



PROFICIENCY TESTING PLAN

ZVP 2019/1 – Mechanical Properties of Plastics
(ZZB 12390, 480-11, 731322, 731324, 731326)

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1 Basic Information about the Proficiency Testing Program

The aim of the Proficiency Testing Program (PTP) is to compare and evaluate the results of tests conducted on plastics in compliance with selected parts of ISO 527-1 [1], ISO 527-2 [2], ISO 178 [3], ISO 179-1 [4], ISO 868 [5], ISO 306 [6], ISO 75-1 [7], ISO 75-2 [8], ISO 1183-1 [9], ISO 11357-1 [10], ISO 11357-3 [11], ISO 1133-1 [12], ISO 1628-1 [13], ISO 1628-5 [14] and ISO 11358-1 [15].

The program strives to provide objective information about the measuring skills of PTP participants. This PT program is running in cooperation with UNIPETROL RPA, s.r.o. - POLYMER INSTITUTE BRNO, odštěpný závod. Within the PT program, POLYMER INSTITUTE BRNO prepares the testing specimens. The PT program is out of the range of accreditation of PT provider Z7008.

The basic criterion for participation is timely registration for the program, and the prerequisites for obtaining the Certificate of Participation and the Final Report on the Results of Interlaboratory Comparison are timely payment of the fee and adherence to the schedule.

Important dates:

Registration deadline:	04/30/2019
Distribution of samples:	06/10/2019 – 06/14/2019
Realization/initiation of testing:	06/17/2019
Results sent to the organizer:	07/31/2019
Evaluation/presentation of Certificate of Participation:	09/30/2019

2 Implementation of the Proficiency Testing Program

2.1 Specifications and Characteristics

Testing laboratories and other institutions interested can register for the PTP.

The minimum number of participants in every part of PT program is 5. If the number of participants is close to the minimum, the coordinator will consider the evaluation of PTP results using Horn's procedure to determine the relative values and the target standard deviation. In that case, the participants will be told before PTP materials are distributed.

The maximum number of participants is 30. If the minimum number of participants is not reached, the PT Provider reserves the right to cancel the PTP. This takes place according to Chapter 3 of the "Cancellation and Complaint Proceedings" instructions available on www.szk.fce.vutbr.cz/en. The program is usually implemented in order to specify the characteristics of plastics – see Table 1.

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Table 1: Specifications and characteristics of testing plastics

No.	Specification	Measured property	Unit	Specimens	Price (CZK)
1	ISO 527-1, -2	Tensile modulus	MPa	5	1500
2	ISO 527-1, -2	Stress at yield Strain at yield	MPa %	5 5	2000
3	ISO 527-1, -2	Stress at yield	MPa	5	1500
4	ISO 178	Flexural modulus	MPa	5	1500
5	ISO 178	Flexural strength Flexural strain at flexural strength	MPa %	5 5	2000
6	ISO 179-1	Charpy unnotched impact strength	kJ/m ²	10	1500
7	ISO 179-1	Charpy notched impact strength (note: notch made by distributor)	kJ/m ²	10	1500
8	ISO 179-1	Charpy notched impact strength (note: notch made by laboratory)	kJ/m ²	10	2000
9	ISO 868	Shore hardness D	°	5	1500
10	ISO 306	Vicat softening temperature VST/A/50	°C	3	1500
11	ISO 306	Vicat softening temperature VST/B/50	°C	3	1500
12	ISO 75-1, -2	Temperature of deflection under load, method A	°C	3	1500
13	ISO 75-1, -2	Temperature of deflection under load, method B	°C	3	1500
14	ISO 1183-1	Density	kg/m ³	3	1500
15	ISO 11357-1, -3	Melting temperature T _m Enthalpy of fusion H _m	°C J/g	3 3	2000
16	ISO 1133-1	Melt mass-flow rate	g/10min	3	1500
17	ISO 1628-1, -5	Viscosity	ml/g	3	1500
18	ISO 11358-1	Filler content	%	3	1500

