FINAL REPORT

Study no.

X02a-146

4 hours Bio-elution Kinetics Study on

Z45 (massive)

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

in a simulated gastric fluid

April 21, 2018

Statement

Title: 4 hours Bio-elution Kinetics Study on **Z45** (massive) at a *2 g/L loading* in a simulated gastric fluid.

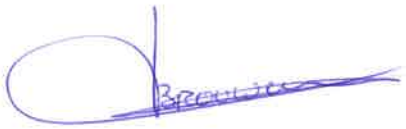
Study no.: **X02a-146**

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 **Z45** (massive) at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-146**

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: - adding test item - experiment, gastric at pH 1.5	 24-01-2018 24-01-2018	18-04-2018
Raw data	18-04-2018	18-04-2018
Draft report	18-04-2018	18-04-2018
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

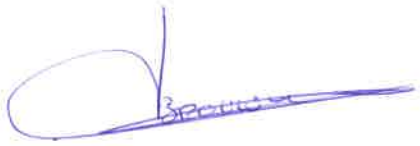
Statement of Authentication

Title: 4 hours Bio-elution Kinetics Study on **Z45** (massive) at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-146**

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 **7**

2.1 STUDY TITLE AND NUMBER 7

2.2 TEST ITEM IDENTIFICATION AND CHARACTERISATION 7

2.3 REFERENCE ITEM IDENTIFICATION AND CHARACTERISATION 8

3 INFORMATION CONCERNING SPONSOR, TEST FACILITY AND TEST SITE **10**

3.1 SPONSOR IDENTIFICATION 10

3.2 TEST FACILITY IDENTIFICATION 10

3.3 TEST SITE IDENTIFICATION 10

4 DATES **11**

5 TEST METHODS **12**

5.1 TEST GUIDANCE 12

5.2 STANDARD OPERATING PROCEDURES (SOPS) 12

6 TEST SYSTEM **13**

6.1 SIMULATED GASTRIC FLUID 13

7 EXPERIMENTAL DESIGN **14**

7.1 MATERIALS 14

7.2 PREPARATION OF THE TEST ITEM 14

7.3 PREPARATION OF THE REFERENCE ITEM 15

7.4 TEST CONDITIONS 16

7.5 PERFORMANCE 16

7.6 DATA ANALYSIS 18

8 RESULTS **19**

9 CONCLUSION **21**

10 DEVIATIONS FROM THE ORIGINAL STUDY PLAN **22**

11 ARCHIVES **23**

12 REFERENCES **24**

ANNEXES **25**

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 copper ions from a **Z45 massive** further noted as **"Z45"** 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 **Z45** (massive) - as placed on the market - was tested in a simulated gastric fluid (pH 1.5; 37 °C) at a loading of 2 g/L loading 'surface equivalent' up to 4 hours (1 hour at an agitation speed of 100 revolutions per minute - rpm - and 3 hours without shaking). A "reference particle size" of 1 mm, similar to the transformation/dissolution protocol, is used to calculate the required exposure surface because the test material is usually available as a massive (i.e. materials >1 mm). The bioelution endpoint was based on the dissolved copper concentrations obtained after 1, 2 and 4 hours of extraction.

Simultaneously, a reference item, copper massive was tested.

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 4 hours sampling point of the test, the pH in the blank control vessels, reference item vessels and test vessels was between 1.50 and 1.51.

The blank control vessels showed no concentrations above the accredited reporting limit of 2 µg/L copper.

The following observations could be made in the reference item vessels containing a nominal loading of **2 g/L** (surface equivalent) **copper massive** showed an average dissolved copper concentration of 232 µg/L Cu ($CV_{between\text{-}vessel}$ = 11 %; N = 3) at the 1-hour sampling point. This amount increased to 2025 µg/L Cu ($CV_{between\text{-}vessel}$ = 6 %; N = 3) at the 4-hour endpoint.
Based on the surface loading of the reference item (13.4 cm^2/L) and the average dissolved copper concentration, a copper release per surface of 173 mg/m^2 and 1511 mg/m^2 could be calculated at the 1-hour sampling point and 4 hours endpoint, respectively.

In the test item vessels with a nominal loading of **2 g/L Z45 massive** (surface equivalent), an average dissolved copper concentration of 192 µg/L Cu ($CV_{between\text{-}vessel}$ = 14 %; N = 3) was measured after 1 hour of exposure to the simulated gastric fluid (pH 1.5). This amount increased to 1018 µg/L Cu ($CV_{between\text{-}vessel}$ = 7 %; N = 3) at the 4-hour endpoint.
Based on the surface loading of the reference item (14.33 cm^2/L) and the average dissolved copper concentration, a copper release per surface of 134 mg/m^2 and 710 mg/m^2 could be calculated at the 1-hour sampling point and 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 **Z45** (massive) at a *2 g/L loading* in a simulated gastric fluid.

