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Original Article

Provision of proficiency test (PT) scheme on proximate and mineral analyses: Philippine experience

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ABSTRACT

Based on ISO Guide 43-1: 1997 and ILAC G-13: 2000 guidelines three (3) proficiency test (PT) Rounds were organized for moisture, fat, protein, ash, iron, calcium and sodium analyses using wheat flour (Round 1), powdered tonic food drink (Round 2) and dried paste shrimp (Round 3) as PT materials. Fifty-three (53), fifty-two (52) and forty-six (46) local and foreign laboratories participated in PT Rounds 1, 2 and 3, respectively. Results were evaluated using the appropriate statistical procedures based on ISO 13528: 2005 and/or IUPAC Technical Report, 2006. The *z*-scores used to evaluate the performance of participant laboratories were computed, applying the standard deviation for proficiency test assessment (σ_p) derived from the CVs/RSDs of previous PTs and the general expression of Horwitz, while the assigned values (robust mean or mode with uncertainty) were obtained from the consensus of PT participants' results. For proximate analyses, 65–90% of PT participant laboratories achieved "satisfactory" ($|z\text{-score}| \leq 2$) performance for the three (3) PT Rounds. For iron and calcium analyses, only 50–70% achieved "satisfactory" performance. The PT test materials' given assigned values (X) and range ($X \pm 2\sigma_p$) were used in method validation and as quality control test materials by the local laboratories. The three (3) PT Rounds provided an effective tool in assessing the laboratory performance of proximate and mineral analyses and in conducting the necessary investigative and corrective action on "questionable" ("Q") and "unsatisfactory" ("U") results.

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1. Introduction

The marketability of Philippine food produce/products for local and global markets has increasingly become critically based on valid and reliable results of food quality and safety laboratory tests. Some rejections and dumping actions at the local and foreign points of entry of local food produce/products may have been due to violative lots that have been passed incorrectly by local tests, their results being oftentimes lacking international comparability. These actions ultimately bring about not only losses in potential revenues/earnings and competitive advantage but also diminished credibility and confidence in local testing laboratories.

Codex Alimentarius, as the reference point in global food trade, recommends that laboratories involved in the testing and inspection of exported and imported food produce/products adopt the following Analytical Quality Assurance (AQA) measures: (a) perform method validation, (b) observe internal quality control (IQC), (c) use certified/standard reference materials (CRMs/SRMs), (d) participate in proficiency tests (PTs), and (e) become accredited to ISO/IEC 17025.

Convinced that PT participation gives laboratories an objective independent measure of their performance and as such is a very useful highly diagnostic tool of the laboratories' quality system, the Philippine Accreditation Office (PAO) strongly recommends participation in PT Schemes for local laboratories to obtain and sustain ISO/IEC 17025 accreditation. Additionally, the PAO also advises on the use of CRMs/SRMs to establish traceability of measurements to national or international standards, endowing these results with international comparability. However, PTs as well as CRMs/SRMs are only provided by a few expert laboratories/suppliers in developed countries (e.g. USA, Australia, and other countries in Europe), and only available upon payment of subscription fees, which are prohibitive to most Philippine laboratories. Furthermore, these available PTs are not always appropriate for specific local needs, in terms of analytes and food matrices.

The need thus became urgent for a local PT provider that will respond to the laboratories' requirement for readily available, accessible, affordable, and internationally acceptable AQA tools to monitor their performance on a continuing basis. The Food and Nutrition Research Institute, Department of Science and Technology

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(FNRI-DOST), took up the challenge of organizing PTs on some local nutrition labeling components using appropriate food matrices. The Institute's confidence to provide credible PT Schemes was based on: (a) the experience gained in the conduct of interlaboratory tests on proximate and mineral analyses in 1989 and 1993 (Portugal et al., 1993) and (b) the in-house capability of its staff in the preparation of RMs and the organization of PTs in nutrient analyses, established through trainings at the Institute for Reference Materials and Measurement (IRMM) in Belgium for preparation of RMs, and the Seibersdorf Laboratories in Vienna, Austria for the conduct of PTs, as organized by the International Atomic Energy Agency (IAEA).

The objectives of the study were: (a) to organize/conduct proficiency tests and evaluate the performance of participant laboratories in the analysis of moisture, fat, protein, ash, iron, calcium and sodium using different food matrices, and (b) to produce batches of homogeneous test materials with assigned values as candidate reference materials (RMs)/quality control test materials (QCTMs) for use in the PTs and subsequently by local food laboratories in their AQA work.

2. Materials and methods

2.1. Preparation of PT test materials/candidate reference materials

Different food matrices chosen to represent foods or ingredients of processed foods that are commonly consumed and routinely analyzed in the Philippine laboratories were used, namely: (a) wheat flour (PT Round 1), (b) powdered tonic food drink (PT Round 2), a mixed processed food containing malted barley, milk, solids cocoa and vegetable oil ingredient, and (c) dried paste shrimp (PT Round 3).

