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Prometia Scientific Conference

SILREF Project – Development of New Certified Reference Materials for Silicon Industry

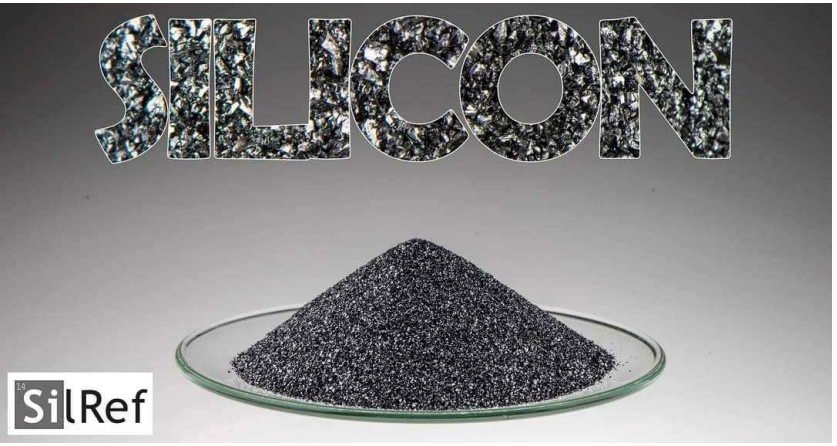
Christa Nimbona

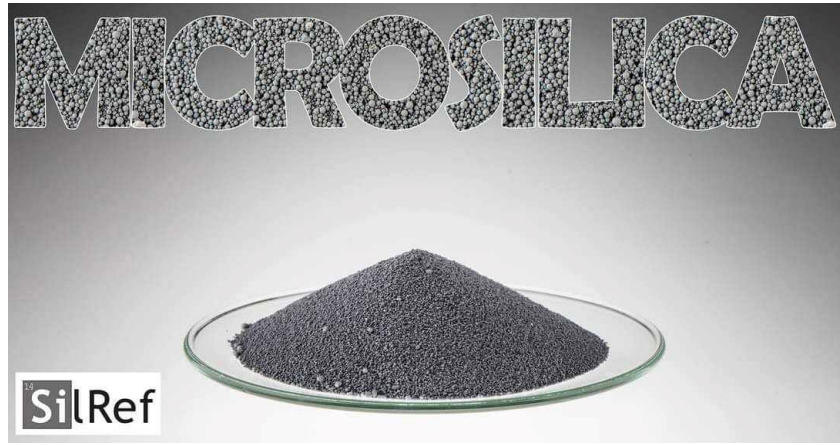
Naples, 26/11/2025

Background & Collaboration

- Contact between Elkem and IMN were established some years ago through the Prometia network
- Production and commercialization of Certified Reference Materials (CRM) for Elkems materials was identified as an opportunity for a joint project with public funding
- An application was submitted through the POLNOR program (bi-lateral progam Norway/Poland)
  - Application was approved, and project started September 2020
- IMN (Institute for non-ferrous metals) is an institute within the Lukasiewicz research network in Poland Services – Łukasiewicz – ORGMASZ
- IMN produce and sell CRM for a wide variety of CRM. They are accredited according to ISO 17034 for CRM production, safeguarding a high technical standard of the finalized CRM products.
- Sales prices of CRM varies depending on the material and market.

- Project title: Development of reference materials for silicon industry - improvement of quality assurance
- Duration: 3 years (from September 2020)
- Scope of work: 8 certified reference materials





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CRM-s for silicon materials – market situation

