Characterisation Report of SCC-05-NP

Processing and characterisation of a microanalytical reference material including its way from precipitate to Nano-Pellets

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Abstract

The microanalytical reference material (MRM) SCC-05-NP is designed for use by laboratories undertaking the determination of major and trace element mass fractions in calcium carbonates ($CaCO_3$) and equivalent matrices with LA-ICP-MS (Laser Ablation Inductively Coupled Plasma Mass Spectrometry). The powder was precipitated from elementally doped solutions of calcium chloride ($CaCl_2$) and sodium carbonate (Na_2CO_3), washed with deionised water (18,2 MΩ) and subjected to a material-specific milling protocol, freeze-dried, homogenised, and split into batches, resulting in the material SCC-05-NP. All batches are sufficient for approximately 1200 units, which should make it available for the foreseeable future. Several Nano-Pellets and Powder from different batches were shipped to collaborating laboratories for analysis. Nano-Pellets were analysed using LA-ICP-MS and micro x-ray fluorescence (micro-XRF). The powders were analysed using wavelength-dispersive XRF (WD-XRF) and ICP-MS following acid and fusion digestions.
This report describes the selection, production, and characterisation of the material.

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1. Introduction

SCC-05-NP is the abbreviation for **S**ynthetic **C**alcium **C**arbonate Number **05** **N**ano-**P**ellet. It is number five because the 4 previous versions were purely experimental and on R&D-scale. This material has higher trace element concentrations, which are also more uniform, than those found in natural carbonates. It is intended to allow signal acquisitions on elements with typically low concentrations.

2. Starting Material

A total amount of 240 g calcium carbonate powder was precipitated from elementally doped solutions of calcium chloride ($CaCl_2$) and sodium carbonate (Na_2CO_3), washed with deionised water (18,2 MΩ).

Fig. 1 Photograph of a SCC-05-NP powder batch and a Nano-Pellet.

This type of precipitation produces a mixture of two $CaCO_3$ crystal varieties, calcite and vaterite. The latter is not stable and subject to change over time. To prevent this, the precipitate was roasted for 48 h at 420 °C, which transforms vaterite to calcite. To verify the identity of the material in accordance with ISO 17034:2016[1] two X-ray diffraction (XRD) analyses were performed. The diffractograms confirm the identity (Fig.2).