Study no.: **X02a-146**

2.2 Test Item identification and characterisation

The test item, **Z45**, as received, is representative of the material as it is, or will be, on the market for use. Further preparation (embedding and polishing) of the test item was 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:	**Z45**
Standardised Test item name:	**CuZn36Pb2As**
CEN number:	EN CW602N
Manufacturer:	Wieland Werke AG Graf-Arco-Straße 36 D-89079 Ulm Germany
Manufacturer contact:	Dr. Stefan Priggemeyer [+49-731-944-2794]
Batch:	D22
ECTX no.:	-

Test Item characterisation

Physicochemical method of production:	n.a.[1]
Element(s) of interest:	Copper
Composition concerning the element of interest:	62.176 % copper [7440-50-8] A certificate of analysis of the tested batch is added to this study report (Annex 7).
Physical form:	Solid (massive form)
Colour:	Copper-yellow
Surface area:	134 mm² of circle disk surface
Homogeneous:	Yes

[1] n.a. not available/applicable

Density:	8.38 g/cm³ (20 °C)

Storage conditions

Room temperature, in the dark.

Expiry date

Stable over time.

Safe Handling Precautions

A safety data sheet on **Wieland Z45** is available.

2.3 Reference Item identification and characterisation

As the reference item, massive copper was used:

Reference Item identification

Reference item name:	**Copper**
Manufacturer:	Wieland Werke AG Graf-Arco-Straße 36 D-89079 Ulm Germany
Manufacturer contact:	Dr. Stefan Priggemeyer [+49-731-944-2794]
Batch:	D22
ECTX no.:	-

Test Item characterisation

Physicochemical method of production:	n.a.
Element(s) of interest:	Copper
Composition concerning the element of interest:	99.99 % copper [7440-50-8] A CEN Technical Specification note, CEN/TS 13388 which summarizes the standardized composition is added to this study report (Annex 6).
Physical form:	Solid (massive form)
Colour:	Copper
Surface area:	143 mm² of circle disk surface
Homogeneous:	Yes
Density:	8.96 g/cm³ (20 °C)

Storage conditions

Room temperature, in the dark.

Expiry date

Stable over time.

Safe Handling Precautions

A safety data sheet on **copper** is not available.

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 **25-01-2018**
	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,
- 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 material, a 40 cm long **Z45** rod (Ø13.05 mm) was provided by the Sponsor.

Massives are tested at particle size, representative of normal handling and use (cfr. Transformation/ Dissolution test guidance). A default diameter value of 1 mm of a spherical particle is used in the 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 **Z45** (density 8.38 mg/mm^3), a Ø 1 mm spherical reference particle has a volume of 0.524 mm^3 and a mass of 4.39 mg. Therefore, a 2 g/L loading is containing 456 reference particles corresponding to a total free metal surface of 1433 mm^2/L. The required loading of 2 g/L is achieved by adding an appropriate amount of test medium to the actual exposed free metal surface after embedding (i.e. 134 mm^2).

The preparation of the samples (cutting, embedding and polishing) is 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 is given below.

Cutting the massive metal

Three slices of **Z45** (Ø13.05 mm x 5.0 mm height) were cut from the original rod of the Z45 alloy by means of a drilling and milling machine. The samples were degreased with ethanol before the epoxy embedding step.

Epoxy embedding

One piece of **Z45** is embedded per epoxy resin sample (EpoFix Kit from Struers). The polymerisation process is done as directed by Struers as follows: To prepare the resin fluid an amount of hardener is mixed into the correct amount of resin (25 parts of resin / 3 parts of hardener by weight). The mixture is mixed well for approx. 2 minutes without introducing too many air bubbles. The mixture is left to rest for 2 minutes. (Pot life of this mixture: about 30 minutes). The epoxy is carefully poured over the specimen in an achieved mould. A vacuum system is used to reduce air bubbles. The embedded samples are left at room temperature for hardening during at least 12 hours.

Surface preparation procedure

Each epoxy sample is 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 is important to apply only a very gentle pressure during the grinding. So each sample is ground only for approximately one minute on each grit size paper with very gentle pressure. After wet grinding, the samples are rinsed with ethanol and dried with nitrogen gas to avoid corrosion during the sample preparation.

Polishing is 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 are held very gently against the polishing cloth with very little applied pressure. Plenty of ethanol is used as a lubricant to obtain a well-defined surface. Each polishing step with diamond paste takes about 5 to 10 minutes, depending on the removal of scratches. After polishing the samples are 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 are 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 is used as the last cleaning medium. After the ultrasonic cleaning, all samples are dried quickly again with cold nitrogen gas.

The polished surface is checked by means of a microscope. The metal surfaces shall be free from scratches and spots. Otherwise, re-polishing is necessary. Finally, the surfaces were free from scratches

Ageing time (after polishing)

The samples were stored for 2 days before testing.