MARKET

CERTIFIED REFERENCE MATERIALS

ISO 17034 17025

- No matching CRMs
- The need to improve fast analytical procedures: XRF

product innovation

Microsilica

Silicon

Silicon alloys

Fast analytical methods

- New ISO standards for CRM producers and accredited laboratories

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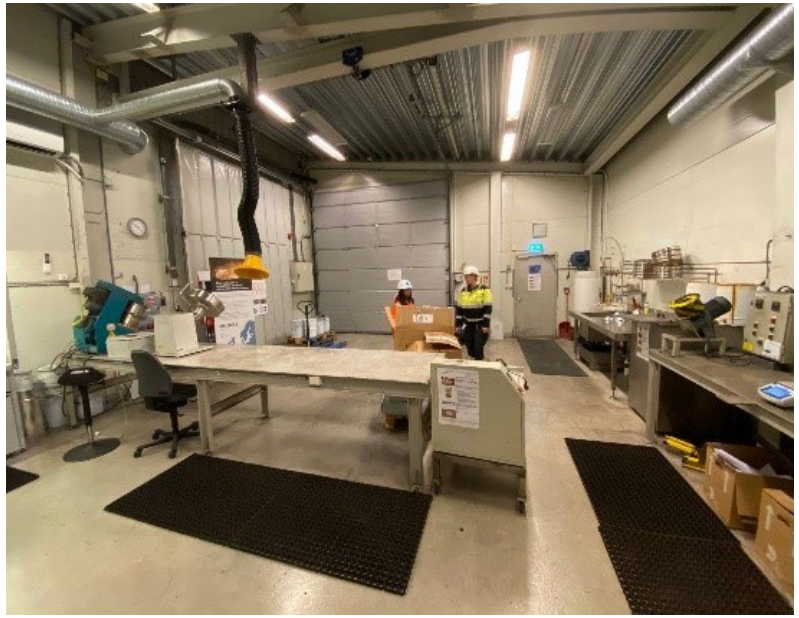
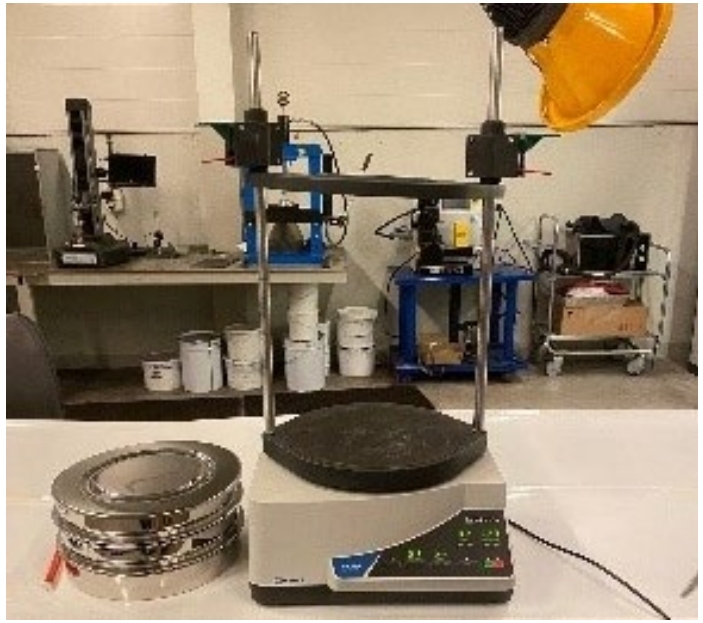
WP1 Acquisition of materials





Material preparation station in Norway

Material (silica dust and silicon) ready for shipment

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WP1 Acquisition of materials

Symbol	Mg, [%]
Si-FSM-1	3.44
Si-FSM-2	7.5
Si-FSM-3	2.35

1st attempt at mixing:

Si-FSM-2 i Si-FSM-3 in proportion 1:1

Time of mixing: 1h

2nd attempt at mixing:

Si-FSM-2 i Si-FSM-3 in proportion 2:1 (22 kg Si-FSM-2, 11 kg Si-FSM-3)

Time of mixing: 11h (divided into three stages: 2 x 4h i 1 x 3h)
3 samples were taken from three extreme locations of the mixer and chemical analyses were carried out

