FINAL REPORT

Study no.

X02a-145

4 hours Bio-elution Kinetics Study on

Stainless Steel 316L Powder

at a *2 g/L loading* in a simulated gastric fluid

April 21, 2018

Statement

Title: 4 hours Bio-elution Kinetics Study on **Stainless Steel 316L Powder** at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-145**

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 WLN Business B.V..

The sponsor remains responsible for the information on the test item that was provided by him/her.

Noted and approved:



21/04/2018

Tony Brouwers

Study Director

Study completion date:

Quality Assurance Statement

Title: 4 hours Bio-elution Kinetics Study on **Stainless Steel 316L Powder** at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-145**

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	22&23-01-2018	23-01-2018
Performance of the study: - weighing test item - experiment, gastric at pH 1.5	 24-01-2018 24-01-2018	17-04-2018
Raw data	17-04-2018	17-04-2018
Draft report	17-04-2018	17-04-2018
Final report	21-04-2018	21-04-2018

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:



21/4/2018.

Marleen Schreurs

Date:

Lead Quality Assurance Manager

Statement of Authentication

Title: 4 hours Bio-elution Kinetics Study on **Stainless Steel 316L Powder** at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-145**

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 WLN Business B.V.. 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:



21/04/2018

Tony Brouwers

Date:

Test Facility Manager

Contents

STATEMENT **2**

QUALITY ASSURANCE STATEMENT **3**

STATEMENT OF AUTHENTICATION **4**

CONTENTS **5**

1 SUMMARY **6**

2 IDENTIFICATION OF THE STUDY, TEST ITEM AND REFERENCE ITEM **8**

2.1 STUDY TITLE AND NUMBER 8

2.2 TEST ITEM IDENTIFICATION AND CHARACTERISATION 8

2.3 REFERENCE ITEM IDENTIFICATION AND CHARACTERISATION 10

3 INFORMATION CONCERNING SPONSOR, TEST FACILITY AND TEST SITE **11**

3.1 SPONSOR IDENTIFICATION 11

3.2 TEST FACILITY IDENTIFICATION 11

3.3 TEST SITE IDENTIFICATION 11

4 DATES **12**

5 TEST METHODS **13**

5.1 TEST GUIDANCE 13

5.2 STANDARD OPERATING PROCEDURES (SOPS) 13

6 TEST SYSTEM **14**

6.1 SIMULATED GASTRIC FLUID 14

7 EXPERIMENTAL DESIGN **15**

7.1 MATERIALS 15

7.2 PREPARATION OF THE TEST ITEM 15

7.3 TEST CONDITIONS 15

7.4 PERFORMANCE 15

7.5 DATA ANALYSIS 19

8 RESULTS **20**

9 CONCLUSION **22**

10 DEVIATIONS FROM THE ORIGINAL STUDY PLAN **23**

11 ARCHIVES **24**

12 REFERENCES **25**

ANNEXES **26**

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 simulated 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".

This study was carried out on request of the Sponsor to answer questions raised during the meeting with ECVAM. The release/dissolution of nickel and iron ions from a **Stainless Steel 316L Powder** further noted as **"SS316L Powder"** in simulated gastric fluid were measured. 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 **SS316L Powder** with a particle size <100 µm was tested in a simulated gastric fluid (pH 1.5; 37 °C) at a loading of 2 g/L up to 4 hours (1 hour at an agitation speed of 100 revolutions per minute - rpm - and 3 hours without shaking). The bioelution endpoint was based on the dissolved nickel and iron concentrations obtained after 1, 2 and 4 hours of extraction.

Simultaneously, reference items, nickel powder and iron powder as also a soluble form of both elements were tested to make that the limit of dissolution was not reached due to the test method (and saturation).

The following deviation from the SOP was made:

- The samples for metal analyses were preserved by adding 0.12 mL concentrated HNO_3 per 12 mL instead of no preservation in the SOP.

The study was performed at ECTX. Analysis of the concentrations of dissolved element concentrations has been performed at WLN Business B.V. (The Netherlands), an ISO 17025 accredited laboratory, as delegated by ECTX.

The temperature of the sampled blank control, reference item and test item vessels was between 36.8 °C and 37.0 °C and corresponded to the required test conditions of 37 °C ± 1 °C.

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. At the 1, 2 and 4 hours sampling points of the test, the pH in the blank control vessels, reference item vessels and test vessels was between 1.47 and 1.51.

The blank control vessels showed no significant concentrations above the accredited reporting limit of 0.5 µg/L Ni for nickel and 10 µg/L Fe for iron.

The following observations could be made in the reference item vessels:

- The reference vessel containing a nominal loading of 2 g/L nickel powder showed an average dissolved nickel concentration of 80.4 mg/L Ni (i.e. 4.0 % of the total nickel content) at the 1-hour sampling point. This amount increased to 351 mg/L Ni (i.e. 18 % of the total nickel content) at the 4-hour endpoint.
- The reference vessel containing a 2 g/L loading of nickel(II)chloride hexahydrate (i.e. 24.69 % Ni) showed an average dissolved nickel concentration of about 453 mg/L Ni throughout the test or 92 % of the total nickel content.
- The reference vessel containing a nominal loading of 2 g/L iron powder showed an average dissolved iron concentration of 320 mg/L Fe (i.e. 16 % of the total iron content) at the 1-hour sampling point. This amount increased to 744 mg/L Fe (i.e. 37 % of the total iron content) at the 4-hour endpoint.

- The reference vessel containing a 2 g/L loading of iron(II) sulfate heptahydrate (i.e. 20.09 % Fe) showed an average dissolved iron concentration of about 369 mg/L Fe throughout the test or 92 % of the total iron content.

In the test item vessels with a nominal loading of **2 g/L SS316L Powder:**

- An average dissolved nickel concentration of 90.1 ± 0.8 µg/L Ni ($CV_{between\text{-}vessel}$ = 1 %; N = 2) was measured after 1 hours of exposure to the simulated gastric fluid (pH 1.5). The average amount of dissolved nickel increased over time to 155 ± 1 µg/L Ni ($CV_{between\text{-}vessel}$ = 0 %; N = 2) at the 4-hours study endpoint.
  Based on the nickel content (10.7 % Ni) in the test item **SS316L Powder** and the average dissolved nickel concentrations in the test solutions, a release of nickel of 0.042 % and 0.073 % of content, could be calculated at the 1-hour sampling point and the 4-hours endpoint, respectively.

- An average dissolved iron concentration of 2202 ± 6 µg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured after 1 hours of exposure to the simulated gastric fluid (pH 1.5). The average amount of dissolved iron increased over time to 4095 ± 35 µg/L Fe ($CV_{between\text{-}vessel}$ = 1 %; N = 2) at the 4-hours study endpoint.
  Based on the iron content (68.61 % Fe) in the test item **SS316L Powder** and the average dissolved iron concentrations in the test solutions, a release of iron of 0.16 % and 0.30 % of content, could be calculated at the 1-hour sampling point and the 4-hours endpoint, respectively.

2 Identification of the Study, Test Item and Reference Item

2.1 Study title and number

Title: 4 hours Bio-elution Kinetics Study on **Stainless Steel 316L Powder** at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-145**

2.2 Test Item identification and characterisation

The test item, an **SS316L Powder**, as received, is representative of the material as it is, or will be, on the market for use. No further preparation of the test item is required to conduct this study. See also Section *7.2 Preparation of the test item*.

The information about the identification and characterisation of the test item has been delivered by the Sponsor at the start of the study.

Test Item identification

Test item name:	**Stainless Steel 316L Powder**
Abbreviation used in the text	**SS316L Powder**
Manufacturer:	Sandvik Osprey Ltd Red Jacket Works Milland Road Neath SA11 1NJ United Kingdom
Manufacturer contact:	+44 (163) 963 41 21
Sample delivered by:	Particle Technology Labs 555 Rogers Street, Downers Grove IL 60515 USA Arnette Schultz [+1-(630)-969-2703 x118]
Sample ID:	N155A.2-PTL
Batch:	14D0439
ECTX no.:	**T0222/01**

Test Item characterisation

Physicochemical method of production:	n.a.[1]
Element(s) of interest:	Iron, nickel
Composition - PIXE (typical range):	16.41 % chromium (16.0 % – 18.0 %) 10.65 % nickel (10.0 % – 14.0 %)

[1] n.a. not available/applicable

	2.10 % molybdenum (2.0 % – 3.0 %) 1.28 % manganese (0.00 % – 2.00 %) 0.46 % silicon (0.00 % – 1.00 %) 0.32 % copper 0.022 % titanium 0.032 % vanadium 0.069 % niobium 68.61 % iron (balance) A certificate of analysis of the tested batch is added to this study report (Annex 14).
Physical form:	Solid (in the powder form)
Colour:	Grey
Particle size distribution (Volume based):	d_{10}: 2.2 µm d_{50}: 3.7 µm d_{90}: 6.2 µm A report of the particle size distribution analysis of this batch is added to this study report. (Annex 15)
Specific surface area (Kr-BET):	0.35 m²/g A report of the Kr-BET analysis of this batch is added to this study report. (Annex 16)
Homogeneous:	Yes
Relative Density:	7.87

Storage conditions

Room temperature, in the dark.

Expiry date

Stable over time.

Safe Handling Precautions

A safety data sheet on **316L** is available.

2.3 Reference Item identification and characterisation

The individual powders (i.e. iron powder and nickel powder) as also a soluble compound of each element of interest were used as reference items:

Reference Item identification (Nickel powder)

Supplier:	Particle Technology Labs 555 Rogers Street, Downers Grove, IL 60515, USA
Supplier contact:	+1-(630)-969-2703 x118

ECTX no.:	Description	PTL no.	Lot no.
T0220/01	Nickel metal powder >99 % ; d_{90}: 1.84 µm	N36F.1.4-PTL	-

Nickel metal powder (ECTX no.: T0220/01) was subject in ECTX study X02a-085.
A certificate of chemical analysis of the tested batch is added to this study report (Annex 7).
Particle Size Distribution (PSD):
D(v, 0.1) = 0.77 µm
D(v, 0.5) = 1.20 µm
D(v, 0.9) = 1.84 µm
A Laboratory report of the PSD is added under Annex 8
Specific Surface Area (N-BET)
2.58 m^2/g
A Laboratory report of the Specific Surface Area (N-BET) is added under Annex 9

Reference Item identification (Iron powder)

Supplier:	Sigma Aldrich BVBA Brusselsesteenweg 288, B-3090 Overijse, Belgium
Supplier contact:	+32 (0)3/899.13.01

ECTX no.:	Description	Acros Art. no.	Lot no.
R0039/01	Iron powder >97 % ; -325 mesh (<44 µm)	209309	STBG7186

CAS no.: 7439-89-6
Molecular weight: 55.85 g/mol
Stored under argon. Moisture sensitive. Safety data sheet is available.
A certificate of analysis of the tested batch is available and will be added to the final study report.

Reference Item identification (soluble compounds)

Supplier:	Acros Organics Janssen Pharmaceuticalaan 3a, B-2440 Geel, Belgium
Supplier contact:	+32 (0)14/59.34.34

ECTX no.:	Description	Acros Art. no.	Lot no.
R0025/01	Nickel(II)chloride hexahydrate, 99.999%	193570050	A0336441
S0014/01	Iron(II) sulfate heptahydrate, 99.5%	201392500	A0344065

3 Information concerning Sponsor, Test Facility and Test Site

3.1 Sponsor identification

Sponsor:	**Eurometaux** Avenue de Broqueville, 12 B-1150 Brussels Belgium
Study Monitor on behalf of the Sponsor:	**Violaine Verougstraete**

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

WLN Business B.V., 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 01-12-2021.	
Contributing Scientist:	Jan Bonnema
Quality Control Manager:	Jeroen Boekenoogen
Test Site Management:	Jeroen van Nuil / Harm de Kleine

4 Dates

Study Initiation Date:	**23-01-2018**
Experimental Starting Date:	**24-01-2018**
Experimental Completion Date:	**02-02-2018** (with specific analyses)
Study Completion Date:	Date of approval of this Final Report by the Study Director
Remarks:	The test was carried out on **24-01-2018**
	The samples were delivered to the analytical lab (WLN) on **27-01-2018**
	Specific analyses were carried out on **01-02-2018**

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 WLN Business B.V.

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,
  - Microbalance Mettler Toledo XP6 (SNR24043156),
  - Analytical balance Mettler Toledo XP204S (SNR1129023848),
  - Balance Mettler Toledo XP8002S (SNR1129020806),
  - Thermometer Testo 875-1i (SNR2738028), pH-meter (HACH HQ40d multimeter SNR080100016916),
  - Thermostatic 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; Supor [polyethersulfone (PES)] membrane (PN 4506),
  - 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 (WLN Business B.V.).

7.2 Preparation of the Test Item

The test item, **SS316L Powder** as received fulfilled the <100 µm size requirement. Therefore, no further preparation of the test item at ECTX was needed. The Sponsor also provided supporting documents which characterise the test item (Certificate of Analysis, analysis reports of the particle size distribution and specific surface area measurements: Annex 14, 15 and 16).

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

250 mL borosilicate glass Erlenmeyer's were used for the blank control vessel, the reference item vessels and the test item vessels at a loading 2 g/L test item.

- In the blank control vessel (3; i.e. one for each sampling time), 50 mL gastric test medium (37 °C ± 1 °C) was added, only.
- In each of three reference item vessel ref A, 50 mL gastric test medium (37 °C ± 1 °C) was added to obtain a loading of 2 g/L nickel.
- In each of three reference item vessel ref B vessel, 50 mL gastric test medium (37 °C ± 1 °C) was added to obtain a loading of 2 g/L nickel(II)chloride hexahydrate.
- In each of three reference item vessel ref C vessel, 50 mL gastric test medium (37 °C ± 1 °C) was added to obtain a loading of 2 g/L iron.
- In each of three reference item vessel ref D vessel, 50 mL gastric test medium (37 °C ± 1 °C) was added to obtain a loading of 2 g/L iron(II) sulfate heptahydrate.
- In each of six test item vessels of 2 g/L loading was prepared by adding 50 mL gastric test medium (37 °C ± 1 °C) to the weighed test item.