7.3 Preparation of the Reference Item

The test material, an 18 cm long **copper** rod (Ø13.50 mm) was provided by the Sponsor.

In the case of **copper** (density 8.96 mg/mm³), a Ø 1 mm spherical reference particle has a volume of 0.524 mm³ and a mass of 4.69 mg. Therefore, a 2 g/L loading is containing 426 reference particles corresponding with a total free metal surface of 1340 mm²/L The required loading of 2 g/L is achieved by adding an appropriate amount of test medium to the actual exposed free metal surface after embedding (i.e. 143 mm²).

Three slices of **copper** (Ø13.50 mm x 5.0 mm height) were cut from the original rod of copper by means of a drilling and milling machine. The samples were degreased with ethanol before the epoxy embedding step.

The same procedure for cutting, epoxy embedding, surface preparation and ageing was applied as noted in the previous paragraph for Z45.

7.4 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).

7.5 Performance

7.5.1 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 (surface equivalent).

- In the blank control vessel, 100 mL gastric test medium (37 °C ± 1 °C) was added, only.
- In each reference item vessel (3) containing 1 embedded copper sample, 107 mL gastric test medium (37 °C ± 1 °C) was added to obtain a loading of 2 g/L (surface equivalent).
- In each test item vessel (3) containing 1 embedded Z45 sample, 94 mL gastric test medium (37 °C ± 1 °C) was added to obtain a 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 in the dark into a thermostatic orbital shaker (37 °C ± 1 °C) at an agitation rate of 100 rpm for one hour. After that, the flasks were settled at 37 °C ± 1 °C for another three hours. Samples are taken after 1, 2 and 4 hours of extraction.

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

Number of replica test vessels	Target loading (g/L)	Pieces of alloy	Test item surface (mm^2)	Test medium added
Blank repl.1	0	**a**	-	100 mL
Ref. item repl.1	2g/L **§**	1 (Ø13.50 mm)	143 mm^2	107 mL
Ref. item repl.2	2g/L **§**	1 (Ø13.50 mm)	143 mm^2	107 mL
Ref. item repl.3	2g/L **§**	1 (Ø13.50 mm)	143 mm^2	107 mL
Test item repl.1	2g/L **§**	1 (Ø13.05 mm)	134 mm^2	94 mL
Test item repl.2	2g/L **§**	1 (Ø13.05 mm)	134 mm^2	94 mL
Test item repl.3	2g/L **§**	1 (Ø13.05 mm)	134 mm^2	94 mL

a blank resin disc
§ surface equivalent loading

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

7.5.2 Sampling during the test

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

After the appropriate extraction time (1, 2 and 4 hours), each vessel was swirled to homogenise the solution before sampling, to avoid a concentration gradient.

Sampling was performed for each test vessel as indicated in the sampling scheme below. In each test vessel, a 10-mL sample was taken with a 10 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 0.10 mL concentrated HNO_3 per 10 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-146-**1** where:

X02a-146 is the study number,

1 is the sample number.

7.5.3 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 (blank) 0 g/L	Reference item + medium Loading 2 g/L	Test item + medium Loading 2 g/L	
1	1*1	1*3	1*3	7
2	1*1	1*3	1*3	7
4	1*1	1*3	1*3	7
Total number of samples:				21

A detailed list is given in Annex 4.

7.5.4 Observations and measurement of temperature and pH

At the end of the test (4h), temperature and pH were measured in the remaining blank control, reference and test solutions after sampling.

The details are presented in Annex 1 and 2.

7.5.5 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*).

7.5.6 Analyses of the samples by the Test Site

The determination of dissolved copper 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 2 µg/L for copper.

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.

7.5.7 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 5).

7.6 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 mg 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 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 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 and 2).

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 1.50 and 1.51. (Annex 1 and 2)

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 5.

The blank measurements of copper were below or close to the corresponding accredited reporting limits of 2 µg/L Cu.

The following observations could be made in the reference item vessels with a nominal loading of **2 g/L copper massive** (surface equivalent):

- At the 1-hour sampling point: An average dissolved copper concentration of 232 ± 26 µg/L Cu (or 116 ± 13 µg/g test item) was measured after 1 hour of exposure to the simulated gastric fluid (pH 1.5) with a between-vessel variation of 11 %.

  Based on the surface loading of the reference item (13.4 cm²/L) and the average dissolved copper concentration, a copper release per surface of 173 mg/m² could be calculated.

  Based on the copper content of the reference item (99.99 %) and the average dissolved copper concentration, a copper release of 0.012 % could be calculated.

- At the 2-hour sampling point: An average dissolved copper concentration of 569 ± 52 µg/L Cu (or 285 ± 26 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 9 %, which met the <20 % quality criterion at the 2 hours endpoint.

  Based on the surface loading of the reference item (13.4 cm²/L) and the average dissolved copper concentration, a copper release per surface of 425 mg/m² could be calculated.