2.2 Notes

- Tensile modulus:** Test specimen type 1A; test speed $v = 1$ mm/min; contact or optical extensometer.
- Stress at yield, strain at yield:** Test specimen type 1A; test speed $v = 50$ mm/min; contact or optical extensometer; limit deformation 20 %.
- Stress at yield:** Test specimen type 1A; test speed $v = 50$ mm/min; without extensometer; up to nominal deformation 20 %.
- Flexural modulus:** Middle part of test specimen type 1A (80x10x4) mm; test speed $v = 2$ mm/min, span between the specimen supports $L = 64$ mm.
- Flexural strength, flexural strain at flexural strength:** Middle part of test specimen type 1A (80x10x4) mm; test speed $v = 2$ mm/min, to measure up to deflection 12 mm, span between the specimen supports $L = 64$ mm.
- Charpy unnotched impact strength:** Test specimen type 1 (80x10x4) mm; without notch; preferentially use pendulum with energy 2 J (alternatively 1 J or 4 J); edgewise; span between the specimen supports $L = 62$ mm.

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7. **Charpy notched impact strength** (note: notch made by distributor): Test specimen type 1 (80x10x4) mm; notch type A (notch tip radius 0.25 mm, remaining width at notch tip 8 mm); preferentially use pendulum with energy 1 J (alternatively 0.5 J or 2 J); edgewise; span between the specimen supports $L = 62$ mm.
8. **Charpy notched impact strength** (note: notch made by laboratory): Test specimen type 1 (80x10x4) mm; distributed without notch - notch will make laboratory: notch type A (notch tip radius 0.25 mm, remaining width at notch tip 8 mm); preferentially use pendulum with energy 1 J (alternatively 0.5 J or 2 J); edgewise; span between the specimen supports $L = 62$ mm, send 1 notched and not tested specimen to POLYMER INSTITUTE BRNO for checking quality of notching.
9. **Shore hardness D** : Test specimen (20x20) mm; time-interval 15 s.
10. **Vicat softening temperature VST/A/50**: Test specimen (20x20) mm; load 10 N; temperature uniform rate 50 °C/h; temperature of the heating equipment shall not be higher 40 °C at the start of each test.
11. **Vicat softening temperature VST/B/50**: Test specimen (20x20) mm; load 50 N; temperature uniform rate 50 °C/h; temperature of the heating equipment shall not be higher 40 °C at the start of each test.
12. **Temperature of deflection under load, method A**: Narrow central part of the multipurpose test specimen A1 (80x10x4) mm; flexural stress at the test-specimen surface 1.80 MPa; start temperature of heating equipment shall be 25 °C; temperature uniform rate 120 °C/h; edgewise; span between lines of contact of the test specimen and the specimen supports $L = 64$ mm.
13. **Temperature of deflection under load, method B**: Narrow central part of the multipurpose test specimen A1 (80x10x4) mm; flexural stress at the test-specimen surface 0.45 MPa; start temperature of heating equipment shall be 25 °C; temperature uniform rate 120 °C/h; edgewise; span between lines of contact of the test specimen and the specimen supports $L = 64$ mm.
14. **Density**: Test specimen (30x10x4) mm; method A-immersion method; recommended immersion liquid - ethanol; temperature 23.0 °C.
15. **Melting temperature T_m , enthalpy of fusion ΔH_m** : Compression molded film (thickness 0.4 mm); purge gas - nitrogen; heat from 50 °C to 180 °C at rate 10 °C/min; crucible closed with lid; baseline: from 60°C till 150°C.
16. **Melt mass-flow rate**: Granules; nominal load 2.16 kg; test temperature 230 °C; time of measurement from 5 s up to 10 s.
17. **Viscosity**: PET granules; solvent is mixture of phenol/1,2-dichlorobenzene (weight out 1 part by mass of phenol (analytical grade) and dissolve in 1 part by mass of 1,2-dichlorobenzene(analytical grade). Work to an accuracy of 1 % or better in the weightings.); warming and dissolving at temperature 70°C; measure viscosity at temperature 25°C.
18. **Filler content**: Central part of test specimen type 1A; material: PP + glass fibers. Procedure:
 - purge gas - nitrogen, heat from 23 °C to 520 °C at rate 40 °C/min,
 - purge gas - change nitrogen to air at 520 °C; heat from 520 °C to 750 °C at rate 20 °C/min
 - purge gas - air: heat from 750 °C to 850 °C at rate 10 °C/min.

