FINAL REPORT

Study no.

X02a-081

2 hours Bio-elution Study on

Stainless Steel 316L Massive

(Epoxy resin embedded)

at a *2 g/L loading (surface equivalent)*

in a simulated gastric fluid

June 18, 2017

Statement

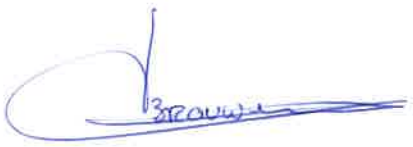
Title: 2 hours Bio-elution Study on **Stainless Steel 316L Massive** (Epoxy resin embedded) at a 2 g/L loading (surface equivalent) in a simulated gastric fluid.

Study no.: **X02a-081**

The study has been performed and conducted according to the OECD Principles of Good Laboratory Practices for experiments carried out by the *Test Facility* ECTX. The raw data and the reported results have been obtained by following the instructions of the study plan and the SOPs concerned. Any planned or unplanned changes to the study plan and SOPs applied have been recorded as an amendment or deviation.

The delegated analytical phase has not been performed in compliance with the OECD Principles of GLP but according to the criteria of the ISO 17025 standard by the accredited Dutch laboratory Waterlaboratorium Noord (WLN).

Noted and approved:



18/06/2017

Tony Brouwers

Study Director

Study completion date:

Quality Assurance Statement

Title: 2 hours Bio-elution Study on **Stainless Steel 316L Massive** (Epoxy resin embedded) at a 2 g/L loading (surface equivalent) in a simulated gastric fluid.

Study no.: **X02a-081**

The study has been audited by the Quality Assurance Unit to assure compliance with the study plan, standard operating procedures and according to the OECD Principles of GLP. The findings of each study phase were reported to management and the Study Director. The audits took place on the following dates:

Audited Period of Project Study phase	Date	Reporting of findings to Study Director and Test Facility Management
study plan	02&09-11-2016	09-11-2016
Performance of the study: - final checking test item - experiment, gastric at pH 1.5	 11-11-2016 11-11-2016	08-01-2017
raw data	08-01-2017	08-01-2017
draft report	08-01-2017	08-01-2017
final report	18-06-2017	18-06-2017

This statement indicates that the reported results accurately and completely reflect the raw data generated by the study.

To the best of our knowledge, there has been no significant deviation from the OECD Principles of GLP that could have adversely affected the integrity of the study.

Noted and approved:



18/6/2017

Marleen Schreurs — Date:

Lead Quality Assurance Manager

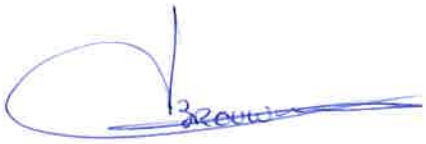
Statement of Authentication

Title: 2 hours Bio-elution Study on **Stainless Steel 316L Massive** (Epoxy resin embedded) at a 2 g/L loading (surface equivalent) in a simulated gastric fluid.

Study no.: **X02a-081**

The experiments of the study have been performed in the *Test Facility* according to the OECD Principles of GLP. The delegated analytical phase has been carried out by the accredited Dutch laboratory Waterlaboratorium Noord (WLN). The SOPs used by the *Test Site* have been accepted by the *Test Facility*. The raw data have been produced according to the instructions of the study plan and procedures. The results accurately reflect the raw data and show that the objectives of the study have been achieved.

Noted and approved:



18/06/2017

Tony Brouwers — Date:

Test Facility Manager

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1 Summary

Bio-elution refers to the *in vitro* extraction methods used to measure the degree to which a substance (e.g., metal or mineral ion) is dissolved in artificial biological fluids. Simulated biological fluids represent relevant exposure routes. The resulting value is the "bio-accessibility", and is defined as the "fraction of a substance that is soluble under physiological conditions and therefore potentially available for absorption into systemic circulation".

The objective of this study was to obtain knowledge about the bio-elution characteristics of **Stainless Steel 316L Massive** further noted as **"SS316L Massive"** in simulated gastric fluid. This study has been conducted according to the recommended Standard Operating Procedure (SOP) for Bioelution Testing of Metals, Inorganic Metal Compounds, and Metal-Containing Complex Materials: Simulated Gastric Fluid (Eurometaux, July 2016) which is based on ASTM D5517-07: Standard Test method for determining the extractability of metals from art materials - ASTM, 2007 (American Society for Testing and Materials). The extent of dissolution of epoxy embedded **SS316L Massive** was tested in a simulated gastric fluid at 37 °C and pH 1.5 during 2 hours (2 g/L loading "surface equivalent") at an agitation speed of 100 revolutions per minute (rpm). A "reference particle size" of 1 mm, similar to the transformation/ dissolution protocol, was used to calculate the required exposure surface because the test material is usually available as a massive (i.e. >1 mm). The bio-elution endpoints were based on the dissolved cobalt (Co) and nickel (Ni) concentrations obtained after 2 hours of extraction.

The following deviations from the SOP were made:

- The samples were preserved by adding 0.12 mL concentrated HNO_3 per 12 mL instead of no preservation in the SOP.
- 250 mL Erlenmeyer test vessels were used in this study (instead of 500 mL vessels according to the SOP) as this fitted best the needs related to the available free metal surface and the added volume of test medium.

The study was performed at ECTX. Analysis of the concentrations of dissolved cobalt and nickel have been performed at Waterlaboratorium Noord (WLN) (The Netherlands), an ISO 17025 accredited laboratory, as delegated by ECTX.

The measured pH of the test medium at the start of the test was 1.50, i.e. within the specifications of pH 1.5 ± 0.1. The pH measured in the additional test item vessel at the start of the test was 1.50 i.e. within the specifications of pH 1.5 ± 0.1. At the 2 hours sampling point of the test, the pH in the blank control vessels and the test vessels was between 1.50 and 1.51.

The temperature of the sampled test solutions including the blank test vessel was between 36.8 °C and 37.1 °C and corresponded to the required test conditions of 37 °C ± 1 °C.

The blank control vessels showed small concentrations of cobalt and nickel above the detection limits of 0.003 µg/L Co and 0.04 µg/L Ni, respectively.

