Planning Proficiency Test FXRV-2026-04 for synthetic gypsum

FLX-2005



Bedburg-Hau, 20th October 2025

Coordinator of PT
Charlotte Winkels-Herding

Statistics and Report
Dr. Rainer Schramm

13th August 2026: Corrections are marked in red.

Introduction

FLUXANA GmbH & Co. KG is a company providing services in the field of X-ray fluorescence analysis (XRF).

In 2011, FLUXANA introduced its own quality management.

In 2020 the accreditation of the FLUXANA Laboratory in Bedburg-Hau, Germany, was updated to DIN EN ISO/IEC 17025:2018 and FLUXANA received accreditation as Producer of Reference materials according to DIN EN ISO 17034:2017, as well.

The performance of proficiency tests is not yet accredited. However, the proficiency tests are conducted following the corresponding norms.

Important Information

Costs for the participants

The participants will be billed a total of **500 Euro** (EXW Bedburg-Hau, Germany) for sample FLX-2005 for this proficiency test (PT).

Analysis

Sample pre-treatment

The samples must be dried at **40°C** to a constant mass before being analysed (2 hours is usually sufficient). All Analyses must refer to the dried sample at 40°C.
Remark: Be aware that drying this gypsum at 105°C is not giving a stable weight, therefore 40°C should not be exceeded.

Parameters to determine

FLX-2005 (synthetic gypsum):

Major and minor elements

Al2O3, BaO, CaO, Cl, Cr2O3, CuO, Fe2O3, K2O, MgO, Mn2O3, Na2O, NiO, P2O5, PbO, SiO2, SO3, SrO, TiO2, V2O5, ZnO, LOI

Traces

Co, As, Se, Br, Rb, Sr, Zr, Mo, Cd, Sn, Sb, La, Ce, Hg, Tl, Bi

- Participation is also permitted even when not all of the parameters can be determined.
- Each sample must be analysed two times, and every value must be reported to four decimal places (0.0000).

Preferred method of analysis

- **XRF with fusion as the sample preparation method (like for cement ISO 29581-2:2010)**
- **LOI (loss on ignition)**
- **Every other traceable method, such as ICP, wet chemistry, etc.**

Other methods, e.g., XRF using “pressed pellets” as the sample preparation method or XRF with the “standardless analysis” method, which are not traceable can also be used. These values will not be included in the evaluation. They will, however, be shown as informational values in the report and laboratory comparison.

Recommended sample preparation for Parameters: LOI (loss on ignition):

The dried material is annealed at 950°C until constant mass. (min. 3 hours)

Recommended analytical method for Al2O3, BaO, CaO, Cr2O3, CuO, Fe2O3, K2O, MgO, Mn2O3, Na2O, NiO, P2O5, PbO, SiO2, SO3, SrO, TiO2, V2O5, ZnO:

Sample preparation with fused bead preparation and measurement with XRF

Typically, for the fused bead preparation of raw materials, the samples are dried and then annealed before use to achieve high precision. In this step, however, elements (Na2O, K2O and SO3) may be lost in gypsum because they are volatile. Therefore, we recommend an alternative method that avoids the risk of loss.

Preparation of fused beads from dried material with additional weight

In these processes, the gypsum is fused from dried material with an additional weight of sample (around the LOI). The LOI takes place during the melting process and components that are otherwise lost during annealing in the muffle furnace are retained because they are bound by the flux.

Examples for the calculation of sample weights:

Your calibration of the XRF requires e.g. a weighing of 1.0000g of annealed sample and 8.0000g of melting flux.

You determine the LOI of the gypsum from a dried sample and obtain e. g. 15% LOI.
This corresponds to a sum of all remaining components of 85% (100% - 15% LOI).

Then you calculate the weighing of the dried sample as follows:

$$weight\ sample\ in\ g = \frac{1.0000g}{85\%} * 100\% = 1.1765$$

In practice, this means that you weigh in 1.1765g dried gypsum and you lose 0.1765g of it during melting, so that the ratio of sample to melting agent is 1.0000 : 8.0000.

Recommended analytical method for Cl

Digestion and titration of dried sample like e.g. DIN EN 196-2.

Recommended analytical method for Co, As, Se, Br, Rb, Sr, Zr, Mo, Cd, Sn, Sb, La, Ce, Hg, Tl, Bi:

Acid digestion followed by ICP-OES or ICP-MS.

Application

To receive the samples, they must be ordered as **FXRV-2026-04** by 30th September 2026.

Shipment

The samples will be shipped in October 2026.

Sending in the results

The results must be submitted by 30th November2026 at the latest. You will receive a link, with which you can download a pre-prepared Excel Table and later upload it (completed) for the submission of your data. We assume that you will use one method for one parameter. Thus, you will receive one lab code. However, if you want to use, e.g., two methods for the same parameter, you must request an additional lab code (at no charge).

Report

We intend to complete the report by the end of December 2026.
All evaluations are performed in agreement with DIN EN ISO/IEC 17043:2023, DIN EN ISO 17034:2017 and DIN ISO 13528:2025.

Proficiency test provider / Address for ordering the samples

FLUXANA GmbH & CO.KG
Borschelstraße 3
47551 Bedburg-Hau, Germany
pt@fluxana.de

Coordinator: Charlotte Winkels-Herding, QM
Responsible for evaluation and data processing: Dr. Rainer Schramm, PT

Subcontractors

Analysis performed by PT Participants.

Proficiency test items

Material was taken directly from the production stream.
The materials were delivered and homogeneously distributed into 30 ml bottles by FLUXANA.