After covering the vessels with a screwcap and swirling the flasks to mix the test item and the medium, the flasks were placed in the dark into a thermostatic orbital shaker (37 °C ± 1 °C) at an agitation rate of 100 rpm for one hour. After that, a set of one blank control vessel, one of each kind of reference vessel and two test item vessels were removed for sampling. The flasks were settled at 37 °C ± 1 °C for another hour. The second set of one blank control vessel, one of each kind of reference vessel and two test item vessels were removed for sampling after the first hour of settling (time: 2 hours of extraction). The remaining flasks (still one blank control vessel, one of each kind of reference vessel and two test item vessels) continued settling for two more hours and then sampled.

An overview of the test set-up is shown in the following table:

Time	Target loading (g/L)	Replica number	Amounts weighed	Test medium added
1 hour	0	blank	-	50 mL
	2	ref A repl1	100.006 mg Ni	50 mL
	2	ref B repl1	100.064 mg $NiCl_2 \cdot 6H_2O$	50 mL
	2	ref C repl1	100.013 mg Fe	50 mL
	2	ref D repl1	100.042 mg $FeSO_4 \cdot 7H_2O$	50 mL
	2	replica1	100.009 mg test item SS316L	50 mL
	2	replica2	100.006 mg test item SS316L	50 mL
2 hours	0	blank	-	50 mL
	2	ref A repl2	100.002 mg Ni	50 mL
	2	ref B repl2	100.019 mg $NiCl_2 \cdot 6H_2O$	50 mL
	2	ref C repl2	100.008 mg Fe	50 mL
	2	ref D repl2	100.051 mg $FeSO_4 \cdot 7H_2O$	50 mL
	2	replica3	100.004 mg test item SS316L	50 mL
	2	replica4	100.011 mg test item SS316L	50 mL
4 hours	0	blank	-	50 mL
	2	ref A repl3	100.000 mg Ni	50 mL
	2	ref B repl3	100.070 mg $NiCl_2 \cdot 6H_2O$	50 mL
	2	ref C repl3	100.008 mg Fe	50 mL
	2	ref D repl3	100.055 mg $FeSO_4 \cdot 7H_2O$	50 mL
	2	replica5	100.005 mg test item SS316L	50 mL
	2	replica6	100.016 mg test item SS316L	50 mL

The test will be performed in a thermostatic shaker in the dark.

7.4.2 Sampling during the test

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

At each sampling time (1, 2 and 4 hours), a set of one blank and one of each reference item and two replica test item vessels were removed from the incubator for sampling. Each vessel was swirled to homogenise the solution before sampling, to avoid a concentration gradient after settling (especially applicable for 2 and 4 hours sampling points).

Sampling was performed for each test vessel as indicated in the sampling scheme below. In each test vessel, a 12-mL sample was taken twice with a 12 mL syringe 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-145-**1** where:

X02a-145 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 was applied:

Sampling time (h)	**Number of samples**						**Total number of samples**
	Medium	RefA	RefB	RefC	RefD	Test item	
	(blank) 0 g/L	Ni Loading 2 g/L	$NiCl_2.6H_2O$ Loading 2 g/L	Fe Loading 2 g/L	$FeSO_4.7H_2O$ Loading 2 g/L	SS316L Loading 2 g/L	
1	2*1	2*1	2*1	2*1	2*1	2*2	14
2	2*1	2*1	2*1	2*1	2*1	2*2	14
4	2*1	2*1	2*1	2*1	2*1	2*2	14
Total number of samples:							42

A detailed list is given in Annex 5.

7.4.4 Observations and measurement of temperature and pH

During the test (at 1h, 2h and 4h), temperature and pH were measured in the 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 contained 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 iron and nickel concentrations in the blank control, reference item 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). Rounded values were used to at least one more decimal place than the corresponding accredited reporting limit of WLN, if relevant. The accredited reporting limit with an accuracy of 90 % and 110 % is 0.5 µg/L for nickel and 10 µg/L for iron.

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 02-02-2018 and is added as an annex to this final report (Annex 6).

7.5 Data analysis

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

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

The dataset of *n* vessels was analysed. The between-vessel mean, standard deviation and coefficient of variation of the measured dissolved element concentrations were calculated using the equations in the table below:

	Mean	Standard deviation	Coefficient of variation
Between	$x_{avg} = \frac{1}{n}\sum_{i=1}^{n} x_i$	$s_{between} = \left(\frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - x_{avg}\right)^2\right)^{1/2}$	$CV_{between} = \frac{s_{between}}{x_{avg}} \cdot 100\%$

A between-vessel coefficient of variation of <20% is a quality criterion for the 2 hours sample point.

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 points was between 36.8 °C and 37.0 °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. At the sampling points of the test, the pH in the blank control vessels, reference item vessels and test vessels was between 1.47 and 1.51. (Annex 1)

The raw data of the dissolved element concentrations in the test samples, measured by the Test Site WLN Business B.V., are presented in Annex 6.

The blank measurements of iron and nickel were below or close to the corresponding accredited reporting limits of 10 µg/L Fe and 0.5 µg/L Ni.

The following observations could be made in the reference item vessels:

- **Reference A (2 g/L nickel powder)**: Remaining, undissolved, metal powder could be observed in the Erlenmeyer flasks at the end of the experiment indicating that the dissolution was not complete.

  At the 1-hour sampling point: An average concentration of dissolved nickel of 80.4 ± 0.1 mg/L Ni ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. A nickel release of 4.0 % related to the total nickel content could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved nickel of 139 ± 1 mg/L Ni ($CV_{between\text{-}vessel}$ = 1 %; N = 2) was measured. A nickel release of 7.0 % related to the total nickel content could be calculated.

  At the 4-hours sampling point: An average concentration of dissolved nickel of 351 ± 17 mg/L Ni ($CV_{between\text{-}vessel}$ = 5 %; N = 2) was measured. A nickel release of 18 % related to the total nickel content could be calculated.

- **Reference B (2 g/L nickel(II)chloride hexahydrate)**: No undissolved material could be observed in the Erlenmeyer flasks at the sampling points indicating that the reference material was completely dissolved.

  At the 1-hour sampling point: An average concentration of dissolved nickel of 460 ± 9 mg/L Ni ($CV_{between\text{-}vessel}$ = 2 %; N = 2) was measured. A nickel release of 93 % related to the total nickel content (i.e. 24.69 % Ni) could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved nickel of 443 ± 1 mg/L Ni ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. A nickel release of 90 % related to the total nickel content could be calculated.

  At the 4-hours sampling point: An average concentration of dissolved nickel of 457 ± 9 mg/L Ni ($CV_{between\text{-}vessel}$ = 2 %; N = 2) was measured. A nickel release of 92 % related to the total nickel content could be calculated.

- **Reference C (2 g/L iron powder)**: Remaining, undissolved, metal powder could be observed in the Erlenmeyer flasks at the end of the experiment indicating that the dissolution was not complete.

  At the 1-hour sampling point: An average concentration of dissolved iron of 320 ± 9 mg/L Fe ($CV_{between\text{-}vessel}$ = 3 %; N = 2) was measured. An iron release of 16 % related to the total iron content could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved iron of 417 ± 0 mg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. An iron release of 21 % related to the total iron content could be calculated.

At the 4-hours sampling point: An average concentration of dissolved iron of 744 ± 11 mg/L Fe ($CV_{between\text{-}vessel}$ = 2 %; N = 2) was measured. An iron release of 37 % related to the total iron content could be calculated.

- **Reference D (2 g/L iron(II) sulfate heptahydrate)**: No undissolved material could be observed in the Erlenmeyer flasks at the sampling points indicating that the reference material was completely dissolved.

  At the 1-hour sampling point: An average concentration of dissolved iron of 364 ± 1 mg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. An iron release of 91 % related to the total iron content (i.e. 20.09 % Fe) could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved iron of 366 ± 11 mg/L Fe ($CV_{between\text{-}vessel}$ = 3 %; N = 2) was measured. An iron release of 91 % related to the total iron content (i.e. 20.09 % Fe) could be calculated.

  At the 4-hours sampling point: An average concentration of dissolved iron of 376 ± 10 mg/L Fe ($CV_{between\text{-}vessel}$ = 3 %; N = 2) was measured. An iron release of 93 % related to the total iron content (i.e. 20.09 % Fe) could be calculated.

The following observations could be made in the test vessels with a nominal loading of **2 g/L SS316L Powder**:

- Remaining test item (grey particles) could be observed on the filter and in the Erlenmeyer flasks at the end of the experiment.
- For nickel:

  At the 1-hour sampling point: An average concentration of dissolved nickel of 90.1 ± 0.8 µg/L Ni ($CV_{between\text{-}vessel}$ = 1 %; N = 2) was measured. A nickel release of 0.042 % related to the total nickel content (i.e. 10.7 % Ni) could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved nickel of 127 ± 3 µg/L Ni ($CV_{between\text{-}vessel}$ = 2 %; N = 2) was measured. The between-vessel coefficient of variation met the <20 % quality criterion at the 2 hours sampling point. A nickel release of 0.059 % related to the total nickel content (i.e. 10.7 % Ni) could be calculated.

  At the 4-hours sampling point: An average concentration of dissolved nickel of 155 ± 1 µg/L Ni ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. A nickel release of 0.073 % related to the total nickel content (i.e. 10.7 % Ni) could be calculated.

- For iron:

  At the 1-hour sampling point: An average concentration of dissolved iron of 2202 ± 6 µg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. An iron release of 0.16 % related to the total iron content (i.e. 68.61 % Fe) could be calculated.

  At the 2-hours sampling point: An average concentration of dissolved iron of 3172 ± 11 µg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was measured. The between-vessel coefficient of variation met the <20 % quality criterion at the 2 hours sampling point. An iron release of 0.23 % related to the total iron content (i.e. 68.61 % Fe) could be calculated.

  At the 4-hours sampling point: An average concentration of dissolved iron of 4095 ± 35 µg/L Fe ($CV_{between\text{-}vessel}$ = 1 %; N = 2) was measured. An iron release of 0.30 % related to the total iron content (i.e. 68.61 % Fe) could be calculated.

All raw data can be found in Annex 1-16 of this study report.

9 Conclusion

During this study on the bio-elution of **SS316L Powder** at a loading of 2 g/L, the following could be observed during 4-hours of exposure to simulated gastric fluid (pH 1.5):

- For nickel, an average value of 90.1 µg/L Ni ($CV_{between\text{-}vessel}$ = 1 %; N = 2) was found after 1 hour of extraction increasing to 155 µg/L Ni ($CV_{between\text{-}vessel}$ = 0 %; N = 2) after 4 hours. The measured concentrations corresponded with a nickel release related to the total nickel content of 0.042 % and 0.073 % at the 1- and 4-hours sampling point, respectively.
- For iron, an average value of 2202 µg/L Fe ($CV_{between\text{-}vessel}$ = 0 %; N = 2) was found after 1 hour of extraction increasing to 4095 µg/L Fe ($CV_{between\text{-}vessel}$ = 1 %; N = 2) after 4 hours. The measured concentrations corresponded with an iron release related to the total iron content of 0.16 % and 0.30 % at the 1- and 4-hours sampling point, respectively.

The reference vessel containing 2 g/L nickel powder showed an average dissolved nickel concentration of 80.4 mg/L Ni (i.e. 4.0 % of the total nickel content) at the 1-hour sampling point increasing to 351 mg/L Ni (i.e. 18 % of the total nickel content) at the 4-hour endpoint.

The reference vessel containing 2 g/L iron powder showed an average dissolved iron concentration of 320 mg/L Fe (i.e. 16 % of the total iron content) at the 1-hour sampling point increasing to 744 mg/L Fe (i.e. 37 % of the total iron content) at the 4-hour endpoint.

Reference item vessels containing a soluble form of nickel ($NiCl_2 \cdot 6H_2O$) and iron ($FeSO_4 \cdot 7H_2O$) showed a constant recovery over time between 90 % and 93 % indicating that no saturation occurred at the tested loadings.

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-145**. The Study Plan, the lab notebook, the raw data, the correspondence and final report has been stored in a locked filing cabinet. A representative sample (10.0353 g) of the reference item, nickel metal powder and of the of the iron powder (R0039/01: 10.1695 g) was already archived earlier in a suitably sealed vial and stored under the storage conditions mentioned (study X02a-085; 11-11-2016 for nickel and study X02a-130; 19-09-2017 for iron). The same was true for the test item where 9.5088 g was archived (study X02a-084; 11-11-2016) 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 Business B.V. 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 D5517-14 (2014): **Standard Test Method for Determining Extractability of Metals from Art Materials**, Philadelphia, PA: American Society for Testing and Materials, 2014.

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 during the 4 hours exposure period in the blank control, reference items and test item exposed to the simulated gastric fluid at pH 1.5.

Time (h)		Blank		Ni powder		$NiCl_2.6H_2O$		Fe powder		$FeSO_4.7H_2O$		SS316L	
		pH	T (°C)	pH	T (°C)	pH	T (°C)	pH	T (°C)	pH	T (°C)	pH	T (°C)
0 hour	Medium	1.50	37.1										
1 hour	Repl1	1.50	37.0	1.50	37.0	1.49	36.9	1.50	37.0	1.47	37.0	1.50	37.0
	Repl2											1.50	37.0
2 hours	Repl1	1.50	36.9	1.51	37.0	1.49	37.0	1.51	36.9	1.48	37.0	1.51	36.9
	Repl2											1.50	37.0
4 hours	Repl1	1.50	37.0	1.50	37.0	1.50	37.0	1.49	37.0	1.47	36.8	1.50	36.9
	Repl2											1.51	36.9

Annex 2:

Data of measured dissolved element concentrations during the 4 hours exposure period of the blank control, reference items and test item exposed to the simulated gastric fluid at pH 1.5.

