

PROFICIENCY TEST PLAN FOR LIMESTONE ANALYSIS

2024



ACCREDITATION

ITAK – Instituto de Tecnologia August Kekulé is an ABNT NBR ISO / IEC 17043 Proficiency Testing provider for the following matrices: Iron Ore, Gold Ore, Copper Ore and Concentrates, Nickel Ore, Silver, Ferroniobium, Phosphate Rock and Phosphate Concentrate and Bauxite, which are accredited by "Coordenação Geral de Acreditação do Inmetro (Cgcre)", ABNT NBR ISO/IEC 17043 (PEP-0021).

Seeking to expand this important work to other matrices, **ITAK** is launching in **2024**, two (2) rounds of the **Proficiency Test Program for Limestone** and invites the laboratories of such market to join the group of participants. We aim to provide continuous improvement practices in performing chemical analyses to our customers worldwide.

BENEFITS

Besides being an impartial tool for assessing laboratory performance, participation in proficiency tests has several benefits, such as:

- ✓ Adherence to one of the requirements of ISO / IEC 17025 for companies already Accredited, or searching for accreditation on this ISO standard or other quality systems;
- ✓ Increased metrological reliability of the results obtained by the laboratory from their levels of precision and accuracy;
- ✓ Comparison with other industry laboratories and finding improvement opportunities to increase performance level (benchmarking);
- ✓ The opportunity for evaluation and comparison of methodologies seeking one that best suits the level of accuracy required for specific parameters and levels;
- ✓ Awareness of the type of deviations that may be done by the laboratory (systematic or random) quiding actions to eliminate/minimize them;
- ✓ Identify staff training needs; and/or normalization or improvement of analytical methods.

CONFIDENTIALITY

According to a confidentiality protocol adopted by ITAK, the labs are identified by specific numerical codes, and the participants have knowledge and access only to their code, avoiding collusion among participants, which in case of occurrence or suspicion, will be properly investigated and handled by the program coordination team.

The performance comments described in the final reports are also confidential and intended to provide important information to the person in charge of the laboratories so that they have a thorough evaluation of the quality aspects of their analysis and know where to act effectively to correct possible deviations.

The experimental results obtained by the Participant Laboratory or even their performance in the Proficiency Test may be disclosed to third parties with the consent of the Participant Laboratory.

All participating laboratories must be aware that their reported results may be used by ITAK in the certification of reference materials used in the PT as test items, preserving data confidentiality.



STATISTICAL TREATMENT

The choice of the method to obtain the assigned value, the standard deviation for proficiency assessment and the estimated uncertainty of the proficiency test will depend on the number of participants and data consistency, using statistical approaches and techniques from the ISO 5725, ISO 17043 and ISO 13528 series standards.

If this Proficiency Test uses a Statistical model with assigned values and Standard deviation from Certified Reference Materials, a minimum number of participants is not required.

If the Proficiency Test uses a Statistical model with Consensus values, a minimum of six participants is required.

If the Proficiency Test uses a Statistical model that involves Consensus values from expert participants after removing outliers, a minimum number of three participants is required for evaluation.

According to the results of accuracy assessments, the performance of the participants can be classified as satisfactory, unsatisfactory, or questionable for each evaluated parameter.

If the participant does not provide complete data for evaluation for some specific analyses, the report will be issued as "insufficient data".

Each statistical technique has its use limited and is conditioned to the number of participants who report numerically valid results and are approved in the Evaluation and Treatment Outliers.

SAMPLES AND ANALYSES

In 2024, ITAK will launch two (02) rounds of the Proficiency Test Program for Limestone Analyses, each one consisting of 10 samples referring to two reference materials of different concentrations (05 sachets containing 10 g each), totaling 10 sachets per round.

The samples used in the Proficiency Testing Program are Reference Materials (RMs) with proven homogeneity and stability. RM's are produced by ITAK following the ISO 9001 Quality Management System and ISO quality protocols (ISO Series Guide 30 to 35) and ABNT NBR ISO 17034 - General requirements for the competence of reference materials producers.

It is the participant's responsibility to follow up and clarify the shipment through customs or carrier when it is required. If samples come back to ITAK because of incorrect address information or because the laboratory has not clarified the shipment through customs, ITAK can charge reshipping expenses.

Test items can be shipped by using customer accounts on FedEx, DHL, and UPS carriers.

Along with the samples will also follow instructions directing the Participating Laboratory on how to treat the samples, carry out the analyses, and report the analytical results.



Any communication about this Proficiency Testing, such as doubts, reporting of results, technical reports, and certificates of participation, is by software available at www.itak.com.br/app for participating laboratories.

In addition to their test results and methodologies, ITAK PT participants should also report the measurement uncertainties of each analyzed parameter (if available). It must be sent as the Standard Uncertainty Combined, without application of any coverage factor, which would make it Expanded Uncertainty. Such data should be reported in specific fields in the ITAK PT Management System.

Invalid analytical results such as "less than" or "zero" are not amenable to statistical evaluation. Therefore, in these situations, ITAK will not do the participant performance evaluation.

The participating laboratory is required to undertake one (1) independent determination in each sample, preferably in five (05) different days, analyzing a couple a day, employing one or more validated analytical methods. The methods used should be reported with the results. The samples must be analyzed as ordinary routine samples of the laboratory, without any special treatment.

