SNAS SLOVENSKÁ NÁRODNÁ AKREDITAČNÁ SLUŽBA

METHODICAL GUIDELINE FOR ACCREDITATION

DETERMINATION OF THE LEVEL AND FREQUENCY OF THE PARTICIPATION IN PROFICIENCY TESTING

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CONTENT

1 INTRODUCTION

The purpose of this MSA is to provide the instruction for the laboratories how to determine the level and frequency of participation in proficiency testing or other types of interlaboratory comparisons that can be used for the purpose of proficiency testing.

2 ABBREVIATIONS USED

APLAC	Asia Pacific Laboratory Accreditation Cooperation
EA	European Co-operation for Accreditation
ILAC	International Laboratory Accreditation Cooperation
IRMM	The Institute for Reference Materials and Measurements
MP	Interlaboratory comparison
MSA	Methodical guideline for accreditation
QA	Providing of quality of results from testing and/or calibrations
PT	Proficiency testing (skúška spôsobilosti)
SM	Management system

Terminology:

Proficiency testing (PT)
evaluation of the performance of participant of testing against the set in advance criteria by means of interlaboratory comparisons.

Interlaboratory testing:
Organizing, carrying out and evaluation of either measurements or testing of the identical or very similar objects by two or more laboratories according to conditions set in advance.

Measuring technique
process of testing/calibration/identifying of the properties, including any preparatory activities required for the current sample received by the laboratory, up to the measuring device (i.e. ICP-MS, PCR, microscopy, measuring of hardness, etc.).

Property
measured parameter, indicator or value (i.e. arsenic, fat, creatinine, length, hardness, etc.).

Object
Material/environment the measuring technique is applied to (e.g. soil, air, vegetables, serum, concrete, working environment, etc.).

Level of participation

number of subareas the organization identifies within the framework of its scope of accreditation (specification of activities) and the resulting number of specific proficiency tests the laboratory should consider its participation in.

Frequency of participation
is a time interval during which the organization takes part in the proficiency testing in one subarea (e.g. once a year, once within the accreditation cycle, etc.). The frequency can be different for various subareas within the laboratory and also among the laboratories within the same subareas.

Subarea
a part of the scope of accreditation, defined at least by one measuring technique, property and object that are related (e.g. determination of arsenic in the soil by means of ICP-MS method).

Area of professional activity
a part from the scope of accreditation, usually defined by one professional competence. The professional competence is usually identified by the need for relevant qualification, training and the use of relevant equipments, knowledge and skills (e.g. microbiology, analytic chemistry, non-destructive testing, measuring of physical factors of the environment, etc.).

3 RELATED DOCUMENTS

ISO/IEC 17025:2005 – General requirements on the competence of testing and calibration laboratories
ISO 15189:2012 - –Medical laboratories – Requirements on the quality and competence
EA-4/18: 2010 – Instructions to determine the level and frequency of participation in proficiency testing
PL- 23: SNAS policy for participation in proficiency testing

4 GENERAL

When evaluating the suitability of the level and frequency of participation in proficiency testing, SNAS takes into account certain aspects below:

1. The level and frequency of participation in proficiency testing should be determined based on the consistent analysis of further activities within ensuring of the quality of results (especially those able to disclose, quantify and follow the development of deviations of the defined size). The participation should take into consideration the extent of application of other QA procedures. Further QA procedures involve. But are not limited to:

    - regular use of (certified) reference materials,

- comparison of measurements/analysis by means of independent techniques/methods,
- participation in the development and validations of methods and/or studies aimed at the characterization of reference materials,
- use of the means of internal quality management,
- other inter/intra-laboratory comparisons, e.g. analysis of standard samplesthe characteristics of which are not known to the operator.