The tendency towards segregation

Time [h]	Si [%]
2	39.95
4	39.98
6	40.10

$$|x_{CRM} - x_{mon}| \leq U_{mon}$$

	[%]	$\lvert X_{CRM} - X_{mon} \rvert$	U_{mon}	Tendency towards segregation
Si	40.15 (certified value)	-	-	-
	39.95	0.20	0.20	NIE
	39.98	0.17	0.20	NIE
	40.10	0.05	0.20	NIE

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WP2 Stability tests

Short-term (transport) stability
u_{trans}

T = 40°C
T = 5°C
T = -19°C

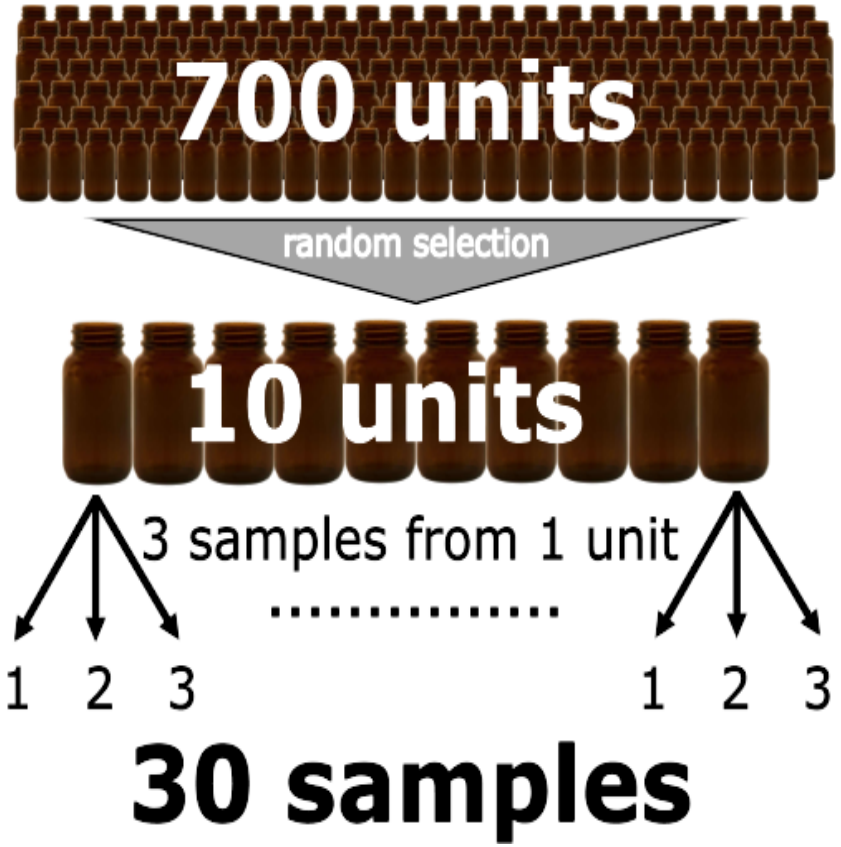
Samples for testing taken every two days

Long-term stability
u_{stab}

Samples for testing taken in January and June from the moment the material is produced until the end of the project and then once a year

	Fe	Al	Ca	Ti	Cr	Ni	V	Mn	Cu	P	B	C
$u_{trans(rel)}$	0.10	0.42	0.27	0.45	0.71	1.4	1.2	0.31	1.6	2.2	0.45	1.1
$u_{stab(rel)}$	0.05	0.10	0.08	0.13	0.25	0.64	0.39	0.14	0.28	0.19	0.21	0.06

WP3 Homogeneity tests



$$N_{min} = \max\left(10, \sqrt[3]{N_{prod}}\right)$$

WP3 Homogeneity tests

Determined relative values of homogeneity uncertainty $u_{h(rel.)}$ for certified elements in Si-FSM-4 material

	Si	Fe	Mg	Ca	Al	Ce	La	Ba	Ti	Cr	Mn	P
$u_{h(rel)}$	0.07	0.15	1.6	2.4	3.0	3.2	3.2	2.5	1.7	1.7	1.7	11.3