Time (h)			Blank		Loading 2g/L					
					Ni powder	$NiCl_2.6H_2O$	Fe powder	$FeSO_4.7H_2O$	SS316L	
			(µg/L Ni)	(µg/L Fe)	(mg/L Ni)	(mg/L Ni)	(mg/L Fe)	(mg/L Fe)	(µg/L Ni)	(µg/L Fe)
1 hour	Repl1	Spl1	<0.5	10	80.3	454	314	365	90.0	2182
		Spl2	<0.5	10	80.5	467	327	363	91.3	2213
		Avg	**<0.5**	**10**	**80.4**	**460**	**320**	**364**	**90.7**	**2198**
		SD	-	0	0.1	9	9	1	1.0	22
		CV (%)	-	3	0	2	3	0	1	1
	Repl2					93%		91%	90.9	2262
						(Recovery)		(Recovery)	88.2	2151
									89.5	**2207**
									1.9	78
									2	4
2 hours	Repl1	Spl1	<0.5	15	138	443	417	359	131	3173
		Spl2	<0.5	12	140	443	417	374	126	3154
		Avg	**<0.5**	**14**	**139**	**443**	**417**	**366**	**129**	**3164**
		SD	-	2	1	1	0	11	3	14
		CV (%)	-	18	1	0	0	3	2	0
	Repl2					90%		91%	124	3168
						(Recovery)		(Recovery)	124	3191
									124	**3180**
									0	16
									0	1
4 hours	Repl1	Spl1	<0.5	11	363	451	752	383	154	4105
		Spl2	<0.5	11	339	463	736	369	156	4135
		Avg	**<0.5**	**11**	**351**	**457**	**744**	**376**	**155**	**4120**
		SD	-	0	17	9	11	10	2	22
		CV (%)	-	3	5	2	2	3	1	1
	Repl2					92%		93%	153	4022
						(Recovery)		(Recovery)	156	4119
									154	**4071**
									3	68
									2	2

Avg = average of 2 test vessels (2 samples per test vessel) (N=4).
SD = within-vessel standard deviation (N=2).
CV = within-vessel Coefficient of Variation (%).
Remark: the measurement results for iron are blank corrected as the blank values exceeded the accredited reporting limit of 10 µg/L.

Figure 1:

Measured dissolved iron and nickel concentrations during the 4 hours test of the reference items (2 g/L) exposed to the simulated gastric fluid at pH 1.5.

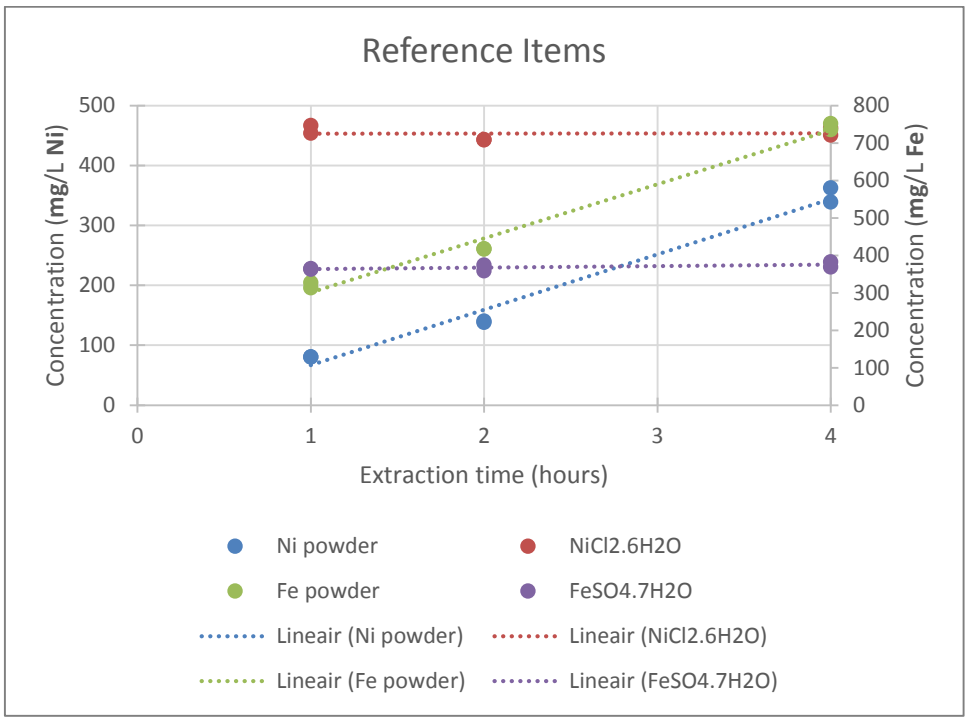
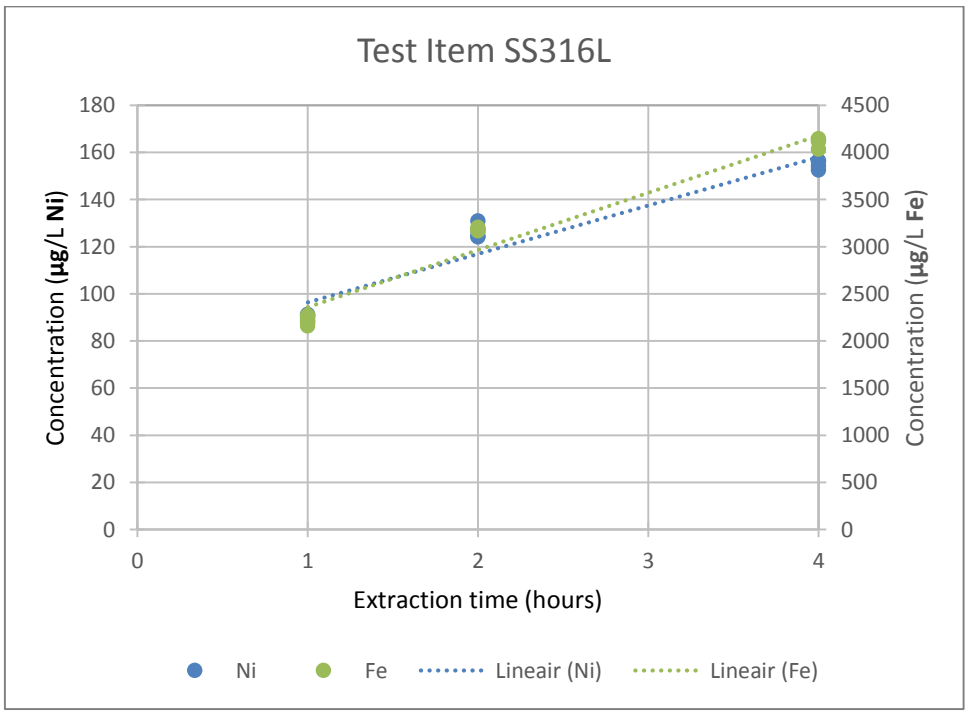


Figure 2:

Measured dissolved iron and nickel concentrations during the 4 hours test of the test item (**2 g/L SS316L Powder**) exposed to the simulated gastric fluid at pH 1.5.



Annex 3:

Bioaccessibility during the 4 hours kinetic study from **2 g/L SS316L Powder** in the simulated gastric fluid at pH 1.5.

		Fe release (µg/g Test Item)	Ni release (µg/g Test Item)
1 hour	replica1	1099	45.3
	replica2	1103	44.8
	x_{avg}	1101	45.1
	$s_{between}$	3	0.4
	$CV_{between}$ (%)	0	1
2 hours	replica1	1582	64.3
	replica2	1590	62.0
	x_{avg}	1586	63.1
	$s_{between}$	6	1.6
	$CV_{between}$ (%)	0	3
4 hours	replica1	2060	77.5
	replica2	2035	77.3
	x_{avg}	2048	77.4
	$s_{between}$	18	0.2
	$CV_{between}$ (%)	1	0

x_{avg} = average of 2 test vessels (N=2).
$s_{between}$ = between-vessel standard deviation (N=2).
$CV_{between}$ = between-vessel Coefficient of Variation (%).

Annex 4:

Summarised results of the average dissolved element concentrations, element release per g test item and the percentage of available element eluted, after 1, 2 and 4 hours of extraction of **2 g/L SS316L Powder** in the simulated gastric fluid at pH 1.5:

	SS316L Powder: 4-h gastric at pH 1.5 at **2 g/L** loading			
	Analyte		$x_{avg} \pm s_{between}$	$CV_{between}$
1 hour	**iron (Fe)**	dissolution	2202 ± 6 µg/L	0 %
		absolute Fe release	1101 ± 3 µg/g	0 %
		% of total available Fe eluted	0.16 %	
	nickel (Ni)	dissolution	90.1 ± 0.8 µg/L	1 %
		absolute Ni release	45.1 ± 0.4 µg/g	1 %
		% of total available Ni eluted	0.042 %	
2 hours	**iron (Fe)**	dissolution	3172 ± 11 µg/L	0 %
		absolute Fe release	1586 ± 6 µg/g	0 %
		% of total available Fe eluted	0.23 %	
	nickel (Ni)	dissolution	127 ± 3 µg/L	2 %
		absolute Ni release	63.1 ± 1.6 µg/g	2 %
		% of total available Ni eluted	0.059 %	
4 hours	**iron (Fe)**	dissolution	4095 ± 35 µg/L	1 %
		absolute Fe release	2048 ± 18 µg/g	1 %
		% of total available Fe eluted	0.30 %	
	nickel (Ni)	dissolution	155 ± 1 µg/L	0 %
		absolute Ni release	77.4 ± 0.2 µg/g	0 %
		% of total available Ni eluted	0.073 %	

x_{avg} = average of 2 test vessels (2 samples per test vessel) (N=4).
$s_{between}$ = between-vessel standard deviation (N=2).
$CV_{between}$ = between-vessel Coefficient of Variation (%).

Annex 5:

A detailed overview of the samples.

Sample no.	Sampling time (h)	pH	Loading (g/L)	Sample treatment	Comment
X02a-145-1	1	1.5	0	filtration (0.2 µm)	Blank replica1 spl1
X02a-145-2	1	1.5	0	filtration (0.2 µm)	Blank replica1 spl2
X02a-145-7	1	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica1 spl1
X02a-145-8	1	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica1 spl2
X02a-145-9	1	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica1 spl1
X02a-145-10	1	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica1 spl2
X02a-145-11	1	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica1 spl1
X02a-145-12	1	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica1 spl2
X02a-145-13	1	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica1 spl1
X02a-145-14	1	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica1 spl2
X02a-145-15	1	1.5	2	filtration (0.2 µm)	T0222/01 replica1 spl1
X02a-145-16	1	1.5	2	filtration (0.2 µm)	T0222/01 replica1 spl2
X02a-145-17	1	1.5	2	filtration (0.2 µm)	T0222/01 replica2 spl1
X02a-145-18	1	1.5	2	filtration (0.2 µm)	T0222/01 replica2 spl2
X02a-145-3	2	1.5	0	filtration (0.2 µm)	Blank replica2 spl1
X02a-145-4	2	1.5	0	filtration (0.2 µm)	Blank replica2 spl2
X02a-145-19	2	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica2 spl1
X02a-145-20	2	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica2 spl2
X02a-145-21	2	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica2 spl1
X02a-145-22	2	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica2 spl2
X02a-145-23	2	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica2 spl1
X02a-145-24	2	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica2 spl2
X02a-145-25	2	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica2 spl1
X02a-145-26	2	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica2 spl2
X02a-145-27	2	1.5	2	filtration (0.2 µm)	T0222/01 replica3 spl1
X02a-145-28	2	1.5	2	filtration (0.2 µm)	T0222/01 replica3 spl2
X02a-145-29	2	1.5	2	filtration (0.2 µm)	T0222/01 replica4 spl1
X02a-145-30	2	1.5	2	filtration (0.2 µm)	T0222/01 replica4 spl2
X02a-145-5	4	1.5	0	filtration (0.2 µm)	Blank replica3 spl1
X02a-145-6	4	1.5	0	filtration (0.2 µm)	Blank replica3 spl2
X02a-145-31	4	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica3 spl1
X02a-145-32	4	1.5	2	filtration (0.2 µm)	T0220/01 Ref A replica3 spl2
X02a-145-33	4	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica3 spl1
X02a-145-34	4	1.5	2	filtration (0.2 µm)	R0025/01 Ref B replica3 spl2
X02a-145-35	4	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica3 spl1
X02a-145-36	4	1.5	2	filtration (0.2 µm)	R0039/01 Ref C replica3 spl2
X02a-145-37	4	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica3 spl1
X02a-145-38	4	1.5	2	filtration (0.2 µm)	S0014/01 Ref D replica3 spl2
X02a-145-39	4	1.5	2	filtration (0.2 µm)	T0222/01 replica5 spl1
X02a-145-40	4	1.5	2	filtration (0.2 µm)	T0222/01 replica5 spl2
X02a-145-41	4	1.5	2	filtration (0.2 µm)	T0222/01 replica6 spl1
X02a-145-42	4	1.5	2	filtration (0.2 µm)	T0222/01 replica6 spl2

Annex 6:

Analysis of the dissolved iron and nickel concentrations at the *Test Site,* WLN

- Analytical Report 18R00782.X02a-145 (8 pages)

WLN Business B.V.
Rijksstraatweg 85
9756 AD Glimmen
telefoon (050) 402 21 21
telefax (050) 409 42 74

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

Analytical Report 18R00782.X02a-145

(02/02/2018)

Project: **17R0152/45**

Determination of iron and nickel in Simulated Gastric Fluid: ECTX Study X02a-145

Submitted to:

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

WLN Business B.V., Rijksstraatweg 85, NL-9851 AD Glimmen, The Netherlands
(ISO 17025 Accreditation certificate n° L075 from 12/11/1993, valid until 01/12/2021)

Scientist: **Jan Bonnema**

Management: **Jeroen van Nuil / Harm de Kleine**

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

Quality Control Manager: **Jeroen Boekenoogen**

CONTENTS

	CONFIDENTIALITY AND RETENTION PERIODS, DATA AND MATERIALS	3
	DISCLAIMER	3
1.	INTRODUCTION	4
2.	ANALYTICAL METHODOLOGY	4
3.	DEVIATIONS FROM THE SOP IN RELATION TO THE EXECUTED ANALYTICAL METHOD	5
4.	COMMENTS CONCERNING THE SAMPLES	5
5.	COMMENTS CONCERNING THE ANALYSIS	5
6.	RESULTS	6
7.	REFERENCES	7
	APPENDIX	8

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 iron (**Fe**) 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 **Fe** and **Ni** in ECTX samples of study X02a-145 as determined by the analytical laboratory of WLN. The sampling was performed by ECTX.