2. The level of risk presented by the laboratory, sector in which it oerates or methodology it uses. SNAS here takes into consideration the lolliwing factors influencing the laboratory activities:

   - number of carried out testing/calibrations/measurements,
   - fluctuation of professional workers,
   - skills and knowledge of the professional workers,
   - sources of traceability (e.g. accessibility of reference materials, national etalons, etc.),
   - knowledge of stability or instability of the measuring technique used,
   - significance and end use of testing/calibration data (e.g. forensic sciences represent the area requiring a high level of trust).

3. SNAS accepts the participation of the laboratory in various types of PT, including the:

   - PT organized by other independent organizations, such as accreditation bodies or ILAC, EA, APLAC and IRMM,
   - inter-laboratory comparison organized with a sufficient number of laboratories whether for one case or continuously,
   - sending of the internal sample or object into another or several external laboratories for the purpose of comparison of data.

4. SNAS is aware of the fact that the participation in PT can be problematic for certain sectors of activities due to the technical characteristics of measurement, character of objects, inaccessibility of PT programs, low number of laboratories in the relevant sector, etc. In some areas the PT can be accessible or economically viable only for one part of carried out testing/calibrations (e.g. EMC testing of the simple object for the limited number of measured values, etc.). In such cases other procedures are important of securing and management of quality.

5. In the case when the requirements on the level and frequency of participation in PT are determined by legislation, SNAS keeps to them.

5 LEVEL AND FREQUENCY OF PARTICIPATION

The first step to determine the level and frequency is to identify the subareas covered by the scope of accreditation.

Ideally the laboratory should participate in specific PT for each measuring technique it uses and for each property and on each object. However, SNAS accepts the fact that such approach would be impossible to realize from both logical and economical point of view. Due to this reason the laboratories have to define the subareas in which it is possible to apply the quality of the results obtained by partcipating in one PT to other techniques, properties and objects within one subarea (see the definition of subarea).

The subarea can contain more than one measuring technique, property and object provided it is possible to prove their equivalency and compatibility. To define the subareas, the first consideration of the laboratory should be that this subarea will not contain any different professional competences (areas of professional activities). Different professional competences are usually identified by the need for different qualification, training and use of different equipments, knowledge and skills.

When defining the subarea, it is appropriate to use the sequence system namely from the measuring technique through properties up to the object. This is due to the higher probability of several products and/or properties being grouped which are linked with one technique rather than vice versa.

(i) With reference to **the technique of measuring**: it is possible, however it is not usual to include different measuring techniques into the same subarea.
(ii) With reference to the measured, determined or identified **property**: It is possible to include more than one property (parameter) into the same subarea.
(iii) With reference to the tested **object**: It is possible to include different objects in the same subarea provided the matrix or materials are equivalent.

If the laboratory determines more than one measuring technique, property or object, SNAS evaluates whether the laboratory is able to prove satisfactorily their equivalency. It can be done by mean of:

- outputs from method validation, or
- using of the same standard method.

By defining the subareas the laboratory determined the “level of participation”. SNAS evaluates also the suitability of the “frequency” of participation with regard to the risks and aspects described in the part 4. The laboratory defines the minimum requirements on the frequency of participation for all subarea; however these can´t be more benevolent than the minimum requirements determined for the participation in PT in PL-23.

In accordance with the requirement 5.9.1 of the standard ISO/IEC 17025: 2005, the laboratory shall have the quality management procedures due to the monitoring of the validity of tests and calibrations carried out. The participation in PT makes part of this monitoring too. These procedures and monitoring must be planned. Due to this reason, when the level and frequency of participation are determined, the laboratory must elaborate its strategy of participation in PT involving the aspects defined in the article 4.

The extent and content of this strategy depends on the circumstances and scope of accreditation of the individual laboratories.

The strategy of participation in PT must be prepared for one accreditation cycle, it means between the accreditation and reaccreditation, or between two subsequent reaccreditations. The strategy shall be verified and reviewed once a year and it is recommended to do so during the management review.

