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PROFICIENCY TESTING PROGRAMME

GYPSUM AND LIMESTONE

Organiser:

**Institute for Chemical Processing of Coal
Proficiency Testing Centre
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1. PRESENTATION OF PROFICIENCY TESTING CENTRE

Proficiency Research Centre (OBB), exists in the organizational structure of the Institute for Chemical Processing of Coal in Zabrze. It was established by the Director of the Institute for Chemical Processing of Coal by decree No. 8A/2008 on 20th of March, 2008. and No. 16A/2010 on 15th of September, 2010.

Institute for Chemical Processing of Coal has many years of experience in the research work. The Institute has implemented a quality management system according to PN - EN ISO 9001:2009 in the following areas: research work and development, expert services and implementation, including the energy industry, cokemaking, use of solid fuels, biomass and waste, processing and use of carbon-based products, energy and environmental services in the field of scientific and technological information as well as standardization.

The team at the Institute's Laboratories, which has implemented a management system, complies with the requirements of PN -EN ISO/IEC 17025:2005. Confirmation of the implementation and proper functioning of the system are the certificates: Quality Management System No. 2008/3/2010 issued by the Polish Centre for Testing and Certification and testing laboratory No. AB 081 issued by the Polish Centre for Accreditation. The Proficiency Testing Centre has implemented a management system which complies with the requirements of PN- EN ISO/IEC 17043. The Centre has a certificate of proficiency organizer PT No. 004, issued by the Polish Centre for Accreditation (<https://www.pca.gov.pl/en/accredited-organizations/accredited-organizations/pt-providers/>). The coordinator is responsible for all stages of the program, on behalf of the Centre. The coordinator cooperates with Accredited Laboratories and the technical experts.

2. CONDITIONS OF PARTICIPATION IN THE PROGRAM

The obtained results will help to evaluate the quality of carried out measurements and possessed proficiency. The program is timed to the organization of individual rounds, according to the schedule for the calendar year. The schedule is available on the website IChPW www.ichpw.pl (OBB tab).


Before the start of each round of the program, the participants are informed, about the exact dates of: declarations, sending samples, testing and transfer of the results. For this opportunity, the Centre develops the *General Information* and puts it on the IChPW website: www.ichpw.pl (OBB tab). The *General Information* is allowed to be transfer to the potential participants of proficiency testing with *Participation Sheet* in a form of written invitation for participation.

Laboratories who want to participate in the particular round PT/ILC are required to familiarize themselves with its program and *General Information*. In order to register to participate in this round of the program, use the platform EBOU (Electronic Participant Service). Access to the login page can be accessed via a bookmark Electronic Shareholder Service Office, located on the website of the Institute for Chemical Processing of Coal, in a Proficiency Testing Centre or by typing the address directly <http://badaniabm.ichpw.pl> in the browser's address field.

Proper application for participation is confirmed by displaying the confirmation window Accession to participate in a round PT / ILC implies the acceptance of the program. Accession to participate in a round PT / ILC implies the acceptance of the program.

Participants are informed by e-mail about any changes in programme schedule and forms, during the round conduction.

Any changes of the programme are placed on the OBB website. The participants should check the latest version.

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The samples and all the necessary forms are sent to the participants after application. Personal receipt of the sample by participant should be confirmed on the form F/OBB/4.6/07/D or F/OBB/4.6/15/D *Confirmation of receipt of samples for testing*.

The Centre requires participants to inform about the damage of the delivered test sample. Participant confirms the protocol form F/OBB/4.6/06/D or F/OBB/4.6/14/D and send it by email to the Centre within three days. The new test sample will be sent immediately.

The test results must be submitted to the Centre within a reasonable time by an Electronic Participant Service. After the submission deadline the participant should print the results transmitted to the results of proficiency testing Sheet (Form F/OBB/4.6/08/D or F/OBB/4.6/16/D). The completed and certified by the signature sheet of the results should be sent by post or electronic scan to the Centre within the prescribed period.

The participant is obliged to adhere strictly to the schedule of the round and report the test results on time. The results, which are not sent by the deadline, will not be taken into account.

Each participant has the right to resign from the participation in proficiency tests. In this case, sending a written notification to the address of the Proficiency Testing Centre up to 3 days before the date of sending samples (in accordance with the schedule) is required. The date of registering the notification by the Centre is the date of submitting the resignation.

3. SELECTION OF MEASURED PARAMETERS

The program includes of interlaboratory comparisons following quality parameters of gypsum and limestone:


gypsum:

- determination of moisture content,
- determination of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ content,
- total content of calcium and magnesium carbonates,
- determination of chloride content,
- determination of Silica and insoluble components content,
- pH,
- whiteness,
- particle size distribution,
- oxide composition (CaO , CaCO_3 , SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , MgCO_3 , K_2O , Na_2O),

limestone:

- determination of moisture content,
- determination of Silica and insoluble components content,
- determination of total carbon content,
- oxide composition (CaO , CaCO_3 , SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , MgCO_3 , K_2O , Na_2O).

Recommended methods are presented in the standards and / or test procedures that are documented in laboratory accreditation or test methods routinely performed by the laboratory of the participant.