2.3 Ensuring Homogeneity and Stability

PT Provider employees and any suppliers whose services they may utilize are aware of the significance of the homogeneity and stability of test specimens for the results of the Proficiency Testing Program. The homogeneity and stability of specimens is ensured in the following ways:

1. the material used for the production of samples is always taken from the same production and is of the same production date; and/or
2. by dividing the specimens produced in different batches in order to ensure specimen homogeneity during testing of physical-mechanical and durability properties,

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3. using a single type of mold-release preparation,
4. using a single type of molds from the same material for one type of test,
5. storing all specimens together under identical conditions,
6. checking all specimens before dispatching to participants.

2.4 Instructions for the Elimination of Main Error Sources

PTP participants have the obligation:

- testing specimens will be conditioned according to EN ISO 11403-1,
- to handle the proficiency testing materials in the same way they handle the majority of routinely tested samples,
- to follow the instructions of the PT Provider employee responsible for the PTP, especially regarding the type of testing carried out, the number of result determinations and the PT schedule,
- to state measurement uncertainties in accordance with their documented procedures, including the corresponding expansion coefficient. Participants will use expansion coefficient 2, which approximately represents the 95
- to adhere to the rules and principles of ethical conduct, as well as to regulations governing health and safety at work and fire safety, and to use exclusively electrical devices and facilities with a valid inspection report,
- to send the test results obtained during proficiency testing, including measurement uncertainties, to the PT Provider by the set deadline the participant received in the confirmation e-mail.

2.5 PTP Schedule

All other information, forms and records not included in this document are accessible in updated form at ptprovider.cz.

3 Procedures used in the Statistical Analysis of Laboratory Results

To describe the accuracy of measuring methods, the terms trueness and precision are used. Trueness refers to the closeness to congruity between the arithmetic mean of a high number of test results and a real or accepted reference value. Precision means the closeness to congruity between test results. The necessity to consider precision is based on the fact that tests generally do not yield the same results even though they are supposed to be carried out on the same material and under the same conditions. This is caused by accidental errors that are impossible to avoid. These errors represent an integral part of every testing procedure and we are unable to control them fully. The comparative analysis of laboratory data does not focus on assessing the trueness of test results, but first and foremost on their precision. Results are thus compared with one another and not with any reference value or real value.

The basis of the statistical analysis is a critical data assessment complying with ISO 5725-2 [16], i.e. the determination of dubious and outlying values, and other irregularities. This assessment is carried out using mainly Grubbs' and Cochran's tests (numerical evaluation) as well as Mandel's statistics (graphical evaluation). Other observed statistical parameters are interlaboratory dispersion, repeatability dispersion, reproducibility dispersion and related characteristics of repeatability and reproducibility. The outcome of PTP is to assess the performance of participating laboratories in compliance with EN ISO/IEC 17043 [17], consisting of the determination of relative values and their uncertainties and a final comparison with the test results of PTP participants.

A prerequisite for using these methods is the unimodal probability distribution of measured data. Furthermore, p will stand for the number of participating laboratories marked by the index A prerequisite for using these methods is the unimodal probability distribution of measured data. Furthermore, p will stand for the number of participating laboratories marked by the index $i = 1, \dots, p$, each of which carried out n number of tests., each of which carried out n number of tests.

3.1 The Numerical Procedure for Determining Outliers

To determine outliers, two basic statistical tests are used. One of them is Cochran's C test, which tests interlaboratory variabilities (in cases when the number of measurements of one quantity in one laboratory > 2) and is used first. If this test marks one participant's results as outlying, the laboratory is excluded and the test repeated. The second test (Grubbs' test) is first and foremost a test of interlaboratory variability and we can also employ it if Cochran's test raises the suspicion that only one of the test results is to blame for the high interlaboratory dispersion. Both tests assume a balanced experiment, i.e. the number of tests at one laboratory for the determination of one quantity must be constant.

