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Original Article

Development of rice reference material and its use for evaluation of analytical performance of food analysis laboratories

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ABSTRACT

Data quality is one of the major concerns in development of food composition database and to editors of many peer-reviewed journals in accepting a scientific paper for publication. Regular use of a reference material and participation in a proficiency testing programme could improve the reliability of the analytical data. The objectives of this project were to prepare rice test material with assigned values and to use it to assess the analytical performance of laboratories which are involved in research and analysis of rice. The international guidelines, ISO Guide 35, ISO 13528 and ISO Guide 43, were followed as much as possible throughout the preparation of the reference material and the laboratory performance study. Brown rice (Jasmine variety) was ground to particle size which passed completely through a sieve with pore size 250 μm and packed in laminated aluminum foil bags under vacuum. Based on the analyses of representative nutrients – moisture, protein, iron, zinc and vitamin B1 – the samples were demonstrated homogeneous. Ten expert laboratories from various countries, 36 laboratories from Thailand, and 16 laboratories from ASEAN and Asia registered for the laboratory performance study. The samples were sent for analysis of selected proximate composition (moisture, protein, dietary fibre and ash), two minerals (iron, zinc), and one labile nutrient (vitamin B1) using routine analytical methods. The assigned values of the nutrients in the test materials, as robust mean $\pm$ robust SD or predicted SD, were established with their uncertainties. For proximate composition, 67–87% of participating laboratories showed good analytical performance. However, many of them showed questionable and unsatisfactory performance on the analyses of dietary fibre (55%) and vitamin B1 (47%). The evaluation of the results of moisture, protein and iron with their uncertainties against the assigned values of the test material using En score was also demonstrated. Finally, the consensus values of nutrients in the rice sample as mean $\pm$ SD were developed from the analytical results of laboratories with good performance for both within- and between-laboratories. This test material can be used as a reference material for internal and external quality control systems to improve the quality of the analytical data.

1. Introduction

Reliable analytical data are required by both food analysts and data users. Standardisation of the analytical methodology and development of a quality control system in a laboratory can help ensure analytical measurement validity and increase data quality and reliability. Reference materials play key roles in the development of the internal quality control system. It is well known that different methods of nutrient analysis or the same analytical methods with some modification are being used by different laboratories, resulting in some discrepancies in the analytical data. Proficiency testing is an external quality assessment designed to assess the laboratory analytical performance which reflects the reliability of the analytical data. It assists in increasing confidence in analyst ability in the case of good performance, or in identifying laboratories with questionable and unsatisfactory results where improvement of the competency in nutrient analysis is required.

Seven rounds of laboratory performance studies were conducted by the Institute of Nutrition, Mahidol University during 1989–2003 (Puwastien and Sungpuag, 1995; Puwastien and Raroengwichit, 2000; Puwastien et al., 1989, 1999, 2001, 2003) using different test materials with consensus assigned values. Two approaches were used to develop assigned values of food components: one from expert laboratories and another from good performance laboratories who participated in laboratory performance studies. With the collaborative study among expert laboratories in Australia, New Zealand, USA, Austria, and laboratories in ASEANFOODS member countries, nine food reference

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materials with consensus values for proximate composition and some minerals were produced. These materials were rice flour (AS-FRM1) and soybean flour (AS-FRM2) (Puwastien et al., 1989); cereal-soy (AS-FRM3) and fish flour (AS-FRM4) (Puwastien and Sungpuag, 1995); weaning food (AS-FRM-5) and fish powder (AS-FRM-6) (Puwastien et al., 1999a); feed (AS-FRM-7) and fish meal (AS-FRM 8) (Puwastien et al., 2001); and milk powder (AS-FRM 9) (Puwastien et al., 2003). Since 2004, the International Year of Rice, various studies on nutritive values of different varieties of rice have been conducted for research and development, generation of food composition data, and for screening and selecting plant varieties. To evaluate performance of laboratories that have analyzed rice, a laboratory proficiency study on nutrient analysis was needed. Thus two main objectives of this study were (1) to prepare a candidate reference material (RM) of rice powder and study its physical and chemical characteristics and (2) to organise a laboratory performance study using the prepared candidate RM as test material.