With regard to the fact that the division of the scope of accreditation into individual subareas and frequency of participation can differ from subject to subject, the laboratories shall have documented the professional arguments to explain the decisions in relation to the participation in PT. These arguments shall be documented.

6 CASE STUDIES

The laboratory is responsible for breaking its scope of accreditation down into subareas (if possible) and defining of the level and frequency of participation in the PT that will be specified more in detail in the strategy of participation in PT. In the following text there are several case studies to illustrate how the laboratory can break down the scope of accreditation into individual subareas. However, these are only examples and not strict and definitive solutions. The strategy of participation in PT is reviewed by SNAS.

Case study 1 - Chemical testing laboratory in the area of environment

Accredited activities carried out by the laboratory:

- Polychlorinated biphenyls (PCB) carried out GC-MS in soils and sludge,
- Polyaromatic hydrocarbons (PAU) carried out GC-MS in soils and sludge,
- Volatile organic substances (VOC) carried out „Purge and Trap“ GC-MS in water,
- Metals carried out ICP-MS in soils, sludge and water,
- pH in the soil, sludge and water.

Factors to be taken into consideration when determining the subareas:

In the case of measuring of pH the laboratory states that it uses the same method according to the ISO standard for all three matrixes (soil, water and sludge). This ISO method is validated against all three matrixes and therefore it is indicated as one subarea.

For the analysis of metals the laboratory indicates that it uses the same measuring technique (ICP-MS) for all three matrixes (soils, water and sludge). Despite this fact, the preparation of testing samples of water is significantly different from the preparation of samples of soil and sludge. In such case the laboratory can’t declare this activity as one subarea although the methodology of soils and sludge is demonstrably similar and therefore included into one subarea. For this reason the laboratory has to determine two subareas.

For the analysis PAU and PCB the laboratory indicates the use of the same measuring technique (GC-MS) and the extraction of matrixes is identical (soils and sludge). However, according to the primary validation of the method it is obvious that PCB and PAU are influenced in different ways when the methodology is changed. Therefore the acceptable or problematic proficiency for PCB doesn´t necessarily mean the same for PAU (or vice versa). For this reason the laboratory identifies two further subareas.

For the methods for VOC the laboratory takes into consideration only one matrix, namely the water. The laboratory is conscious that the methods of analysis of various properties of water may potentially react differently. By validating its method of analysis the laboratory demonstrates that when changing the given method, various properties react in comparable way. For this reason the laboratory considers one subarea.

The resulting subareas from the above procedure are as follows:

- Polychlorinated biphenyls (PCB) carried out GC-MS in soils and sludge,
- Polyaromatic carbohydrates (PAU) carried out GC-MS in sols and sludge,
- Volatile organic substances (VOC) „Purge and Trap“ GC-MS in water,
- Metals carried out ICP-MS in soils and sludge,
- Metals carried out ICP-MS in water,
- pH in soils, sludge and water.

Case study 2 -
Microbiology testing laboratory

Accredited activities carried out by the laboratory:

- Detection of *Escherichia coli* in meat,
- Detection of salmonella in meat,
- Detection of *Escherichia coli* in vegetables,
- Detection of salmonella in vegetables,
- Detection of *Escherichia coli* v dairy products,
- Detection of *Escherichia coli* in drinking water,
- Detection of *Escherichia coli* in swimming pool water.

Factors to be taken into consideration when determining the subareas:

When determining the number of *Escherichia coli* the laboratory identifies that it uses the same method to analyse the samples of meat and vegetables. This method was validated for both these matrixes and therefore the laboratory identifies these cases as one subarea only. As this was not validated for dairy products, the laboratory uses a different method for these sample types. For this reason there is another subarea determined for this case.

The method the laboratory uses to determine the number of salmonellas is different from the one used for *Escherichia coli*. However, this method was validated for meat and vegetables and for this reason the laboratory determines this case as another subarea.

Although various methods of sampling and processing of samples are used to detect the *Escherichia coli* in water, the method used (different from the method used for foodstuff) was validated for both drinking and swimming pool water and therefore this case is determined as another subarea.