In the blank corrected test item vessels with a loading of 2 g/L (surface equivalent) epoxy embedded **SS316L Massive**, the following average dissolved cobalt and nickel concentration of:

- 0.127 ± 0.006 µg/L Co ($CV_{between\text{-}vessel}$ = 5 %; N = 3),
- 3.11 ± 0.17 µg/L Ni ($CV_{between\text{-}vessel}$ = 5 %; N = 3),

were measured after 2 hours of exposure to the simulated gastric fluid (pH 1.5).

Based on the specific surface area of epoxy embedded **SS316L Massive** (i.e. 0.000749 m²/g), this corresponds with:

- a cobalt release per surface of 0.085 mg/m²,
- a nickel release per surface of 2.1 mg/m².

Based on the cobalt content (≤0.18 %) and nickel content (11.2 %) in the test item and the average dissolved cobalt and nickel concentrations in the test solutions:

- a cobalt release of ≥0.0035 %,
- a nickel release of 0.0014 %,

could be calculated at the 2 hours endpoint.

The results of this study can be used as a tool to correct the hazard classification, to establish categories for grouping or to justify test waivers.

2 Identification of the Study, Test Item and Reference Item

2.1 Study title and number

Title: 2 hours Bio-elution Study on **Stainless Steel 316L Massive** (Epoxy resin embedded) at a 2 g/L loading (surface equivalent) in a simulated gastric fluid.

Study no.: **X02a-081**

2.2 Test Item identification and characterisation

The test item, **SS316L Massive**, as received, is representative of the material as it is, or will be, on the market for use. Further preparation of the test item was required to conduct this study (embedding and polishing). See also Section 7.2 Preparation of the test item.

The information about the identification and characterisation of the test item was supplied by the Sponsor at the start of the study.

Test Item identification

Test item name:	**Stainless Steel 316L Massive**
Abbreviation used in the text	**SS316L Massive**
Supplier/Manufacturer:	Outokumpu Pte Riihitontuntie 7 Espoo, FI 02201 Finland
Supplier/Manufacturer contact:	Dr. V. Schulz-Klemp [+358 3 5894211]
Sample delivered by:	Particle Technology Labs 555 Rogers Street, Downers Grove IL 60515 USA Arnette Schultz [+1-(630)-969-2703 x118]
Sample ID:	N152.1.RE.3-PTL
ECTX no.:	**T0221/02**

Test Item characterisation

Physicochemical method of production:	n.a.[1]
Element(s) of interest:	Cobalt, nickel
Composition measured by PIXE (typical range):	68.1 % iron (Balance) 16.2 % chromium (16 % – 28 % W) 11.2 % nickel (2.5 % – 23 % W) 2.3 % molybdenum (1 % – 6.5 % W) 1.1 % manganese (0 % – 2 % W)

[1] n.a. not available/applicable

	0.65 % silicon (0 % – 1 % W) 0.25 % copper (0 % – 1 % W) 0.049 % titanium (0 % – 0.7 % W) 0.12 % aluminium <0.18 % cobalt (0 % – 0.6 % W) A certificate of analysis of the tested batch is added to this study report. (Annex 6)
Physical form:	Solid
Colour:	Grey
Particle size distribution:	4 samples, 10 mm x 10 mm x 2 mm of 2.5 g - 2.6 g
Surface area (free metal):	**T0221/02-1**: 101 mm² **T0221/02-2**: 101 mm² **T0221/02-3**: 101 mm² **T0221/02-4**: 101 mm²
Homogeneous:	n.a.
Relative Density:	8.0

Storage conditions

Room temperature, in the dark.

Expiry date

Stable over time.

Safe Handling Precautions

A safety data sheet on **Chromium-Nickel-Molybdenum-Alloyed Stainless Steel grades** is available.

2.3 Reference Item identification and characterisation

No reference item was used in this study.

3 Information concerning Sponsor, Test Facility and Test Site

3.1 Sponsor identification

Sponsor:	**Nickel Producers Environmental Research Association (NiPERA)** 2525 Meridian Parkway, Suite 240 Durham, North Carolina 27713 USA
Study Monitor on behalf of the Sponsor:	**Katherine Heim**

3.2 Test Facility identification

ECTX bvba, Havenstraat 46/0.01, B-3500 Hasselt, Belgium.	
Study Director:	**Tony Brouwers**
Lead Quality Assurance Manager:	Marleen Schreurs
Test Facility Manager:	Tony Brouwers

3.3 Test Site identification

Waterlaboratorium Noord b.v. (WLN), Rijksstraatweg 85, NL-9756 AD Glimmen, The Netherlands. (ISO 17025 Accredited) RVA (Raad Voor Accreditatie) Accreditation certificate no. L075 valid from 12-11-1993 to 01-12-2017.	
Contributing Scientist:	Jan Bonnema
Quality Control Manager:	Jeroen Boekenoogen
Test Site Management:	Jan van der Kooi / Harm de Kleine

4 Dates

Study Initiation Date:	**09-11-2016**
Experimental Starting Date:	**11-11-2016**
Experimental Completion Date:	**05-12-2016** (with specific analyses)
Study Completion Date:	Date of approval of this Final Report by the Study Director
Remarks:	The test was carried out on **11-11-2016**
	The samples were delivered to the analytical lab (WLN) on **21-11-2016**
	Specific analyses were carried out on **22-11-2016**

5 Test methods

5.1 Test guidance

The conduct of the study is based on the Standard Operating Procedure (SOP) for Bioelution Testing of Metals, Inorganic Metal Compounds, and Metal-Containing Complex Materials: Simulated Gastric Fluid (Eurometaux, July 2016).

5.2 Standard Operating Procedures (SOPs)

5.2.1 SOPs of ECTX

The SOPs versions used in the study at the Test Facility were valid at the start of the experiment and were listed below:

- SOP IN-01: Instrumentation – µpipettes,
- SOP IN-02: Instrumentation – pH, temperature, dissolved oxygen and conductivity measurements,
- SOP AB-09: Recipe book,
- SOP IA-01: Instrumentation – balances,
- SOP TX-02: bio-elution tests,
- SOP AB-10: Cleaning and maintenance,
- SOP AB-11: Waste management,
- SOP AB-14: Analytical measurements by an external laboratory.