When determining divergent or outlying values, three situations can occur:

- If the test statistic is within or equal to 5% of the critical value, the tested entity is considered to be *correct*;
- If the test statistic diverges from the critical value by more than 5%, but is within or equal to 1% of the critical value, the tested entity is considered to be *divergent*;
- If the test statistic diverges from the critical value by more than 1%, the tested entity is considered to be *outlying*.

3.1.1 Cochran's test

The Cochran's C statistic is given by the equation:

$$C = \frac{s_{max}^2}{\sum_{i=1}^p s_i^2} \quad (1)$$

where s_{max} is the highest sample standard deviation, s_i are sample standard deviations determined according to the results from all laboratories and p means the number of laboratories participating in the PT program.

The sample standard deviation is determined from the equation

$$s_i = \sqrt{\frac{1}{n_i - 1} \sum_{k=1}^{n_i} (y_k - \bar{y})^2}, \quad (2)$$

where n_i is the number of test results from the determination of one quantity in i -th laboratory, y_k is the k -th value and \bar{y}_i is the average value measured in the i -th laboratory. If only two results were measured for the relevant quantity, we can use the simplified equation:

$$s_i = \frac{|y_1 - y_2|}{\sqrt{2}}. \quad (3)$$

3.1.2 Grubbs' test – One Outlying Observation

From the given set of x_i data for $i = 1, 2, \dots, p$, ordered upward according to size, Grubbs' statistic G_p is calculated in order to use Grubbs' test to determine whether the largest observation is an outlier:

$$G_p = \frac{x_p - \bar{x}}{s}, \quad (4)$$

whereby \bar{x} is the arithmetic mean of the observed feature. The observed feature can be the average value of the quantity determined within the laboratory. Furthermore, s is a sample standard deviation of the observed feature, which in this case is a standard deviation calculated for all the laboratories.

For significance testing of the smallest observation the test statistic is calculated:

$$G_p = \frac{\bar{x} - x_p}{s}. \quad (5)$$

3.2 Mandel's Statistics

In order to determine data consistency, two values called Mandel's h and k statistics were used. These indicators are commonly used for the graphical evaluation of laboratories in a similar way to a description of variability.

3.2.1 Interlaboratory Consistency Statistic h

For each laboratory, the interlaboratory consistency statistic h was evaluated according to the formula

$$h_i = \frac{\bar{y}_i - \bar{\bar{y}}}{\sqrt{\frac{1}{p-1} \sum_{i=1}^p (\bar{y}_i - \bar{\bar{y}})^2}}, \quad (6)$$

where \bar{y}_i is the average value for the i -th laboratory, $\bar{\bar{y}}$ is the arithmetic mean of all values and p is the number of laboratories. The values of the h_i statistics were plotted on graphs.

3.2.2 Intralaboratory Consistency Statistic k

The intralaboratory consistency statistic k is calculated from the equation

$$k_i = \frac{s_i \sqrt{p}}{\sqrt{\sum_{i=1}^p s_i^2}}, \quad (7)$$

where s_i is a sample standard deviation of values measured at the i -th laboratory. Just as with h statistics, the k values are plotted on graphs.

Study of the graphs displaying h and k values may indicate that certain laboratories show a significantly different ordering of results than other studied laboratories. This is caused by a permanently large and/or permanently small dispersion of results or extreme averages of results across all levels.

3.3 Calculation of Variances Estimates

After the elimination of outliers (of laboratories), we can proceed to the calculation of basic variability characteristics, i.e. repeatability dispersion, interlaboratory dispersion and reproducibility dispersion. These characteristics are stated in the form of standard deviations, i.e. after extracting the root. It is advantageous when the variability characteristics and the observed quantity are of the same physical dimensions.