The subareas resulting from the above procedure are the following:

- Detection of *Escherichia coli* in meat and vegetables,
- Detection of *Escherichia coli* in dairy products,
- Detection of salmonella in vegetables and meat,
- Detection of *Escherichia coli* in drinking and swimming pool water.

Case study 3 -
Clinical testing laboratory

The accredited activities carried out by the laboratory:

- Screening of occurrence of drugs in the blood by means of the method ELISA and fluid EIA,
- Screening of occurrence of drugs in the urine by means of the method ELISA and fluid EIA,
- Evidence of amphetamine in the blood by means of GC-MS,
- Evidence of amphetamine in the urine by means of GC-MS,
- Evidence of codeine in the blood by means of GC-MS,
- Evidence of codeine in the urine by means of GC-MS,
- Evidence of diazepam in the blood by means of LC-GC-MS,
- Evidence of diazepam in the urine by means of LC-GC-MS,
- Evidence of cocaine in the blood by means of LC-GC-MS,
- Evidence of cocaine in the urine by means of LC-GC-MS,
- Evidence of EDDP in the blood by means of LC-GC-MS,
- Evidence of EDDP in the urine by means of LC-GC-MS,
- Evidence of buprenorphine in the blood by means of GC-MS-MS,
- Evidence of buprenorphine in the urine by means of GC-MS-MS,
- Evidence of tetrahydrocanabinole in the blood by means of GC-MS-MS,
- Evidence of tetrahydrocanabinole in the urine by means of GC-MS-MS.

Factors to be taken into consideration when determining the subareas:

Two methods used for screening of the use of drugs are different but both of them were validated for investigation of blood and urine samples. In such case the laboratory determines two subareas.

Three techniques used for the evidence of use of diverse drugs are very different although each of them was validated for both matrixes, it means for the blood and urine. In addition it is assumed that various detection systems are included into independent subareas. The drugs, although they belong to various groups, are considered to be equivalent from competence point of view. For this reason the laboratory identifies that the tests to prove them belong to three complementary subareas.

The subareas resulting from the above are as follows:

- Screening of the use of drugs in the blood and urine by means of ELISA method
- Screening of the use of drugs in the blood and urine by means of fluid EIA method
- Evidence of amphetamine and codeine in the blood and urine by means of GC-MS*
- Evidence of diazepam, cocaine and EDDP in the blood and urine by means of LC-GC-MS*
- Evidence of buprenorphine and tetrahydrocanabinole in the blood and urine by means of GC-MS-MS*

Note:
Although with regard to the equivalence of various drugs from competence point of view these were put into one subarea, it doesn´t mean that they are equivalent from the point of view of the methods and laboratory activities. Therefore it is expected that the laboratory will participate on the regular basis on the PT for all drugs it has in the scope of accreditation. This fact must be analyzed more in detail in the strategy of laboratory participation in PT.

Case study 4 - Laboratory carrying out physical tests

Accredited activities carried out by the laboratory:

- Fracture toughness and increase of fatigue failure in metals and metal alloys (ASTM E 399),
- Tensile and pressure tests of materials from metals and metal alloys (e.g. EN 10002 part 1),
- Tensile and pressure tests of plastic materials (ISO 527-1),
- Hardness test according to Brinell (ISO 6506), according to Vickers (ISO 6507) and Rockwell (ISO 6508),
- Charpy's impact test according to ISO 148-1,
- Determination of grain size (thickness) (ISO643),
- Optical emission spectrometry (quantification of chemical elements in the steel matrixes; own procedure of the laboratory).

Factors to be taken into consideration when determining the subareas:

Many laboratories carry out the above activities in the area of mechanical tests. The methods are described in the ISO, EN or ASTM Standards. The standard usually specifies

the equipment necessary as well as other relevant and related parameters. The featured testing activities are carried out with the equipment of either same or different type requiring a specific calibration and specific knowledge of the staff to be able to perform the above tests.