2. ANALYTICAL METHODOLOGY

The analyses of iron and nickel is 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.

WLN Business B.V. used a Thermo Fisher Scientific iCAP Q ICP-MS (inductively-coupled plasma-mass spectrometer) to determine the total dissolved concentrations for the elements iron (**Fe**) and nickel (**Ni**) in the as-received test and blank matrix solutions. To prepare blanks, calibration and control solutions/dilutions we always used de-ionized water (d.i. H_2O) at a purity level of 18 MΩ.

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: **^{56}Fe and ^{60}Ni**

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

- Reporting limit: **0.5 µg/L** for Ni; and **10 µg/L** for Fe (= according to our SOP)
- Repeatability: **≤5%** (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 Fe and Ni in sample solution X02a-145-1 to X02a-145-42 (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 Fe and Ni in sample solution X02a-145-1 to X02a-145-42 (in µg/L).

Parameter	Description	Standard	
Procedure	Accreditation		
Fe	Iron	WLN-CM.W.11.1	RD
Ni	Nickel	WLN-CM.W.11.1	RD

Sample Number	Description (ECTX)	Sampling Date	Date of Analysis	Fe (µg/L)	Ni (µg/L)
1804689	X02a-145-1	24-01-2018	01-02-2018	10	0.2
1804690	X02a-145-2	24-01-2018	01-02-2018	10	0.1
1804691	X02a-145-3	24-01-2018	01-02-2018	15	0.3
1804692	X02a-145-4	24-01-2018	01-02-2018	12	0.3
1804693	X02a-145-5	24-01-2018	01-02-2018	11	0.2
1804694	X02a-145-6	24-01-2018	01-02-2018	11	0.4
1804695	X02a-145-7	24-01-2018	01-02-2018	-	80255
1804696	X02a-145-8	24-01-2018	01-02-2018	-	80463
1804697	X02a-145-9	24-01-2018	01-02-2018	-	453722
1804698	X02a-145-10	24-01-2018	01-02-2018	-	466581
1804699	X02a-145-11	24-01-2018	01-02-2018	313674	-
1804700	X02a-145-12	24-01-2018	01-02-2018	327009	-
1804701	X02a-145-13	24-01-2018	01-02-2018	365038	-
1804702	X02a-145-14	24-01-2018	01-02-2018	363116	-
1804703	X02a-145-15	24-01-2018	01-02-2018	2193	90.0
1804704	X02a-145-16	24-01-2018	01-02-2018	2223	91.3
1804705	X02a-145-17	24-01-2018	01-02-2018	2272	90.9
1804706	X02a-145-18	24-01-2018	01-02-2018	2162	88.2
1804707	X02a-145-19	24-01-2018	01-02-2018	-	138091
1804708	X02a-145-20	24-01-2018	01-02-2018	-	140141
1804709	X02a-145-21	24-01-2018	01-02-2018	-	443274
1804710	X02a-145-22	24-01-2018	01-02-2018	-	442515
1804711	X02a-145-23	24-01-2018	01-02-2018	416924	-
1804712	X02a-145-24	24-01-2018	01-02-2018	417497	-
1804713	X02a-145-25	24-01-2018	01-02-2018	358577	-
1804714	X02a-145-26	24-01-2018	01-02-2018	374285	-
1804715	X02a-145-27	24-01-2018	01-02-2018	3187	130.9
1804716	X02a-145-28	24-01-2018	01-02-2018	3167	126.5
1804717	X02a-145-29	24-01-2018	01-02-2018	3182	124.2
1804718	X02a-145-30	24-01-2018	01-02-2018	3205	124.4
1804719	X02a-145-31	24-01-2018	01-02-2018	-	362643
1804720	X02a-145-32	24-01-2018	01-02-2018	-	339146
1804721	X02a-145-33	24-01-2018	01-02-2018	-	450782
1804722	X02a-145-34	24-01-2018	01-02-2018	-	463098
1804723	X02a-145-35	24-01-2018	01-02-2018	751689	-
1804724	X02a-145-36	24-01-2018	01-02-2018	735803	-
1804725	X02a-145-37	24-01-2018	01-02-2018	382737	-
1804726	X02a-145-38	24-01-2018	01-02-2018	368766	-
1804727	X02a-145-39	24-01-2018	01-02-2018	4116	154.1
1804728	X02a-145-40	24-01-2018	01-02-2018	4146	156.5
1804729	X02a-145-41	24-01-2018	01-02-2018	4033	152.5
1804730	X02a-145-42	24-01-2018	01-02-2018	4130	156.3

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

Annex 7:

Certificate of Analysis of the **Nickel powder** (PIXE Test Report - Elemental Analysis, Inc., 4 pages).

Element Analysis Corporation
101 Venture Ct. Ste. B-1
Lexington, KY 40510
Tel: 1-800-282-PIXE(7493)
Email Technical: PIXE@IGLOU.COM

Date: 12/19/01
Run: 267 267
Job: 8701-01
Name: Particle Technology Labs
Project: Kelli Hubert
Target: Nickel Powder

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
* O	----------		0.001 % ±	
Na	1.041	0.131 %		
Mg	1.254	422.300 ppm		
Al	1.485	204.000 ppm		
Si	1.740	137.000 ppm		
P	2.014	103.600 ppm		
S	2.308	91.780 ppm		
Cl	2.623	79.680 ppm		
K	3.314	44.690 ppm		
Ca	3.692	41.460 ppm		
Sc	4.091	45.400 ppm		
Ti	4.511	44.010 ppm		
V	4.952	41.840 ppm		
Cr	5.415	47.950 ppm		
Mn	5.899	75.600 ppm		
Fe	6.404	100.300 ppm		
Co	6.930	119.600 ppm		
Nickel	7.478	154.200 ppm	99.999 % ±	1.000 %
Cu	8.048	369.300 ppm		
Zn	8.639	118.700 ppm		
Ga	9.250	70.640 ppm		
Ge	9.887	88.120 ppm		
As	10.544	55.260 ppm		
Se	11.222	77.310 ppm		
Br	11.924	130.200 ppm		
Rb	13.395	133.700 ppm		
Sr	14.165	179.500 ppm		
Y	1.922	263.900 ppm		
Zr	2.043	234.300 ppm		
Nb	2.166	218.800 ppm		
Mo	2.294	207.000 ppm		
Tc	2.424	177.500 ppm		
Ru	2.559	179.000 ppm		
Rh	2.697	154.500 ppm		
Pd	2.839	133.800 ppm		
Ag	2.984	123.800 ppm		
Cd	3.133	122.700 ppm		
In	3.286	128.400 ppm		
Sn	3.444	121.900 ppm		
Sb	3.604	122.500 ppm		
Te	3.768	106.700 ppm		
I	3.937	114.200 ppm		
Cs	4.288	122.600 ppm		
Ba	4.466	134.300 ppm		
La	4.648	124.700 ppm		
Ce	4.841	120.300 ppm		
Pr	5.034	137.000 ppm		
Nd	5.230	144.800 ppm		
Pm	5.431	160.700 ppm		
Sm	5.632	319.200 ppm		
Eu	5.841	263.800 ppm		
Gd	6.050	191.900 ppm		
Tb	6.271	220.400 ppm		
Dy	6.492	270.900 ppm		
Ho	6.725	234.100 ppm		
Er	6.945	316.000 ppm		
Tm	7.182	610.100 ppm		
Yb	7.416	0.776 %		
Lu	7.655	0.262 %		
Hf	7.899	486.000 ppm		
Ta	8.149	0.269 %		
W	8.398	0.530 %		
Re	8.652	312.000 ppm		

* It should be noted that the low atomic number elements (C,O,H,N,etc) represent an unanalyzed component to the sample. Please see interpretation sheet for more information.

Element Analysis Corporation
101 Venture Ct. Ste. B-1
Lexington, KY 40510
Tel: 1-800-282-PIXE(7493)
Email Technical: PIXE@IGLOU.COM

Date: 12/19/01
Run: 267 267
Job: 8701-01
Name: Particle Technology Labs
Project: Kelli Hubert
Target: Nickel Powder

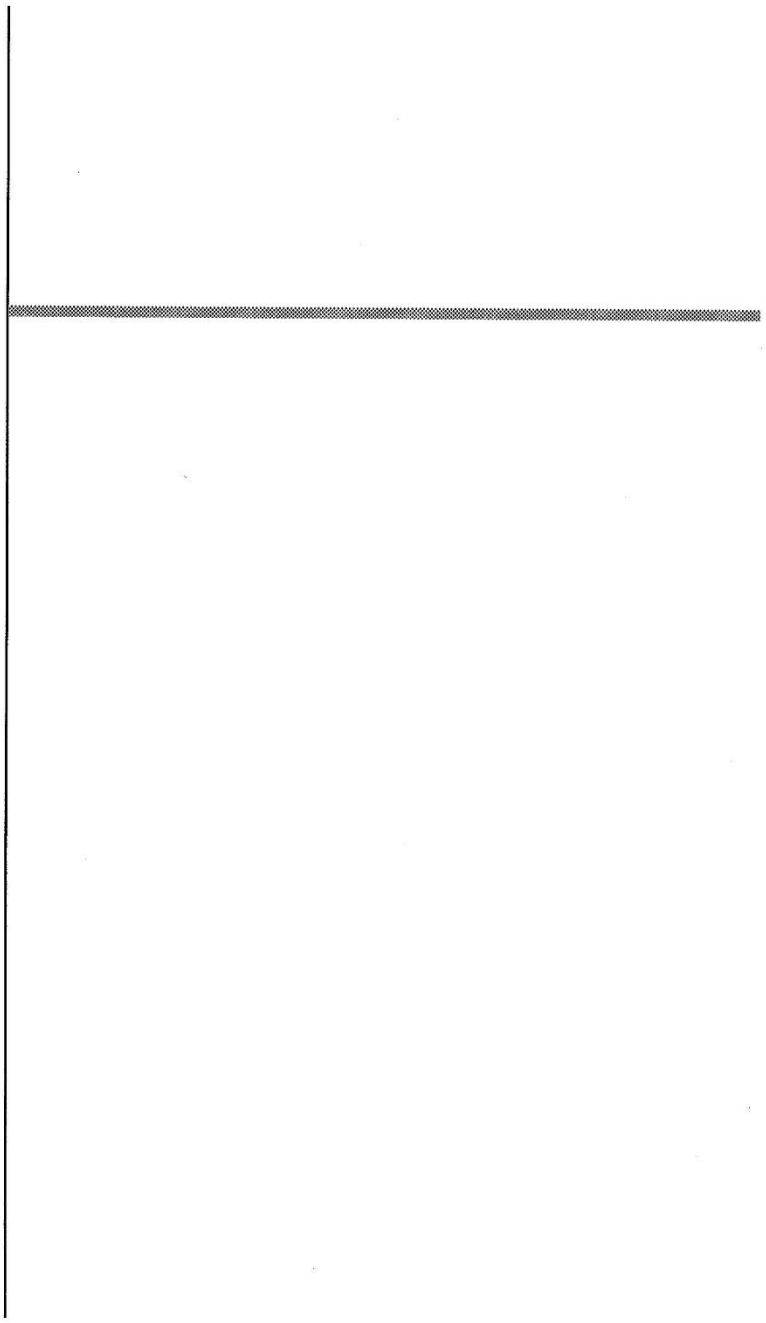
Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Os	8.911	228.900 ppm		
Ir	9.174	212.500 ppm		
Pt	9.443	179.100 ppm		
Au	9.712	191.300 ppm		
Hg	9.989	208.600 ppm		
Tl	10.267	195.200 ppm		
Pb	10.551	145.300 ppm		
Bi	10.838	226.200 ppm		
Th	12.968	306.900 ppm		
U	13.616	385.400 ppm		