  Based on the copper content of the reference item (99.99 %) and the average dissolved copper concentration, a copper release of 0.028 % could be calculated.

- At the 4-hour sampling point: An average dissolved copper concentration of 2025 ± 117 µg/L Cu (or 1012 ± 58 µg/g test item) was measured after 4 hours of exposure to the simulated gastric fluid (pH 1.5) with a between-vessel variation of 6 %.

  Based on the surface loading of the reference item (13.4 cm²/L) and the average dissolved copper concentration, a copper release per surface of 1511 mg/m² could be calculated.

  Based on the copper content of the reference item (99.99 %) and the average dissolved copper concentration, a copper release of 0.10 % could be calculated.

The following observations could be made in the test item vessels with a nominal loading of **2 g/L Z45 massive** (surface equivalent):

- At the 1-hour sampling point: An average dissolved copper concentration of 192 ± 28 µg/L Cu (or 96 ± 14 µg/g test item) was measured after 1 hour of exposure to the simulated gastric fluid (pH 1.5) with a between-vessel variation of 14 %.

  Based on the surface loading of the test item (14.33 cm²/L) and the average dissolved copper concentration, a copper release per surface of 134 mg/m² could be calculated.

Based on the copper content of the test item (62.176 % Cu) and the average dissolved copper concentration, a copper release of 0.015 % could be calculated.

- At the 2-hour sampling point: An average dissolved copper concentration of 406 ± 36 µg/L Cu (or 203 ± 18 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 9 %, which met the <20 % quality criterion at the 2 hours endpoint.

  Based on the surface loading of the test item (14.33 cm²/L) and the average dissolved copper concentration, a copper release per surface of 283 mg/m² could be calculated.

  Based on the copper content of the test item (62.176 % Cu) and the average dissolved copper concentration, a copper release of 0.033 % could be calculated.

- At the 4-hour sampling point: An average dissolved copper concentration of 1018 ± 66 µg/L Cu (or 509 ± 33 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 7 %.

  Based on the surface loading of the test item (14.33 cm²/L) and the average dissolved copper concentration, a copper release per surface of 710 mg/m² could be calculated.

  Based on the copper content of the test item (62.176 % Cu) and the average dissolved copper concentration, a copper release of 0.082 % could be calculated.

All raw data can be found in Annex 1-7 of this study report.

9 Conclusion

During this study on the bio-elution of **Z45 massive** at loading of 2 g/L, the following could be observed during 4 hours of exposure to simulated gastric fluid (pH 1.5): An average value of 192 µg/L Cu ($CV_{between-vessel}$ = 14 %; N = 3) was found after 1 hour of extraction increasing to 1018 µg/L Cu ($CV_{between-vessel}$ = 7 %; N = 3) after 4 hours. The measured concentrations corresponded with a copper release related to the total copper content of 0.015 % (or 134 mg/m²) and 0.082 % (or 710 mg/m²) at the 1-hour sampling point and 4-hours endpoint, respectively.

The reference vessel containing 2 g/L copper massive showed an average dissolved copper concentration of 232 µg/L Cu ($CV_{between-vessel}$ = 11 %; N = 3) at the 1-hour sampling point increasing to 2025 µg/L Cu ($CV_{between-vessel}$ = 6 %; N = 3) at the 4-hour endpoint. The measured concentrations corresponded with a copper release related to the total copper content of 0.012 % (or 173 mg/m²) and 0.10 % (or 1511 mg/m²) at the 1-hour sampling point and 4-hours endpoint, respectively.

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

10 Deviations from the original Study Plan

Section *7.5.2 Sampling during the test* of the study plan incorrectly stated that samples are taken in duplicate *("…a 10-mL sample will be taken with a 10 mL syringe from the test vessel…"*. However, only one sample was taken from each test vessel at each sampling time as otherwise the available test volume was too strongly influenced. The sampling was correctly noted in section *7.5.3 Sampling scheme*.

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-146**. The Study Plan, the lab notebook, the raw data, the correspondence and final report has been stored in a locked filing cabinet. One piece of embedded reference and test item was archived in a suitable sealed container and stored under the storage conditions mentioned (02-02-2018).

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 and dissolved element concentrations during the 4 hours exposure period of the reference item (2 g/L loading surface equivalent **copper metal**) exposed to the simulated gastric fluid at pH 1.5).

