Journal of Food Composition and Analysis 23 (2010) 828–836

Contents lists available at ScienceDirect

Journal of Food Composition and Analysis

journal homepage: www.elsevier.com/locate/jfca

Original article

# Quality control materials development for proximate composition determination in baby foods to enhance the Portuguese food composition database: Packaging conditions

I. Castanheira *, C. António, C. Mota, C. André, A. Sanches-Silva

*Department of Food Safety and Nutrition (INSA), National Institute of Health Dr. Ricardo Jorge, Av. Padre Cruz, 1649-016 Lisbon, Portugal*

ARTICLE INFO

*Article history:*
Received 31 July 2009
Received in revised form 11 January 2010
Accepted 9 March 2010

*Keywords:*
Reference materials
Quality control materials
QCM
Certified reference materials
CRM
Foodstuffs
Data quality
EuroFIR
Food composition database
Infant formula
Food contact materials
Homogeneity
Stability
Food data management

ABSTRACT

Food matrix reference materials play an important role in the quality of data used in food composition databanks concerning the precision, trueness and accuracy of analytical values. In this work the feasibility studies according to ISO 34 (ISO, 2000) of two commercial baby foods (ready-to-eat baby soup and milk-based powdered infant formula) were evaluated to check for their suitability as quality control materials for the analysis of moisture, protein, fat, ash and acidity. The suitability of plastic packaging materials to guarantee the reference materials' characteristics is discussed. Official methods of analysis were used to evaluate the homogeneity and to monitor short-term and long-term stability studies. ANOVA was carried out to confirm homogeneity within and between samples. ISO 34 method was applied to monitor stability at different temperatures. The coefficients of variation obtained between sachets for milk-based powdered infant formula were less than 4.4% for moisture and 0.5% for fat. The stability data over 8.5 months storage period at 20 °C, 5 °C and −20 °C and −70 °C indicate that both materials are stable depending on the parameter studied and temperature. According to results, quality control materials (QCMs) prepared in agreement with ISO 34 requirements are a valuable tool in food composition analysis.



## 1. Introduction

In Europe the interest in compiling food data goes back to the 19th century when in 1878 the German scientist Joseph König published the first food composition table (Church, 2006). Since then, food composition tables have passed through an intense development process in which the use of computers plays an important role. The interest of food composition databanks (FCDBs) in many fields of work like clinical practice, research, public health, education and food industry have contributed to this improvement (Egan et al., 2007; Williamson and Buttriss, 2007). Although food data upgrading and updating of databases has been remarkable over the last few decades, there were still some difficulties with respect to the comparability of values among different countries.

In view of the need for internationalization and the probable globalization of FCDBs, the standardization and harmonization of food data have become the target for increasing interest by data compilers and users, as can be seen by an increasing number of papers on this subject (Deharveng et al., 1999; Vaask et al., 2004; Egan et al., 2007; Slimani et al., 2007).

Within this context, in 2005 the EuroFir Project (European Food Information Resource Network of Excellence), funded by the European Commission, was started with the aim of harmonising FCDBs in Europe (Williamson and Buttriss, 2007; EuroFIR, 2009). The final goal is to obtain a single European FCDB with accessible and reliable information which could support, for example, more epidemiological studies regarding diet/health relationship.

The quality of analytical data is an essential underpinning tool of FCDBs. Therefore, many efforts have been made to create a system to evaluate and improve the quality of data in FCDBs (Southgate, 2002; Castanheira et al., 2007a,b, 2009; Phillips et al., 2007; Haytowitz et al., 2008). EuroFIR have recently developed a system to evaluate data quality from scientific literature, which allows the producers of food composition databases to evaluate the quality of data according to common guidelines. The system, described elsewhere (Castanheira et al., 2007b; Castanheira et al., 2008), requires the use of reference materials (RMs) from

* Corresponding author.
*E-mail address:* isabel.castanheira@insa.min-saude.pt (I. Castanheira).

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doi:10.1016/j.jfca.2010.03.009

production of analytical data to evaluate the laboratory performance up to compilation process in order to define the quality of nutrient values that enter in food composition databanks. RMs are used at three stages of the overall process. The first is during the analytical process where RMs are tools for evaluating the quality of analytical data, the second is during compilation of data where they play an important role for selection of data and the last is during the interchange of data across databanks from different countries, to decide about comparability of values (Castanheira et al., 2009).

