and Documents**

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

8. **Sample description of the test item**

Product name:	Diniobium pentaoxide	Chemical name:	Diniobium pentaoxide
Empirical formula:	Nb ₂ O ₅	Molecular mass:	265.8 g/mol
CAS-No:	1313-96-8	Batch No.:	AD/4199
Content:	99.2 %		
Date of arrival at test site:	see table	Sample no. of the formulations:	see table

9. **Test Method**

9.1 The determination of the content of niobium in the water formulations

Method no.: 2011-0366401-92D

Supervisor: Dr Schweer

9.1.1 Principle of the method

The water of the whole sample was evaporated to nearly dryness. The residue was digested with hydrofluoric and nitric acid and diluted to measuring concentration. The analysis of niobium was performed by the ICP-MS technique (inductively coupled plasma – detection by mass spectrometry) using rhodium as internal standard.

9.1.2 Sample preparation

The samples are suspensions of the solid test item in water. To avoid inhomogeneity during sampling the whole sample was used for determination and the following procedure was applied:

1. The volume of the whole sample was determined.
2. The whole sample was transferred into a glass carbon beaker, acidified with hydrofluoric and nitric acid and the water was evaporated carefully to nearly dryness.
3. The residue was digested with hydrofluoric and nitric acid and diluted to measuring concentration.
4. Analysis of the measuring solutions by ICP-MS using rhodium as internal standard.

9.1.3 Method parameters

Equipment: Varian 820-MS

Plasma: Nebulizer flow: 0.90 L/min

Power: 1.35 kW

Pump rate: 6 rpm

Scan time: 95 msec.

9.1.4 Results

Stability testing

Sample code according to label	Sample code according to study plan	Sampling / Sampling date (arrival at test site)	Volume of the sample in ml	High dosis 100 g/l Start (0 h)		High dosis 100 g/l End (6 h)		Low dosis 25 g/l Start (0 h)		Low dosis 25 g/l End (6 h)	
				Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)
17a	17	Week 1 (2009-09-24)	10.3	63.9 (64.2 / 63.7)	91.5 g/l (91.5 %)						
18a	18	Week 1 (2009-09-24)	10.4			40.6 (40.4 / 40.7)	58.0 g/l (58.0 %)				
19a	19	Week 1 (2009-09-24)	10.2					18.2 (18.3 / 18.2)	26.1 g/l (104.4 %)		
20a	20	Week 1 (2009-09-24)	10.2							11.5 (11.6 / 11.4)	16.5 g/l (65.8 %)

Homogeneity testing

Sample code according to label	Sample code according to study plan	Sampling / Sampling date (arrival at test site)	Volume of the sample in ml	High dosis 100 g/l Top		High dosis 100 g/l Middle		High dosis 100 g/l Bottom		Low dosis 25 g/l Top		Low dosis 25 g/l Middle		Low dosis 25 g/l Bottom	
				Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)
21a	21	Week 1 (2009-09-24)	3.2	69.0 (69.9 / 68.1)	98.7 g/l (98.7 %)										
22a	22	Week 1 (2009-09-24)	3.1			66.8 (66.2 / 67.5)	95.6 g/l (95.6 %)								
23a	23	Week 1 (2009-09-24)	3.1					64.1 (64.1 / 64.1)	91.7 g/l (91.7 %)						
24a	24	Week 1 (2009-09-24)	3.2							20.9 (20.9 / 20.9)	29.9 g/l (119.7 %)				
25a	25	Week 1 (2009-09-24)	3.2									14.3 (14.5 / 14.1)	20.5 g/l (81.8 %)		
26a	26	Week 1 (2009-09-24)	3.1											14.7 (14.7 / 14.6)	21.0 g/l (84.0 %)