2 g/L reference item	1 hour	2 hours	4 hours		
pH 1.5 (1 h at 100 rpm)	µg/L Cu	µg/L Cu	µg/L Cu	T (°C)	pH
Accred. Reporting Limit	2	2	2		
test medium at 0 h				37.1	1.50
blank	<2	<2	<2	37.0	1.50
replica1	203	518	1915	36.9	1.50
replica2	237	567	2012	36.9	1.51
replica3	255	622	2147	36.8	1.50
x_{avg}	232	569	2025		
$s_{between}$	26	52	117		
$CV_{between}$ (%)	11	9	6		

x_{avg} average of 3 test vessels (1 sample per test vessel) (N=3)
$s_{between}$ between vessel Standard Deviation (N=3)
$CV_{between}$ between vessel Coefficient of Variation (N=3)

Annex 2:

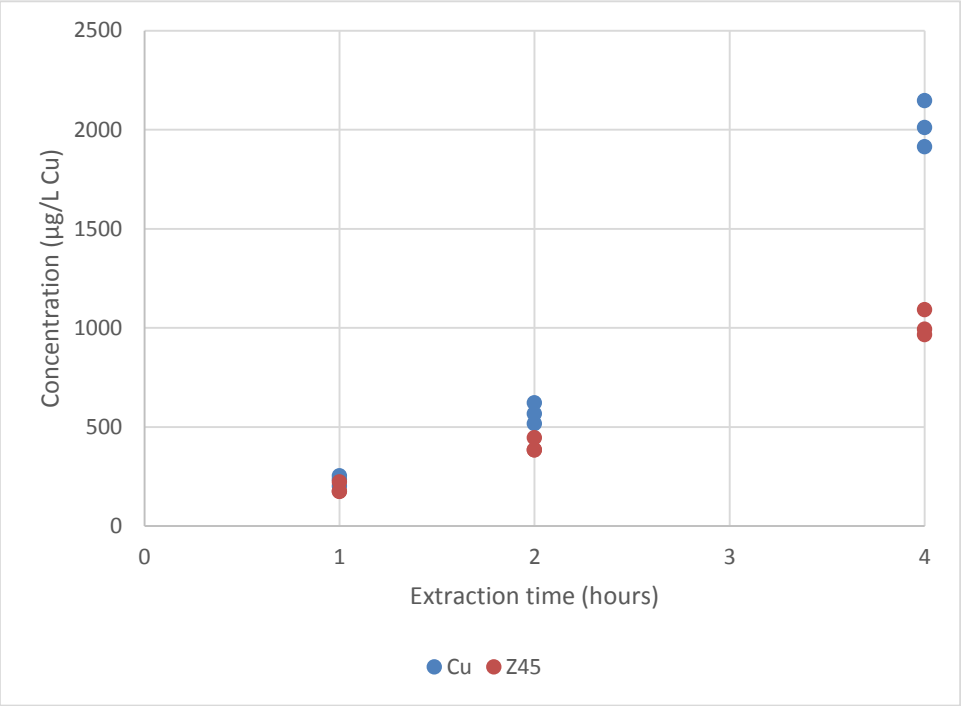
Data of measured temperature, pH and dissolved element concentrations during the 4 hours extraction period of **2 g/L Z45** (surface equivalent) in the simulated gastric fluid at pH 1.5.

2 g/L test item	1 hour	2 hours	4 hours		
pH 1.5 (1 h at 100 rpm)	µg/L Cu	µg/L Cu	µg/L Cu	T (°C)	pH
Accred. Reporting Limit	2	2	2		
test medium at 0 h				37.1	1.50
blank	<2	<2	<2	37.0	1.50
replica1	177	385	966	37.0	1.50
replica2	175	385	995	36.9	1.51
replica3	224	447	1092	37.0	1.51
x_{avg}	192	406	1018		
$s_{between}$	28	36	66		
$CV_{between}$ (%)	14	9	7		

x_{avg} average of 3 test vessels (1 sample per test vessel) (N=3)
$s_{between}$ between vessel Standard Deviation (N=3)
$CV_{between}$ between vessel Coefficient of Variation (N=3)

Figure 1:

Measured dissolved copper concentrations during the 4 hours test of the reference (**copper** massive) and test item (**Z45** massive) exposed to the simulated gastric fluid at pH 1.5.



Annex 3a:

Summarised results of the average dissolved copper concentrations, copper release per g test item (surface equivalent) and the percentage of available element eluted, after 1, 2 and 4 hours of extraction of **2 g/L Copper massive** (surface equivalent) in the simulated gastric fluid at pH 1.5:

	Cu massive: 4-h gastric at pH 1.5 at **2 g/L** surface equivalent loading			
	Analyte		$x_{avg} \pm s_{between}$	$CV_{between}$
1 hour	**copper (Cu)**	dissolution	232 ± 26 µg/L	11 %
		absolute Cu release	116 ± 13 µg/g	11 %
		% of total available Cu eluted	0.012 %	
		Cu release/surface	173 mg/m²	
2 hours	**copper (Cu)**	dissolution	569 ± 52 µg/L	9 %
		absolute Cu release	285 ± 26 µg/g	9 %
		% of total available Cu eluted	0.028 %	
		Cu release/surface	425 mg/m²	
4 hours	**copper (Cu)**	dissolution	2025 ± 117 µg/L	6 %
		absolute Cu release	1012 ± 58 µg/g	6 %
		% of total available Cu eluted	0.10 %	
		Cu release/surface	1511 mg/m²	

x_{avg} = average of 3 test vessels (1 sample per test vessel) (N=3).
$s_{between}$ = between-vessel standard deviation (N=3).
$CV_{between}$ = between-vessel Coefficient of Variation (%).

