Planning Proficiency Test RV-2017-01 for Used Auto Catalytic Converters

FLX-CRM 132, FLX-CRM 133

Bedburg-Hau, 02 February 2017

Coordinator of PT
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Head of Laboratory
Dr. Barbara Schäfer

Statistics and Report
Dr. Rainer Schramm

Important Information

Costs for the participants

The participants will be billed a total of **400 Euro** (EXW Bedburg-Hau, Germany) for all 2 samples for this proficiency test (PT).

Analysis

Sample pretreatment

The samples, as received, should be dried at 105 °C until constant mass (could take up to 8 hours). **All results must be based on dried sample.**

Parameters to determine

Pd, Pt, Rh

- **Participation is only possible when all parameters can be determined.**
- **Each sample must be analyzed at least two times; a maximum of six individual measurements are allowed; every value must be provided in mg/kg with one decimal place (0.0).**

Preferred method of analysis

- **XRF (handheld, portable or stationary) combined with bulk powder or pressed pellets**
- **Every other suitable method, such as ICP, wet chemical, etc. (only as informational values)**

The concentrations for these samples have already been determined in a previous proficiency test. This means that the returned values will be evaluated with regard to the certified concentrations.

Application

To receive the samples, they must be ordered as **RV-2017-01** by 28 April 2017.

Shipment

The samples will be shipped in May 2017.

Sending in the results

The results must be submitted by July 15, 2017, at the latest. You will receive a pre-prepared Excel Table for the submission. 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.

Report

We intend to complete the report by the end of September 2017.

Introduction

X-ray fluorescence analysis is a frequently used technique for the analysis of oxidic materials.

However, for the calibration of XRF instruments, dedicated standard material is needed. As a worldwide supplier for XRF laboratories, FLUXANA has developed a number of services to support XRF users. One of these services is the production of new reference materials and the organization of proficiency tests (PT).

In 2011, FLUXANA introduced its own quality management.

In February 2014, FLUXANA received accreditation from the German DAKKS according to DIN EN ISO/IEC 17025 for the test laboratory in Bedburg-Hau.

The production of reference materials and the performance of proficiency tests is not yet accredited. However, FLUXANA has applied for the accreditation process at DAKKS.

Nevertheless, all evaluations are performed in agreement with DIN EN ISO/IEC 17043:2010-05, ISO Guide 34:2009, ISO Guide 31:2000 and ISO Guide 35:2006.

Proficiency test provider / Address for ordering the samples

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

Coordinator: Charlotte Winkels-Herding, QM
Responsible for evaluation and data processing: Dr. Rainer Schramm, CEO
Responsible for in-house analytical tests: Dr. Barbara Schäfer, Head of test laboratory

Proficiency test items

This reference material sample was produced from commercial products. Material was taken directly from the production stream.
Analysis performed by PT Participants.

Proficiency test items

About 9 kg of each material were delivered and homogeneously distributed into 50 ml bottles by FLUXANA. The bottles were then vacuum packed for storage.

Test item	Description
FLX-CRM 132	Used Catalytic Converter
FLX-CRM 133	Used Catalytic Converter

Homogeneity and stability

The material was used as delivered. A homogeneity and stability study of the materials was performed based on ISO Guide 35:2006 and DIN ISO 13528:2009-01.

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 the previous proficiency test had to be traceable. Other methods were not permitted.
In this proficiency test, only XRF 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

Potential major sources of errors

- The sample must be dried and homogenized before the analysis

Recommended rules for the analysis

Approx. 4 g of the dried (1 h, 105 °C) and homogenized sample should be filled into a suitable sample cup and measured.
Alternatively, a pressed pellet (4 g sample and 1g Cereox for 32 mm diameter) can be made and measured.

Evaluation

According to DIN EN ISO/IEC 17043:2010-05, we will use robust statistical methods in agreement with DIN ISO 13528:2009-01, ISO/TS 20612:2007 and DIN 38402-45:2014-06.

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). Outliers shown in the evaluation are only based on z-scores and marked with yellow or red colors.

Number of measurements

All participants are requested to perform two measurements, for some methods up to six measurements are recommended. 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) based on the laboratory means µ.

Calculation of reproducibility standard deviation s_R

The reproducibility standard deviation s_R is calculated using the Q-method (ISO/TS 20612:2007 9.2.3).

Calculation of repeatability standard deviation s_r

The repeatability standard deviation s_r is also calculated using the Q-method.

Uncertainty of Mean U

The **uncertainty** 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):

(1) $$U = 2 * 1.25 * \frac{s_R}{\sqrt{p}}$$

Laboratory performance

The laboratory proficiency assessment is based on z-scores.
From all laboratory means µ, the **z-score** z is calculated:

(2) $$z = \frac{m - \mu}{s_R}$$

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

Assessment of 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

All laboratory means µ with 3 >= $|z|$ >= 2 are highlighted with a yellow color, z-scores with $|z|$ >= 3 are highlighted with a red color.