5.2.2 SOPs of Waterlaboratorium Noord

The SOPs versions used in the study at the Test Site were valid at the start of the experiment and were listed below:

- SOP WLN-MHB.07: Receipt, handling and storage of samples,
- SOP WLN-CM.W.11.1: Elemental determination by ICP-MS technique, SFS-EN ISO 17294-2,
- SOP WLN-OK.KHB.05 5.10: Reporting of results,
- SOP WLN-OK.P.39.1: Discarding of waste/samples.

6 Test System

6.1 Simulated gastric fluid

Simulated gastric fluid (0.07 N HCl) was prepared according to recipe 63 of SOP AB-09 (recipe book). The pH of the medium was brought to pH 1.50, using a NaOH solution. The chemical composition of the test medium is shown in the following table:

HCl (30 %)	7.41 mL
Ultrapure water	Up to 1 L
NaOH solution	Few drops to adjust pH to pH 1.5 ± 0.1 (as reported in ASTM 5517)

7 Experimental Design

7.1 Materials

- Usual laboratory equipment,
- Analytical balance Mettler Toledo XP204S (SNR1129023848),
- Balance Mettler Toledo XP8002S (SNR1129020806),
- Thermometer Testo 875-1i (SNR2738028),
- pH-meter (HACH HQ40d multimeter SNR080100016916),
- Thermostatic laboratory shaker Thermo Scientific SHKE8000-8CE (SNR109245-80) (stroke length: 2.54 cm),
- New, pre-cleaned and acid rinsed closed borosilicate 3.3 Erlenmeyer flasks (250 mL),
- Demineralised water was prepared by a MilliQ Integral 5 System: conductivity 18 MΩ; Total Organic Carbon = 3 ppb C,
- Simulated gastric fluid (SOP AB-09: Recipe book: recipe 63),
- 10N NaOH solution,
- Dispensette, Brand Dispensette III digital 5-50 mL (SNR08K21110),
- 0.2 µm Pall Acrodisc® 25 mm syringe filters (PN 4612),
- 12 mL Latex-and oil-free syringes of for sampling (HSW Norm-Ject Ref 4100.000Spec),
- 15 ml 120x17 mm conical PP sample tubes, (Deltalab Ref 429910),
- Automatic pipettes (Brand Transferpette®, Eppendorf Multipette®),
- Concentrated HNO_3 (67-69%), PlasmaPur, SCP SCIENCE 250-037-175PL,
- ICP-MS: Thermo Scientific iCAP™ Q ICP-MS (Waterlaboratorium Noord).

7.2 Preparation of the Test Item

The test material, 4 slices of **SS316L Massive** (approx. 10 mm x 10 mm x 2 mm), was delivered by Particle Technology Labs on request of the Sponsor.

The preparation of the samples (cutting, embedding and polishing) was beyond the scope of this study plan and was not performed according to the principles of GLP. However, a brief description of the sample preparation was given below.

Massives were tested at a particle size, representative of normal handling and use (cfr. Transformation/ Dissolution test guidance). A default diameter value of 1 mm of a spherical particle was used in absence of this information. A spherical particle with a diameter of 1 mm has a surface of 3.14 mm^2 ($4 \cdot \pi \cdot r^2$) and a volume of 0.524 mm^3 ($\frac{4}{3} \cdot \pi \cdot r^3$). In the case of **SS316L Massive** (density 8.0 mg/mm^3), a Ø 1 mm spherical reference particle has a volume of 0.524 mm^3 and a mass of 4.19 mg. Therefore, a 2 g/L loading was containing 477 reference particles corresponding with a total free metal surface of 1498 mm^2/L. The required loading of 2 g/L was achieved by adding an appropriate amount of test medium to the actual exposed free metal surface after embedding (i.e. 101 mm^2).

The test item embedding, grinding and polishing was performed at ECTX using, as close as possible, the following procedure:

Cutting the massive metal

No cutting was needed. Four pieces of **SS316L Massive** plate were delivered by the sponsor and were ready to prepare four epoxy resin discs.

Epoxy embedding

One piece of **SS316L Massive** was embedded per epoxy resin sample (EpoFix Kit from Struers). The polymerisation process was done as directed by Struers as follows: To prepare the resin fluid an amount of hardener was mixed into the correct amount of resin (25 parts of resin / 3 parts of hardener by weight). The mixture was mixed well for approx. 2 minutes without introducing too many air bubbles. The mixture was left to rest for 2 minutes. (Pot life of this mixture: about 30 minutes). The epoxy was carefully poured over the specimen in an achieved mold. A vacuum system was used to reduce air bubbles. The embedded samples were left at room temperature for hardening during at least 12 hours.

Surface preparation procedure

Each epoxy sample was wet ground with 400 grit, 800 grit, 1200 grit and finally 2000 grit SiC paper in sequence. In order to avoid maintaining a memory imprint of the grinding scratches deeper into the bulk material matrix, it was important to apply only a very gentle pressure during the grinding. So each sample was ground only for approximately one minute on each grit size paper with very gentle pressure. After wet grinding, the samples were rinsed with ethanol and dried with nitrogen gas to avoid corrosion during the sample preparation.

Polishing was performed using 3, 1 and 0.25 µm diamond paste (DP-Paste M) in turn with ethanol as a lubricant, and with utmost care. To obtain smooth scratch-free surfaces, the samples were held very gently against the polishing cloth with very little applied pressure. Plenty of ethanol was used as a lubricant to obtain a well-defined surface. Each polishing step with diamond paste took about 5 to 10 minutes, depending on the removal of scratches. After polishing the samples were cleaned with ethanol and quickly dried with cold nitrogen gas.

To remove any residual particles left on the surface after grinding and polishing, all samples were cleaned ultrasonically first in ethanol for 6 minutes followed by ultrasonic cleaning in isopropyl alcohol for 6 min, and a final rinsing with isopropyl alcohol. Isopropyl alcohol was used as the last cleaning medium. After the ultrasonic cleaning, all samples were dried quickly again with cold nitrogen gas.

The polished surface was checked by means of a microscope. Finally, the metal surfaces were free from scratches and spots.

Aging time (after polishing)

The samples with an average free metal surface of about 100 mm² were stored for 24 ± 1 hours before testing. The Sponsor also provided supporting documents which characterises the test item (Certificate of Analysis: Annex 6).

7.3 Test conditions

- Temperature controlled at 37 °C ± 1 °C, in the dark,
- pH initially set at pH 1.5 ± 0.1,
- Agitation speed 100 rpm in a temperature controlled orbital laboratory shaker (orbital size: 1 inch, 2.54 cm).