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4. TEST OBJECTS AND DISTRIBUTION

Object of study are samples of gypsum and limestone.

For each test item separate rounds of interlaboratory comparisons are held. Each round of proficiency testing in addition to the name: PROFICIENCY TESTING: GYPSUM AND LIMESTONE has appropriate identification number consisting of the initial letters derived from the name of the object, the number of the next round and the year in which this round is done, for example:

- PROFICIENCY TESTING. GYPSUM AND LIMESTONE GiKW/1/2017 what means the first round in the year 2017, in which the object of the research is gypsum and limestone.

Before preparing the test samples the homogeneity of the samples is checked.

Test objects are prepared in accordance with the standard and / or documented test procedure applicable at the Laboratory of Fuels and Activated Carbons IChPW.

Packaging, labelling and distribution of test is performed in accordance with the Operating Procedures PO/OBB/4.6/D - *Realisation of proficiency testing* and instructions I/OBB/4.6/02/D - *Packaging samples*.

The testing samples should be treated by participants as usual and routine test.

5. SUB-CONTRACTING

Various parts of the proficiency testing program may be sub-contracted. When subcontracting takes place, the work is outsourced to competent subcontractors and Proficiency Testing Centre is responsible for this work. Subcontracting is regulated by the procedure PO/OBB/5.5/A - *Subcontracting services*.

6. HOMOGENEITY AND STABILITY EVALUATION OF TEST OBJECTS

The assessment of homogeneity and stability of testing samples is carried out in accordance to the Operating Procedures PO/OBB/4.7/D - *Data analysis and evaluation of proficiency testing* and instructions I/OBB/4.6/01/D - *Material homogeneity and stability check*.

6.1. The criteria for homogeneity


The assessment of homogeneity of prepared samples of coke is carried out basing on the determination one or few of the parameters: particle size distribution, whiteness, determination of chloride content, oxide composition. The number of sample units were randomly collected from samples prepared for testing. Computational methods used to assess the homogeneity of test items conform to ISO 13528:2015 Annex B. Due to Annex B of ISO 13528:2015 following steps are determined:

- Cochran test for results in pairs,
- The average ash content of individual samples \bar{x}
- The average standard deviation s_x
- Standard deviation of the individual sample pairs s_w
- The standard deviation between sample unit s_s
- variance σ_{all}^2 (connected to standard deviation of proficiency testing σ_{pt}),
- c value (on the basis of statistics χ^2 and F-Snedecor).

The samples are considered sufficiently homogeneous if the criteria are met (confidence level 95%):

$$s_s \leq \sqrt{c}$$

The standard deviation of proficiency testing σ_{pt} is calculated according to ISO 13528:2015.

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Above mentioned conditions are met, confirms samples homogeneity. Selected statistical model is approved by Coordinator.

5.2 The criteria for stability

The stability of the samples is determined according to the average value of the above mentioned parameter of individual samples. Computational methods used to evaluate the stability of the samples are carried out in accordance to ISO 13528:2015 Annex B. The samples are considered as sufficiently stable when the criterion, presented below, is met:

$$|\bar{y}_1 - \bar{y}_2| \leq 0,3\sigma_{pt}$$

where:

- \bar{y}_1 - the mean value of the parameter obtained during the homogeneity test for the tested samples,
- \bar{y}_2 - the mean value of the parameter obtained during the stability test for the tested samples,
- σ_{pt} - the standard deviation of proficiency testing

Standard deviation of proficiency testing σ_{pt} is calculated according to ISO 13528:2015. Selected statistical model is approved by Coordinator. This requirement confirms the stability of the samples.

7. STATISTICAL METHODS

Centre uses statistical models and methods for analysing data with a description of the criteria in accordance to the Operating Procedures *Data analysis and evaluation of proficiency test* (PO/OBB/4.7/D) and PN-EN ISO / IEC 17043:2011 and ISO 13528:2015.

7.1. The determination of assigned value and standard uncertainty

The assigned value for each round of proficiency testing is the value agreed with the results of the participants. The way to determine the assigned value and the estimation of the uncertainty of its designation is approved by the coordinator.

To determine the assigned value and its uncertainty applies robust statistical method compatible with ISO 13528:2015 is applied. For all the assigned values standard uncertainty of their determination u_{x_i} is estimated (according to ISO 13528:2015).

If the condition $u_x \leq 0,3\sigma_{pt}$ (determined in accordance with ISO 13528:2015) is met then the uncertainty of the assigned value is negligible and does not need to be included into the calculation.


For check the normal distribution Shapiro-Wilk test is used. For the identification of outliers Grubbs test is used.

The method of determining the assigned value and the applicable criteria shall be included in the report from proficiency testing.