Annex 3b:

Summarised results of the average dissolved copper concentrations, copper release per g test item (surface equivalent) and the percentage of available element eluted, after 1, 2 and 4 hours of extraction of **2 g/L Z45 massive** (surface equivalent) in the simulated gastric fluid at pH 1.5:

	Z45 massive: 4-h gastric at pH 1.5 at **2 g/L** surface equivalent loading			
	Analyte		$x_{avg} \pm s_{between}$	$CV_{between}$
1 hour	**copper (Cu)**	dissolution	192 ± 28 µg/L	14 %
		absolute Cu release	96 ± 14 µg/g	14 %
		% of total available Cu eluted	0.015 %	
		Cu release/surface	134 mg/m²	
2 hours	**copper (Cu)**	dissolution	406 ± 36 µg/L	9 %
		absolute Cu release	203 ± 18 µg/g	9 %
		% of total available Cu eluted	0.033 %	
		Cu release/surface	283 mg/m²	
4 hours	**copper (Cu)**	dissolution	1018 ± 66 µg/L	7 %
		absolute Cu release	509 ± 33 µg/g	7 %
		% of total available Cu eluted	0.082 %	
		Cu release/surface	710 mg/m²	

x_{avg} = average of 3 test vessels (1 sample per test vessel) (N=3).
$s_{between}$ = between-vessel standard deviation (N=3).
$CV_{between}$ = between-vessel Coefficient of Variation (%).

Annex 4:

A detailed overview of the samples.

Sample no.	Sampling time (h)	pH	Loading (g/L) §	Sample treatment	Comment
X02a-146-1	1	1.5	0	filtration (0.2 µm)	Blank replica1 spl1
X02a-146-4	1	1.5	2	filtration (0.2 µm)	Cu replica1 spl1
X02a-146-5	1	1.5	2	filtration (0.2 µm)	Cu replica2 spl1
X02a-146-6	1	1.5	2	filtration (0.2 µm)	Cu replica3 spl1
X02a-146-13	1	1.5	2	filtration (0.2 µm)	Z45 replica1 spl1
X02a-146-14	1	1.5	2	filtration (0.2 µm)	Z45 replica2 spl1
X02a-146-15	1	1.5	2	filtration (0.2 µm)	Z45 replica3 spl1
X02a-146-2	2	1.5	0	filtration (0.2 µm)	Blank replica1 spl1
X02a-146-7	2	1.5	2	filtration (0.2 µm)	Cu replica1 spl1
X02a-146-8	2	1.5	2	filtration (0.2 µm)	Cu replica2 spl1
X02a-146-9	2	1.5	2	filtration (0.2 µm)	Cu replica3 spl1
X02a-146-16	2	1.5	2	filtration (0.2 µm)	Z45 replica1 spl1
X02a-146-17	2	1.5	2	filtration (0.2 µm)	Z45 replica2 spl1
X02a-146-18	2	1.5	2	filtration (0.2 µm)	Z45 replica3 spl1
X02a-146-3	4	1.5	0	filtration (0.2 µm)	Blank replica1 spl1
X02a-146-10	4	1.5	2	filtration (0.2 µm)	Cu replica1 spl1
X02a-146-11	4	1.5	2	filtration (0.2 µm)	Cu replica2 spl1
X02a-146-12	4	1.5	2	filtration (0.2 µm)	Cu replica3 spl1
X02a-146-19	4	1.5	2	filtration (0.2 µm)	Z45 replica1 spl1
X02a-146-20	4	1.5	2	filtration (0.2 µm)	Z45 replica2 spl1
X02a-146-21	4	1.5	2	filtration (0.2 µm)	Z45 replica3 spl1

§ surface equivalent loading

Annex 5:

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

- Analytical Report 18R00782.X02a-146 (8 pages)

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Analytical Report 18R00782.X02a-146

(02/02/2018)

Project: **17R0152/45**

Determination of copper in Simulated Gastric Fluid: ECTX Study X02a-146

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 copper (**Cu**) 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 **Cu** in ECTX samples of study X02a-146 as determined by the analytical laboratory of WLN. The sampling was performed by ECTX.

2. ANALYTICAL METHODOLOGY

The analyses of copper 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 element copper (**Cu**) 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: **^{63}Cu**

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

- Reporting limit: **2 µg/L** for Cu (= 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 Cu in sample solution X02a-146-1 to X02a-146-21 (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 Cu in sample solution X02a-146-1 to X02a-146-21 (in µg/L).

