INSTITUTO DE PESQUISAS TECNOLÓGICAS

PROFICIENCY TESTING SCHEME FOR PULP

CYCLE 2026

PROTOCOL

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PROTOCOL

1 INTRODUCTION

A laboratory can be considered the main environment for the practice of metrology and it is expected that it delivers results with assured quality. For that, it needs a quality system which ensures the emission of metrologically reliable results and an external evidence of its proficiency.

The participation in a Proficiency Testing Scheme (PT Scheme) is a manner an external evidence of proficiency of a laboratory, as mentioned in ISO/IEC 17025 These PT Schemes consist of measurement of one or more parameters performed independently by a group of laboratories in samples of a material. Their application requires a provider and participating laboratories. Among the functions of the provider are: to prepare instructions, to send the samples (test items) to the participants and to statistically treat the results obtained from participating laboratories. The mean role of the participant is to follow the instructions given by the coordinator.

The mean steps of a PT Scheme are shown in **Figure 1**.

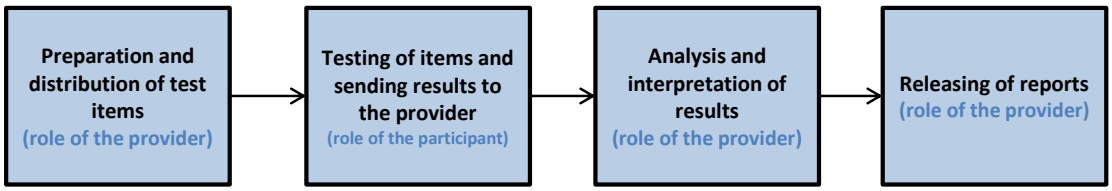

Figure 1 – Mean steps of a PT Scheme

IPT - Institute of Technological Research - has a large experience in coordinating PT Schemes, in which the first PT Scheme offered for tests in pulp began in the 1980's.

The cordination of PT Scheme for Pulp is the researcher Patrícia Kaji Yasumura, from *Laboratório de Celulose, Papel e Embalagem* (Pulp, Paper and Packaging

Laboratory), who, together with her team, offers a scheme that allows participating laboratories to observe their performance against a group of laboratories and to identify the nature of deviations of their results, as well as some problems such as calibration of equipment and technicians trainings.

The PT Scheme for Pulp focuses unbleached and bleached hardwood pulps. This PT Scheme is offered yearly and consists of three rounds, except for two of the tests offered, which are executed in two rounds (see item 3).

2 TARGET TYPE OF PARTICIPANT

Laboratories that perform tests in pulp, whether they are from industries, private companies, associations, research institutes or universities.

3 TESTS OFFERED

Table 1 – Tests offered

Test	Pulp	Standard	Related Brazilian Standard	No. of rounds
GROUP I				
Limiting viscosity number	Bleached and unbeaten	ISO 5351:2010	ABNT NBR ISO 5351:2012	3
Kappa number	Unbleached	ISO 302:2015 TAPPI/ANSI T 236 om-13	ABNT NBR ISO 302:2018	3
Water retention value	Bleached and beaten	ISO 23714:2014	-	3
Drainability, *Schopper-Riegler*	Bleached and beaten	ISO 5267-1:1999	ABNT NBR 14031:2004	3
GROUP II				
Milling evaluation (PFI mill)	Bleached and unbeaten	ISO 5264-2:2011 TAPPI/ANSI T 248 sp-15	ABNT NBR ISO 5264-2:2012	2
Handsheet evaluation (conventional sheet-former)	Bleached and beaten	ISO 5269-1:2005	ABNT NBR ISO 5269-1:2006	2

ABNT = *Associação Brasileira de Normas Técnicas* (Brazilian Association of Technical Standards).
ISO = International Organization for Standardization.
NBR = *Norma Brasileira* (Brazilian Standard).
NM = *Norma Mercosul* (Mercosul Standard).

4 APPLICATION

The laboratory interested in participating in this PT Scheme should fill in the Application Form included in the *Invitation Letter*. Also available on: (https://ipt.br/papel-e-celulose).