Sample code according to label	Sample code according to study plan	Sampling / Sampling date (arrival at test site)	Volume of the sample in ml	High dosis 100 g/l		High dosis 100 g/l		High dosis 100 g/l		Low dosis 25 g/l		Low dosis 25 g/l		Low dosis 25 g/l	
				Top	Middle	Middle	Bottom	Top	Middle	Middle	Bottom				
27a	27	Week 5 (2009-11-10)	3.3	47.8 (47.9 / 47.7)	68.4 g/l (68.4 %)										
28a	28	Week 5 (2009-11-10)	3.0			56.4 (56.1 / 56.6)	80.6 g/l (80.6 %)								
29a	29	Week 5 (2009-11-10)	3.0					72.1 (72.0 / 72.2)	103.1 g/l (103.1 %)						
30a	30	Week 5 (2009-11-10)	3.4							12.5 (12.4 / 12.5)	17.9 g/l (71.4 %)				
31a	31	Week 5 (2009-11-10)	3.0									15.8 (15.7 / 15.9)	22.6 g/l (90.3 %)		
32a	32	Week 5 (2009-11-10)	3.4											18.7 (18.7 / 18.7)	26.7 g/l (106.8 %)

Determination of the nominal concentration (accompanying analysis)

Sample code according to label	Sample code according to study plan	Sampling / Sampling date (arrival at test site)	Volume of the sample in ml	Control sample (blank test)		High dosis		Medium dosis		Low dosis	
				Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	100 g/l	50 g/l	50 g/l	25 g/l	25 g/l	25 g/l
				Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)
1a	1	Week 1 (2009-09-24)	10.2	< 0.001 (< 0.001 / < 0.001)	< 0.001 g/l						
2a	2	Week 1 (2009-09-24)	10.1			64.3 (64.8 / 63.8)	91.9 g/l (91.9 %)				
3a	3	Week 1 (2009-09-24)	7.5					42.1 (41.6 / 42.6)	60.2 g/l (120.5 %)		
4a	4	Week 1 (2009-09-24)	10.2							15.6 (15.4 / 15.7)	22.3 g/l (89.1 %)
5a	5	Week 3 (2009-11-10)	10.3	0.002 (0.002 / 0.002)	0.003 g/l						
6a	6	Week 3 (2009-11-10)	10.4			58.5 (58.1 / 58.9)	83.7 g/l (83.7 %)				
7a	7	Week 3 (2009-11-10)	10.2					30.2 (30.1 / 30.2)	43.1 g/l (86.3 %)		
8a	8	Week 3 (2009-11-10)	10.0							16.2 (16.2 / 16.2)	23.2 g/l (92.8 %)
9a	9	Week 5 (2009-11-10)	10.0	0.003 (0.003 / 0.003)	0.005 g/l						

Sample code according to label	Sample code according to study plan	Sampling / Sampling date (arrival at test site)	Volume of the sample in ml	Control sample (blank test)		High dosis 100 g/l		Medium dosis 50 g/l		Low dosis 25 g/l	
				Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)	Content of Niobium in g/l mean value (repeated test)	Content of Diniobium pentaoxide g/l mean value (recovery in %)
10a	10	Week 5 (2009-11-10)	10.0			53.6 (53.8 / 53.4)	76.7 g/l (76.7 %)				
11a	11	Week 5 (2009-11-10)	10.0					31.6 (31.7 / 31.6)	45.2 g/l (90.5 %)		
12a	12	Week 5 (2009-11-10)	10.2							15.3 (15.3 / 15.3)	21.9 g/l (87.5 %)
13a	13	Week 7 (2009-11-10)	10.0	< 0.001 (<0.001 / <0.001)	< 0.001						
14a	14	Week 7 (2009-11-10)	10.0			69.1 (69.2 / 68.9)	98.8 g/l (98.8 %)				
15a	15	Week 7 (2009-11-10)	10.0					32.5 (32.5 / 32.6)	46.5 g/l (93.1 %)		
16a	16	Week 7 (2009-11-10)	10.0							16.0 (16.0 / 16.1)	22.9 g/l (91.7 %)