2. Materials and methods

2.1. Preparation of candidate reference material (RM)

2.1.1. Test material for mineral analysis

Test material used was brown rice, Jasmine variety, obtained from Center of Excellence for Rice Molecular Breeding and Product Development, Kasetsart University, Thailand. Three kilograms of the test material were frozen using liquid nitrogen in order to make rice to be brittle and facilitate the grinding process and then ground in a stainless steel grinding machine (to avoid iron and zinc contamination) until the fine particles passed completely through sieve No. 60 mesh (250 μm). The sample was mixed thoroughly manually, in a humidity controlled air conditioned room and then packed under vacuum in aluminum foil bags, about 10 g each. Bags were randomly divided into 2 sets, A and B, and then labeled with sample code number. These prepared samples were used for analyses of minerals (iron, zinc) and kept in a freezer at −20 °C.

2.1.2. Test material for analyses of proximate composition and vitamin B1

Another set of the test material, 15 kg, was ground using the Cyclotec sample mill until the fine particles passed completely through sieve No. 60 mesh (250 μm). The sample was then mixed thoroughly by a rotating mixer for 5 h in a humidity controlled air conditioned room and then packed under vacuum in aluminum foil bags, about 30 g each. Bags were randomly divided into 2 sets, A and B, and then labeled with sample code number. They were used for the analysis of proximate composition and vitamin B1. The samples were kept in a freezer at −20 °C.

2.2. Characteristics of the candidate reference material

2.2.1. Particle size distribution

The particle size distribution of the test material for nutrient analysis (Section 2.1.2) was manually studied by sieve analysis. One hundred gram of the test material was passed through 3 standard sieves 60, 80 and 120 mesh, with pore sizes of 250, 180 and 125 μm, respectively. Each fraction was collected, weighed and recorded. Percent distribution at each fraction was calculated.

2.2.2. Homogeneity study

Ten packages (5 from set A and 5 from set B) each of the prepared sub-samples for mineral analysis and for proximate analysis were selected at random. Homogeneity of the candidate material was evaluated by analyses of selected representative nutrients, i.e. iron and zinc (representative of trace elements), moisture and protein (representatives of proximate composition) and vitamin B1 (representative of labile nutrient). The analyses were performed in two test portions from each package, in a random order. Each analysis was performed in one setting under repeated conditions, i.e. by competent analysts, on the same day using the same set of reagents and conditions. The results were statistically evaluated.

2.2.3. Stability study

Since Vitamin B1 is the most labile nutrient in rice, its stability was checked throughout the storage period. The prepared candidate reference materials were kept at −20 °C. Five packages of the prepared sub-samples were randomly selected at 1, 6 and 12 months intervals for the first year and every 6 months thereafter. At each period, single analysis of vitamin B1 by HPLC in each sample was conducted. The stability of the vitamin was evaluated by comparing the results obtained at each period with the levels analysed at 0 month (using data from homogeneity study).

2.2.4. Chemical analyses of the components in the test materials

Ten expert laboratories from different countries in OCEANIA, Europe, and North America collaborated to develop assigned values of nutrients in the test material. Each laboratory registered to analyse several and none of them analysed all of the assigned components. Since the number of derived data from expert laboratories would not be a sufficient number for reliable assigned values, the better assigned values for the measurands in the test material were derived from the analytical values of both expert laboratories and the participants of the performance study according to the ISO 13528, 2005.

2.3. Laboratory performance study

Following closely the ISO Guide 43 (ISO Guide 43-1, 1997), a laboratory performance study for nutrient analyses was conducted.

2.3.1. Participants

Fifty-six laboratories from various countries, mainly from ASEAN, registered to participate in the performance study. However, not all laboratories registered for analyses of all assigned measurands and four of them did not submit the report.

2.3.2. Distribution of samples and documents

Two packages each of 10 g test material (package A and B with random number) for analysis of minerals and 30–40 g (package A and B with random number) for analysis of other components were sent to expert laboratories and oversea participants via airmail and by post for local laboratories. Five documents – instruction to the participants, report form, uncertainty form, questionnaire for method used and questionnaire for in-house quality control system – were sent electronically as attached files with a secret laboratory code number assigned to each laboratory.

2.3.3. Analytical components and methods of analysis

Participating laboratories were assigned to analyse proximate composition (moisture, protein, dietary fibre, ash), minerals (iron and zinc), and vitamin B1 using their routine test methods. They were requested to analyse vitamin B1 within two weeks of receiving the samples. Two individual values (A and B) of each component, one from each package of the test material, were requested to be reported in the report form where unit of expression and number of significant decimal places were indicated. For the first trial, the participants were requested to report values for moisture, protein and iron with their uncertainty values (expanded uncertainty with a coverage factor of $k = 2$).