3.3.1 Repeatability Variance

$$s_r^2 = \frac{\sum_{i=1}^p (n_i - 1) s_i^2}{\sum_{i=1}^p (n_i - 1)} \quad (8)$$

3.3.2 Interlaboratory Variance

$$s_L^2 = \frac{s_d^2 - s_r^2}{\bar{n}}, \quad (9)$$

where

$$s_d^2 = \frac{1}{p-1} \sum_{i=1}^p n_i (\bar{y}_i - \bar{\bar{y}})^2 \quad (10)$$

and

$$\bar{n} = \frac{1}{p-1} \left[\sum_{i=1}^p n_i - \frac{\sum_{i=1}^p n_i^2}{\sum_{i=1}^p n_i} \right]. \quad (11)$$

3.3.3 Reproducibility Variance

$$s_R^2 = s_r^2 + s_L^2, \quad (12)$$

where s_r^2 is repeatability variance and s_L^2 is interlaboratory variance.

3.4 Repeatability and Reproducibility

Repeatability expresses the fact that the difference between two test results from the same sample from tests carried out by the same person at the same facility and within the shortest time interval possible will not exceed the repeatability value r on average more than once in 20 cases if the method is employed in the common and correct manner.

The repeatability value is expressed by the relation

$$r = 2, 8s_r, \quad (13)$$

where $s_r = \sqrt{s_r^2}$ stands for the standard deviation of repeatability.

Reproducibility expresses the fact that the reproducibility value R for test results from one sample obtained in the shortest time interval possible by two persons who used their own devices will not differ on average more than once in 20 cases if the method is employed in the common and correct manner.

The reproducibility value is expressed by the relation

$$R = 2, 8s_R, \quad (14)$$

where $s_R = \sqrt{s_R^2}$ stands for the standard deviation of reproducibility.

3.5 Assigned Values

The PT Provider will ensure the determination of assigned value X and its uncertainty for every PTP. Assigned values are always only imparted to PTP participants after they have submitted their PTP results so that they cannot obtain any benefit from the premature revelation of the values.

The assigned values are determined by the PT Provider as consensual values derived from the results of participants in compliance with Appendix B of EN ISO/IEC 17043 [17] using the statistical methods described in ISO 13528 [18] and ISO 5725-5 [19]. The assigned value X is therefore determined as a robust estimate of the average value x^* (the A algorithm mentioned in [18] and [19]):

Initial values x^* and s^* (robust standard deviation) are calculated as

$$x^* = \text{median } x_i, \quad (15)$$

$$s^* = 1, 483 \cdot \text{median } |x_i - x^*|, \quad (16)$$

where $i = 1, \dots, p$. The values of x^* and s^* are then processed as follows. First, $\varphi = 1, 5 \cdot s^*$ is computed. For every x_i ($i = 1, \dots, p$) value, the following is calculated

$$x_i^* = \begin{cases} x^* - \varphi & \text{if } x_i < x^* - \varphi, \\ x^* + \varphi & \text{if } x_i > x^* + \varphi, \\ x_i & \text{in other cases.} \end{cases} \quad (17)$$

New values of x^* and s^* are calculated from the following equations

$$x^* = \sum_{i=1}^p \frac{x_i^*}{p}, \quad (18)$$

$$s^* = 1, 134 \cdot \sqrt{\sum_{i=1}^p \frac{(x_i^* - x^*)^2}{p - 1}}. \quad (19)$$

Robust estimates are derived by iteration until the estimate changes between calculations become small.

The standard uncertainty u_X of an assigned value determined in this manner is calculated from the relation

$$u_X = 1, 25 \cdot \frac{s^*}{\sqrt{p}}. \quad (20)$$

In the case of a small number of PTP participants, the PT Provider sets the assigned values as consensual values obtained from expert participants who have proven their competence to determine the measured quantity that is the subject of testing.