The tests of fracture toughness and increase of fatigue failure are carried out with the same testing device and the method (ASTM E 399) was validated for metals and metal alloys. Therefore the laboratory identifies this activity as one subarea.

Tensile and pressure tests of materials from metals and metal alloys are based on one testing method. As the test for the increase of fatigue failure involves the capabilities of tensile and pressure tests, the laboratory identified that it didn't need to participate further in the additional tests of capability for metals and alloys (note: participation in the tests of capability in tensile and pressure tests doesn't need to be sufficient for testing of the increase of fatigue failure). Usually, specific testing equipment is used with application of different loading on either flat or round testing samples. The basic requirements are on the measuring of load, class 1 (±1%) and measuring of elongation (±1%). The calculation of the result of this testing method is in reality done by means of computer system adjusted either by the device producer or by the user who has an access to the software. In principle the given test of steel sample determines the strength and elongation. For specific materials, their behaviour and relevant results it is critical to work the sample.

Similar testing systems are used for pressure and tensile tests of plastic materials; however the power load is smaller. The complementary testing device is different with regard to higher tensibility of plastic materials. In addition to this, according to ISO 527 the definitions of parameters determined by testing differ. Testing device must be calibrated once a year, the use of reference materials is limited to a small number of laboratories. For this reason the laboratory identifies such test as further subarea, because it uses a different method.

The hardness tests according to Brinell (ISO 6506), Vickers (ISO 6507) are using as intrusive body the ball or pyramid in order to create a hole in the surface of metallic material. The dimensions are measured after this step of the diagonal of this hole and the hardness of the material is calculated. In the relevant standards from the range ISO 6506-1 a 6507-1 there is a description of the requirements on the direct calibration state of loading (load, intrusive body, device to measure the length). The calibration must be repeated once a year and the use is mandatory before testing of the certified reference materials from whence it follows that the laboratory defines another subarea for these two methods.

When determining the Rockwell hardness (ISO 6508-1) the measuring procedure is different from the one of Brinell or Vickers. According to ISO 6508 it is possible to use different intrusive bodies to create a hole in the measured material surface under the pre-defined loading conditions. By this test the depth of the intrusion is measured in a specific procedure. The ISO standard requires the calibration and use of the certified reference material. Due to this reason the laboratory identifies one more subarea.

The Charpy's impact test according to ISO 148-1 prescribes the sample dimensions. The testing device must be calibrated once a year and the standard requires a specific reference material for the indirect control of the adjustment of the whole equipment. The size is measured of the impact energy. The laboratory identifies it as other subarea.

The grain size determination (ISO 643) is done on the surface of steel sample prepared in a specific way, such as grinding, polishing, etching in order to show off the edge of grains in the tested material. The following step is to measure the grain dimensions under the microscope with calibrated magnification from which it is possible to calculate the relevant parameters in accordance with the procedure specified in the standard. The laboratory identifies this test as the next subarea.

The optical emission spectrometry is used in many laboratories to determine the steel alloys. To calibrate the equipment it is necessary to use the certified reference materials and secondary own standards. The laboratory identifies this activity as another subarea.

The subareas resulting from the above are as follows:

- Fracture toughness and increase of fatigue failure in metals and metal alloys,
- Tensile and pressure tests of plastic materials,
- Hardness test by Brinell or Vickers,
- Rockwell's hardness test,
- Charpy's impact test,
- Grain size determination,
- Optical emission spectrometry.