Test item	Description
FLX-2005	Synthetic gypsum

Homogeneity and stability

The material was used as delivered. A homogeneity study of the materials was performed based on ISO 33405:2024 and DIN ISO 13528:2025.
The stability is not critical for the time frame of the Proficiency Test and transport, since the material is pre-treated before use.

Metrological traceability

In agreement with internationally valid standards, the analytical procedures (e.g., ICP or any other wet chemical procedure) used by the participants to determine the certified values in this proficiency test have to be traceable. Other methods are not permitted.
In this proficiency test, only traceable results will be used to determine the statistical data. Concentration values determined with other methods can also be submitted; they will be shown in the evaluation and, as with all other values, the z-scores calculated.

Participant accreditation

It is important to know whether the participant laboratory works under ISO 17025 accreditation. Therefore, we will ask this information for each parameter. Which values were determined under accreditation will be shown anonymously in the final report.

Number of participants

The minimum number of participants is 10. For fewer participants, it will be decided on a case-by-case basis whether a statistical evaluation will be conducted.

Potential major sources of errors

Care must be taken to ensure that the material is dried before the analysis.

Evaluation

According to DIN EN ISO/IEC 17043:2023, we will use robust statistical methods in agreement with DIN ISO 13528:2025, ISO/TS 20612:2007 and DIN 38402-45:2024.

Advantages of using robust statistics

Statistical methods are robust in the sense that any outliers have only a limited effect on the overall result. Steps were taken to ensure that the results are still meaningful, even if the proportion of outliers is 1/3. Robust statistics are also preferable for small populations.

Outliers

Outliers in the statistical sense are typically not detected when using robust statistical methods because the robust A+S algorithms were found to work better than the classical approach (which is outlier detection plus arithmetic mean and classical s.d. formula).

If the **reproducibility standard deviation s_R** of a mean value is higher than 50% relative of the mean, all statistical data are only shown as information to the participant. In such a case there is no serious assessment of z-scores possible.

Number of measurements

All participants are requested to perform two measurements. This is necessary to perform the repeatability standard deviation for the laboratories. Participants who send only one or more than two values must first ask for permission. Otherwise, they will be excluded.

Publication of the results

All participants will be informed about the results of the PT with a report. Which results were delivered by which laboratory will be kept confidential. All laboratories are encoded, and the code is only known to the organizer and the individual laboratory. The final report will be published on the FLUXANA website. First, a preliminary report will be sent out for verification by the participants. Within one month, the final report will be published.

Laboratory performance

Each participant will receive a performance evaluation report based on z-scores. The diagram shows the relative difference to the assigned values

Further Information

For this proficiency test, the participants' results must be submitted to the organizer using only the "Result Sheet" Excel table, which must not be altered. Paper sheets or other Excel tables will only be accepted in special cases in prior agreement with the organizer. In this way, we want to improve the data quality and avoid any transmission errors.

Statistical Evaluation used for this PT

Calculation of Mean m

The mean m for all laboratories is calculated using the Hampel estimator (ISO/TS 20612:2007 9.2.3 + DIN ISO 13528 C.5.3.2) based on the laboratory means µ using traceable methods only.

Calculation of reproducibility standard deviation s_R

The reproducibility standard deviation s_R is calculated using the Q-method (ISO/TS 20612:2007 9.2.2 + DIN ISO 13528 C.5.2.2).

Calculation of repeatability standard deviation s_r

The repeatability standard deviation s_r is also calculated using the Q-method (ISO/TS 20612:2007 Annex B).

Calculation of reproducibility standard deviation s*

The reproducibility standard deviation s* is calculated from the laboratory means µ using the Q-method (ISO/TS 20612:2007 9.2.2 + DIN ISO 13528 C.5.2.2).

Calculation of uncertainty U_{s_R} (according to Nordtest TR 537 ed 3.1.)

The **uncertainty U_{s_R}** for a confidence interval of P=95% (k=2) can be calculated from the **reproducibility standard deviation** s_R (factor 1.25 for average median, robust statistics) and the number of participating laboratories p:

$$U_{s_R} = 2 * 1.25 * \frac{s_R}{\sqrt{p}}$$

Calculation of uncertainty U_{s^*} (according to DIN ISO 13528:2025 7.7.7)

The **uncertainty** $\boldsymbol{U_{s^*}}$ for a confidence interval of P=95% (k=2) can be calculated from the **robust standard deviation** s* (factor 1.25 for average median, robust statistics)) and the number of participating laboratories p:

$$\boldsymbol{U_{s^*} = 2 * 1.25 * \frac{s^*}{\sqrt{p}}}$$

The **uncertainty** $\boldsymbol{U_{s^*}}$ only takes the between laboratories uncertainty into account while the **uncertainty** $\boldsymbol{U_{s_R}}$ also includes the within laboratories uncertainty. Therefore $\boldsymbol{U_{s_R}}$ is recommended for use in accredited laboratories.

Laboratory performance

Laboratory proficiency assessment is based on z-scores.
The **z-score** z is calculated from all laboratory means µ:

$$z = \frac{m - \mu}{s_R}$$

m	Mean value for all laboratories (assigned value)
µ	Mean value of individual laboratory
s_R	Reproducibility standard deviation

Assessment on z-scores:

$\|z\|$ <= 2.0	indicates “satisfactory” performance = generates no signal
2.0 < $\|z\|$ < 3.0	indicates “questionable” performance = generates a warning signal
$\|z\|$ >= 3.0	indicates “unsatisfactory” performance = generates an action signal

Z-scores with 3 >= $|z|$ >= 2 are highlighted with a yellow color, z-scores with $|z|$ >= 3 are highlighted with a red color.

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