7.4 Performance

7.4.1 Test set-up

One borosilicate Erlenmeyer flask of 250 mL was used for the blank control vessel containing a disc of epoxy resin without test item. In the test vessels, 101 mm² test item ‘free metal surface’ (epoxy resin

containing 1 piece of **SS316L Massive**) were introduced into three separate 250 mL Erlenmeyer flasks. A fourth replica with test item (named replica X) was prepared only for the measurement of the initial pH (at the start of the test). This additional test vessel was set up to avoid cross contaminations and did not have further utility in the test. An appropriate amount of extraction fluid (37 °C ± 1 °C) was added to the blank control vessel and each test item vessel (see table below) resulting in a final loading of 2 g/L 'surface equivalent'. After covering the vessels with a screwcap and swirling the flasks to mix the test item and the medium, the flasks were placed into a thermostatic orbital shaker (37 °C ± 1 °C) at an agitation rate of 100 revolutions per minute for one hour. After that, the flasks were settled at 37 °C ± 1 °C for another hour. An overview of the test set-up is shown in the following table:

Time	Target loading (g/L)	Number of replica test vessels	Test item ID	Dimensions of the test item (mm)	Test item surface (mm^2)	Test medium added
0 hours	2	Replica X*	T0221/02-4	10.0 x 10.1 mm	101 mm^2	67 mL
2 hours	0	Blank control	a	-	-	67 mL
	2	Test item repl.1	T0221/02-1	10.0 x 10.1 mm	101 mm^2	67 mL
	2	Test item repl.2	T0221/02-2	10.0 x 10.1 mm	101 mm^2	67 mL
	2	Test item repl.3	T0221/02-3	10.0 x 10.1 mm	101 mm^2	67 mL

a blank resin disc
* replica X was added to check the initial impact of the test item to the pH of the test system and was discarded after the start of the test.

The test was performed in a thermostatic shaker in the dark.

7.4.2 Sampling during the test

The following sampling procedure was used at the sampling time to collect and preserve the samples for ICP-MS analyses:

At the end of the incubation period (2 hours = 1 hour shaking at 100 rpm and 1 hour settling), each vessel was swirled to homogenise the solution prior to sampling, in order to avoid a concentration gradient after settling.

Sampling was performed for the blank control and each test item vessel as indicated in the sampling scheme below. In each test vessel, a 12-mL sample was taken three times with a 12 mL syringe from the test vessel at a depth of two-thirds of the supernatant. The samples were filtrated through a 0.2 µm syringe filter and transferred to uniquely labelled 15-mL PP sample tubes. The samples were preserved by adding 0.12 mL concentrated HNO_3 per 12 mL. The samples were covered (to avoid evaporation and concentration) and stored at room temperature in the dark until shipment to the Test Site (see *3.3 Test Site identification*).

Each sample was labelled with a unique number and with an expiry date that is one month after sampling:

e.g. X02a-081-**1** where:

X02a-081 is the study number,

1 is the sample number.

7.4.3 Sampling scheme

For the test vessels with simulated gastric fluid the following sampling scheme has been applied:

Sampling time (h)	Number of samples		Total number of samples
	Medium (blank) 0 mg/L	Medium + test item 2 g/L	
2	3*1	3*3	12
total number of samples:			12

A detailed list is given in Annex 4.

7.4.4 Observations and measurement of temperature and pH

At the start of the test (0h), pH was measured in the additional (fourth) replica with test item (named replica X). The addition of the test item to the test system had no impact on the initial pH of the test system, so pH adjustment using an HCl or NaOH solution was not necessary.

After sampling, temperature and pH were measured in the remaining blank control and test solutions after sampling.

The details are presented in Annex 1.

7.4.5 Shipment of the samples to the Test Site

The samples and datasheet DSH026 Sample Transfer (external), which contains a chain of custody and a detailed sample list with storage conditions were transferred to the Test Site (see *3.3 Test Site identification*) in an appropriate manner according to SOP AB-14 (see *5.2.1 SOPs of ECTX*).

7.4.6 Analyses of the samples by the Test Site

The determination of dissolved cobalt and nickel concentrations in the blank control and test item vessels were carried out at the Test Site (see *3.3 Test Site identification*), using an ICP-MS (SOP WLN-CM.W.11.1 Elemental determination by ICP-MS technique, SFS-EN ISO 17294-2). A detection limit for cobalt and nickel was determined as the rounded up value of three times the standard deviation of all blank measurements (N = 6) sampled in this study (3 samples) and the blank sampled in study X02a-082->085 (3 samples). Study X02a-082->085 ran with the same batch of test medium under the same test conditions but did not contain a blank epoxy sample. For reporting, values below the detection limit (DL) are reported as '<0.003 µg/L' for cobalt and '<0.04 µg/L' for nickel, otherwise rounded values were used, if relevant. To describe the dissolution behaviours even below the accredited reporting limits of WLN and to allow more accurate toxicity calculations, raw data were used to perform calculations. For values between the detection limit and the accredited reporting limits (i.e. 0.5 µg/L for Co and Ni), an accuracy of 90 % - 110 % cannot be guaranteed.

The analyses were not performed in compliance with the GLP principles, but according to the ISO 17025 standard.

Each sample, possibly after proper dilution, was measured once.

7.4.7 Reports from the Test Site

An analytical report of the delegated phase including the raw data has been written and approved by the Test Site Scientist. The analytical report does not contain a QA Statement because the tests have been carried out according to the criteria of the ISO 17025 standard which does not require a QA Statement.

The analytical report of the delegated phase was sent to the Study Director on 05-12-2016 and is added as an annex to this study report (Annex 5).

7.5 Data analysis

The data from the analytical laboratory are expressed in µg element per L solution.

These data are recalculated (blank corrected) and expressed in µg element per g test item and as percent metal release of the total available element.

The results are also expressed as released element per surface (mg/m²).