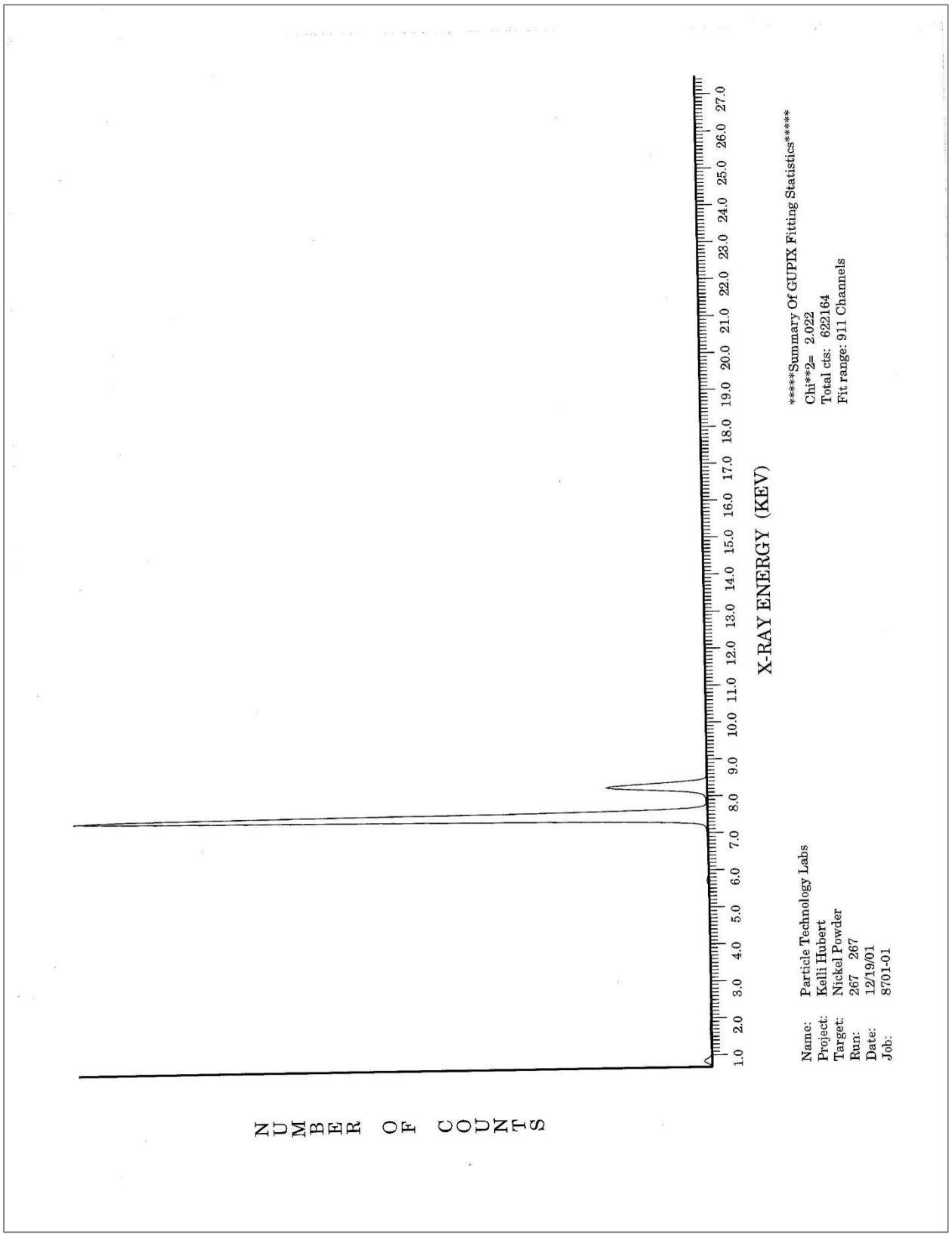
Name: Particle Technology Labs
Project: Kelli Hubert
Target: Nickel Powder

Run: 267 267
Date: 12/19/01
Job: 8701-01

Histogram: Square Root Scaled
Normalization: Internal

Na	[0.131	%]		
Mg	[422.300	ppm]		
Al	[204.000	ppm]		
Si	[137.000	ppm]		
P	[103.600	ppm]		
S	[91.780	ppm]		
Cl	[79.680	ppm]		
K	[44.690	ppm]		
Ca	[41.460	ppm]		
Sc	[45.400	ppm]		
Ti	[44.010	ppm]		
V	[41.840	ppm]		
Cr	[47.950	ppm]		
Mn	[75.600	ppm]		
Fe	[100.300	ppm]		
Co	[119.600	ppm]		
Nickel	99.999	% ±	1.000	
Cu	[369.300	ppm]		
Zn	[118.700	ppm]		
Ga	[70.640	ppm]		
Ge	[88.120	ppm]		
As	[55.260	ppm]		
Se	[77.310	ppm]		
Br	[130.200	ppm]		
Rb	[133.700	ppm]		
Sr	[179.500	ppm]		
Y	[263.900	ppm]		
Zr	[234.300	ppm]		
Nb	[218.800	ppm]		
Mo	[207.000	ppm]		
Tc	[177.500	ppm]		
Ru	[179.000	ppm]		
Rh	[154.500	ppm]		
Pd	[133.800	ppm]		
Ag	[123.800	ppm]		
Cd	[122.700	ppm]		
In	[128.400	ppm]		
Sn	[121.900	ppm]		
Sb	[122.500	ppm]		
Te	[106.700	ppm]		
I	[114.200	ppm]		
Cs	[122.600	ppm]		
Ba	[134.300	ppm]		
La	[124.700	ppm]		
Ce	[120.300	ppm]		
Pr	[137.000	ppm]		
Nd	[144.800	ppm]		
Pm	[160.700	ppm]		
Sm	[319.200	ppm]		
Eu	[263.800	ppm]		
Gd	[191.900	ppm]		
Tb	[220.400	ppm]		
Dy	[270.900	ppm]		
Ho	[234.100	ppm]		
Er	[316.000	ppm]		
Tm	[610.100	ppm]		
Yb	[0.776	%]		
Lu	[0.262	%]		
Hf	[486.000	ppm]		
Ta	[0.269	%]		
W	[0.530	%]		
Re	[312.000	ppm]		
Os	[228.900	ppm]		
Ir	[212.500	ppm]		
Pt	[179.100	ppm]		
Au	[191.300	ppm]		
Hg	[208.600	ppm]		
Tl	[195.200	ppm]		
Pb	[145.300	ppm]		
Bi	[226.200	ppm]		
Th	[306.900	ppm]		
U	[385.400	ppm]		




NUMBER OF COUNTS
1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0
X-RAY ENERGY (KEV)
*****Summary Of GUPIX Fitting Statistics*****
Chi**2= 2.022
Total cts: 622164
Fit range: 911 Channels
Name: Particle Technology Labs
Project: Kelli Hubert
Target: Nickel Powder
Run: 267 267
Date: 12/19/01
Job: 8701-01

Annex 8:

Laboratory report of the particle size distribution analysis of the **Nickel powder** (Particle Technology Labs, 1 page).

MALVERN INSTRUMENTS MASTERSIZER

Result: Histogram Report

Sample Details

Sample ID: N36-PTL
Sample File: 4968
Sample Path: A:\
Sample Notes: NiPERA Nickel Powder SNP-122
Carrier/Dispersant: Canola Oil
Ultrasonic: 1 Minute
Particle Technology Labs

Run Number: 2
Record Number: 3

Analyst: AG
PTL: N36-PTL

Measured: Thu Nov 15 2001 11:37AM
Analysed: Thu Mar 14 2002 10:40AM
Result Source: Analysed

System Details

Range Lens: 300RF mm
Presentation: 3_NIPOW
Analysis Model: Polydisperse
Modifications: None

Beam Length: 2.40 mm
[Particle R.I. = (1.9800, 0.1000); Dispersant R.I. = 1.4712]

Sampler: MS1

Obscuration: 22.1 %
Residual: 2.090 %

Result Statistics

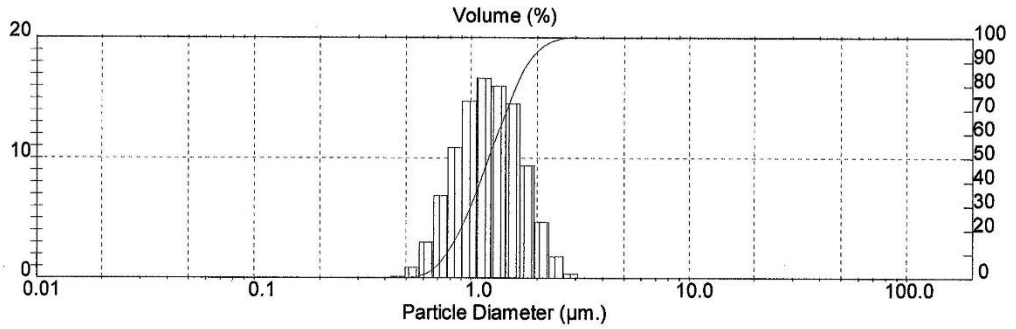
Distribution Type: Volume
Mean Diameters:
D [4, 3] = 1.26 um

Concentration = 0.0029 %Vol
D (v, 0.1) = 0.77 um
D [3, 2] = 1.13 um

Density = 1.000 g / cub. cm
D (v, 0.5) = 1.20 um
Span = 8.916E-01

Specific S.A. = 5.3068 sq. m / g
D (v, 0.9) = 1.84 um
Uniformity = 2.826E-01

Size (um)	Volume Under %	Size (um)	Volume Under %	Size (um)	Volume Under %	Size (um)	Volume Under %
0.055	0.00	0.635	2.60	7.31	100.00	84.15	100.00
0.061	0.00	0.700	5.57	8.06	100.00	92.79	100.00
0.067	0.00	0.772	10.26	8.89	100.00	102.3	100.00
0.074	0.00	0.851	16.56	9.80	100.00	112.8	100.00
0.082	0.00	0.938	24.53	10.81	100.00	124.4	100.00
0.090	0.00	1.03	34.04	11.91	100.00	137.2	100.00
0.099	0.00	1.14	44.54	13.14	100.00	151.3	100.00
0.109	0.00	1.26	55.19	14.49	100.00	166.8	100.00
0.121	0.00	1.39	65.45	15.97	100.00	183.9	100.00
0.133	0.00	1.53	75.39	17.62	100.00	202.8	100.00
0.147	0.00	1.69	84.20	19.42	100.00	223.6	100.00
0.162	0.00	1.86	90.70	21.42	100.00	246.6	100.00
0.178	0.00	2.05	95.03	23.62	100.00	271.9	100.00
0.196	0.00	2.26	97.69	26.04	100.00	299.8	100.00
0.217	0.00	2.49	99.14	28.72	100.00	330.6	100.00
0.239	0.00	2.75	99.80	31.66	100.00	364.6	100.00
0.263	0.00	3.03	99.99	34.92	100.00	402.0	100.00
0.290	0.00	3.34	100.00	38.50	100.00	443.3	100.00
0.320	0.00	3.69	100.00	42.45	100.00	488.8	100.00
0.353	0.00	4.07	100.00	46.81	100.00	539.0	100.00
0.389	0.00	4.48	100.00	51.62	100.00	594.3	100.00
0.429	0.00	4.94	100.00	56.92	100.00	655.4	100.00
0.473	0.08	5.45	100.00	62.76	100.00	722.7	100.00
0.522	0.31	6.01	100.00	69.21	100.00	796.9	100.00
0.576	1.05	6.63	100.00	76.32	100.00	878.7	100.00



Malvern Instruments Ltd.
Malvern, UK
Tel:=+[44] (0)1684-892456 Fax:+[44] (0)1684-892789

Mastersizer S long bed Ver. 2.18
Serial Number:

p. 2
14 Mar 02 10:41

Annex 9:

Laboratory report of the specific surface (N-BET) analysis of the **Nickel powder** (Particle Technology Labs, 2 pages).

Date: 11/27/2001 Page 1

Particle Technology Labs, Ltd.
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.16

Sample ID	N36-PTL				
Description	NIPERA				
Comments	4968 N36-PTL Nickel Powder SNP-122				
Sample Weight	1.3649 g				
Adsorbate	NITROGEN	Outgas Temp	200.0 °C	Operator	ED
Cross-Sec Area	16.2 Å²/molecule	Outgas Time	5.0 hrs	Analysis Time	41.5 min
NonIdeality	6.580E-05	P/Po Toler	4	End of Run	11/19/01 07:13PM
Molecular Wt	28.0134 g/mol	Equil Time	2	File Name	AS0B1501.RAW
Station #	1	Bath Temp.	0.00		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
1.1300e-01	0.6301	1.618E+02
1.6300e-01	0.6814	2.287E+02
2.1300e-01	0.7327	2.956E+02