5 TEST ITEMS

5.1 Preparation

In each round, the laboratory receives, for each test subscribed, one pair of samples (**Sample A** and **Sample B**).

IPT ensures that every sample received by the participants has the same variability, because they are sent only after the verification of homogeneity. The parameters chosen for homogeneity check are shown below.

- Bleached pulp:
  - Beaten: drainability, *Schopper-Riegler*;
  - Unbeaten: Limiting viscosity number;
- Unbleached pulp: Kappa number.

To verify the homogeneity of samples, a defined number of specimens to be tested is extracted from both lots of samples A and B, considering the sort of pulp (unbleached and bleached) and its condition (beaten and unbeaten). The obtained values are treated by Analysis of Variance (ANOVA) single factor, whose result shows whether or not the lot is homogeneous.

5.2 Analysis and results sending

The participants perform tests in the samples received following the guidelines outlined in the Instruction Manual sent by IPT. In this manual is also indicated how participants should send their results to IPT.

The veracity of test results is a responsibility of the participant.

6 STATISTICAL ANALYSIS OF RESULTS

The statistical treatment applied to the participants' results aims to determine consensus values and evaluate laboratory performance, according to ISO 13528:2022 — *Statistical methods for use in proficiency testing by interlaboratory comparison*.

Calculations are performed using robust methods that reduce the influence of discrepant values, ensuring stable estimates representative of the dataset.

6.1 Group I

6.1.1 For tests with fewer than 6 participants

When there are fewer than six valid results, no robust statistical treatment is applied, as the reduced number of observations does not allow for reliable estimation of variability. In these cases, the results are presented in tables or graphs only, without performance statistics.

This procedure also applies to tests for which statistical treatment is not applicable (for example, surface strength – Dennison wax test).

6.1.2 For tests with six or more participants (robust Q/Hampel method)

For datasets with six or more valid results, the **robust Q/Hampel method** is applied, which combines two main estimators:

- **Robust standard deviation (s*)**: calculated using the **Qn estimator** (Croux & Rousseeuw, 1992), defined as the first quartile of all pairwise absolute differences multiplied by 2.2219. This estimator is resistant to up to 50 % of extreme values.
- **Consensus value (x*)**: calculated using the **Hampel M-estimator**, obtained iteratively from the median and weighted according to the robust dispersion s^*.

This procedure down-weights distant results and converges to a stable consensus value.

For each result xi, the **robust z-score** is computed as:

$$zi = \frac{xi - x^*}{s^*}$$

Interpretation of performance follows the criteria of ISO 13528:2022:

$|z| \leq 2$ → Satisfactory performance;

$2 < |z| < 3$ → Questionable performance;

$|z| \geq 3$ → Unsatisfactory performance.

6.1.3 Construction of the Youden Plot

Laboratory performance for samples A and B is represented graphically through the **Youden plot**. Each point on the plot corresponds to a laboratory, with coordinates representing its results for samples A (X Axis) and B (Y Axis).

The plot center is given by the consensus (x_A^**,x_B^*), obtained by the robust Q/Hampel method.

The joint dispersion of results is represented by a **95 % confidence ellipse**, constructed from the covariance matrix between samples A and B. The ellipse boundary corresponds to:

$$(x-\mu)'\sum^{-1}(x-\mu) = \chi^2_{2;0,95}$$

where $\mu = (x_A^*, x_B^*)$, and Σ is the covariance matrix, and $\chi^2_{2;0,95} = 5{,}991$ is the critical value of the chi-square distribution with 2 degrees of freedom.

In addition, the graph presents an elongated region in the direction of the largest eigenvector of Σ, representing the zone where systematic errors predominate.
Shifts approximately parallel to this direction indicate common trends between samples A and B (consistently high or low values).
Dispersions perpendicular to this direction characterize random error.

Rectangular zones centered at the consensus point, corresponding to ± 2σ and ± 3σ along each axis, are added to help interpret the magnitude of individual deviations, approximating the z-score.

