



## Validation of a sampling plan to generate food composition data



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### ABSTRACT

A methodology to develop systematic plans for food sampling was proposed. Long life whole and skimmed milk, and sunflower oil were selected to validate the methodology in Argentina. Fatty acid profile in all foods, proximal composition, and calcium's content in milk were determined with AOAC methods. The number of samples ( $n$ ) was calculated applying Cochran's formula with variation coefficients  $\leq 12\%$  and an estimate error ( $r$ ) maximum permissible  $\leq 5\%$  for calcium content in milks and unsaturated fatty acids in oil.  $n$  were 9, 11 and 21 for long life whole and skimmed milk, and sunflower oil respectively. Sample units were randomly collected from production sites and sent to labs. Calculated  $r$  with experimental data was  $\leq 10\%$ , indicating high accuracy in the determination of analyte content of greater variability and reliability of the proposed sampling plan. The methodology is an adequate and useful tool to develop sampling plans for food composition analysis.

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### 1. Introduction

Food composition data are widely used to monitor nutrient intake within populations, to develop national and regional nutrition policies and dietary guidelines, to plan and conduct health research related to dietary intake, and to support food trade. Composition data for foods must be appropriate in level of detail, documentation, statistical parameters and other information to support the needs of those organizations and investigators who will use the data for diverse applications. Today there is a demand for reliable food composition information that can be validated (Chu et al., 2009; Noncioli, 2013).

A food composition database (FCDB) is an electronic repository of food names and descriptions, food component values, and related documentation which are generated or acquired from various sources, including scientific literature. Whether the purpose is to generate data for a new database or to expand/upgrade the quality of existing data in a database, a well-constructed approach to sampling the key foods is required to ensure overall usefulness and quality of the database. The lists of foods that contribute in approximately the 80% of the intake of any one specific nutrient of the diet are identified as key foods (Haytowitz, Pehrsson, & Holden, 2000).

Food sampling methods are critical to generate reliable data that represent the composition of the food of interest. A sampling

plan is a specific procedure for the selection, extraction, preservation, transportation and preparation of the parts to be removed from a population to serve as samples (Horwitz, 1990). The process of defining all types of units of food that constitute the population (i.e., foods) of interest is a primary step. A portion of a material selected from a larger quantity of material is called a sample (Greenfield & Southgate, 2003, chap 6; Holden & Davis, 1997, chap 12).

When a food is collected specifically to generate results for a FCDB it is of primary importance that samples are representative of the food item that is consumed or sold. Analysis of the nutritional composition of a food material depends on the successful completion of a number of different steps. The final report of the Third International Conference Food Data (1999), incorporated a list of the critical steps in the food sampling process. The list included: set up goals; foods to sample; food components to analyze; required number of samples; selection, preparation and transportation of samples; analytical procedure; statistical analysis and data reporting. The sampling plan also seeks to determine a representative average value for each analyte of interest, and to estimate variability of nutrients and foods.

After determining the ranked list of foods to be sampled (key-foods) and the nutrients of interest, it will be important to determine where the food sample units will be selected. The systematic approach to determine where to sample will be called the sampling frame (Cochran, 1977), and must give information related to what, where, how much, and when to sample. The sampling frame must be actualized to secure reliable estimates with known

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variability for the nutrient content of food and beverages consumed by the population (Pehrsson, Perry, & Daniel, 2013; Haytowitz, Pehrsson, & Holden, 2008).

To determine the required number of units ( $n$ ), statistical levels of confidence must be set in order to reflect the variability in the nutrient food composition.

The design of a food sampling protocol requires a general knowledge of the food type or class, as well as information about the specific foods or products within that class (e.g., poultry products, parts, with/without skin). It is important to know the way(s) the food(s) are available (raw, refrigerated, frozen), prepared (fried, baked, cooked dish), distributed, and consumed. The decisions for foods, forms, nutrients, and sampling options should be driven by the sampling objective(s) (Haytowitz, Pehrsson, & Holden, 2000). The definition of the objectives will depend upon the resources available as well as the project goals to be achieved. The sampling plan to generate food composition data may have different goals. They can be applied to determine nationally representative estimates of the composition of foods (Galeazzi, Lima, Colugnati, Padovani, & Rodriguez-Amaya, 2002; Pehrsson, Haytowitz, Holden, Perry, & Beckler, 2000; Nickle & Pehrsson, 2013); to conduct a pilot study to determine the magnitude of variances associated with specific parameters (e.g., cultivar, feed, breed) (Davey et al., 2007); to generate comprehensive and representative data for a specific nutrient (Holden et al., 2005), among others.