The within-vessel, between-vessel and overall means, standard deviations, coefficients of variation of the measured dissolved element concentrations will be calculated using the equations in the table below:

	Mean	Standard deviation	Coefficient of variation
Within (for each vessel i)	$x_{avg,i} = \frac{1}{m}\sum_{j=1}^{m} x_{ij}$	$s_{within,i} = \left(\frac{1}{m-1}\sum_{j=1}^{m}\left(x_{ij} - x_{avg,i}\right)^2\right)^{1/2}$	$CV_{within,i} = \frac{s_{within,i}}{x_{avg,i}} \cdot 100\%$
Between	$x_{avg} = \frac{1}{n}\sum_{i=1}^{n} x_{avg,i}$	$s_{between} = \left(\frac{1}{n-1}\sum_{i=1}^{n}\left(x_{avg,i} - x_{avg}\right)^2\right)^{1/2}$	$CV_{between} = \frac{s_{between}}{x_{avg}} \cdot 100\%$
Overall	$x_{avg} = \frac{1}{n \cdot m}\sum_{i=1}^{n}\sum_{j=1}^{m} x_{ij}$	$s = \left(\frac{1}{n \cdot m - 1}\sum_{i=1}^{n}\sum_{j=1}^{m}\left(x_{ij} - x_{avg}\right)^2\right)^{1/2}$	$CV = \frac{s}{x_{avg}} \cdot 100\%$

A within-vessel coefficient of variation of <10 % for each vessel and a between-vessel coefficient of variation of <20 % are quality criteria for the 2 hours endpoint.

With very low concentrations close to the detection limit of the analytical method, such as may be with blanks or with a substance that releases element concentrations close to the detection limit, the between-vessel coefficient of variation can be expected to exceed this target coefficient of variation. Moreover, should the substance be characterised by a wide particle size range, the possibility exists that this coefficient of variation target may not be readily met.

8 Results

The temperature of the test solutions at the sampling point was between 36.8 °C and 37.1 °C, which was in line with the test conditions of 37 °C ± 1 °C (Annex 1).

The measured pH of the test medium at the start of the test was 1.50, i.e. within the specifications of pH 1.5 ± 0.1. The pH measured in the additional test item vessel at the start of the test was 1.50 i.e. within the specifications of pH 1.5 ± 0.1. At the 2 hours sampling point of the test, the pH in the blank control vessels and the test vessels was between 1.50 and 1.51. (Annex 1).

The raw data of the dissolved cobalt and nickel concentrations in the test samples, measured by the Test Site Waterlaboratorium Noord, are presented in Annex 5.

Measurements showed that values below the reporting limit of 0.5 µg/L for cobalt and nickel were very reproducible. To describe the dissolution behaviors even below the reporting limits of WLN and to allow more accurate toxicity calculations, detection limits were determined as the rounded-up values of three times the standard deviation of the cobalt and nickel measurements of all blank control samples in this study and studies X02a-082->085 (total of blank measurements: N = 6). These detection limits, 0.003 µg/L Co for cobalt and 0.04 µg/L Ni for nickel are applied in this study report as reporting limits. The blank control vessels during the test showed low concentrations above the detection limit of cobalt (i.e. 0.003 µg/L Co) and nickel (i.e. 0.04 µg/L Ni) (Annex 1).

The following observations could be made in the test vessels with a loading of 2 g/L (surface equivalent) epoxy embedded **SS316L Massive**:

- No visible changes of the test material could be observed at the end of the experiment. The test solution remained colourless.
- An average dissolved cobalt concentration of 0.127 ± 0.006 µg/L Co (or 0.063 ± 0.003 µg/g test item) was measured after 2 hours of exposure to the simulated gastric fluid (pH 1.5) with a between-vessel variation of 5 % which meets the 20 % quality criterion at the 2 hours endpoint. The within-vessel coefficients of variation met the <10% quality criteria for all test vessels at the 2 hours endpoint.

  Based on the specific surface area of epoxy embedded **SS316L Massive** (0.000749 m²/g test item) a cobalt release per surface of 0.085 mg/m² was calculated.

  Based on the cobalt content of the test item (≤0.18 %) and the average dissolved cobalt concentration, a cobalt release of ≥0.0035 % could be calculated at the 2 hours endpoint.
- An average dissolved nickel concentration of 3.11 ± 0.17 µg/L Ni (or 1.55 ± 0.08 µg/g test item) was measured after 2 hours of exposure to the simulated gastric fluid (pH 1.5) with a between-vessel variation of 5 % which meets the 20 % quality criterion at the 2 hours endpoint. The within-vessel coefficients of variation met the <10% quality criteria for all test vessels at the 2 hours endpoint.

  Based on the specific surface area of epoxy embedded **SS316L Massive** (0.000749 m²/g test item) a nickel release per surface of 2.1 mg/m² was calculated.

  Based on the nickel content of the test item (11.2 %) and the average dissolved nickel concentration, a nickel release of 0.0014 % could be calculated at the 2 hours endpoint.

All raw data can be found in Annex 1-6 of this study report.

9 Conclusion

During this study on epoxy embedded **SS316L Massive** at a loading of 2 g/L (surface equivalent) in simulated gastric fluid (pH 1.5), it was shown that following amounts of cobalt and nickel were measured.

- For cobalt an average value of 0.127 µg/L ($CV_{between\text{-}vessel}$ = 5 %; N = 3) was found after 2 hours of extraction, corresponding with a cobalt release of ≥0.0035 % (or 0.085 mg/m²).
- For nickel an average value of 3.11 µg/L ($CV_{between\text{-}vessel}$ = 5 %; N = 3) was found after 2 hours of extraction, corresponding with a nickel release of 0.0014 % (or 2.1 mg/m²).

The results can be assumed reliable since the test conditions stayed constant during the experiment.

10 Deviations from the original Study Plan

None.

11 Archives

All the materials and records necessary to reconstruct the study have been maintained in the archives of ECTX in a file coded **X02a-081**. The Study Plan, the lab notebook, the raw data, the correspondence and final report has been stored in a locked filing cabinet. The used, embedded test item samples were archived in a suitably sealed vial and stored under the storage conditions mentioned.

The stock solutions, specimens and test and calibration solutions used during the study have not been archived and have been disposed of by ECTX and WLN according to the instructions of their corresponding SOPs when their expiry date has been exceeded or when their original characteristics could not be guaranteed anymore.

The duration of archiving is five (5) years after the date of approval of the Final Report by the Study Director. After this period the Sponsor decides if:

- The archiving of the records and materials should be continued;
- The archived material should be sent to the Sponsor;
- The archived material should be destroyed.