The Youden Plot is generated individually for each test and allows immediate evaluation of:

- consistency between samples A and B
- presence of systematic trends
- random dispersion of results
- relative position of each participant compared to the consensus

6.2 Group II

6.2.1 Milling evaluation (PFI mill)

For each pair of test items received by the laboratory, the refining in PFI mill shall be performed to obtain four points of different refining degrees, in which the first one does not require refining. For each point, drainability is determined and seven handsheets are formed and sent to IPT together with refining data (number of revolutions and drainability). IPT determines tensile and tear indices for the sent handsheets.

The number of revolutions and the results received for tensile and tear indices are plotted in graphs against drainability and the values of these properties are interpolated for 38 SR using the regression equation corresponding to each graph. These interpolated values are tabled for tensile and tear indices.

6.2.2 Handsheet evaluation (conventional sheet-former)

From the pair of samples received by the participant, fourteen handsheets shall be formed. Seven of them stay at the participant laboratory and the remaining seven are sent to IPT. Both IPT and the participant laboratory perform the following tests: grammage; thickness; air permeance, *Gurley*; opacity; bursting strength; tensile strength and tearing resistance. The data obtained by IPT for each test are treated statistically according to item 6.1 for Group I and the data obtained by the laboratories are just tabled.

7 CONFIDENTIALITY

Absolut secrecy is ensured to the participant, that is identified by a code known only by itself and IPT. The documents issued by IPT contain only the codes of the laboratories an there is no information that can identify these laboratories.

NOTE Participants can choose to waive confidentiality within the proficiency testing program for the purpose of discussion and mutual assistance, for example, to improve performance. Confidentiality may also be waived by participants for the purpose of regulation or recognition. In most cases, the results its the proficiency test can be provided by the participants themselves to a competent authority.

When an interested party requires the results of the proficiency test to be directly provided by the proficiency test provider, it will only be possible after approval by the participant.

8 PRESENTATION OF PT SCHEME RESULTS

By the end of each round, the participant receives one custom-made report where it can observe its position in relation to the group of participant laboratories. The report shows information and comments for comprehension of results obtained and advices in case of non-satisfactory performances.

By the end of last round, the participant receives a document that summarizes its performance on this PT Scheme.

9 COMPLAINT

To register a complaint, the participant must contact the IPT Ombudsman's Office via email ouvidoria@ipt.br.

The Ombudsman's Office will receive the complaint and register it in the system designed for this purpose. The participant will be notified of the receipt of their manifestation, the actions that will be taken and the deadline for responding.

The Ombudsman will evaluate the origin of the complaint with the area complained of and will monitor the service until the process is completed and the problem is corrected.

After completing the process, the Ombudsman will contact the participant to check on their satisfaction.

10 APPEAL

To appeal against the performance evaluation in the program, contact by email interlab@ipt.br. The deadline for appeal will be 15 calendar days after the submission of the report.

The appeal will be sent to the Quality Representative who will receive and record the request on the form intended for this purpose. The participant will be notified of the receipt of their manifestation, the actions that will be taken and the deadline for responding.

The Quality Representative will evaluate the origin of the complaint and will monitor the service until the process is completed and the problem is corrected.

Upon completion of the process, the Quality Representative will contact the participant to check on their satisfaction.

11.1 Of activities

FIRST ROUND

STEP	MARCH				APRIL				MAY			
International samples dispatch	06											
National samples dispatch		13										
Performing of tests by the participant and sending of results to IPT							20					
Performing of tests on handsheets, formed by the participants, by IPT											22	
Composing of the report and sending to the participants												12/06

SECOND ROUND

STEP	JUNE				JULY				AUGUST			
International samples dispatch		12										
National samples dispatch			19									
Performing of tests by the participant and sending of results to IPT								27				
Composing of the report and sending to the participants												28

THIRD ROUND

STEP	SEPTEMBER				OCTOBER				NOVEMBER			
International samples dispatch	04											
National samples dispatch		11										
Performing of tests by the participant and sending of results to IPT							19					
Performing of tests on handsheets, formed by the participants, by IPT											19	
Composing of the report and sending to the participants											04/12	
Sending of performance summary and declaration of participation												11/12

11.2 Of payment

Charge	Months								
	Mar.	Apr.	May	Jun.	Jul.	Aug.	Sep.	Oct.	Nov.
Single payment									

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