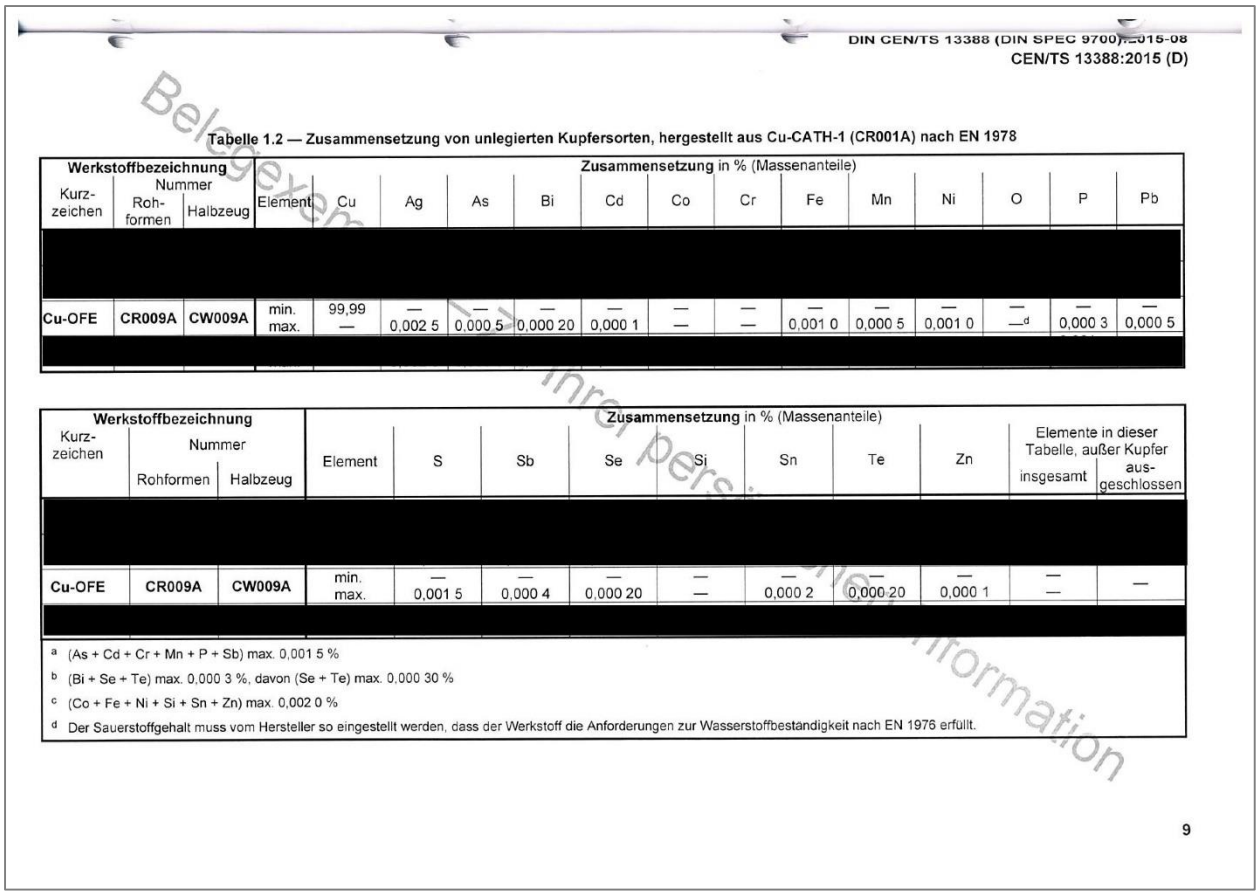
Parameter	Description	Standard Procedure	Accreditation
Cu	Copper	WLN-CM.W.11.1	RD

Sample Number	Description (ECTX)	Sampling Date	Date of Analysis	Cu (µg/L)
1804731	X02a-146-1	24-01-2018	01-02-2018	0.9
1804732	X02a-146-2	24-01-2018	01-02-2018	1.2
1804733	X02a-146-3	24-01-2018	01-02-2018	1.3
1804734	X02a-146-4	24-01-2018	01-02-2018	203.1
1804735	X02a-146-5	24-01-2018	01-02-2018	237.2
1804736	X02a-146-6	24-01-2018	01-02-2018	254.9
1804737	X02a-146-7	24-01-2018	01-02-2018	517.9
1804738	X02a-146-8	24-01-2018	01-02-2018	567.2
1804739	X02a-146-9	24-01-2018	01-02-2018	622.2
1804740	X02a-146-10	24-01-2018	01-02-2018	1914.6
1804741	X02a-146-11	24-01-2018	01-02-2018	2012.4
1804742	X02a-146-12	24-01-2018	01-02-2018	2147.0
1804743	X02a-146-13	24-01-2018	01-02-2018	176.7
1804744	X02a-146-14	24-01-2018	01-02-2018	175.0
1804745	X02a-146-15	24-01-2018	01-02-2018	223.9
1804746	X02a-146-16	24-01-2018	01-02-2018	384.8
1804747	X02a-146-17	24-01-2018	01-02-2018	385.4
1804748	X02a-146-18	24-01-2018	01-02-2018	446.8
1804749	X02a-146-19	24-01-2018	01-02-2018	966.0
1804750	X02a-146-20	24-01-2018	01-02-2018	994.6
1804751	X02a-146-21	24-01-2018	01-02-2018	1092.4