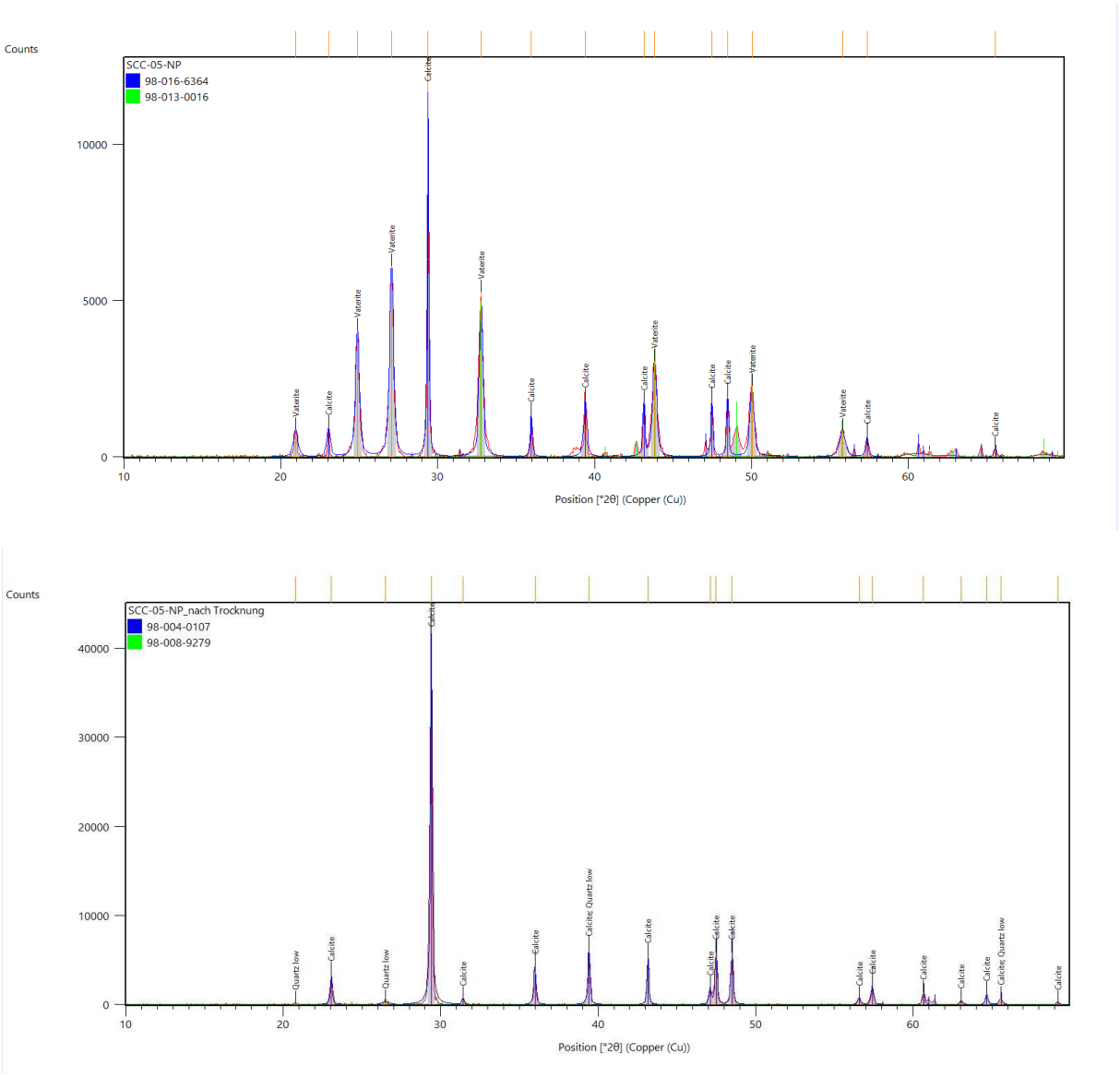

Fig. 2 X-ray diffractograms of precipitate before (top) and after roasting (bottom). The diffractogram confirms the identity of the material as calcite.

3. Sample Preparation

The final grinding step was performed using our patented milling technique. The resulting slurry was freeze-dried, homogenised in a mixer-mill, and split into batches of about 4.5 g using a rotary sample splitter and filled into hydrolytic class 1 glass vials for storage (Fig.1). A total of 36 batches was prepared, 8 of which were used for the analyses.

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4. Homogeneity & Stability Testing

The homogeneity test was performed on the pellets (final packaged form). For this purpose, various batches were randomly selected, and the powders were pressed into Nano-Pellets.

Homogeneity testing was performed adapting a procedure from the ASTM Guide E-826-14[2]. To transfer this procedure to LA-ICP-MS, the pellet surface was divided into seven analytical zones (Fig. 3). However, instead of making three repeated measurements on the same spot, three repeated measurements in close proximity were made (n=21). In total the homogeneity test is comprised of 105 spot measurements. The three results (a, b, c) in each analytical zone are then averaged, so each pellet contributes 7 results for the calculation of the uncertainty component pertaining to homogeneity. One collaborator extended this protocol by analysing 12 analytical zones in triplicate. The uncertainty component resulting from homogeneity was calculated following Annex C.1 in ISO Guide 35:2017[3]. (This guide was replaced by ISO 33405:2024[4], data evaluation had already begun under the previous document and was continued.)

A formal stability test was not performed, however previous experience from 2013 until today with $CaCO_3$ Nano-Pellets has shown long-term stability over several years.

Ultimately, the usability of an uncertainty is determined by the manufacturer and the reference materials' intended use. Therefore, the Horwitz equation[5] and Horwitz Ratio (HORRAT) (eq. 1 & 2) was used to provide a mathematical basis for determining the usability of an uncertainty.

$$RSD_{Horwitz} = 2 \times Concentration^{(-0,1505)} \quad (1)$$

$$HORRAT = \frac{RSD_{Meausured}}{RSD_{Horwitz}} \quad (2)$$

A HORRAT of ≤ 1 is ideal, between 1 and 2 acceptable and > 2 unacceptable.

A working example of such evaluations can be found here:

Example of our Data Evaluation for Certified Values

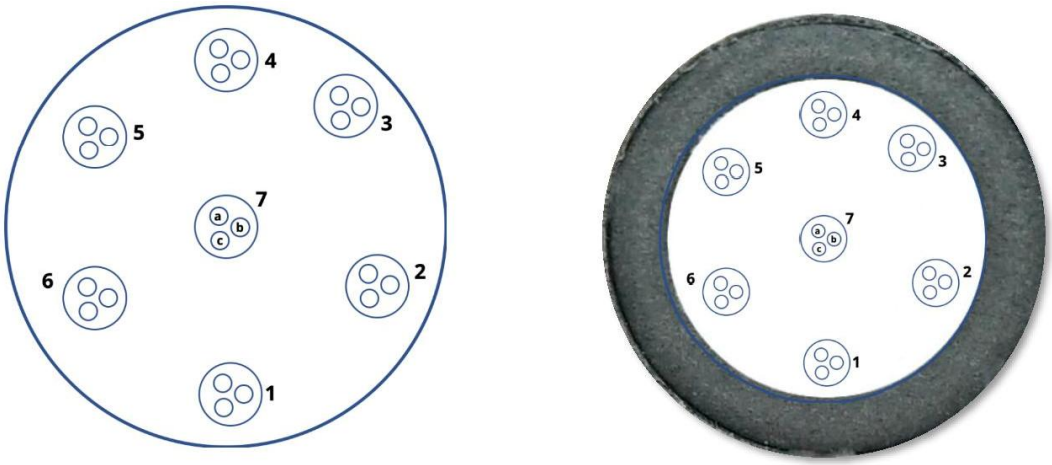

Fig. 3 Ablation pattern adapted from ASTM Guide E826-14.

5. Characterisation & Value Assignment

For characterisation of major and trace elements the characterisation approach "Characterisation of a non-operationally defined measurand using two or more methods of demonstrable accuracy in one or more competent laboratories" was applied.

Solution and fusion analyses were performed using WD-XRF and ICP-MS at several reputable research institutions. Reference and information values (including the list of methods) can be found in the product information sheet here:

Product information sheet

The assigned values and the uncertainty component resulting from the characterisation were calculated using equation 3 and 4.

$$Assigned\ Value = \frac{\sum accepted\ data\ set\ means}{number\ of\ data\ sets} \quad (3)$$

$$Uncertainty_{Characterisation} = \frac{Std.\,Dev.\,of\ data\ set\ means}{\sqrt{number\ of\ data\ sets}} \quad (4)$$

To use equations 3 & 4 the data set means must show an approximately normal distribution. This prerequisite was tested using the software XLSTAT.

Finally, each reference value needs to be accompanied by a combined expanded uncertainty at the 95 % CL. The uncertainty is calculated (eq. 5) by combining the uncertainty components from characterisation and homogeneity testing.

$$Unc._{Final} = k \times \sqrt{Unc._{Homog.}^2 + Unc._{Char.}^2} \quad (5)$$

The expansion factor k is determined by the effective degrees of freedom and their corresponding value in Student's t-distribution. The coverage factor was applied to reach a confidence level of 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (GUM)[6]. According to ISO Guide 35:2017 it is permissible to use an expansion factor of 2 if there are ≥ 10 degrees of freedom. If there are < 10 degrees of freedom the appropriate factor should be taken from a Student's-t table, i.e., the distribution at the desired confidence level.

Here (eq. 6 & Tab. 1) is an unrelated example on how the final uncertainty is calculated:

Tab. 1 Calculation of the final combined and expanded uncertainty, using the analyte Uranium as an example.

	Reference Value	**Unit**
Uranium (U)	31.9	µg/g
Uncertainty$_{Chacaterisation}$	0.2	µg/g
Uncertainty$_{Homogeneity}$	0.9	µg/g
Expansion factor (k)	2	[-]

$$Unc._{Final} = 2 \times \sqrt{0.2^2 + 0.9^2} = 1.8 \quad (6)$$

Analyte [µg/g]	**Reference Value**	**Expanded Uncertainty @ 95 % CL**
Uranium (U)	31.9	1.8

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6. Minimal Sample Size

Every MRM must have a statement of its minimal sample size. Minimal sample size for powdered reference materials is typically given in milligrams (mg). LA-ICP-MS analyses only take subsamples of e.g. a pressed powder pellet, meaning, that the mass of the powder used to make the pellet becomes irrelevant.

The minimal sample size is the smallest beam diameter used for which the homogeneity test still passed. In this case it was 55 µm.

Tab. 2 Ablation and signal acquisition parameters during homogeneity- and stability-testing.

Laser fluence	5 J/cm^2
Repetition rate	15 Hz

7. Acknowledgements

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Tomas Naeraa	University of Lund, Sweden
Dany Savard	Université du Quebec á Chicoutimi (UQAC), Canada
Andrew Zipkin	EAG Eurofins Laboratories Syracuse, USA

8. References

[1] EN ISO 17034:2016 (D/E), *General requirements for the competence of reference material producers*

[2] ASTM E826-14, *Standard Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectroscopy.* ASTM International, West Conshohocken, PA, 2014. www.astm.org

[3] ISO Guide 35:2017 (E), *Reference materials – Guidance for characterization and assessment of homogeneity and stability*

[4] ISO 33405:2024, *Reference materials — Approaches for characterization and assessment of homogeneity and stability*

[5] Horwitz, W., Albert, R. (1995), *Precision in analytical measurements: Expected values and consequences in geochemical analyses.* Fresenius Journal of Analytical Chemistry. 351:507-513

[6] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

9. Document History

Version	*Date*	*Changes applied*
1.0	25.07.2025	First publication