Area = 2.582E+00 m²/g

Slope = 1.338E+03

Y - Intercept = 1.059E+01

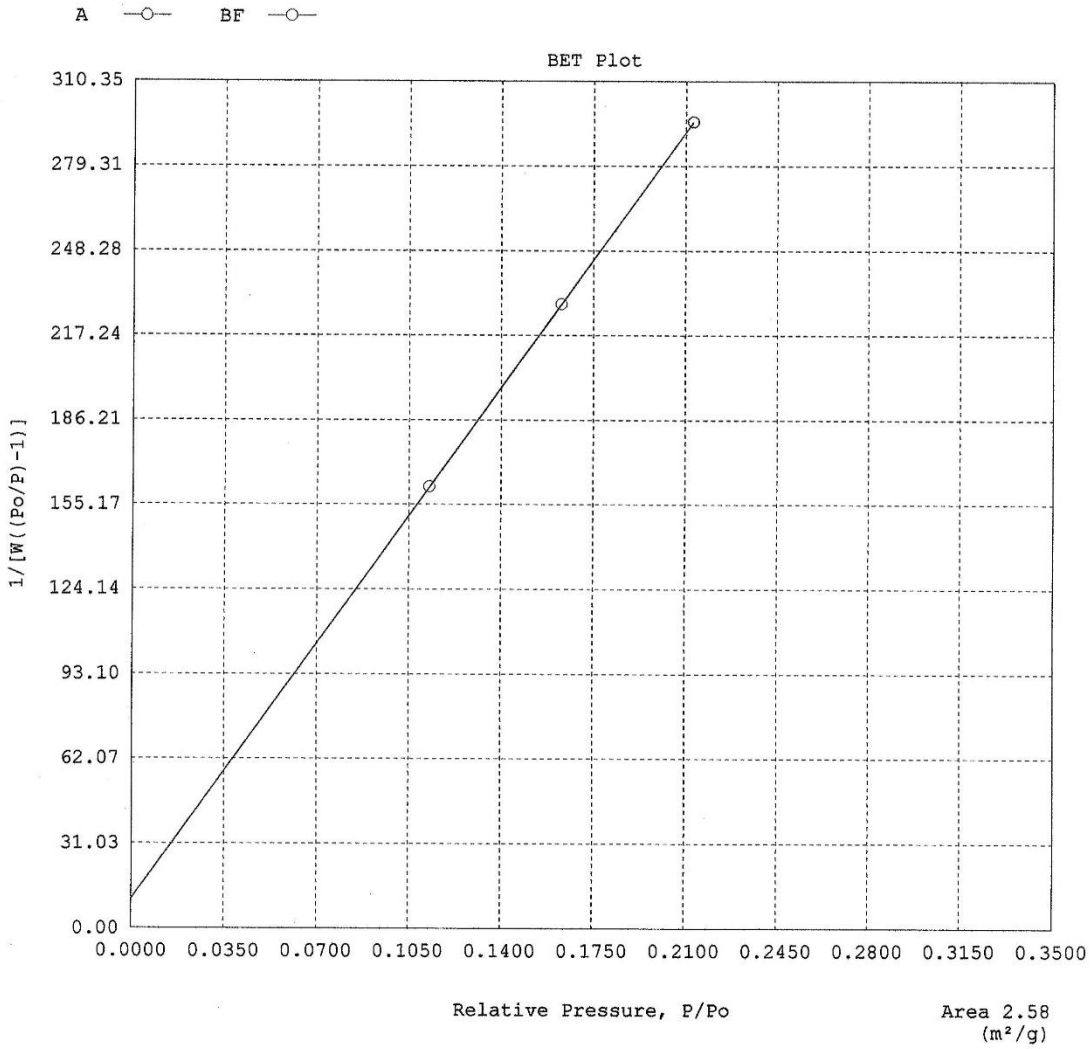
Correlation Coefficient = 1.000000

C = 1.273E+02

Date: 11/27/2001

Particle Technology Labs, Ltd.
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.16

Sample ID	N36-PTL				
Description	NIPERA				
Comments	4968 N36-PTL Nickel Powder SNP-122				
Sample Weight	1.3649 g				
Adsorbate	NITROGEN	**Outgas Temp**	200.0 °C	**Operator**	ED
Cross-Sec Area	16.2 Å²/molecule	**Outgas Time**	5.0 hrs	**Analysis Time**	41.5 min
NonIdeality	6.580E-05	**P/Po Toler**	4	**End of Run**	11/19/01 07:13PM
Molecular Wt	28.0134 g/mol	**Equil Time**	2	**File Name**	AS0B1501.RAW
Station #	1	**Bath Temp.**	0.00		



Annex 10:

Certificate of Analysis of **$NiCl_2.6H_2O$** (Acros Organics, 1 page).

Certificate of Analysis **Page 1 of 1**

ACROS ORGANICS

Version	00
Molecular weight	237.71
Quality Test / Release Date	06/18/2013
Molecular Formula	Cl2 Ni . 6 H2 O
CAS No	7791- 20- 0
Linear Formula	NiCl2.6H2O
Flash Point (°C)	

Certificate of Analysis

This is to certify that units of the above mentioned lot number were tested and found to comply with the specifications of the grade listed. Certain data have been supplied by third parties. Acros Organics expressly disclaims all warranties,expressed or implied, including the implied warranties of merchantability and fitness for a particular purpose. Unless otherwise stated, these products are not intended for dialysis, parenteral, or injectable use without further processing. The following are the actual analytical results obtained:

Catalog Number	19357	**Quality Test / Release Date** 06/18/2013
Lot Number	A0336441	
Description	Nickel(II) chloride hexahydrate,99.999%,(trace metal basis)	
Country of Origin	UNITED STATES OF AMERICA	
Declaration of Origin	synthetic	

BSE/TSE comment 1	
Chemical Comment	

Result name	Units	Specifications	Test Value
Appearance		green crystals, crystalline powder and/or chunks	green crystals, crystalline powder and/or chunks
Nickel (Ni)		24 to 26 %	24.1 %
Total trace metal imp.		=<0.001 %	0.00022 %



L. Van Den Broek, QA Manager Issued: 07- 24- 2013

Acros Organics
ENA23, zone1, nr 1350, Janssen Pharmaceuticalaan 3a, B- 2440 Geel, Belgium
Tel +32 14/57.52.11 - Fax +32 14/59.34.34 Internet: http://www.acros.com
1 Regent Lane, Fair Lawn, NJ 07410, USA Fax 201- 796- 1329

Annex 11:

Certificate of Analysis of the **Iron powder** (Sigma-Aldrich, 1 page).

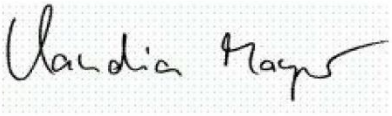
SIGMA-ALDRICH®

3050 Spruce Street, Saint Louis, MO 63103 USA
Email USA: techserv@sial.com Outside USA: eurtechserv@sial.com

Certificate of Analysis

Product Name:	IRON powder, –325mesh, 97 %
Product Number:	209309
Batch Number:	STBG7186
Brand:	Aldrich
CAS Number:	7439-89-6
Formula:	Fe
Formula Weight:	55.85
Quality Release Date:	07 DEC 2016

TEST	SPECIFICATION	RESULT
APPEARANCE (COLOR)	GREY	GREY
APPEARANCE (FORM)	POWDER	POWDER
THIOSULFATE TITRATION	96.5 - 103.5 %	98.5 %
PARTICLE SIZE	-325 MESH	CONFIRMED
X-RAY DIFFRACTION	CONFORMS TO STRUCTURE	CONFORMS



Claudia Mayer
Manager Quality Control
Steinheim, Germany

Sigma-Aldrich warrants that at the time of the quality release or subsequent retest date this product conformed to the information contained in this publication. The current specification sheet may be available at Sigma-Aldrich.com. For further inquiries, please contact Technical Service. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

Annex 12:

Certificate of Analysis of the specific surface (N-BET) analysis, the particle size distribution (PSD) analysis of the **Iron powder** (Umicore analytical Competence Center, 2 pages).

Umicore
Analytical Competence Center
Watertorenstraat 33
B-2250 OLEN

CoA 2017-0106

umicore
Research & Development

CERTIFICATE OF ANALYSIS

Page No. 1 of 2

Certificate Date	**03/10/2017**	Certificate No.	Lab LIMS No.	Your Ref. / Order No.
Date of Receipt	**29/09/2017**	**2017-0106**	**B-2017-0053149**	**Tel from 18/09/2017**

Supplier:
Tony Brouwers
ECTX bvba
Havenstraat 46/0.01
3500 Hasselt
Belgium

Sample Identification	R0039/01 Iron powder SA 209309

Analysis result of BET and PSD analysis:

BET: 0.30 m²/g

Particle size distribution: on Page 2

Abstract Tests & Conditions:

- **BET analysis:**
  The determination of the specific surface, expressed in m²/g, is based on the adsorption of N2 molecules on the surface of powder particles. At a relative pressure of 30% and at liquid N2 temperature, the amount of adsorbed N2 atoms is related to the total available surface of all powder particles.
  The used equipment to measure the resulting pressure drop around a certain mass of powder is the 'Tristar 3000' from Micromeritics with measuring range: 0.02-215 m²/g.
- **PSD analysis:**
  Particle size distribution on sample as such using Sympatec Helos laser diffraction. Laser diffraction is an optical measuring process, where the diffraction pattern caused by the presence of particles in the path of a laser beam is measured. The dispersion of the particles in the laser beam is realised by compressed air on dry powder.
  The diameter of the diffraction ring generated is inversely proportional to the size of the particles present, while the distribution of the light energy in the diffraction pattern contains the information on their quantity.

Certificate signed by

☒ Eddy Boydens
Lab Manager

Liesbet Jongen
Lab Manager

Marc Sterckx
Lab Manager

Signed:



The results of analyses in this report are only valid for the tested sample(s).
This analysis report may be reproduced only in its full form and only by ACC Olen and the supplier.

UMICORE n.v.
Analytical Competence Center Olen
Watertorenstraat 33,
B – 2250 Olen,
BELGIUM

Tel +32-14 24 51 49
Fax +32-14 24 56 79

VAT (BE) 0 401 574 852
Bank 210-0053806-23
HR Turnhout 64 124
Registered Office Broekstraat 31 Rue du Marais, B-1000 Brussels, Belgium

Umicore
Analytical Competence Center
Watertorenstraat 33
B-2250 OLEN

CoA 2017-0106

umicore
Research & Development

CERTIFICATE OF ANALYSIS

Particle size distribution

Sympatec GmbH
System-Partikel-Technik

HELOS Particle Size Analysis
WINDOX 5

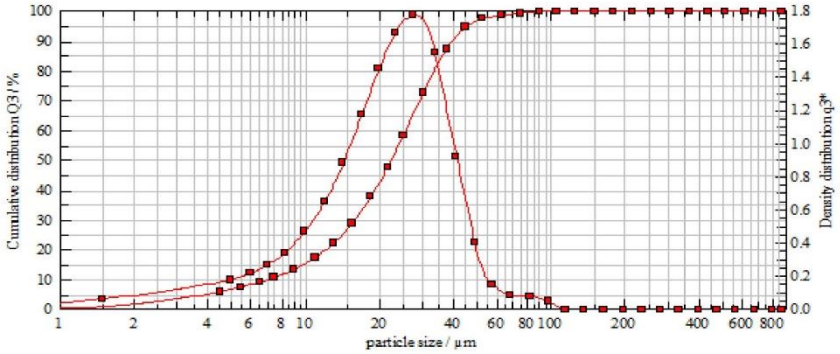
UMICORE

HELOS (H1920) & RODOS, R5: 0.5/4.5...875µm **2017-10-04, 14:02:09,441**

Divers B-2017-0053149 R0039-01

x_1 = 1,18 µm	x_{16} = 10,31 µm	x_{95} = 46,15 µm
x_2 = 1,86 µm	x_{50} = 22,34 µm	x_{98} = 58,79 µm
x_5 = 3,91 µm	x_{84} = 35,85 µm	x_{99} = 73,48 µm
x_{10} = 7,12 µm	x_{90} = 40,28 µm	x_{100} = 105,00 µm

Optische concentratie: 13,13 %



comment:
Iron powder SA 209309

user parameters:
Gebruiker: jvdv
Identifier: B-2017-0053149 R0039-01
Parameter 3:

cumulative distribution

x_0/µm	Q_3/%	x_0/µm	Q_3/%	x_0/µm	Q_3/%	x_0/µm	Q_3/%
4,50	5,86	18,50	37,87	75,00	99,08	305,00	100,00
5,50	7,41	21,50	47,38	90,00	99,70	365,00	100,00
6,50	8,98	25,00	58,30	105,00	100,00	435,00	100,00
7,50	10,63	30,00	72,33	125,00	100,00	515,00	100,00
9,00	13,33	37,50	87,30	150,00	100,00	615,00	100,00
11,00	17,42	45,00	94,59	180,00	100,00	735,00	100,00
13,00	22,13	52,50	97,29	215,00	100,00	875,00	100,00
15,50	28,84	62,50	98,42	255,00	100,00		

evaluation: WINDOX 5.7.1.0, *HRLD*
revalidation:
reference measurement: 10-04 14:01:53
contamination: 0,00 %

Measurement duration
Measurement duration: 1,31 s

C_{opt}= 13,13 %

trigger condition: opt conc 2%
time base: 100,00 ms
start: c.opt >= 2,1%
valid: always
stop: 5s c.opt <= 1,9% or 99s real time

dispersing method: 3.0bar;100%;1.0mm;delay20s
cascade: 0
pressure: 3,03 bar, vacuum: 99,00 mbar
revolution: 100,00 %
doser: VIBRI, feed rate: 100,00 %

UMICORE n.v.
Analytical Competence Center Olen
Watertorenstraat 33,
B – 2250 Olen,
BELGIUM

Tel +32-14 24 51 49
Fax +32-14 24 56 79

VAT (BE) 0 401 574 852
Bank 210-0053806-23
HR Turnhout 64 124
Registered Office Broekstraat 31 Rue du Marais, B-1000 Brussels, Belgium

Annex 13:
Certificate of Analysis of **$FeSO_4.7H_2O$** (Acros Organics, 1 page).