For PT Round 1, approximately twenty-five (25) kg hard wheat flour, purchased from a flour milling company, was passed through a Retsch stainless steel 100-mesh (150 μm) sieve. For PT Round 2, twenty (20) packs (1 kg each) of a local commercial tonic food drink of the same lot, provided by an established multinational food company and stored in a freezer (−20 °C) overnight was ground in a Retsch SP 200 Rotor Beater Mill, and passed through a Retsch stainless steel 60-mesh (250 μm) sieve. For PT Round 3, approximately thirty-two (32) kg dried Jawal paste shrimp (*Acetes indicus*), purchased from one dealer in a Metro Manila wet market was cleaned free of stones and other foreign materials and pre-dried at 60 °C in a Memmert air oven to a moisture level ≤4%. The material was ground and sieved manually in a Retsch stainless steel 100-mesh (150 μm) sieve. The shells and other parts that did not pass the 100-mesh sieve were discarded.

Each PT bulk powder material was mixed thoroughly in a fabricated rolling drum mixer for 2 h at 30-min intervals. Subsamples of 50–60 g portions were weighed in pre-labeled and pre-numbered (e.g. name of test material, sample code) polyethylene-aluminum foil bags, which were sealed with a Technotrip vacuum sealer and resealed with a Dongfeng heat sealer to ensure minimal moisture absorption. The randomly selected labeled test materials were kept in a freezer (−20 °C) until tested for homogeneity, stability or distributed to PT participants for analysis of moisture, fat, protein, ash, iron, calcium and sodium.

2.2. Characterization of PT materials/candidate reference materials

2.2.1. Homogeneity testing

Two (2) test portions of twelve (12) randomly selected packs of the three (3) test materials were analyzed at random using validated methods of analysis under repeatability conditions (same analysts, equipment and supplies, laboratory conditions). The powdered tonic food drink in PT Round 2 and dried paste shrimp in PT Round 3 were analyzed for moisture, fat, protein, ash, iron, calcium and sodium. For wheat flour in PT Round 1, the homogeneity test was limited to moisture, protein, ash and sodium analyses.

2.2.2. Stability testing

Five (5) randomly selected packs of each PT material stored for 0.5 month (ambient temperature) and 1.5 months (frozen) were analyzed in duplicate for moisture, protein, ash and sodium using the same methods for homogeneity test. Storage at 0.5 month and 1.5 months reflects the stability of the PT material from distribution up to analysis and report submission by the PT participants. The stability per analyte was evaluated by comparing the stability test mean ($N = 5$) at final storage period (1.5 months) with the homogeneity test mean ($N = 12$) per analyte obtained at 0.0 month.

2.3. Conduct of proficiency testing

The three (3) proficiency tests were carried out from 2006 to 2009, based on ISO Guide 43-1: 1997 and ILAC G-13: 2005 guidelines. Technical inputs were provided by an Advisory Group composed of: (a) two consultants: a statistician and a chemist, and (b) a multi-agency Technical Working Group (TWG) of chemists from government and private laboratories with expertise in the relevant laboratory analysis and Analytical Quality Assurance (AQA).

2.3.1. Invitation of participants

Laboratories from the government and private local commercial testing services, industry quality control units, the academe and ASEAN as well as Australian laboratories were invited to participate in the three (3) FNRI-DOST PT Schemes. The PT Schemes were also promoted by the Philippine Accreditation Office (PAO) to local ISO/IEC 17025-accredited food testing laboratories.

2.3.2. Distribution of PT materials

Two (2) packs of wheat flour in PT Round 1 while one pack each of powdered tonic food drink, and dried shrimp in PT Rounds 2 and 3, respectively were given to local and foreign PT participants. The test materials were distributed through courier or air/surface mail to the local regional and foreign participants with the following documents: Instructions to Participants, Analytical Procedures Form; and Results Sheet. Each laboratory was assigned a laboratory code number to maintain confidentiality of test results.

2.3.3. Conduct of analysis and reporting of results

The PT participants were instructed to analyze any or all of the following analytes: proximate (moisture, fat, protein and ash) and minerals (iron, calcium and sodium) in their respective laboratories using their own routine methods, treating the test material in the same way samples were routinely tested in their own laboratories.

The participants were given four (4) weeks after receipt of test materials to finish the analyses, report the results of the analyses on the material "as received" and record the results on the Result Sheet, using the prescribed number of significant digits and units of expression for each analyte. The participants were also requested to report the estimated measurement uncertainty (MU).

2.3.4. Collation and verification of PT results

The participants were requested to return the results and other documents on or before the closing date for submission of results. The individual participant's tabulated results (with method

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