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MEĐULABORATORIJSKO USKLAĐIVANJE POSTUPAKA ISPITIVANJA FIZIKALNO KEMIJSKIH SVOJSTAVA

Sažetak

Sposobnost provedbe ispitivanja u ispitnom laboratoriju može se vrednovati putem ispitivanja osposobljenosti, tzv. Proficiency Testing Shemes – PTS. Te aktivnosti su, osim za procjenu osposobljenosti ispitnih i kalibracijskih laboratorija, također i dodatna tehnika u procesu ovlašćivanja laboratorija od ustanove za ovlašćivanje laboratorija prema normi EN 45001:1989.

Glavni ciljevi PT su održanje i povećanje tehničke osposobljenosti ispitnih laboratorija, te povećanje pouzdanosti i usporedivosti u procesu ispitivanja/kalibracije. Ako su rezultati provedenog PTS nezadovoljavajući, PT organizator uz predstavnika laboratorija mora pokušati pronaći razloge loše izvedbe PTS, a sam laboratorij mora poduzeti efektivne korektivne mjere da se poboljša kvaliteta ispitivanja. Postoje različiti tipovi PT shema, a njihov izbor ovisi o područjima ispitivanja, koja su od specifičnih interesa ustanova za ovlašćivanje i laboratorija koji sudjeluje u PT. Prije faze dizajniranja PT sheme, potrebno je definirati njezinu svrhu. Iskusni PT organizator trebao bi izabrati tehničkog koordinatora i savjetodavnu grupu, koja će mu pomoći u obavljanju prijeko potrebnih poslova prije nego što se PT shema distribuira laboratorijima sudionicima. Također je potrebno definirati politiku za sudionike u toj PT shemi, politiku za uporabu rezultata, politiku za povjerljivost, te definirati politiku kakvoće.

UVOD

ISPITIVANJE OSPOSOBLJENOSTI (Proficienciensy testing shemes - PTS) - primjena međulaboratorijskih usporednih ispitivanja za utvrđivanje načina izvedbi postupaka ispitivanja ili kalibracije u pojedinačnim laboratorijima. Također se nadziru podudarnost i usporedivost laboratorijskih ispitivanja i kalibracijskih podataka.

Cilj PT shema je poboljšati usporedivost u postupcima ispitivanja/ kalibracije, uvesti djelotvornost i usporedivost novih metoda ispitivanja, te pomoć u održavanju kalibracije instrumenata. Izvedbom PTS demostrira se osposobljenost na određenom polju ispitivanja što vodi do komercijalnog poboljšanja i dodatnog povjerenja kupaca. Analizom rezultata PT shema mogu se otkriti uzroci razlika u ispitivanjima između laboratorija, te predložiti efektivne korektivne radnje za njihovo uklanjanje. Jedna od mogućih primjena PTS je u procesu ovlašćivanja od ustanove za ovlašćivanje prema normi EN 45001:1989 (slika 1). U točki 6.3. tog standarda preporuča se suradnja sa drugim laboratorijima putem korištenja PTS. Međutim, rezultati PTS nisu jedina osnova za dodjelu ili održanje akreditacija nekog laboratorija. Ipak, u slučaju nezadovoljavajućih rezultata PTS može biti upitan proces ovlašćivanja. Ustanova za ovlašćivanje mora imati politiku za korištenje PTS. Izvedba PTS mora biti u skladu s ISO uputama 43-1.

TIPOVI PT SHEMA

Postoje različiti tipovi PTS, a izbor ovisi o prirodi uzorka ili materijala za ispitivanje, metodi ispitivanja i broju sudionika.

- Prema vrsti uzorka/materijala i metodi ispitivanja:

1. Shema mjerenja usporedivosti – uzorak za ispitivanje ili kalibraciju kruži od jednog do drugog laboratorija sudionika. Uzorak se periodički vraća u laboratorij koji organizira PTS radi provjere svojstava. Nedostaci ovog tipa PTS jest to što je potrebno veće vremensko razdoblje za završetak, a u svezi s tim se javljaju problemi sa stabilnošću uzorka, kao i problemi s nadgledanjem cirkulacije uzorka.
2. Shema međulaboratorijskog ispitivanja – uzorak se homogenizira i selektira u poduzorke koji se distribuiraju sudionicima PT sheme. Svaki sudionik obavi potpunu analizu uzorka. Rezultati ispitivanja se vraćaju laboratoriju organizatoru i koordinatoru na obradbu, nakon čega se sudionici izvještavaju o rezultatima PTS.
3. Shema ispitivanja podijeljenog uzorka – uzorci materijala za ispitivanje

podijele se na dva ili više dijelova. Svaki laboratorij sudionik ispituje jedan dio svakog uzorka. Ostale dijelove istog uzorka ispituju drugi laboratoriji sudionici. Za razliku od drugog tipa PTS ova shema ima obično limitiranu kontrolu, a homogenost uzorka se mora dodatno provjeravati.