Under the FAO project TCP/RLA/3107 “Developing Food Composition Data Base for Argentina, Chile and Paraguay to strengthen the international trade and consumers’ protection”, a probability-based methodology to systematically develop sampling plans for food analysis was proposed. The methodology discussed and approved in a Workshop of the FAO project, was included in the Sampling Workbook for Latin American Countries in process of publishing (Holden, Pehrsson, Perry, & Greenfield, 2012). It has a structure of five steps (Samman, Masson, de Pablo, & Ovelar, 2011) and was developed in order to ensure that the data are truly representative of the national food supply.

Usually a large number of samples, depending on the food type and variables affecting their composition, must be analyzed to obtain representative data (Galeazzi et al., 2002; Greenfield et al., 2009; Tarley, Visentainer, Matsushita, & de Souza, 2004). The objective of the proposed methodology is to select the main variables that influence the composition of each food, discarding the less important in order to reduce variability, and thereby the samples number analyzed, without losing its statistical significance.

Fig. 1 summarizes the main steps of the proposed methodology and Fig. 2 includes two approaches used to define where to sample according to production or consumption conditions. Food samples could be selected in shops, supermarkets, street vendors, farms or homes, or any other location in every region, or province in the country. Types of production or consumption of food may determine the decision of where to sample. In many countries of Latin America, including Argentina, statistics are unavailable on consumption data or sales distribution in supermarkets. It is therefore necessary to look for other alternative sources of information on where to sample.

If production is concentrated in a defined region or in few factories it would be convenient to take samples in manufacture/production places. In this case  $n$  could be distributed proportionally to production volume of each company. If production is widespread throughout the country, it would be desirable to take samples at points of consumption.

Cochran uses the human population as a model system. When addressing the development of the sampling frame, Cochran recommended that “the population must be divided into parts that are called sampling units or simply units”. These units must cover

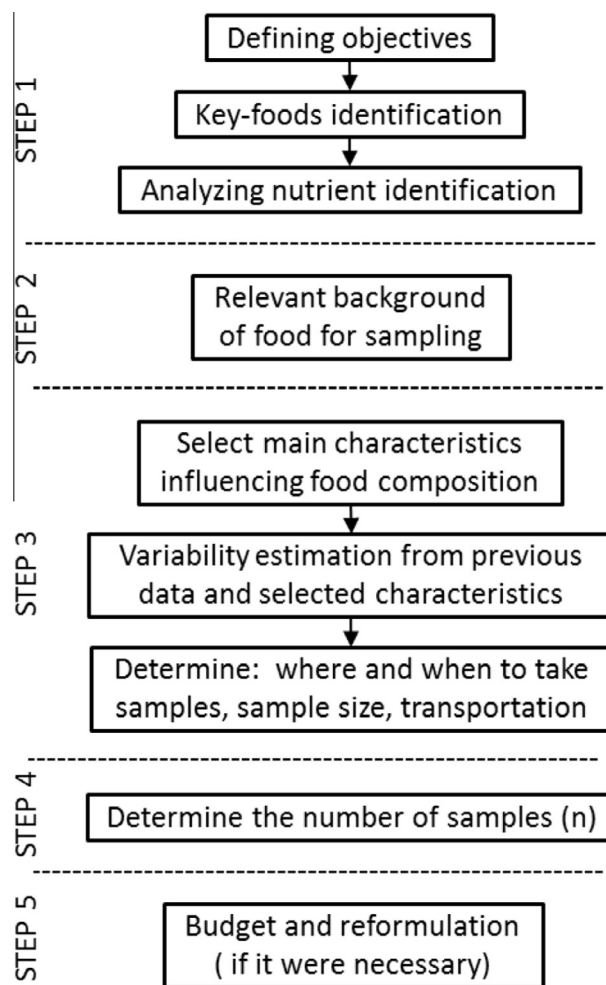


Fig. 1. Main steps of the proposed methodology to systematically develop sampling plans for food analysis in Latin America.

the whole population but they must not overlap, in the sense that every element in the population belongs to one and only one unit (Cochran, 1977). Therefore, it will be necessary to use a systematic approach to identify the list of all possible locations. From this list it will be necessary to select a subset of locations where the actual selection of the food sample units can take place. The number of locations could be determined, in part, by the objectives and scope of the study and in part, by the amount of funding and other resources available. In this case “ $n$ ” is distributed randomly in selected cities with probability proportional to their population. For example, Perry, Pehrsson, and Holden (2003) obtained the U.S. National Census Data and identified the U.S. County as the unit of interest for the sampling frame of the National Food and Nutrient Analysis Program (NFNAP). The US Department of Agriculture’s (USDA) Nutrient Data Laboratory develops a second revision of the NFNAP sampling plan implemented in 2012, for the national collection of food samples from retail outlets for nutrient analysis. In that case, Chromy’s Procedure, a probability minimum replacement probability proportional to size sampling scheme was used (Pehrsson et al., 2013). In this way, in TACO project (Tabela Brasileira de Composição de Alimentos), foods were selected from nine cities in the five official Brazilian geopolitical regions (Galeazzi et al., 2002).