The parameters to be determined in each sample are listed below:

Al₂O₃ (%), CaO (%), Fe₂O₃ (%), K₂O (%), MgO (%), MnO (%), P₂O₅ (%), SiO₂ (%), TiO₂ (%) and (LOI) (%).

The samples will be sent by the ITAK from João Monlevade / MG, under the guidance of the Participant Laboratory, using an appropriate way to transport them. In case of loss or damage to any test item, ITAK will evaluate the possibility of replacement, which will depend on how it can be transported and the delivery deadline so as not to compromise the schedule.

ANALYTICAL METHODS

The Participant should use its routine procedure in Limestone Analyses. ITAK suggests the following analytical methods for this Proficiency Test:

- ✓ Al₂O₃, CaO, Fe₂O₃, K₂O, MgO, MnO, P₂O₅, SiO₂ and TiO₂: Fusion and determination by XRF
- LOI: Gravimetric method or TGA.

The methodologies used must be informed following, whenever possible, the "Digestion Technique / Reading Instrument" standard, using simplified codes as per the following examples:

• Fusion and XRF: FUS-XRF;

• Gravimetry: **GRA**;

• Thermogravimetric Analyser: TGA.



SCHEDULE FOR 2024 ROUNDS

The proposed schedule for 2024 is presented in the table below but can be adjusted as required. The official schedule should be visited through the PTP Management System.

Steps of the Program	1st Round 2024	2nd Rodada 2024
1- Sending invitations	15/Aug/2024	
2- Confirmation of participants	13/Sep/2024	
3- Sending samples to the participants	13/Sep/2024	30/Sep/2024
4- Receiving samples by the participant	30/Sep/2024	18/Oct/2024
5- Completion of the Analyses and report the results by the participants	21/Oct/2024	15/Nov/2024
6- Confecção e entrega do Relatório de Desempenho aos participantes.	08/Nov/2024	29/Nov/2024

Note: the dates in bold (item 5) must be met to avoid delays in issuing the results, which can compromise the subsequent rounds of the program.

If delays occur, the Program Coordinator must be contacted to assess the possibility of extending the deadline without compromising the schedule or loss to the other participants.

If there are unjustified delays, ITAK may close the rounds without the missing results.

For each round, the instructions will be sent along with the samples, containing guidelines on how the laboratory should proceed, and specific information about the samples such as expected levels of the analyte (s).

REQUIREMENTS FOR PARTICIPATION

The Laboratory interested in participating in the Proficiency Testing Program for Limestone Analysis - ITAK - 2024 should be a legally responsible organization with the technical capacity and equipment to carry out the chemical analysis of its scope; complete the registration form on the ITAK website at www.itak.com.br/app, confirming its participation until the deadline set in the schedule.



PERFORMANCE REPORT

For each round of the Proficiency Test Program for Limestone Analyses – 2024, ITAK will issue a personalized digital Interlaboratory Performance Evaluation Report under confidentiality identification for the internal evaluation of the Participant Laboratory and identifying improvement opportunities.

The Performance Report is structured following the ISO / IEC 17043 requirements.

If the participant wants to receive the report of another unit of the same business group, it must be requested directly to the person in charge, or through a formal authorization from the person in charge. Preliminary reports will not be issued.

If required, ITAK may rectify or ratify reports provided that such corrections do not require further statistical processing of data. All rectification generates a new version and new report number that will replace the previous version when published and communicated.

If required, by law, the performance report can be delivered or presented to the Regulatory Entity or Public Prosecutor's Office without the participant's permission and will be formally and previously communicated.

If it is not possible to deliver the performance report on the date planned in the schedule, the participants will be promptly notified about the possible delay and the new date delivery.

ITAK Interlaboratory Performance Report is accepted as an ISO/IEC 17025 requirement.

APPEAL

The participant of ITAK's Proficiency Test has the right to appeal against its performance evaluation on the Technical Report.

The participants who have doubts or disagree with the performance evaluation must register their appeal in the "Appeals" field in the Proficiency Test Management System.

Meeting a requirement of ABNT NBR ISO/IEC 17043 - Conformity assessment — General requirements for proficiency testing, ITAK has a procedure that manages this process.

INVESTMENT

Request a proposal by email: interlab@itak.com.br

ITAK has an attractive discount policy such as participation of several units of companies belonging to the same economic group, discounts for laboratories participation in more than one PT, discounts for purchase of ITAK's CRM, etc.

Find out more by contacting us at: interlab@itak.com.br



All customs duties are participants' responsibility.

ITAK must receive the total amount without any deductions such as bank fees or taxes applied. It is necessary to take them into account before sending us the payment.

SUBCONTRACT ACTIVITIES

For this PT, homogeneity test analyses of Reference Materials used as samples (test items) can be subcontracted from competent laboratories.

All laboratories are evaluated and qualified in the ITAK's Quality Management System for these analyses providing results with which, after appropriate statistical treatment, the designated values, standard deviations, and uncertainties are obtained for the Proficiency Test.

COORDINATION TEAM

Bráulio de Freitas Pessoa Technical Director

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