- Prema broju sudionika:

1. Multilateralna PT shema – u njoj sudjeluje više laboratorija sudionika i ona služi kao alat u postupcima ovlašćivanja u skladu s ISO uputama 43-1, kao minimumu. Cilj je da sudionici usporede vlastitu izvedbu s referentnim laboratorijima ili laboratorijima sa sličnim područjima ispitivanja. Uzorak mora biti zadovoljavajuće homogeniziran i stabilan. U svojim ispitivanjima laboratoriji sudionici mogu koristiti standardne procedure prema nacionalnim ili međunarodnim standardima i metode za koje traže ili već posjeduju akreditaciju.
2. Bilateralna PT shema – u njoj sudjeluju dva sudionika: laboratorij koji traži akreditaciju i ocjenjivač. Ocjenjivač sam nabavlja PT materijal od treće strane, kompetentnih ustanova, zaostali materijal od prijašnjih PT shema i sl. Takav uzorak mora biti potpuno karakteriziran prije starta PT sheme.

PROVEDBA PT SHEME

U provedbi PT sheme razlikujemo dvije faze: faza planiranja i definiranja PT sheme, te faza dizajniranja i provedbe PT sheme.

1. FAZA PLANIRANJA I DEFINIRANJA PT SHEME (slika 2). Na početku faze planiranja potrebno je izabrati tip PT sheme, koji će se koristiti. Taj izbor ovisi o materijalu koji će se ispitivati, svojstvima materijala, metodi ispitivanja i predviđenom broju sudionika. PT shemu je potrebno provoditi na onim područjima ispitivanja, koja su od specifičnog interesa laboratorija koji sudjeluje i ustanove za ovlašćivanje. Zatim se moraju definirati ciljevi PT sheme koji mogu biti: provjera kakvoće ispitivanja na specifičnim područjima ispitivanjima, u slučaju nezadovoljavajućih rezultata PT sheme mogu se otkriti razlozi i predložiti efikasne korektivne mjere. Cilj, također, može biti dobivanje ili zadržavanje akreditacije u postupku ovlašćivanja.

Za sve faze PT sheme temeljna je povjerljivost, a o politici povjerljivosti treba obavijestiti sudionike prije početka PTS. Svakom laboratoriju sudioniku dodijeli se kodni broj, koji se koristi u svim dokumentima vezanim za PTS. Treba pripremiti listu s vezom broj – sudionik s kojom je upoznat minimalni broj osoba uključenih u rad PTS. Prilikom davanja uputa sudionicima

potrebno je razjasniti da je falsificiranje rezultata oprečno znanstvenom pristupu i vodi do krivih procjena osposobljenosti laboratorija. Laboratoriji sudionici ne smiju razmjenjivati rezultate ispitivanja međusobno niti pokušati dobiti informacije o svojstvima PT materijala od organizatora. Za provedbu PT sheme odabire se tehnički koordinator. On mora imati dugogodišnje iskustvo na područjima ispitivanja, znanja o svim relevantnim značajkama organizacije PTS, imati temeljno znanje o statističkim metodama, kao i biti upoznat s nacionalnim i internacionalnim standardima. PT shemu ne može izvesti samo jedna osoba. Pomoć pri planiranju, izvedbi, procjeni i obradbi rezultata tehnički koordinator može zatražiti od odabrane savjetodavne grupe. PT organizator treba dokumentirati politiku za sudjelovanje u PTS. Ona treba sadržavati: učestalost sudjelovanja u PTS, kriterij uzet za procjenu da li je izvedba zadovoljavajuća, kako će se rezultati PT koristiti u procesu ovlašćivanja, te pripremiti listu mogućih sudionika. Također je potrebno definirati politiku o kakvoći (prema ISO Uputama 43-1). Ustanova za ovlašćivanje traži od laboratorija ogranizatora PT sheme najmanje istu razinu sustava kakvoće, koju oni traže od ovlaštenih laboratorija.