Careful handling of food samples from the time of acquisition to the time of analysis is critical to ensure the integrity of the samples and subsequent generation of accurate nutrient values (Trainer et al., 2010; Westenbrink, Oseredczuk, Castanheira, & Roe, 2009).

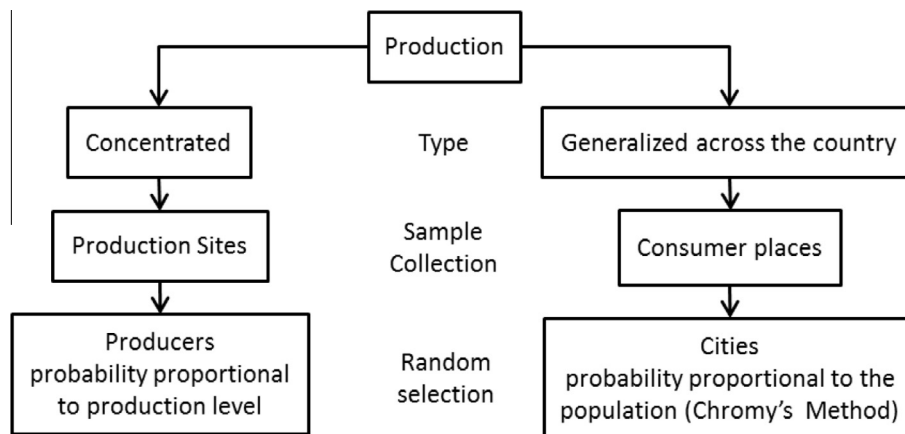


Fig. 2. Different ways to decide where to sample.

During the project sampling plans for 30 key-foods previously determined for each country (Argentina, Chile and Paraguay) were developed. Five of them were selected to implement the developed sampling plan and subsequent analysis in each country.

The main objective of this work was to validate experimentally the proposed methodology for the case of foods sampled in production/industrialization locations.

## 2. Materials and methods

### 2.1. Applying the sampling plan

#### 2.1.1. Step 1: Defining the purpose of sampling, selecting the food(s) and nutrient(s) to be analyzed

The objective of sampling plans showed in this work was to generate data for National TCA. The selected foods were long life whole and skimmed milk, and sunflower oil. These foods were chosen because sampling was conducted in production areas, one of the alternatives proposed in Fig. 2. The determined nutrients were proteins, lipids and fatty acid profiles, ash, dry matter and calcium in milks and fatty acid profile in sunflower oil. All the analytical determinations were performed using AOAC methods (AOAC, 1995). Dry matter was determined by drying in convection oven AOAC 925.23 method. Lipid content was determined according to alkaline hydrolysis method, AOAC 905.02. Total protein content was determined using Kjeldahl (BUCHI DIGESTIÓN UNIT K-435) procedure with a nitrogen-to-protein conversion factor of 6.38, AOAC 991.20 method. Ash analysis used a carbonization at 550 °C (Muffle furnace), AOAC 945.46 method. Total calcium content was analyzed using atomic absorption spectrometer. Calibration of the measurements was performed using commercial standards, AOAC 991.25 method. The fatty acid profile was determined using gas chromatography after methylation acid process UNE 55-037-73 (AENOR, 1991). Analyses were performed in triplicate for each primary sample received at laboratory.

#### 2.1.2. Step 2: Relevant background of food for sampling plan development

**2.1.2.1. Whole and skimmed milk.** Argentina produce around 10,000 million liters of milk per year, rank 18 among the producing countries, with a contribution of 1.7% of the global production, and 2.6% to the world exported milk. 80% of the total milk production is used to manufacture dairy products. The remainder 20% is used to process different types of fluid milk. The main milk producing provinces are Santa Fe (33%), Córdoba (32.5%), Buenos Aires (28%), Entre Ríos (4%), La Pampa (1.5%), and other provinces (1%). About 99% of the processing plants are located in the Pampa region.

All the milk production is transported to large processing companies located in the provinces of Buenos Aires, Santa Fe, and Córdoba. In Argentina there are about 12 large milk companies, of which the three most important are SANCOR, Mastellone and Milkaut, that processed about 80% of total production (CIL, 2013).