Case study 5 -
Taking into account of the matrix (Clinical chemistry)

Accredited activities carried out by the laboratory:

- FSH by means of chemiluminiscence in blood,
- LH by means of chemiluminiscence in blood,
- Folic acid by means of chemiluminiscence in blood,
- Calcium electrochemically in urine and blood,
- Potassium electrochemically in blood and urine,
- Cryoglobulins by means of electrophoresis in blood,
- Carbamazepine immunologically in blood,
- Cyclosporine immunologically in blood,
- Transferine nephelometrically in blood and urine,
- α2 macroglobulin nephelometrically in blood and urine,
- ALAT UV- by means of visible spectroscopy in blood,
- ASAT UV – by means of visible spectroscopy in blood,
- Magnesium UV – by means of visible spectroscopy in blood and urine.

Factors to be taken into consideration when determining the subareas:

The laboratory shall prepare the list of all its measuring techniques it uses within its scope of accreditation, list of all properties representing the individual parameters or groups of equivalent parameters and the list of all materials (matrixes) as mentioned below.

Measuring techniques:
Chemiluminiscence
Elektrochemistry
Electrophoresis
Immunology test
Nephelometry
UV-visible spectroscopy

Properties:
Medicaments (carbamazepine, cyclosporine)
Electrolytes (calcium, kalium, magnesium)
Enzymes (ALAT, ASAT)
Hormones (FSH, LH)
Specific proteins (cryoglobuline, transferine, α2 macroglobuline)
Vitamins (folic acid)

Materials
Blood
Urine

List of analysis:
The laboratory should link each parameter from the above list with one measuring technique, one property and object (material).

Parameter	Measuring technique	Property	Material
FSH	Chemiluminiscence	Hormons	Blood
LH	Chemiluminiscence	Hormons	Blood
Folic acid	Chemiluminiscence	Vitamins	Blood
Calcium	Electrochemistry	Electrolytes	Blood
Calcium	Electrochemistry	Electrolytes	Urine
Kalium	Electrochemistry	Electrolytes	Blood
Kalium	Electrochemistry	Electrolytes	Urine
Cryoglobulines	Electrochemistry	Specific proteins	Blood
Carbamazepine	Immunology test	Medicaments	Blood
Cyclosporine	Immunology test	Medicaments	Blood
Transferine	Nephelometry	Specific proteins	Blood
Transferine	Nephelometry	Specific proteins	Urine
α2 macroglobuline	Nephelometry	Specific proteins	Blood
α2 macroglobuline	Nephelometry	Specific proteins	Urone
ALAT	UV-visible spectroscopy	Enzymes	Blood
ASAT	UV-visible spectroscopy	Enzymes	Blood

Magnesium	UV-visible spectroscopy	Electrolytes	Blood
Magnesium	UV-visible spectroscopy	Electrolytes	Urine

Resulting matrix:

Based on the list of analysis the laboratory can specify the matrix that will clarify the subarea as mentioned below. If the number of materials (objects) is limited, they can be set into the matrix. If not, the evaluation of materials can be carried out separately.

Property Measuring technique	**Medicaments**		**Electrolytes**		**Enzymes**		**Hormones**		**Specific proteins**		**Vitamins**	
Material	K	M	K	M	K	M	K	M	K	M	K	M
Chemiluminiscence							x				x	
Electrochemistry			x	x								
Electrophoresis									x			
Immunology test	X											
Nephelometry									x	x		
UV-visible spectroscopy			x	x	x							

K - blood, M- urine

The resulting subareas from the above are as follows:

- Hormones by means of chemiluminiscence in blood,
- Vitamins by means of chemiluminiscence in blood,
- Electrolytes electrochemically in blood and urine,
- Specific proteins by means of electrophoresis in blood,
- Medicaments by means of immunology test in blood,
- Specific proteins nephelometrically in blood and urine,
- Electrolytes UV – visible spectroscopy in blood and urine,
- Enzymes UV – visible spectroscopy in blood.

Note:
Although various materials were linked as equivalent into one subarea for each detection system from competence point of view, it doesn't mean that they are equivalent from both method and activity of laboratory point of view. Due to the above it is expected from the laboratory to participate in such PT that cover especially all the objects (materials) and scope of accreditation. This fact must be stated in detail in the strategy of participation.

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