2. FAZA DIZAJNIRANJA I PROVEDBE PT SHEME. Osoblje uključeno u izvedbu PT sheme treba imati tehničke, statističke i administrativne kvalifikacije. Svaki član organizacijske grupe mora imati jasno definiranu odgovornost. Oprema korištena od laboratorija koji organizira PT shemu za karakterizaciju PT materijala, kao i oprema laboratorija sudionika, mora biti umjerena. Za obradbu rezultata PT sheme poželjno je koristiti kompjutorske programe. Svi aspekti planiranja, razvoja i provedbe PTS moraju biti dokumentirani. Plan provedbe PT sheme može biti u formi protokola. On će sadržavati podatke o sudionicima, razloge provedbe PTS, detalje metoda i procedura koji sudionici trebaju koristiti pri izvedbi, temeljne statističke metode koje će biti korištene, te tehnike za procjenu izvedbe laboratorija. Ako laboratorij ima proveden sustav kakvoće potrebno je u postojeći implementirati PTS (politika, dokumentacija...).

Metode ispitivanja korištene u PT shemi mogu biti po izboru laboratorija sudionika. Izabrane metode moraju biti konzistentne s procedurama korištenim u laboratorijima. Mogu biti korištene nacionalne i internacionalne metode temeljene na ISO i EN standardima. Kriterij za procjenu izvedbe pojedinačnog laboratorija definira PT organizator, a ocjenjivač u postupku ovlašćivanja mora prihvatiti dani kriterij. U slučaju nezadovoljavajuće izvedbe PT organizator može predložiti korektivne mjere, čiju provedbu ocjenjivač

može provjeriti na sljedećoj procjeni. Izbor i priprema materijala za PT jedan je od najzahtjevnijih dijelova PTS (slika 3).

Uzorak je potrebno homogenizirati i zatim provesti ispitivanje homogenosti i stabilnosti. Nakon uzorkovanja potrebno je uzorak pripremiti za PTS. To uključuje sušenje, zatim reduciranje do željene veličine i ponovno ispitivanje homogenosti i stabilnosti. Nakon toga vrši se detaljna karakterizacija uzorka. Često je slučaj da je potrebno selektirati iz više kandidiranih PT materijala onaj koji zadovoljava postavljene kriterije PTS.

Kod obradbe rezultata potrebno je izabrati prikladan statistički model, koji će uključiti prethodno definirani kriterij za procjenu izvedbe. Uzorak nakon karakterizacije se mora pravilno identificirati, označiti i transportirati do laboratorija sudionika prema prethodno utvrđenim procedurama. Ako je PT organiziran na međunarodnoj razini, potrebno je osigurati sve prijeko potrebne dokumente za carinu. Svi koraci u planiranju, organiziranju i provođenju PTS moraju biti planirani vrlo pažljivo. Potrebno je definirati vremenske rokove za sve faze izvedbe PTS. Minimalno trajanje PTS je od 9 – 12 mjeseci. Sudionicima prije provedbe PT sheme daju se detaljne upute. U okviru poziva za sudjelovanje navodi se tip PTS, ciljevi provedbe PTS, korištene metode ispitivanja, vremenski rokovi, način izvješćivanja i troškovi PTS. Poželjno je izraditi formulare za izvješćivanje o rezultatima PTS. Nakon primitka rezultata PTS od laboratorija i obradbe rezultata sastavlja se završno izvješće svih sudionika. U njemu se koriste kodni brojevi laboratorija. Svaki laboratorij prima pismenim putem izvješće, koje sadržava i komentar na rezultate. Svaki laboratorij nakon završene PT sheme dobiva certifikat o sudjelovanju u PT shemi, a PT organizator i savjetodavna grupa može ogranizirati završni sastanak na koji mogu pozvati predstavnike laboratorija sudionika.

ZAKLJUČAK

Na temelju rezultata provedene PT sheme PT organizator i savjetodavni odbor izvlače zaključke, koji utječu na stalno poboljšanje u izvedbi PT shema i poboljšanje u izvedbi fizikalno kemijskih ispitivanja u laboratorijima.