Therefore the availability of RMs is very important to produce food composition data that fits the quality requirements for comparability at international level overtime. Moreover the intended use of an RMs for assessment of a measure procedure, assigning values and quality control need to be clarified to guarantee that RMs are relevant in terms of matrix and concentration. Terminology, definitions and sub-groups of RMs have been discussed and reviewed recently (Emons, 2006). This classification is derived from the traceability and metrological requirements that RMs have to fulfil, considering that a RM can only be used for a single purpose. In the new definition RM is characterised as "material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process". RMs may be grouped into three categories: (a) quality control materials (QCMs), a subgroup of RMs which fulfil requirements only for homogeneity and stability for the intended use; (b) calibrants, usually designated as analytical standard; and (c) certified reference materials (CRMs), defined as "a reference, characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of a specified property, its associated uncertainty, and a statement of metrological traceability". CRMs have by definition the highest metrological grade and they are produced in Europe by IRMM (Institute of Reference Materials and Measurements) and in North America by NIST in the US (National Institute for Standards and Technology) and NRC in Canada (National Research Council Canada). CRMs produced by US and Canadian Institutes are designated as SRM (Standard Reference Materials). QCMs are extremely important in day-to-day analysis. In the field of food composition analysis, few laboratories in the world have demonstrated their capabilities for QCM production (Phillips et al., 2005, 2006). The publication of a new edition of ISO Guide 34 and 35 (ISO, 2000, 2006) will help the producers and users of QCM to demonstrate or check the quality of materials used alongside routine samples. The accreditation of QCM producers by national accreditation bodies recognised by ILAC allows the preparation of QCM for representative real samples that match the foods in terms of matrix and concentration.

Addressing this issue is one of the objectives of Project for Food Matrix Reference Materials (LMARSA) in Portugal. One of priorities is to support food composition analysis through the production of a range of reference materials relevant to determine prioritised nutrients in selected foods. The project includes the development of specific quality management requirements to guarantee that QCMs perform the purpose of EuroFIR quality requirements.

The familiarisation with the NP EN ISO/IEC 17025:2005 standard (2005), in combination with ISO Guide 34, was considered fundamental to demonstrate that QCMs produced in the country can be used in the Complier Certification Scheme carried out by EuroFIR. The programme includes a series of QCMs in combination with CRMs to be used by contract laboratories. The first phase of the project aims with the development of QCMs for analysis of baby foods that match the selected foods in terms of matrix and nutrient concentration.

The present paper describes the strategies carried out to evaluate the suitability of ready-to-eat baby soup and milk-based powdered infant formula as QCM for the purpose of analytical quality control of proximate analysis. The assessment is based on the homogeneity and stability of the matrices, including the suitability of packaging materials which are required conditions for the production of QCMs.

## 2. Materials and methods

### 2.1. Samples

#### 2.1.1. Food samples

Selected foods were milk-based powdered infant formula and ready-to-eat soup containing a mix of vegetables: carrots, pumpkin, white beans, turnip and cabbage. Both foods were selected to represent baby food. Furthermore to guarantee that the candidate QCMs have both a relevant matrix and nutrient concentration, commercial materials, purchased at local supermarket were selected according to label information for fat 22 g (soup) and 3 g(milk) and for protein 1 g (soup) and 13 g (milk). Fig. 1 shows a sampling plan according to EuroFir Guidelines.

#### 2.1.2. Packaging materials

High density polyethylene (HDPE) wide-mouthed bottles EU approved food contact materials were used for bottling ready-to-eat soup.

Two kinds of low-density polyethylene (LDPE) bags were tested for compliance with EU directives for food contact materials and for assessing the impact of packaging on stability testing of powdered milk. Plastic A is a film for preservation of foodstuffs and plastic B is a plastic to freeze foodstuffs. Bags on roll are open at one end and pre-sealed at other end. Bags were taken for analysis at regular intervals according to a suitable cutting plan to trace back every single unit (Fig. 2).

### 2.2. Preparation of the food materials

Materials purchased at local supermarket were delivered to laboratory. Twenty four units of 250 g soup package and ten units of 1 kg milk powder were stored till processing at room temperature. The homogenisation of materials was done with a blender for 2 h in a polyethylene high-density container. Milk batches were dispensed as 50 g units in plastic sachets. After filling each sachet was sealed in a single chamber packaging machine (Multivac GB- prüfzert) For homogeneity and stability studies random sachets were collect during first middle and last third of dispensing process. The bottling process of soup consisted in hand-filling the HDPE bottles to a standardised volume 50 mL, after filling bottles were closed with a HDPE insert and screw cap a random scheme was applied to collect bottles during filling process.

### 2.3. Analytical procedures

The moisture content was determined by dry oven method at $102 \pm 3$ °C for about 2 h and the sample was weighted every 30 min to constant weight (IDF 26A, 1993). Total nitrogen was determined by Kjeldahl method using a Kjeldahl analyzer and total protein was calculated applying 6.38 (milk) or 6.25 (soup) Jones' factor (AOAC Method 991.20).

Ash content was determined by dry ashing samples in a muffle furnace at $525 \pm 5$ °C for 2–3 h (AOAC Method 930.30). Fat was determined by the acid-butirometric method with slight changes. In brief, a solution is prepared with the milk-based powdered infant formula sample, according to the level of fat excepted. In this study