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

Annex 6:

CEN Technical Specification note, CEN/TS 13388 which summarizes the standardized composition of the reference item, **Copper massive** (1 page).



Belegexemplar – nur zum persönlichen Gebrauch – Information

Tabelle 1.2 — Zusammensetzung von unlegierten Kupfersorten, hergestellt aus Cu-CATH-1 (CR001A) nach EN 1978

Werkstoffbezeichnung Kurzzeichen	Nummer Rohformen	Nummer Halbzeug	Element	Zusammensetzung in % (Massenanteile) Cu	Ag	As	Bi	Cd	Co	Cr	Fe	Mn	Ni	O	P	Pb
[redacted]																
Cu-OFE	CR009A	CW009A	min.	99,99	—	—	—	—	—	—	—	—	—	—	—	—
			max.	—	0,002 5	0,000 5	0,000 20	0,000 1	—	—	0,001 0	0,000 5	0,001 0	—[d]	0,000 3	0,000 5
[redacted]																

Werkstoffbezeichnung Kurzzeichen	Nummer Rohformen	Nummer Halbzeug	Element	Zusammensetzung in % (Massenanteile) S	Sb	Se	Si	Sn	Te	Zn	Elemente in dieser Tabelle, außer Kupfer insgesamt	Elemente in dieser Tabelle, außer Kupfer ausgeschlossen
[redacted]												
Cu-OFE	CR009A	CW009A	min.	—	—	—	—	—	—	—	—	—
			max.	0,001 5	0,000 4	0,000 20	—	0,000 2	0,000 20	0,000 1	—	
[redacted]												

[a] (As + Cd + Cr + Mn + P + Sb) max. 0,001 5 %

[b] (Bi + Se + Te) max. 0,000 3 %, davon (Se + Te) max. 0,000 30 %

[c] (Co + Fe + Ni + Si + Sn + Zn) max. 0,002 0 %

[d] Der Sauerstoffgehalt muss vom Hersteller so eingestellt werden, dass der Werkstoff die Anforderungen zur Wasserstoffbeständigkeit nach EN 1976 erfüllt.

Annex 7:

Certificate of Analysis of the **Z45 massive** (Wieland -Werke AG, 1 page).

Untersuchungsbericht **890000959717**

Wieland

Wieland-Werke AG
Unternehmensbereich Metall
Zentrallabor und Entwicklung
Abteilung Prüflaboratorien

LIMS Prüflaboratorien

Z45 brass sample for bio-elution	Anzahl Proben: 1 Probenart: Stange

Kostenträger:	4450002773
Kunde:	
Ersteller:	Vogt, Hermann Manfred - MFLB/Vo - 3426
Auftraggeber:	Ebner, Michael - MFL - 3706
Empfänger:	Ebner, Michael - MFL - 3706

Verantwortlich: Dr. Michael Ebner
Tel.: 0731 / 944-3706
Fax.: 0731 / 944-4617
email: michael.ebner@wieland.de

Eingangsdatum: 16.08.2016 / 15:24
Abschlußdatum: 17.08.2016 / 17:37
Revision 0: 17.08.2016 / 17:37

Probenbeschreibung / Bemerkung
3 x 40cm Stange mit Durchmesser 13mm

Prüfergebnisse:

	Probe:		Leg.:	Ort	Ergebnis	Einheit	Toleranz / Prüfbemerkung
	D22		Z45				
	LR-Nr.:	**817795**					
01. Probenvorbereitung in MFL-B							
E	PBLAB				durchgeführt		

02. Metall-Analyse

AG		0,00200 %	AL	<	0,00050 %	AS		0,048 %	B	<	0,00050 %
BE	<	0,00050 %	BI		0,00063 %	CD	<	0,00050 %	CO	<	0,00050 %
CR	<	0,00050 %	CU		62,176 %	FE		0,086 %	MG	<	0,00050 %
MN		0,00061 %	NI		0,01583 %	P	<	0,00050 %	PB		1,977 %
S		0,00166 %	SB		0,00100 %	SE	<	0,00050 %	SI		0,00099 %
SN		0,059 %	TE	<	0,00050 %	TI	<	0,00050 %	ZN		35,631 %
ZR	<	0,00050 %									

Geprüft als: Z45

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