Slika 1: PT shema kao dio procesa ovlašćivanja
Figure 1: PT SHEMA - part of the accreditation process

Slika 2: Temeljne točke u planiranju PTS/ Basic points of planning PTS
Figure 2: Basic points of PTS planning

Slika 3: Izbor materijala i uzorka za ispitivanje
Figure 3: Selection of material or test item

INTERLABORATORY COMPARISON OF TEST PROCEDURES INVOLVING PHYSICO-CHEMICAL PROPERTIES

Abstract

The capacity of performing tests in test laboratories may be evaluated by testing the equipement level - the so called Proficiency Testing Schemes - PTS. Apart from evaluating the proficiency of testing and calibration laboratories, these activities constitute also an additional technique in the process of laboratory accreditation on the part of competent bodies, in compliance with the EN 45 001:1989 standard.

The main PT goals are the maintenance and increasing of test laboratories' equipment level, as well as increasing reliability and comparability in the testing/calibration process. If the results of PTS are not satisfactory, the PT Organizer must, together with a laboratory representative, try and find out the reasons for this. The laboratory must, on its part, undertake efficient corrective measures in order to improve its testing quality.

There are different PTS types, while their selection is dependent on specific interests of the laboratory accreditation bodies, as well as of the labs taking part in PT. Before entering into the PTS designing phase, its purpose must be determined first. An experienced PT Organizer should choose a Technical Co-ordinator and an Advisory Board, to help him/her perform tasks that must precede PTS distribution to participating laboratories. It is also necessary to define the policy for participants in the said PTS, the policy of results utilization, confidentiality policy, as well as to set the Quality Assurance policy.

INTRODUCTION

EFFICIENCY LEVEL TESTING (Proficiency testing schemes - PTS) - the application of comparative interlaboratory tests for the purpose of identifying procedures of testing or calibration performance at individual laboratories.

The matching and comparability of laboratory tests and calibration data is also being monitored.

PTS goals are as follows: improve comparability of testing/calibration procedures, introduce efficiency and comparability of new test methods, offer assistance in maintaining instrument calibration. PTS reveals efficiency level in a given field of testing, leading to commercial improvements and increased customer trust. PTS results may reveal the causes of differences among tests performed at individual laboratories, and hence suggest efficient corrective measures for their elimination. One among possible PTS applications is in the accreditation process on the part of a competent body, in compliance with the EN 45 001:1989 standard (Figure 1). Item 6.3. of the said Standard recommends co-operation with other laboratories, through the use of PTSs. However, PTS results are not the only basis for laboratory accreditation granting or maintenance. Still, if PTS results prove to be unsatisfactory, the accreditation-granting process may become questionable. The accreditation body must develop a policy of using PTS. PTS must be performed in compliance with the ISO Guide 43-1.

TYPES OF PTS

There are different PTS types. Their choice is dependent on the nature of test sample or material, test method, and number of participants.

- According to the sample/material kind and test method:

1. **The Comparability Measuring Scheme** - testing or calibration sample goes from one participating laboratory to another. The sample is being periodically returned into the lab organizing the PTS, for its properties to be checked. The drawback of this particular PTS type lies in the fact that it is rather time-consuming, resulting in problems associated with sample stability, as well as with sample circulation monitoring.
2. **Interlaboratory Test Scheme** - the sample is homogenized ans selected into sub-samples and distributed to PTS participants. Every participant performes complete sample analysis. The test results are returned to the lab organizing and co-ordinating the procedure for processing, after which the participants are informed of PTS results.
3. **The Split Sample Testing Scheme** - test material samples are split into two or more parts. Each participating laboratory tests a part of each sample and the rest are tested by other participants. Unlike the second PTS type, this particular scheme usually has only limited control, while the sample homogeneity has to be ckecked.

- According to the number of participants:

1. **Multilateral PTS** - with several participating laboratories. The Scheme serves as a tool in accreditation procedures in compliance with ISO Guide 43-1, setting the minimum. The purpose is for the participants to compare their own performance with that of a reference laboratory, or with that of laboratories in similar testing areas. The sample must have a satisfactory level of both homogeneity and stability. While performing their tests, the laboratories may use standard procedures in compliance with national or international standards, and already accredited methods, or those which are currently under accreditation process.
2. **Bilateral PTS** - involving two participants: The laboratory seeking accreditation and the evaluator. The latter obtains PT material on its own from a third party, from competent institutions, or takes left-over material from former PTSs... The properties of such a sample must be fully identified before launching a PTS.

PTS IMPLEMENTATION

There are two phases of PTS implementation: PTS planning and definition phase, & PTS designing and implementation phase.

1. **PTS PLANNING AND DEFINITION PHASE** (Figure 2). At the beginning of the planning phase, one must choose PTS to be used. The choice is dependent on the material to be tested, its properties, the test method, and the envisaged participant number. PTS must be performed in the areas of testing of specific interest for both the participating laboratory and the accreditation body.