12 References

a) Eurometaux (2016): **Standard Operating Procedure (SOP) for Bioelution Testing of Metals, Inorganic Metal Compounds, and Metal-Containing Complex Materials: Simulated Gastric Fluid** (Eurometaux Brussels, July 2016).

b) ASTM D5517-07 (2007): **Standard Test Method for Determining Extractability of Metals from Art Materials**, Philadelphia, PA: American Society for Testing and Materials, 2007.

c) Henderson R.G., V. Verougstraete, K. Anderson, J.J. Arbildua, T.O. Brock, T. Brouwers, D. Cappellini, K.Delbeke, G. Herting, G. Hixon, I. Odnevall Wallinder, P.H. Rodriguez, F. Van Assche, P. Wilrich and A.R. Oller (2014). **Interlaboratory Validation of Bioaccessibility Testing for Metals.** Regul Toxicol and Pharmacol. 70(1): 170-181.

d) OECD (1981-2016) **OECD Series on Principles of Good Laboratory Practice and Compliance Monitoring.**

Annexes

Annex 1:

Data of measured temperature, pH and dissolved cobalt and nickel concentrations during the 2 hours extraction period of 2 g/L (surface equivalent) epoxy embedded **SS316L Massive** in simulated gastric fluid at pH 1.5. (COBALT AND NICKEL VALUES OF THE TEST ITEM SAMPLES ARE BLANK CORRECTED)

2 g/L test item (surf.equiv.)		2h			
pH 1.5 (1 h at 100 rpm)		µg/L Co	µg/L Ni	T	
Detection Limit		0.003	0.04	(°C)	pH
test medium				37.1	1.50
replicaX				37.0	1.50
blank repl1	spl1	0.021	0.23	37.1	1.51
	spl2	0.020	0.21		
	spl3	0.021	0.21		
	$x_{avg,1}$	0.021	0.22		
	$s_{within,1}$	0.001	0.01		
	$CV_{within,1}$ (%)	4	6		
replica1	spl1	0.123	2.89	37.0	1.50
	spl2	0.125	2.92		
	spl3	0.132	3.02		
	$x_{avg,1}$	0.126	2.94		
	$s_{within,1}$	0.005	0.07		
	$CV_{within,1}$ (%)	4	2		
replica2	spl1	0.120	3.14	36.8	1.50
	spl2	0.125	3.15		
	spl3	0.120	3.02		
	$x_{avg,2}$	0.122	3.10		
	$s_{within,2}$	0.003	0.08		
	$CV_{within,2}$ (%)	2	2		
replica3	spl1	0.133	3.26	36.9	1.51
	spl2	0.134	3.24		
	spl3	0.132	3.33		
	$x_{avg,3}$	0.133	3.28		
	$s_{within,3}$	0.001	0.04		
	$CV_{within,3}$ (%)	1	1		
	x_{avg}	0.127	3.11		
	$s_{between}$	0.006	0.17		
	$CV_{between}$ (%)	5	5		
	s	0.006	0.15		
	CV (%)	5	5		

$x_{avg,v}$	within vessel average of 3 samples where v is the replica test vessel number (N=3)
$s_{within,v}$	within vessel Standard Deviation where v is the replica test vessel number (N=3)
$CV_{within,v}$	within vessel Coefficient of Variation where v is the replica test vessel number (N=3)
x_{avg}	average of 3 test vessels (3 samples per test vessel; blank corrected) (N=9)
$s_{between}$	between vessel Standard Deviation (N=3)
$CV_{between}$	between vessel Coefficient of Variation (N=3)
s	overall Standard Deviation (N=9)
CV	overall Coefficient of Variation (N=9)

Annex 2:

Bioaccessibility of cobalt and nickel at 2 hours released from 2 g/L (surface equivalent) epoxy embedded **SS316L Massive** in simulated gastric fluid (blank corrected, if the blank values are greater than the detection limit, otherwise blank assumed as 0).

Time	2h	
	Co release (µg/g Test Item)	Ni release (µg/g Test Item)
replica1	0.063	1.47
replica2	0.061	1.55
replica3	0.067	1.64
x_{avg}	0.063	1.55
$s_{between}$	0.003	0.08
CV (%)	5	5

x_{avg} = average of 3 test vessels (blank corrected) (N=3).
$s_{between}$ = standard deviation (N=3).
CV = Coefficient of Variation (%).

Annex 3:

Summarized results of the average dissolved concentration for cobalt and nickel the release per g test item, the percentage of cobalt and nickel eluted, and the corresponding release per surface, after 2 hours of extraction of 2 g/L (surface equivalent) epoxy embedded **SS316L Massive** in simulated gastric fluid at pH 1.5:

SS316L Massive, loading 2 g/L (surface equivalent) (= 0.001498 m²/L surface loading)			
		2-h gastric at pH 1.5	
Analyte		$x_{avg} \pm s_{between}$	CV
cobalt (Co)	dissolution	0.127 ± 0.006 µg/L	5 %
	absolute Co release	0.063 ± 0.003 µg/g	5 %
	% of total available Co eluted	≥0.0035 %	
	Co release/surface	0.085 mg/m²	
nickel (Ni)	dissolution	3.11 ± 0.17 µg/L	5 %
	absolute Ni release	1.55 ± 0.08 µg/g	5 %
	% of total available Ni eluted	0.0014 %	
	Ni release/surface	2.1 mg/m²	

x_{avg} = average of 3 test vessels (3 samples per test vessel; blank corrected).

$s_{between}$ = between vessel standard deviation (N=3).

CV = Coefficient of Variation (%).

Annex 4:

A detailed overview of the samples.