In Argentina dairy cattle breeds are Holstein-Argentina and Jersey. Historically, there was a reduction in milk production in autumn and especially in winter, but currently the effect of seasonality has practically disappeared by the addition of fodder reserves in cattle feeding scheme.

Raw milk is gathered directly to the manufacture plants where milk is analyzed and classified. The final destination is defined according to its quality: manufacture of dairy products or fluid milk. Thermal process versatility has generated different types of fluid milk, such as pasteurized, ultra pasteurized and Ultra High Temperature (UHT) pasteurized milk. Jointly with the modernization process of fat stabilization, different kinds of fluid milk are available: whole (3% of fat), partially skimmed (1.5–2% of fat), or totally skimmed (0–0.3% of fat) milk. In the national market milk is available as: milk with vitamins A and D, milk with conjugated linoleic acid (CLA), milk with fiber, milk with iron, milk with extra calcium, lactose-free milk, and flavored milks (CIL, 2013).

**2.1.2.2. Sunflower oil data.** Sunflower oil was selected because it is the one most consumed in the country. The average national consumption is 9.6 kg/person/year, including fresh oil and processed products with sunflower oil. Argentina has a greater production of soybean oil but very little is used for direct consumption; most of it is used to produce margarine, dressings, shortening, or it is exported. The product of interest for the sampling is pure sunflower oil. This type of oil is produced in two varieties: high oleic and medium oleic sunflower oil. The first one is used only by the food industry and the medium oleic sunflower oil is commercialized in the retail market.

There are three production areas in Argentina: North (Formosa, Chaco, and Salta Provinces); Center (Entre Ríos, Córdoba, and Santa Fe Provinces) and South (Buenos Aires and La Pampa Provinces). The installed processing capacity of these areas is 480, 26,000, and 17,000 Tn/24 h, respectively. About 80% of the processing plants and the production area of sunflower oil are located in the Pampa region. The main seed production is seasonal with one crop a year, which is stored and processed at manufacturing plants throughout the year.

All production of sunflower oil is distributed throughout the country in bottles of 500, 900, 1500, 3000, and 5000 mL. They are available in all supermarkets and small stores. The main commercial brands of sunflower oil are: Cocinero, Natura, Patito, and Ideal,

which cumulatively constitute over 90% of the total market (Franco, 2010).

### 2.1.3. Step 3. Developing the sampling frame

- (a) The main characteristic that influence the food composition were selected by analysis of the information carefully acquired from the bibliography (Step 2).

**Milk:** It was decided to take samples of whole and partially skimmed milk, both in UHT or long life condition. In all cases milk samples were fortified with A and D vitamins.

**Oil:** Pure sunflower oil bottles of different brands were sampled, without differentiating between them because each factory brand respond to different positions in the market, with the same composition.

- (b) Variability estimation: The coefficient of variation and estimation error (difference between sample mean and population mean) for calcium content in whole and skimmed milk and unsaturated fatty acid content in sunflower oil were calculated from previous data from LATINFOODS TCA (LATINFOODS, 2012) and ASAGA (unpublished data). The accepted limit of estimation error was 10% (Holden and Davis, 1997, chap 12).
- (c) Where and when to take samples, sample size:

It was decided to sample long life fluid milk, in tetra bricks packaging, of different brands, processed by the main dairy companies, SANCOR, Mastellone and Milkaut. The number of samples was proportional to their production volume (Table 1). The processing volume of these three companies represents approximately 80% of the national production. Sampling was performed at any time since there was no effect of seasonality in fluid milk production. The size of each primary sample was defined as 4 tetra bricks packaging of 1000 mL each one because it should cover the needs for analytical determinations. The samples were taken from the processing line in each company, at random after filling, and before packing in packs.

Sunflower oil samples were taken from the five companies with the largest pure sunflower oil production, at any time throughout the year including all brands belonging to the selected factories. They represent approximately 80% of the Argentine annual production volume. Table 1 shows the selected companies, all of them located at Pampas and Northeast regions. The sample unit was

defined as 4 bottle of 900 mL each one. Samples were taken in stowage deposits of each company. Four pallets were chosen randomly, a box was taken, and the center bottle was extracted. These make up the sample unit. The process was repeated until the total number of samples was completed.

In every company a record similar to an official sample routine inspection was completed when samples were taken. Each unit sample (composed of 4 individual packages for both, milk or oil) was wrapped in a plastic bag, identified and placed in a box labeled for shipping. The transport service withdrew samples from each sampling site the scheduled day and shipped them immediately to the Food Laboratory, Faculty of Engineering, National University of Jujuy. The 4 packages contents, which constitute a unit sample, were mixed in a container and homogenized by manual shaking. Then, aliquots were removed for analysis. Aliquots were also immediately sent to the other participating laboratories.