Components $C_{\%}$ >1% – allowable value of relative uncertainty of homogeneity <5%

Components $C_{\%}$ <1% – allowable value of relative uncertainty of homogeneity <15%

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WP4 Development of analytical methods and validation analyses

Material	Analytical technique				
	ICP-OES		WDXRF		Elemental analysis with infrared detection (IR)
	Open system digestion (HNO_3 + HF)	Microwave digestion	Preparation for the form of borate beads	Preparation for the form of pellets	-
Silicon	Stability, homogeneity and chemical composition tests	-	-	-	Stability, homogeneity and chemical composition tests
FSM		-	Chemical composition tests	Si, Fe - Stability, homogeneity and chemical composition tests	
Silica fume	-	Chemical composition tests		Stability, homogeneity and chemical composition tests	Stability, homogeneity and chemical composition tests

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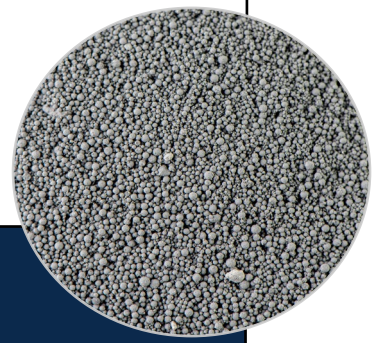
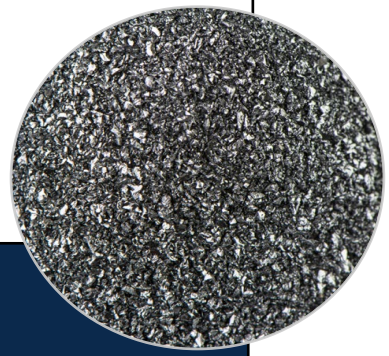
WP5 Certification and verification of CRMs in industrial conditions

Si
Fe
Al
Ca
Na
K
Mg
Cl
P
S
Zn
C
LOI



Silica fume

Fe
Al
Ca
Ti
Cr
Ni
V
Mn
Cu
P
B
C



Silicon

Si
Fe
Mg
Al
Ca
Ce
La
Ba
Ti
Cr
Mn
P

FSM

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WP5 Certification and verification of CRMs in industrial conditions

Participating laboratories:

- **Łukasiewicz Research Network – Institute of Non-Ferrous Metals, Centre of Analytical Chemistry,** Laboratory of Emission Spectrometry and Chromatography, Gliwice, Poland
- **Łukasiewicz Research Network – Institute of Non-Ferrous Metals, Centre of Analytical Chemistry,** Laboratory of Atomic Spectrometry, Gliwice, Poland
- **Łukasiewicz Research Network – Institute of Non-Ferrous Metals, Centre of Analytical Chemistry,** Laboratory of Classical Analysis, Gliwice, Poland
- **Łukasiewicz Research Network – Institute of Ceramics and Building Materials,** Gliwice, Poland
- **Elkem ASA,** Kristiansand, Norway
- **Central Mining Institute – National Research Institute,** Katowice, Poland
- **Centre for Energy Research,** Hungary
- **Eurofins EAG Materials Science,** France
- **REC Solar Holdings AS, Kristiansand,** Norway
- **Elkem Metal,** Canada

Analytical methods used:

- Inductively coupled plasma optical emission spectrometry (ICP-OES)
- Elemental analysis with infrared (IR) detection
- Atomic absorption spectrometry (AAS)
- Prompt-gamma neutron activation analysis (PGAA)
- Neutron activation analysis (NAA)
- X-ray fluorescence spectrometry (XRF)
- Gravimetric method
- Volumetric analysis
- Argentometric titration with potentiometric detection

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Coefficient of variation (for results obtained within 1 laboratory): < 15% - accepted results, > 15% - rejected results