After that, it is necessary to determine PTS goals, which may be as follows: Testing quality evaluation in specific test areas. Should the PTS results prove unsatisfactory, it is possible to reveal the reasons and suggest efficient correction measures. The purpose may also be to obtain or maintain accreditation in the certification process.

Confidentiality lies in the basis of all PTS phases. The participants must be notified of it before the PTS starts. Each participating laboratory is assigned a code, used in all documents associated with PTS. A list must be prepared, associating the code with the participant in question. Only a minimal number of persons involved in PTS are to be aware of it. While providing guidelines to the participants, it must be made clear that any falsification of results is contrary to the scientific approach and leads to false laboratory proficiency results. The participating laboratories must not exchange test results among themselves or try to obtain information on PT material properties from the Organizer. A Technical Co-ordinator is appointed for PTS

implementation. He/she must have a long-term experience in the test area in question, as well as be versed in all relevant characteristics of PTS organization, statistical methods, and be familiar with both national and international standards. PTS may not be implemented by a single person. The Technical Co-odinator may require assistance in planning, implementation, evaluation, and processing of results from a selected Advisory Board.

PT organizer must document the PTS participation policy. The said policy must include: PTS participation frequency, evaluation criteria, how are PT results to be used in the accreditation process, and a list of possible participants. It is also necessary to determine the Quality Assurance Policy (in compliance with the ISO Guide 43-1). The accreditation body requires from the laboratory organizing the PTS at least the same Quality Assurance System level as the one requested by it from accredited laboratories.

2. **PTS DESIGNING AND IMPLEMENTATION PHASE**. Personnel involved in PTS implementation must have the necessary technical, statistical, and administrative qualifications. Each Organization Group member must have strictly defined responsibilities. The equipment used by the laboratory organizing the PTS for the characterization of PT material, as well as that by the participating laboratory, must be gauged. It is advisable to use computer software for processing PTS results. All the aspects of PTS planning, development, and implementation, need to be documented. The PTS implementation plan may be structured as a protocol. It shall contain data on participants, reasons for PTS implementation, method and procedure-related details to be used by the participants during implementation, basic statistical methods to be used, as well as techniques for evaluating laboratory performance. If the laboratory in question already has an implemented Quality Assurance System, then PTS (policy, documentation) needs to be incorporated into it.

The test methods used in PTS may be chosen by the participating laboratory. The selected methods must be consistent with procedures used in the labs. Both national and international methods based on ISO and EN standards may also be used. The criterion for evaluating the performance of each individual laboratory is determined by the PT Organizer, while the Evaluator has to adopt it in the accreditation procedure. In cases of unsatisfactory performance, the PT Organizer may suggest correction measures, the implementation of which may be checked by the Evaluator in

the following evaluation process. The choice and preparation of PT materials is one among the most demanding parts of PTS (Figure 3).

The sample needs to be homogenized, and homogenity and stability consequently tested. After sampling, the sample needs to be prepared for PTS. This includes drying, reduction down to a desired size, and rechecking of homogenity and stability. What follows is a detailed sample characterization. It frequently occurs that one must select the sample matching the set PTS requirements out of several PT materials offered.

While processing results, one must select a proper statistical model, including the previously determined criterion for performance evaluation. After its characteristics have been identified, the sample must be properly identified, labelled, and transported to the participating lab, according to previously determined procedures. If PT has been organized on an international level, all the necessary customs office documents must also be provided. All the steps in PTS planning, organization, and implementation must be very carefully planned. Time limits must be set for all the PTS implementation phases. Minimum PTS duration is from 9-12 months. Before the implementation itself, PTS participants are given detailed instructions. The sollicitation for their participation indicates PTS type in question, its implementation goals, test methods to be used, deadlines, reporting manner, and PTS costs. It is advisable to develop forms for providing PTS results. Once the PTS results are obtained from the laboratories and processed, a final report of all participants is being drawn up, featuring individual labs' codes. Each laboratory receives a written report including a comment of the results obtained by it. Once PTS is completed, each lab obtains a Certificate of Participation in the PTS. The PT Organizer may also arrange for a final meeting, featuring all the participating laboratories' representatives.

CONCLUSION

Based on PTS results, the PT Organizer and the Advisory Board draw up conclusions resulting in permanent improvements in the implementation of PTSs, as well as in the performance of physico-chemical lab tests.

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