Sample no.	Sampling time (h)	pH	Loading (g/L)	Sample treatment	Comment
X02a-081-1	2	1.5	0	filtration (0.2 µm)	Blank replica1 spl1
X02a-081-2	2	1.5	0	filtration (0.2 µm)	Blank replica1 spl2
X02a-081-3	2	1.5	0	filtration (0.2 µm)	Blank replica1 spl3
X02a-081-4	2	1.5	2	filtration (0.2 µm)	T0221/02 replica1 spl1
X02a-081-5	2	1.5	2	filtration (0.2 µm)	T0221/02 replica1 spl2
X02a-081-6	2	1.5	2	filtration (0.2 µm)	T0221/02 replica1 spl3
X02a-081-7	2	1.5	2	filtration (0.2 µm)	T0221/02 replica2 spl1
X02a-081-8	2	1.5	2	filtration (0.2 µm)	T0221/02 replica2 spl2
X02a-081-9	2	1.5	2	filtration (0.2 µm)	T0221/02 replica2 spl3
X02a-081-10	2	1.5	2	filtration (0.2 µm)	T0221/02 replica3 spl1
X02a-081-11	2	1.5	2	filtration (0.2 µm)	T0221/02 replica3 spl2
X02a-081-12	2	1.5	2	filtration (0.2 µm)	T0221/02 replica3 spl3

Annex 5:

Analysis of the dissolved element concentrations at the *Test Site,* WLN

- Analytical Report 16R08794.X02a-081 (8 pages)

wln
water | onderzoek | advies

Postbus 26
9470 AA Zuidlaren
Rijksstraatweg 85
9756 AD Glimmen
telefoon (050) 402 21 21
telefax (050) 409 42 74

e-mail info@wln.nl
website www.wln.nl

Analytical Report 16R08794.X02a-081

(05/12/2016)

Project: **11R0653/110**

Determination of cobalt and nickel in Simulated Gastric Fluid: ECTX Study X02a-081

Submitted to:

ECTX bvba
(Ecotoxicology & Biodegradation)
Havenstraat 46/0.01
B-3500 Hasselt, Belgium

Waterlaboratorium Noord (WLN), Rijksstraatweg 85, NL-9851 AD Glimmen, The Netherlands
(ISO 17025 Accreditation certificate n° L075 from 12/11/1993, valid until 01/12/2017)

Scientist: **Jan Bonnema**

Management: **Jan van de Kooi / Harm de Kleine**

Analyst (ICP-MS): **Jan Bonnema**

Quality Control Manager: **Jeroen Boekenoogen**

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CONFIDENTIALITY AND RETENTION PERIODS, DATA AND MATERIALS

WLN supplies data and results to the client only. Information to third parties will only take place after receiving a written consent from the client. Samples will be stored under controlled conditions until the research has been fully completed. This period can be extended on the clients' request. All information regarding the analyses or/and research, including rough analysis data, will be kept over a period of at least 5 years. A copy of the raw analysis data will be handed to the study director of ECTX after finalisation of the analytical report.

DISCLAIMER

Any checks, opinions and interpretations in the report fall outside of the RvA accreditation. The results, if relevant, only concern the analysed items.

Any determination and/or reference made in this report with respect to any specific commercial product, process or service by trade name, trademark, manufacturer or otherwise shall be considered to be opinion; WLN makes no, and does not intend to make any, representations or implied warranties of merchantability or fitness for a particular purpose nor is it intended to endorse, recommend or favour any specific commercial product, process or service. The views and opinions of authors expressed herein do not necessarily state or reflect those of WLN and may not be used for advertising or product endorsement purposes.

1. INTRODUCTION

At the request of ECTX, we have determined the concentrations of cobalt (**Co**) and nickel (**Ni**) in Simulated Gastric Fluid solutions (0.07N HCl) that they sent to us for analysis. The data may be a component of the dossiers submitted to the REACH (Registration, Evaluation and Authorisation of Chemicals) registry, an environmental protection measure within the framework of the European Union (EU), reporting provisions of which producers, importers, users and distributors are obligated to meet in order to maintain access to their EU markets.

In this report, we present the analyses for **Co** and **Ni** in ECTX samples of study X02a-081 as determined by the analytical laboratory of WLN. The sampling was performed by ECTX.

2. ANALYTICAL METHODOLOGY

The analyses of cobalt and nickel was performed according to our Standard Operating Procedure "WLN-CM.W.11.1: Determination of the metal content in water by means of an inductively coupled plasma and mass-spectrometry", which is conform NEN-EN-ISO 17294-2:2016 Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: "Determination of selected elements including uranium isotopes" and which was valid at the time when the analyses were performed.

Waterlaboratorium Noord (WLN) used a Thermo Fisher Scientific iCAP Q ICP-MS (inductively-coupled plasma-mass spectrometer) to determine the total dissolved concentrations of cobalt (**Co**) and nickel (**Ni**) in the gastric and blank matrix solutions received from ECTX. To prepare blanks, calibration and control solutions/dilutions of de-ionized water (d.i. H_2O) at a purity level of 18 MΩ was used.

The ICP-MS was calibrated at the start of each run. Recalibration was performed every 25 samples and again at the end of the run. These recalibrations were also used for instrument drift corrections.

The linearity (2x calibration level) and the factors of interference were determined and checked after the initial calibration.

The calibration standard solution used was a custom made multi-element standard (iV inorganic Ventures).

Matrix corrections were performed by the online addition of an internal standard.

Each run also contained at least one blank procedure.

All measurements were checked for "carry over" as also the RSD of the three-integrations of each measurement.

For each measurement the 'drift' of the internal standard was checked.

The calibrations were checked by means of an independent control standard (another supplier, either SCP Science or J.T. Baker), which was prepared using single element standards.

All measured values of the control standards were evaluated against the control charts. These values were expected to be within 2 sigma levels of the control.

The last control sample value was transferred to the WLN laboratory information management system (LIMS).

Table 1: Operating conditions for Thermo Fisher Scientific iCAP Q ICP-MS

- Instrument: **Thermo Fisher Scientific iCAP Q ICP-MS (serial no.: 730R, WLN no.: 730000 / serial no.: 731R, WLN no.: 731000).**
- Forward power: **1550** W
- Cool gas flow rate: **14.00** L/min
- Auxiliary gas flow rate: **0.80** L/min
- Number of replicates analyses: **1 (average of 3 integrations)**
- Isotope monitored: **^{59}Co, ^{60}Ni**

Table 2: Performance characteristics - Measured parameters accredited for drinking water, groundwater, surface water

- Reporting limit: **0.5 µg/L** for Co and Ni (= according to our SOP)
- Repeatability: **≤10%** (at the reporting limits)
- Reproducibility: **≤10%** (at the reporting limits)
- Accuracy: **90-110%** (at the reporting limits)

The operating conditions of each run are described in a performance report which is printed and archived.

All data are handled and stored according to our quality system .

3. DEVIATIONS FROM THE SOP IN RELATION TO THE EXECUTED ANALYTICAL METHOD

On request of the study director, raw data instead of rounded data (according to our SOP) were reported as also values below the validated reporting limit. For values below the validated reporting limits an accuracy between 90-110% cannot be guaranteed.