7.2. The criteria for evaluation of results

If the condition $u_x \leq 0,3\sigma_{pt}$ to the statement of the achievements of participants is met, the indicator z-score is used:

$$Z = \frac{X_i - X_{pt}}{\sigma_{pt}}$$

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where:

x_i - average value for specific parameter

x_{pt} - assigned value,

σ_{pt} – the standard deviation for proficiency assessment,

Interpretation of the assessment results obtained by the participants is as follows:

$|z\text{-score}| \leq 2$ = satisfactory

$2 < |z\text{-score}| < 3$ = doubtful

$|z\text{-score}| \geq 3$ = unsatisfactory

If the above condition is not met, then to the statement of Participants achievements z'-score index is used (according to ISO 13528:2015):

$$z' = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u(x_{pt})^2}}$$

where:

x_i - average value for specific parameter,

x_{pt} - assigned value,

σ_{pt} – the standard deviation of proficiency testing,

$u(x_{pt})$ – uncertainty of the assigned value

Interpretation of the assessment results obtained by the participants is as follows:

$|z'\text{-score}| \leq 2$ = satisfactory

$2 < |z'\text{-score}| < 3$ = doubtful

$|z'\text{-score}| \geq 3$ = unsatisfactory

In addition, the results of which are given by the participants, including the uncertainty, will be assessed by indicators:

$$\zeta_i = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}}$$

where:

x_i - average value for specific parameter,

x_{pt} - assigned value,

$u(x_i)$ – standard uncertainty of x_i

$u(x_{pt})$ – uncertainty of the assigned value

Interpretation of the assessment results obtained by the participants is as follows:

$|\zeta_i| \leq 2$ = satisfactory

$2 < |\zeta_i| < 3$ = doubtful

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$I_{\Sigma} \geq 3$ = unsatisfactory

If the obtained results are doubtful and unsatisfactory, the budgets of the uncertainty should be re-estimated.

Participants results are presented in the Reports of proficiency testing in a tabular and graphical form.

8. PROFICIENCY TEST REPORT

Centre prepares a detailed report of every test round program. The report contains all the essential points of comparisons, including:

- The name and address of the organizer,
- Name of the Coordinator,
- The issue date of the report,
- The name and code number of the round,
- Descriptions of the subcontracted parts of the round,
- Description of the test object with information about preparation of the samples, the homogeneity and stability study,
- Results of the homogeneity and stability of the tested object,
- Code numbers of participants and their results,
- Statistical data and a summary, including assigned values and range of acceptable results,
- The procedures used to determine the assigned value,
- Tabular and graphical representation of the participants' results,
- The procedures used for the statistical analysis of the data,
- The procedures used for designing and implementation of the program,

A copy of the report (in the form of a secured PDF file before editing) will be available on the EBOU platform (Electronic Participant Service) up to three months.

9. PUBLICATION OF TEST RESULTS

Centre reserves the copyright to all the data taken from the published reports.


Centre uses the results of proficiency testing in the studies-studies, statistical and research work, without the consent of the participant preventing identification of participants.

10. CONFIDENTIALITY

Centre ensures the confidentiality of all informations related to participation in the program for all participants of the proficiency testing by following PO/OBB/4.10/D Operating Procedures - *Ensuring the confidentiality and intellectual property participant proficiency testing*.

The centre does not provide information about the test results obtained by the laboratories involved in the program to third parties. In exceptional circumstances, Coordinator is asked by decision-making authority or Accreditation for direct delivery of proficiency testing results to these units. In this particular situation the participants are informed.

The participant is required to perform research and to report the results to the Centre, without any communication with other laboratories that participate in the program.

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11. COLLUSION AND FALSIFICATION

The Centre ensures that proficiency testing programs are designed to exclude the possibility of collusion or falsification of results by proficiency testing participants.

In case of suspicion of collusion or falsification of results by participants of proficiency testing, OBB Coordinator asks in writing to the participants about the copy of the source documents proving obtained results and asks to send it to the Centre.

If collusion or falsification of results is detected, participant or group of participants will be excluded without refunding of participation cost.

However, it should be noted that avoiding collusion and falsification of results is the responsibility of the participating laboratories.

12. CONTACTING THE PARTICIPANT

The centre offers to the participants a detailed information concerning the program, of every single round. All the necessary forms and documents are sent to the participants, either in person or by mail.

Technical Manager provides with the information about the planned program of proficiency testing, fees and the criteria of participation, by posting information to the website.

The basic correspondence regarding the implementation of the program, includes:

1. General Information for each round of PT / ILC.
2. The required forms provided with the samples for testing.
3. The report - sent to participants at the end of the round
4. Poll client assessment service level Proficiency Testing Centre- send to the participants after finishing each round.

If the participant finds the non-compliance of his sent results, Centre should be informed about it and the participant should provide the Centre with proper explanation. In this situation, the results already submitted will be replaced by the new ones. Admission by the Centre for new studies to provide them with an explanation in a timely manner.

Centre will take into account the new results, only if they are sent before the final report is published.

Every participant have the right to submit written comments / complaints to the report within 14 days Centre considers comments / complaints in accordance to the Operating Procedures PO/OBB/5.8/D - *Complaints and appeals*.

On doubtful issues that require explanation or justification as well as any other matter which may contribute to the improvement of the organization of proficiency testing Laboratories are encouraged to contact with the Centre.

13. CONTACT WITH THE CENTR

On behalf of the Centre for the realization of this program corresponds Coordinator::

prof. Marek Sciazko

Institute for Chemical Processing of Coal

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