Dixon's Q test - performed on results obtained within one laboratory and then on the means obtained from individual laboratories to show that none of the results differ statistically significantly from the others, or to reject outliers

Graphical evaluation in the form of a chart with the median for all results plotted in a straight line, the range separated by the values of twice the standard deviation for all results from the mean for all results, and the average results obtained in the laboratories along with their standard deviations as error bars

WP5 Certification and verification of CRMs in industrial conditions

- average of results for each of the analyzed elements for the set of results from each laboratory separately
- standard deviation for each of the analyzed elements for the set of results from each laboratory separately
- average of results for each of the analyzed elements from the averages calculated for the results from the individual laboratories
- standard deviation for each of the analyzed elements for all results

$$u_{char} = \frac{s_{char}}{\sqrt{n}}$$

WP5 Certification and verification of CRMs in industrial conditions

- PN-EN ISO 17034:2017
- PN-EN ISO 17025
- ISO Guide 35:2017
- ISO Guide 31:2017

Audit March 2024

Certified Reference Material
Silica Fume

Si-SF-3

Łukasiewicz
IMN

Lot 1
Unit no. 1
70 g

Expire date: 2054
Produced in 2023 in accordance with ISO 17034

Łukasiewicz
Instytut Metali Nieżelaznych

PCA
Polskie Centrum Akredytacji
RM 006

CERTIFICATE
OF REFERENCE MATERIAL

Si-FSM-4
Magnesium Ferrosilicon

The assigned certified values[1] and uncertainties[2]

	Si	Fe	Mg	Al	Ca	Ce	La	Ba	Ti	Cr	Mn	P
	%	%	%	%	%	%	%	mg/kg	%	%	%	%
Si-FSM-4	**44.94**	**44.13**	**5.65**	**0.649**	**2.00**	**0.401**	**0.226**	**49.4**	**0.0512**	**0.0442**	**0.375**	**0.0172**
	±0.99	±0.54	±0.22	±0.041	±0.12	±0.027	±0.017	±3.9	±0.0028	±0.0024	±0.017	±0.0039

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination.
[2] The certified uncertainty is the expanded uncertainty with a coverage factor k=2, corresponding to a level of confidence of about 95 %.

The certified reference material was developed within the SILREF project funded by Norway and state budget and realized by consortium of:
Łukasiewicz Research Network – Institute of Non-Ferrous Metals,
Elkem ASA

Signature

SIEĆ BADAWCZA ŁUKASIEWICZ-
INSTYTUT METALI NIEŻELAZNYCH
DYREKTOR
dr inż. Barbara Juszczyk, MBA