Certificate of Analysis **Page 1 of 1**

ACROS ORGANICS

Version	00
Molecular weight	278.01
Quality Test / Release Date	02/10/2014
Molecular Formula	Fe O4 S . 7 H2 O
CAS No	7782- 63- 0
Linear Formula	FeSO4.7H2O
Flash Point (°C)	

Certificate of Analysis

This is to certify that units of the above mentioned lot number were tested and found to comply with the specifications of the grade listed. Certain data have been supplied by third parties. Acros Organics expressly disclaims all warranties,expressed or implied, including the implied warranties of merchantability and fitness for a particular purpose. Unless otherwise stated, these products are not intended for dialysis, parenteral, or injectable use without further processing. The following are the actual analytical results obtained:

Catalog Number	20139	**Quality Test / Release Date** 02/10/2014
Lot Number	A0344065	
Description	Iron(II) sulfate heptahydrate,99.5%,for analysis	
Country of Origin	SPAIN	
Declaration of Origin	synthetic	

BSE/TSE comment	
Chemical Comment	

Result name	Units	Specifications	Test Value
Appearance		slightly greenish to blue crystals	slightly blue crystals
Assay		>=99.5 %	101.3 %
Subst not pptd by NH3		=<0.05 % (as SO4)	=<0.05 % (as SO4)
Total nitrogen compounds		=<0.001 %	=<0.001 %
Trace analysis		Type: Cl measure =< 5 ppm	Type: Cl measure =< 5 ppm
Trace analysis		Type: PO4 measure =< 10 ppm	Type: PO4 measure =< 10 ppm
Trace analysis		Type: Cu measure =< 10 ppm	Type: Cu measure 7 ppm
Trace analysis		Type: Zn measure =< 50 ppm	Type: Zn measure 6 ppm
Trace analysis		Type: Pb measure =< 5 ppm	Type: Pb measure =< 1 ppm
Trace analysis		Type: Fe3+ measure =< 100 ppm	Type: Fe3+ measure =< 100 ppm



L. Van den Broek, QA Manager Issued: 10- 02- 2014

Acros Organics
ENA23, zone1, nr 1350, Janssen Pharmaceuticalaan 3a, B- 2440 Geel, Belgium
Tel +32 14/57.52.11 - Fax +32 14/59.34.34 Internet: http://www.acros.com
1 Regent Lane, Fair Lawn, NJ 07410, USA Fax 201- 796- 1329

Annex 14:

Certificate of Analysis of **SS316L Powder** (PIXE Test Report - 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:	9/26/2016
Run:	409
Job:	3352-16
Name:	Particle Testing Lab
Project:	Arnette Schultz
Target:	N155A.3-PTL

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Na	1.041	893.600 ppm		
Mg	1.254	324.600 ppm		
Al	1.485	191.500 ppm		
Silicon	1.740	164.300 ppm	0.46%	0.02%
P	2.010	163.400 ppm		
S	2.308	0.110%		
Cl	2.623	66.460 ppm		
K	3.314	31.500 ppm		
Ca	3.692	71.770 ppm		
Sc	4.091	42.950 ppm		
Titanium	4.511	95.990 ppm	0.022%	0.007%
Vanadium	4.952	92.050 ppm	0.032%	0.007%
Chromium	5.415	113.900 ppm	16.41%	0.16%
Manganese	5.899	0.136%	1.28%	0.05%
Iron	6.405	490.800 ppm	68.61%	0.69%
Co	6.930	0.214%		
Nickel	7.478	206.500 ppm	10.65%	0.11%
Copper	8.048	244.700 ppm	0.32%	0.03%
Zn	8.639	38.800 ppm		
Ga	9.250	32.440 ppm		
Ge	9.887	33.490 ppm		
As	10.544	47.910 ppm		
Se	11.222	31.320 ppm		
Br	11.924	59.360 ppm		
Rb	13.395	94.690 ppm		
Sr	14.165	55.960 ppm		
Y	14.959	55.680 ppm		
Zr	15.775	103.900 ppm		
Niobium	16.615	160.600 ppm	0.069%	0.015%
Molybdenum	2.293	197.500 ppm	2.10%	0.03%
Tc	18.367	124.800 ppm		
Ru	19.279	173.800 ppm		
Rh	2.697	118.700 ppm		
Pd	2.839	119.400 ppm		
Ag	2.984	96.600 ppm		
Cd	3.133	83.920 ppm		
In	3.286	85.770 ppm		
Sn	3.444	108.100 ppm		
Sb	3.604	171.900 ppm		
Te	3.768	135.200 ppm		
I	3.937	106.000 ppm		
Cs	4.288	117.800 ppm		
Ba	4.466	139.800 ppm		
La	4.648	429.200 ppm		
Ce	4.841	187.900 ppm		
Pr	5.034	232.900 ppm		
Nd	5.230	655.000 ppm		
Pm	5.431	0.329%		
Sm	5.632	344.800 ppm		
Eu	5.841	0.112%		
Gd	6.050	987.000 ppm		
Tb	6.271	0.281%		
Dy	6.492	0.396%		

Page 1 of 2

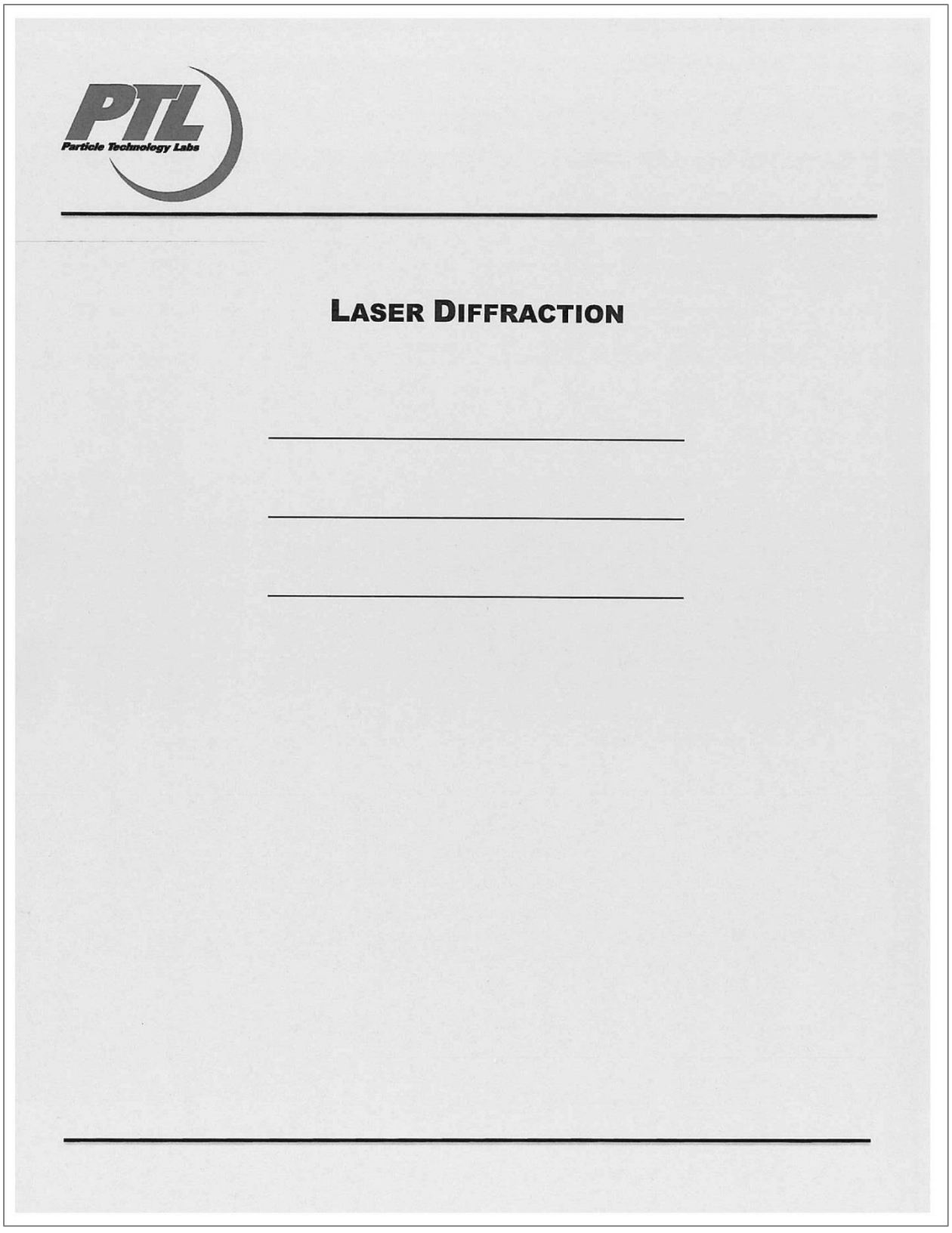
PIXE Test Report
Elemental Analysis, Inc.
2101 Capstone Dr. Ste. 110
Lexington, KY 40511
Tel: 1-800-563-PIXE(7493)

Date:	9/26/2016
Run:	409
Job:	3352-16
Name:	Particle Testing Lab
Project:	Arnette Schultz
Target:	N155A.3-PTL

Element Name	Energy (keV)	Det. Limit 95% Conf.	Concentration Mass	Error
Ho	6.725	318.500 ppm		
Er	6.945	0.152%		
Tm	7.182	0.220%		
Yb	7.416	0.264%		
Lu	7.655	711.900 ppm		
Hf	7.899	280.500 ppm		
Ta	8.149	809.000 ppm		
W	8.398	739.900 ppm		
Re	8.652	109.100 ppm		
Os	8.911	232.400 ppm		
Ir	9.174	86.410 ppm		
Pt	9.443	75.500 ppm		
Au	9.712	77.240 ppm		
Hg	9.989	113.500 ppm		
Tl	10.267	95.360 ppm		
Pb	10.551	78.440 ppm		
Bi	10.838	76.750 ppm		
Th	2.992	177.300 ppm		
U	3.166	151.300 ppm		

Annex 15:

Laboratory report of the particle size distribution analysis of **SS316L Powder** (Particle Technology Labs, 3 pages).



555 Rogers Street, Downers Grove, IL 60515
630-969-2703 ph 630-969-2745 fx
www.particletechlabs.com

THE PARTICLE EXPERTS

INTERPRETING YOUR MALVERN FINE PARTICLE ANALYSIS

INTRODUCTION

The particle size analysis of your sample(s) has been conducted on a Malvern® MasterSizer LASER diffractor. This instrument is considered an ensemble analyzer that calculates a volume distribution from the LASER (**L**ight **A**mplification by **S**timulated **E**mission of **R**adiation) diffraction pattern of a suspension of particles. This raw scatter data are then processed using a complex algorithm and presented on the basis of **EQUIVALENT SPHERICAL DIAMETER**. The Malvern equipment currently in use at Particle Technology Labs (PTL) are the *MasterSizer S*, the *MasterSizer 2000, and the MasterSizer 3000.*

THE DATA

The header section contains various user-entered information including client name, sample identification, and analysis notes. Each project submitted to PTL is given a unique seven digit code (PTL Project #) which can be found on these data pages, although its location is dependent on the instrumentation used. On the MasterSizer S data, the PTL Project # is identified as Sample File. On the MasterSizer 2000 and 3000 data, the PTL Project # appears at the bottom of the page and is identified as File Name or within the File. Please refer to this PTL Project # when contacting us with any questions regarding the analysis.

The data output also provides the parameters specific to the instrument being used, as well as the parameters specific to the analysis. The optical model (composed of the sample's refractive index as well as the imaginary absorption value) is required if determining the particle size using Mie theory. This theory can be used on any size particles, but is specifically recommended over the use of the Fraunhofer approximation according to ISO 13320:2009 for material in the size region less than approximately 50 µm. Please note that, unless otherwise requested or provided by the client, the analysis of an unknown material is generally conducted using the standard instrument default optical values as detailed below.

SYSTEM DEFAULT VALUES

Settings	MASTERSIZER S	MASTERSIZER 2000 / MASTERSIZER 3000	
	Wet	Wet	Dry
Analysis Model	Polydisperse	General Purpose	General Purpose
Presentation	30HD	Default	Default
Sample RI Value	1.5295	1.520	1.520

The Fraunhofer approximation, which does not require specific knowledge of the optical properties of the sample, can be applied for large particles if a known refractive index of the material is not available or the sample is composed of multiple components. If the actual RI of the sample material is provided at a later date, the raw data can be recalculated to adjust for the refractive index.

Please note the following commonly reported values when reviewing your data:

- **TABULATED DATA:** ALWAYS appears as a Cumulative % less than (*Volume Under %*) unless otherwise requested.
- **SPAN:** Value related to the width of the curve, expressed as $\frac{[D(v,0.90) - D(v,0.10)]}{D(v,0.50)}$
- **D[3,2]:** Surface-weighted mean diameter (Sauter diameter)
- **D[4,3]:** Volume-weighted MEAN
- **10%, 50%, 90% size values:** Indicates the size median which 10%, 50%, or 90% of the particles within the distribution is smaller than (example: D(0.9): 140 µm, this means that 90% of the particulate is smaller than 140 µm on a volume basis).
- **Specific Surface Area:** If a calculated Specific Surface Area value is reported, consider this value only as an approximate surface area since calculations are based upon **non-porous spheres**. It is not a replacement for a result produced from an actual gas adsorption instrument due to the above assumption.

Also included is the Particle Size Distribution DIFFERENTIAL HISTOGRAM. This histogram shows the Tabulated Data as a **Differential Volume Percent Less Than Indicated Size**. Please note that the histograms for the *MasterSizer S* and *MasterSizer 3000* also generally include a CUMULATIVE Curve representative of the Tabulated Data.

For additional questions specific to your sample results, please contact us directly.