3. PROCEDURES USED IN THE STATISTICAL ANALYSIS OF LABORATORY RESULTS

Furthermore, if the number of participants is small ($4 \leq p \leq 20$), the PT Provider can consider determining the relative values by using what is called **Horn's method**. This method consists in the determination of so-called pivots used as a basis for estimating location and variability. First, the assessed data are ordered upwards. The low pivot is then determined from the equation

$$x_D = x_{(H)}, \quad (21)$$

where H is an ordinal index given by the equation $H = \frac{\text{int}(\frac{p+1}{2})}{2}$ or $H = \frac{\text{int}(\frac{p+1}{2}+1)}{2}$.

The upper pivot is then determined from the equation

$$x_H = x_{p+1-H}. \quad (22)$$

Using Horn's method, the assigned value is determined as a location estimate, i.e. as the so-called pivot half sum:

$$x^* = \frac{x_D + x_H}{2}. \quad (23)$$

The variability estimate is determined as the so-called pivot range

$$R_L = x_H - x_D \quad (24)$$

and the uncertainty of an assigned value calculated in this way is determined as a 95% interval estimate of the mean value

$$u_X = R_L \cdot t_{L;0,95}(p), \quad (25)$$

where $t_{L;0,95}(p)$ is the $(1 - \alpha)$ quantile of the T_L probability distribution with p degrees of freedom.

3.6 Calculation of Performance Statistics

Proficiency test results often need to be transformed into performance statistics in order to aid interpretation and to allow comparison with defined objectives. The aim is to express the divergence from the assigned value in a way that enables its comparison with performance criteria. In compliance with the EN ISO/IEC 17043 standard [17], the performance of participating laboratories is evaluated according to the so-called z-score and ζ -score (zeta-score).

For every non-outlying laboratory (participant), the z-score is calculated according to the equation

$$z_i = \frac{|\bar{x}_i - x^*|}{s^*}. \quad (26)$$

ζ -score is calculated using the equation

$$\zeta_i = \frac{|\bar{x}_i - x^*|}{\sqrt{u_i^2 + u_X^2}}, \quad (27)$$

where u_i is a combined standard uncertainty of the i -th laboratory. Combined standard measurement uncertainties can be arrived at by dividing the extended uncertainty U by the extension coefficient k , which for normal probability division has the value $k = 2$. If the participant does not state the extended measurement uncertainty in their test result protocol, it is impossible to determine the ζ -score. For more about measurement uncertainties see document [20].

The following scales are applied for the z-score and ζ -score (to simplify the matter, only the z-score is shown):

$$z\text{-score} = \begin{cases} |z| \leq 2 & \text{shows that the laboratory performance is } \mathbf{satisfactory} \text{ and generates no signal;} \\ 2 \leq |z| \leq 3 & \text{shows that the laboratory performance is } \mathbf{questionable} \text{ and generates an action signal;} \\ 3 \leq |z| & \text{shows that the laboratory performance is } \mathbf{unsatisfactory} \text{ and generates an action signal.} \end{cases} \quad (28)$$

4 Certificate of Participation and the Final Report on the Results of Interlaboratory Comparison

The PT Provider gives expert commentary on participant efficiency evaluation in the Final Report as part of training courses the PT Provider organises. The Final Report preserves the anonymity of the PTP participants. Each participant, or the participant's test results, is represented by an ID number. The Certificate of Participation in the PT programme is part of the Final Report. The Certificate is unique to each participant and includes the participant's ID number.

5 Safeguards for Confidentiality

The identity of PTP participants is confidential and only known to persons/subjects involved with the PTP. All participant information is considered confidential by the PT Provider. The participant may renounce this confidentiality for the purposes of discussion and mutual assistance until the PTP results are obtained. The PT Provider reveals the proficiency testing results to no third party with the sole exception of a written request by a regulatory authority submitted prior to the commencement of the PTP and which has been granted a written consent by the PTP participants.

6 Related Documents

- Quality Handbook of the PT Provider at the SZK FAST
- Cancellation and Complaint Proceedings available at www.szk.fce.vutbr.cz [21]
- MPA 20 – 01 - . . . for application of EN ISO/IEC 17043 Concordance Assessment – General Requirements for Proficiency Testing in the Accreditation System of the Czech Republic.

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