4. COMMENTS CONCERNING THE SAMPLES

None.

5. COMMENTS CONCERNING THE ANALYSIS

None.

6. RESULTS

The Table of analytical determinations are in the Appendix as follows:

- □ Annex 1: Concentrations of Co and Ni in sample solution X02a-081-1 X02a-081-12 (in µg/L).

7. REFERENCES

a) WLN-CM.W.11.1 Determination of the metal content in water by means of an inductively coupled plasma and mass-spectrometry.

b) NEN-EN-ISO 17294-2:2016 : Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: Determination of selected elements including uranium isotopes.

APPENDIX

Annex 1: Concentrations of Co and Ni in sample solution X02a-081-1 X02a-081-12 (in µg/L).

Parameter	Description	Standard Procedure	Accreditation
Co	Cobalt	WLN-CM.W.11.1	RD
Ni	Nickel	WLN-CM.W.11.1	RD

Sample Number	Description (ECTX)	Sampling Date	Date of Analysis	Co (µg/L)	Ni (µg/L)
1654121	X02a-081-1	11-11-2016	22-11-2016	0.021	0.230
1654122	X02a-081-2	11-11-2016	22-11-2016	0.020	0.209
1654123	X02a-081-3	11-11-2016	22-11-2016	0.021	0.207
1654124	X02a-081-4	11-11-2016	22-11-2016	0.144	3.107
1654125	X02a-081-5	11-11-2016	22-11-2016	0.145	3.136
1654126	X02a-081-6	11-11-2016	22-11-2016	0.152	3.233
1654127	X02a-081-7	11-11-2016	22-11-2016	0.140	3.355
1654128	X02a-081-8	11-11-2016	22-11-2016	0.146	3.369
1654129	X02a-081-9	11-11-2016	22-11-2016	0.141	3.231
1654130	X02a-081-10	11-11-2016	22-11-2016	0.154	3.474
1654131	X02a-081-11	11-11-2016	22-11-2016	0.155	3.460
1654132	X02a-081-12	11-11-2016	22-11-2016	0.153	3.542

RD: raw data, not rounded as mentioned in our standard operating procedures.

Annex 6:

Certificate of Analysis of **Stainless Steel 316L Massive** (Elemental Analysis Inc., 2 pages).

PIXE Test Report
Elemental Analysis, Inc.
2101 Capstone Dr. Ste. 110
Lexington, KY 40511
Tel: 1-800-563-PIXE(7493)

Date:	9/1/2016
Run:	187
Job:	3238-16
Name:	Particle Technology Labs
Project:	Arnette Schultz
Target:	N152.1-PTL/PTL ID#225748-10

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Na	1.041	828.500 ppm		
Mg	1.254	307.800 ppm		
Aluminium	1.487	282.500 ppm	0.12%	0.02%
Silicon	1.740	163.100 ppm	0.65%	0.02%
Phosphorus	2.010	150.200 ppm	0.024%	0.011%
S	2.308	0.101%		
Cl	2.623	67.490 ppm		
K	3.314	40.840 ppm		
Ca	3.692	72.990 ppm		
Sc	4.091	35.160 ppm		
Titanium	4.511	74.870 ppm	0.049%	0.008%
Vanadium	4.952	94.560 ppm	0.039%	0.008%
Chromium	5.415	175.900 ppm	16.21%	0.16%
Manganese	5.899	0.117%	1.10%	0.06%
Iron	6.405	351.500 ppm	68.05%	0.68%
Co	6.930	0.180%		
Nickel	7.478	191.600 ppm	11.19%	0.11%
Copper	8.048	206.200 ppm	0.25%	0.03%
Zn	8.639	30.550 ppm		
Ga	9.250	33.310 ppm		
Ge	9.887	27.870 ppm		
As	10.544	41.190 ppm		
Se	11.222	32.390 ppm		
Br	11.924	56.350 ppm		
Rb	13.395	80.230 ppm		
Sr	14.165	56.240 ppm		
Y	14.959	63.620 ppm		
Zr	15.775	48.100 ppm		
Nb	16.615	68.830 ppm		
Molybdenum	2.293	302.000 ppm	2.27%	0.03%
Tc	18.367	118.100 ppm		
Ru	19.279	187.000 ppm		
Rh	2.697	112.200 ppm		
Pd	2.839	120.900 ppm		
Ag	2.984	95.740 ppm		
Cd	3.133	81.730 ppm		
In	3.286	110.000 ppm		
Sn	3.444	102.200 ppm		
Sb	3.604	162.200 ppm		
Te	3.768	132.700 ppm		
I	3.937	93.180 ppm		
Cs	4.288	113.400 ppm		
Ba	4.466	177.700 ppm		
La	4.648	380.400 ppm		
Ce	4.841	186.200 ppm		
Pr	5.034	211.600 ppm		
Nd	5.230	550.000 ppm		
Pm	5.431	0.287%		
Sm	5.632	302.700 ppm		
Eu	5.841	920.800 ppm		
Gd	6.050	836.800 ppm		
Tb	6.271	0.242%		
Dy	6.492	0.336%		

Page 1 of 2

PIXE Test Report
Elemental Analysis, Inc.
2101 Capstone Dr. Ste. 110
Lexington, KY 40511
Tel: 1-800-563-PIXE(7493)

Date:	9/1/2016
Run:	187
Job:	3238-16
Name:	Particle Technology Labs
Project:	Arnette Schultz
Target:	N152.1-PTL/PTL ID#225748-10

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Ho	6.725	270.000 ppm		
Er	6.945	0.130%		
Tm	7.182	0.187%		
Yb	7.416	0.227%		
Lu	7.655	614.900 ppm		
Hf	7.899	226.400 ppm		
Ta	8.149	692.700 ppm		
W	8.398	627.200 ppm		
Re	8.652	84.230 ppm		
Os	8.911	181.700 ppm		
Ir	9.174	85.440 ppm		
Pt	9.443	69.500 ppm		
Au	9.712	84.860 ppm		
Hg	9.989	79.150 ppm		
Tl	10.267	74.770 ppm		
Pb	10.551	195.200 ppm		
Bi	10.838	64.430 ppm		
Th	2.992	186.700 ppm		
U	3.166	159.000 ppm		