Version 1 from 1st of June 2024

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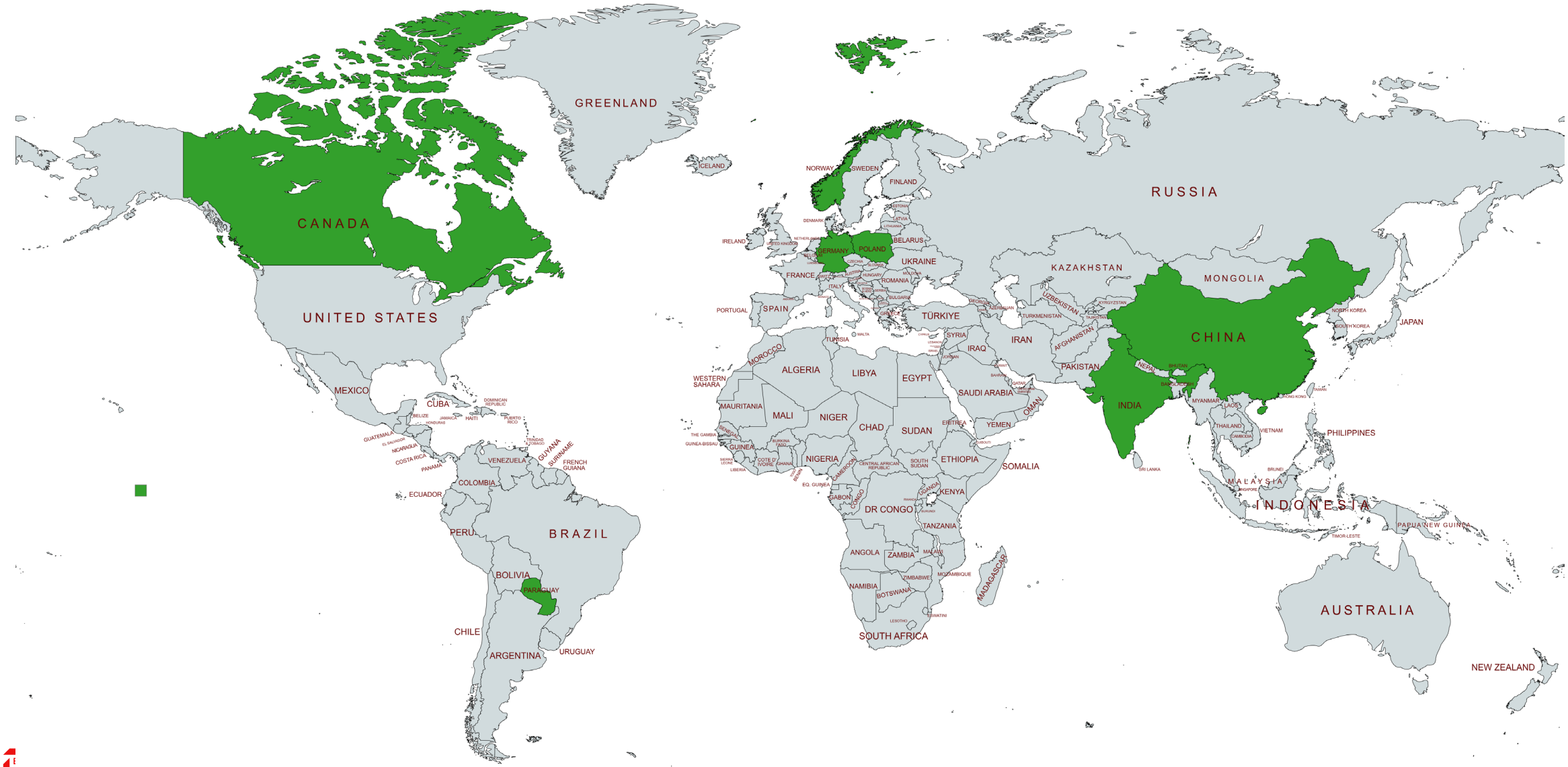

WP5 Certification and verification of CRMs in industrial conditions

SUMMARY

- As part of the SILREF project, nine new CRMs were developed and manufactured for three types of silicon materials: silicon, silica fume and magnesium ferrosilicon.
- This process was carried out in accordance with the requirements of the ISO 17034 standard, which enabled the inclusion of new CRMs in the scope of accreditation of the Polish Centre of Accreditation with number RM006.
- As a part of the work, homogeneity confirmation, short-term and long-term stability tests, as well as the process of characterizing materials on CRM were carried out.
- The results obtained at each stage were statistically evaluated, and the standard uncertainties of each stage were determined. Finally, in silicon CRMs twelve elements: Fe, Al, Ca, Ti, Cr, Ni, V, Mn, Cu, P, B, C; in silica fume thirteen elements: Si, Fe, Al, Ca, Na, K, Mg, Cl, P, S, Zn, C, LOI; and in FSM twelve elements: Si, Fe, Mg, Al, Ca, Ce, La, Ba, Ti, Cr, Mn, P were certified.
- Based on the accepted results, certified values and the accompanying expanded uncertainty values were determined.
- Based on the information collected, a certificate and label were set for each new material (for details see: www.reference-materials.com).
- The developed CRMs were implemented in industrial laboratories

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