Istituto Superiore di Sanità

Preparation of Materials for Proficiency Testing in the field of official controls: a key point for the organizer

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According to Regulation (EU) 2017/625 on official controls and other official activities performed to ensure the application of food and feed law [1], the participation in proficiency testing (PT) is a key activity for laboratories dealing with the official controls aimed at assuring the food safety in the EU.
Based on this premise, the European Union Reference Laboratories (EURLs) and the National Reference Laboratories (NRLs) play a key role as PT provider. The main scope of their scheme is to meet the needs of official laboratories (OLs), taking into account the novelty in regulations setting maximum limits (MLs). In the field of chemical elements in food, many PTs are commercially available, but most of these are based on freeze-dried materials or/and the concentrations of analytes are not adequate to practice the methods at levels close to the limits set in the EU Regulation [2]. To cover this gap, in 2009 the Unit for PTs at the Italian National Institute of Health was granted accreditation as PT provider based on the requirements in ISO Guide 43 and subsequently based on ISO/IEC 17043. It acted as European Union Reference for Chemical Elements in food of animal origin until the end of 2018, thus organising exercises for the benefit of the relevant NRLs. As of 2019, the Unit has been providing the NRL for metals and nitrogenous compound in food with accredited PT (metals in food) addressed to the Italian official laboratories. The main objectives of PT provider and the benefits that OLs have by participating are listed below.

PTs Organization for OLs: Main Objectives of PT providers

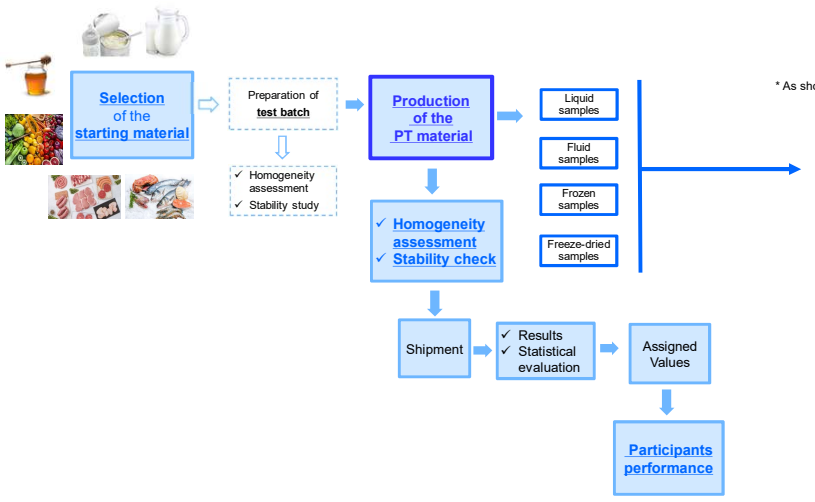
- ✓ to check/improve the performance of analytical methods used by the OLs around MLs
- ✓ to assist participants in finding the cause of analytical problems leading to unsatisfactory performance (follow-up activity)
- ✓ to verify the effectiveness of corrective actions (repetition of PTs)
- ✓ to assess the long-term performance of participants
- ✓ to promote the improvement of QCs
- ✓ to harmonize the performance of the network (z score) and method parameters (*e.g.* LoQ; U)
- ✓ to verify and harmonize the interpretation of results in terms of the sample acceptance, in accordance with the relevant legislation in the field of competence (*e.g.* CR (EC) 333/2007)

PTs Participation: Main features for OLs

- ✓ to evaluate the laboratories performance by using interlaboratory comparisons (the best choise according to ISO/IEC 17025)
- ✓ to assess the ability of laboratories to competently perform specific tests and/or measurements at levels close to the limits set in the EU Regulation
- ✓ to provide independent evidence that the laboratory produces technically valid and reliable results
- ✓ to implement the laboratory internal quality control procedures as it can be considered as an additional external audit of the laboratory's capability (basis for continuous improvement)
- ✓ to have an independent reassurance that all the activities performed are subjected to controls
- ✓ to allow accreditation bodies to assess the ability of laboratories to competently perform tests (ISO/IEC 17025)

Production of PT test items

Over the years, the Unit for PTs implemented different procedures to produce material in physical form and with level of metals meeting the requests of the OLs. The main and most critical steps necessary for the production of liquid/fluid samples (e.g., milk, fruit juice, vegetable drinks, honey), frozen items (e.g. meat, fish, offal, processed meat) and freeze-dried samples are outlined as follows:



Main critical steps of PT material preparation

Preparation of standard solutions (chemical elements) ➡ Fortification ➡ Homogenisation* ➡ Sampling

* As shown in the example below by pictures, additional steps are necessary for freeze-dried sample preparation: homogenization of the fresh fortified material batch, freezing, freeze-drying, homogenisation of the freeze-dried bath, sieving, quartering

Homogeneity and Stability of PT test items

Specific procedures, developed, documented and applied for each different kind of material preparation, ensured that each critical step was kept under control. The effectiveness of these procedures has been demonstrated over the years by the production of PT materials that were always homogeneous even when using a strict σ_{pt} (lower than Horwitz/Thompson) for the assessment (example in figure 1). Furthermore, the criteria on stability set out in ISO 13528 [3] were always met for the duration of the PT. Some materials were analysed over time and the results were in good agreement with the homogeneity data taking into account the measurement uncertainty (table 1).

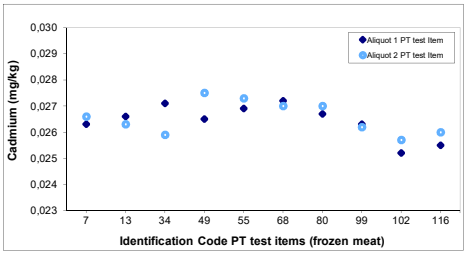


Fig. 1 Homogeneity test for Cadmium (Cd) relative to a PT on frozen meat. Concentration (mg/kg) of Cd is measured in 10 randomly selected bottles identified by a numerical code. The analysis is performed in duplicate, and both the values obtained for the first (squares) and the second (circles) test portion are reported

Table 1 Results coming from the homogeneity test on two different PTs on milk are compared to the results obtained after certain times. The PTs have been produced by Unit for PTs and both data have been obtained using the same accredited method. Each value of concentration is reported in µg/kg together with the relevant expanded uncertainties (coverage factor k = 2)

Analyte	13th PT Homogeneity	Stability test value after 7 months	12th PT Homogeneity	Stability test value after 18 months
As	80.6 ± 11.3	76.0 ± 10.6	125 ± 17.5	122 ± 17.1
Cd	7.8 ± 0.8	7.0 ± 0. 7	5.5 ± 0.6	5.0 ± 0.5
Pb	37.9 ± 3.0	36.0 ± 2.9	28.9 ± 2.3	30.0 ± 2.4

As future perspective, the inclusion in Commission Regulation (EU) 2023/915 [4] of new matrices, the lowering of certain maximum levels and the possible inclusion of new metals could represent next challenges to be faced by the reference laboratories and the next tasks in the role of PT provider.

Bibliography

1. Regulation (EU) 2017/625, Official Journal of the European Union L 95, 7.4.2017, p. 1–142
2. Commission Regulation (EU) 2023/915, Official Journal of the European Union L 119, 5.5.2023, p. 103–157
3. ISO 13528:2022 – Statistical methods for use in proficiency testing by interlboratorycomparison
4. Commission Regulation (EU) 2023/915 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006