MASTERSIZER 2000

Result Analysis Report

Operator notes:

NIPERA *SS316L Powder*
Carrier: Non-aqueous *N-155A*
Chemist: LMR
Particle Technology Labs *PTL ID#: 225747-10*

SOP Name:

Result Source:
Averaged

Number of this Measurement:
0

Measured:
Wednesday, August 31, 2016 11:27:51 AM

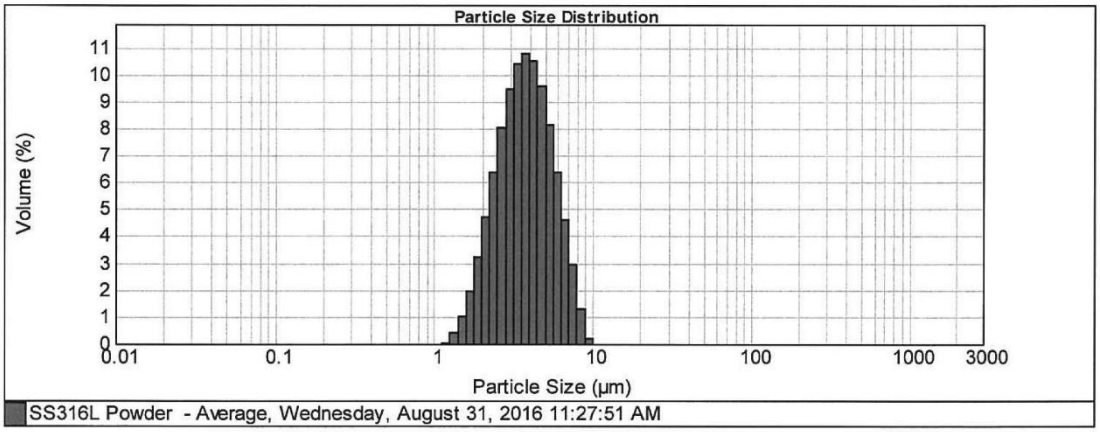
Analyzed:
Wednesday, August 31, 2016 11:27:53 AM

Number of Measurements:
0

Particle Name: Default-1.0	**Accessory Name:** Hydro 2000S (A)	**Pump Speed:** 2000 rpm	**Analysis model:** General purpose	**Sensitivity:** Normal
	Particle RI: 1.520	**Absorption:** 1	**Size range:** 0.020 to 2000.000 µm	**Obscuration:** 11.75%
Dispersant Name: In-house hydrocarbon (1.42)	**Dispersant RI:** 1.420	**Average Result Records:** "1,2,3,4"	**Weighted Residual:** 0.430 %	**Result Emulation:** Off

Concentration: 0.0055 %Vol	**Span :** 1.079	**Uniformity:** 0.33	**Result units:** Volume
Specific Surface Area: 1.76 m²/g	**Surface Weighted Mean D[3,2]:** 3.413 µm	**Vol. Weighted Mean D[4,3]:** 3.987 µm	

d (0.1) : 2.177 µm **d (0.5) : 3.727 µm** **d (0.9) : 6.199 µm**



Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.020	0.00	0.142	0.00	1.002	0.00	7.096	95.57	50.238	100.00	355.656	100.00
0.022	0.00	0.159	0.00	1.125	0.00	7.962	98.51	56.368	100.00	399.052	100.00
0.025	0.00	0.178	0.00	1.262	0.04	8.934	99.83	63.246	100.00	447.744	100.00
0.028	0.00	0.200	0.00	1.416	0.47	10.024	100.00	70.963	100.00	502.377	100.00
0.032	0.00	0.224	0.00	1.589	1.50	11.247	100.00	79.621	100.00	563.677	100.00
0.036	0.00	0.252	0.00	1.783	3.46	12.619	100.00	89.337	100.00	632.456	100.00
0.040	0.00	0.283	0.00	2.000	6.67	14.159	100.00	100.237	100.00	709.627	100.00
0.045	0.00	0.317	0.00	2.244	11.39	15.887	100.00	112.468	100.00	796.214	100.00
0.050	0.00	0.356	0.00	2.518	17.78	17.825	100.00	126.191	100.00	893.367	100.00
0.056	0.00	0.399	0.00	2.825	25.80	20.000	100.00	141.589	100.00	1002.374	100.00
0.063	0.00	0.448	0.00	3.170	35.23	22.440	100.00	158.866	100.00	1124.683	100.00
0.071	0.00	0.502	0.00	3.557	45.64	25.179	100.00	178.250	100.00	1261.915	100.00
0.080	0.00	0.564	0.00	3.991	56.42	28.251	100.00	200.000	100.00	1415.892	100.00
0.089	0.00	0.632	0.00	4.477	66.91	31.698	100.00	224.404	100.00	1588.656	100.00
0.100	0.00	0.710	0.00	5.024	76.46	35.566	100.00	251.785	100.00	1782.502	100.00
0.112	0.00	0.796	0.00	5.637	84.59	39.905	100.00	282.508	100.00	2000.000	100.00
0.126	0.00	0.893	0.00	6.325	90.98	44.774	100.00	316.979	100.00		

LMR
2016-08-31
EF 2016-08-31

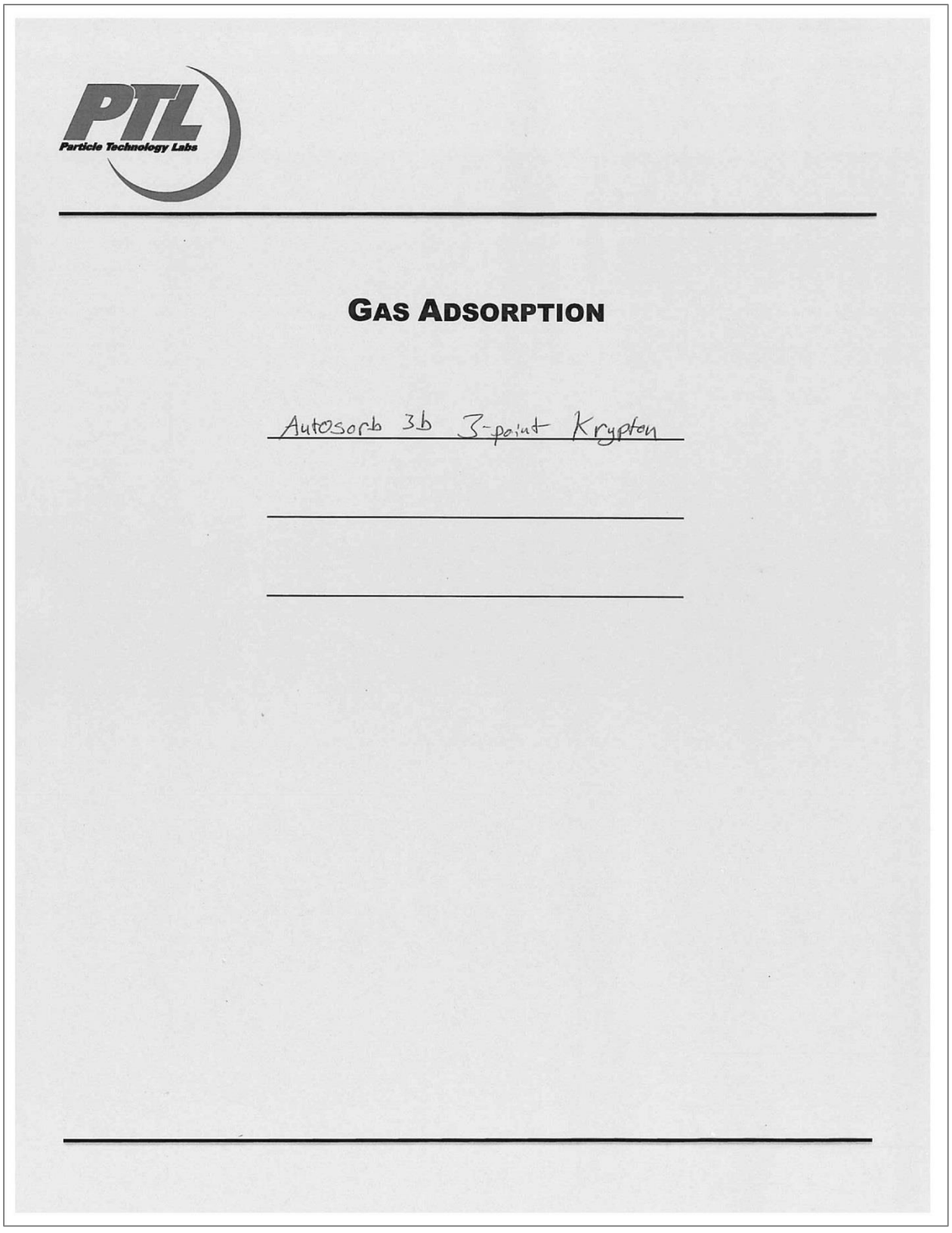
Particle Technology Labs
555 Rogers Street
Downers Grove, IL 60515

Mastersizer 2000 Ver. 5.60
Serial Number : MAL100023

File name: 36640-10
Record Number: 5
8/31/2016 11:43:44 AM

Annex 16:

Laboratory report of the specific surface (Kr-BET) analysis of **SS316L Powder** (Particle Technology Labs, 5 pages).



Particle Technology Labs

BET SURFACE AREA DATA SUMMARY

SAMPLE ID	SPECIFIC SURFACE AREA (m^2/g)	DEGASSING CONDITIONS			ADSORBATE
		DURATION	TEMPERATURE	TECHNIQUE	
SS316L Powder					
N-155A	0.35	1.5 hours	Room temperature	Vacuum	Krypton

JF 2016-08-31
CP 2016-08-31.

PRELIMINARY RESULTS **PTL Project #36640-10**

555 Rogers Street, Downers Grove, IL 60515
630-969-2703 ph 630-969-2745 fx
www.particletechlabs.com

THE PARTICLE EXPERTS

INTERPRETING YOUR BET SPECIFIC SURFACE AREA ANALYSIS BY GAS PHYSISORPTION (STATIC VOLUMETRIC METHOD)

INTRODUCTION

The surface area of your sample(s) has been measured via gas physisorption analysis, using the static volumetric method. For a typical multipoint determination, the amount of inert gas that adsorbs to the surface of a sample is measured at varying relative pressures. The surface area is calculated using the Brunauer–Emmett–Teller (BET) equation below:

$$\frac{1}{V_a\left(\left(\frac{P}{P_0}\right)-1\right)} = \frac{C-1}{V_m C} \; x \; \frac{P}{P_0} + \frac{1}{V_m C}$$

where $\frac{P}{P_0}$ = relative pressure,
V_a = volume of gas adsorbed,
V_m = volume of gas adsorbed to produce an apparent monolayer on the surface,
C = BET constant.

For the BET transform, the quantity $\frac{1}{V_a\left(\left(\frac{P}{P_0}\right)-1\right)}$ is plotted against the relative pressure (P/P_0). The BET plot should yield a straight line around the relative pressures where monolayer adsorption occurs, typically in the range of 0.05-0.3. The volume of gas needed to produce a monolayer on the sample surface (V_m) and the BET constant (C) are derived from the slope and y-intercept of the line from the BET plot. The specific surface area is determined using the calculated V_m, the cross sectional area of the adsorbate gas and the mass of the test powder.

The approximate BET surface area can also be determined at one single relative pressure only (commonly at $P/P_0 = 0.3$), either assuming the y-intercept of the BET plot is zero or using certain previously established parameters through multipoint determination for the sample type.

SAMPLE PREPARATION

As the amount of gas adsorbed onto the sample surface is critical to the analysis, moisture and other impurities must be removed from the sample surface prior to analysis. This process, called degassing, may involve pulling vacuum on, flowing inert gas over the sample, or both. Degassing can also be done at different temperatures depending on the thermal stability of the material. It is extremely important to choose a temperature and technique such that the sample is not altered physically or chemically since either could affect the surface area of the sample. The degassing conditions used in your analysis are included on the preliminary results summary page and the final report.

THE DATA

The header section contains various user-entered information specific to each project. It generally includes the client name, sample identification, and analysis notes. Each project is given its own unique seven digit code (PTL Project #) which can be found on these data pages. Please refer to this PTL Project # when contacting us with any questions regarding the analysis.

The data includes a BET tabular report and the BET plot. A table of the relative pressure and quantity of gas adsorbed at that relative pressure is provided in the tabular portion of the data. The last column on the right is the BET transform of the data, which can also be visualized in the BET plot.

Commonly reported values from your surface area data are explained below:

- **BET Specific Surface Area**: the surface area calculated based on the BET model normalized by the sample mass.
- **C Value (also called the BET constant)**: a value related to the affinity of the solid with the adsorbate gas. Since a negative C value has no physical meaning, the C value must be positive. A very low C value indicates the adsorbate gas did not interact well with the surface, suggesting the gas may not have formed a complete monolayer. On the other hand, a high C value may indicate a strong interaction between the surface and adsorbate gas, e.g. chemisorption sites or presence of micropores.
- **Correlation Coefficient**: a value that indicates the linear transform fit of the adsorption data. The BET surface area should only be evaluated over the linear region of the BET plot. At PTL, as per the current USP <846>, the correlation coefficient must be ≥ 0.9975 for data to be considered acceptable.

For additional questions specific to your sample results, please contact us directly.

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.16

Sample ID	NIPERA SS316L Powder PTL Project# 36640-10				
Description	N-155A PTL ID: 225747-10				
Comments					
Sample Weight	7.9991 g				
Adsorbate	KRYPTON	Outgas Temp	23.0 °C	Operator	JF
Cross-Sec Area	20.5 Å²/molecule	Outgas Time	1.5 hrs	Analysis Time	422.3 min
NonIdeality	3.000E-05	P/Po Toler	0	End of Run	08/30/2016 23:36
Molecular Wt	83.8000 g/mol	Equil Time	4	File Name	36640A.RAW
Station #	1	Bath Temp.	77.40		

MULTIPOINT BET

P/Po	Volume [cc/g] STP	1/(W((Po/P)-1))
9.9161e-02	0.0653	4.508E+02
2.0024e-01	0.0787	8.510E+02
2.9912e-01	0.0883	1.292E+03

Area = 3.481E-01 m²/g

Slope = 4.207E+03

Y - Intercept = 2.532E+01

Correlation Coefficient = 0.999400

C = 1.672E+02

JF 2016-08-31

QP 2016-08-31.

Date: 08/31/2016

Quantachrome Corporation
Quantachrome Autosorb Automated Gas Sorption System Report
Autosorb for Windows® Version 1.16

Sample ID	NIPERA SS316L Powder PTL Project# 36640-10				
Description	N-155A PTL ID: 225747-10				
Comments					
Sample Weight	7.9991 g				
Adsorbate	KRYPTON	**Outgas Temp**	23.0 °C	**Operator**	JF
Cross-Sec Area	20.5 Å²/molecule	**Outgas Time**	1.5 hrs	**Analysis Time**	422.3 min
NonIdeality	3.000E-05	**P/Po Toler**	0	**End of Run**	08/30/2016 23:36
Molecular Wt	83.8000 g/mol	**Equil Time**	4	**File Name**	36640A.RAW
Station #	1	**Bath Temp.**	77.40		

A —○— BF —○—