### 2.1.4. Step 4: Calculating the number of samples

The number of samples to analyze ( $n$ ) is critical in order to estimate the mean and the magnitude of variances. The methodology proposed for the sampling plan is to calculate  $n$  with Cochran's formula, which is applied iteratively until convergence of two consecutive values (Cochran, 1977; Holden and Davis, 1997, chap 12). The nutrient with greater variability for each food was considered. They were calcium for milk and polyunsaturated fatty acid for sunflower oil.

$$\text{Cochran's formula: } n \geq \left( t_{\alpha(n-1)} \right)^2 \frac{(SD)^2}{r^2 \bar{y}^2}$$

$n$ : Number of samples

$\frac{SD}{\bar{y}}$ : Variation coefficient of most variable nutrient

$t$ : Student's  $t_{(n-1)} (1-\alpha)$  – desired level of confidence

$r$ : desired relative error limit

#### 2.1.4.1. Example: Calculating the number of samples ( $n$ ) for whole milk UHT.

- From previous data: nutrient with highest variability: Ca; variation coefficient of Ca content in whole milk UHT: 0.047; desired relative error limit  $r = 0.03$
- Assuming  $n = 7$  to start iterations in Cochran's formula, from table of Student's  $t$ -distribution  $\rightarrow t_{(7-1)} (1-0.05) = 1.9432$
- Substituting the above values in the right side term in Cochran's formula, a new  $n \geq 10$  could be calculated.

$$\text{With } n = 10 \rightarrow t_{(9; 0.05)} = 1.833 \text{ Replacing } \rightarrow n \geq 9$$

**Table 1**  
Sample distribution by company.

Companies	Factories location	Brands	Processing volume	N° samples ( $n$ )	
				Whole	Partially skimmed
<i>(a) Sampling for milk by type</i>			10 <sup>6</sup> (L/day)		
Mastellone	Buenos Aires	Serenísima	6.0	4	5
Sancor	Buenos Aires	Sancor	6.0	2	3
	Santa Fe			2	2
Milkaut	Santa Fe	Milkaut	2.0	1	1
Total			14.0	9	11
<i>(b) Sampling for sunflower oil</i>			Tn/24 h	Sunflower oil	
Molinos Río de la Plata SA	Buenos Aires	Cocinero. Patito. Lira.	980	4	
	Santa Fe			4	
Aceitera Gral. Deheza SAICA	Córdoba	Natura, Familiar, Cada día	500	4	
Nidera S.A.	Buenos Aires	Legítimo	500	4	
Molino Cañuelas	Buenos Aires	Cañuelas, Comodín	300	3	
Vicentín S.A.	Santa Fe	Vicentín	220	2	
Total			2.500	21	

With  $n = 9 \rightarrow t_{(8; 0.05)} = 1.860$  Replacing  $\rightarrow n \geq 9$

With three iterations in Cochran's formula  $n$  converge in 9 so, this is the number of samples.

### 2.1.5. Step 5: Budget and reformulation if necessary

All samples were donated by companies in which foods were sampled. The samples transport and analysis costs were covered with funds from the FAO project and resources of the participating laboratories.

The sampling plan was implemented through the National Network of Food Protection (RENAPRA), Ministry of Agriculture, Livestock and Fisheries of Argentina, inspectors of the network collected the samples. A guideline for sampling and samples submission was prepared (Sammán & Kleiman, 2009). It details all the important aspects for sampling implementation, including methods for sample handling and transportation.

**2.1.5.1. Selecting qualified laboratories and analytical methods.** The selected laboratories for samples analysis were: Department of Food Science, Faculty of Pharmacy and Biochemistry, University of Buenos Aires to analyze sunflower oil, Food National Institute (INAL) for milk, and Food Laboratory, Faculty of Engineering, National University of Jujuy for both foods.

Previously, in order to determine the laboratories quality, an Analytical Proficiency Laboratory Test was performed under the Swedish Food Agency direction. Three reference standard food matrices were used: (1) Meat based food for proximate analysis and fatty acid composition, including trans fatty acids. (2) Cereal for dietary fiber, sodium and iron. (3) Juice for vitamin C and beta-carotene.

## 3. Results and discussion

### 3.1. Samples number calculation

Theoretical values of variation coefficient and relative error for whole and partially skimmed milk and sunflower oil used in the

**Table 2**  
Indicators of sampling variability.

Variability	Food	Used	Estimate	Results
Coefficient of variation (%)	Sunflower oil	12.0	5.7	High precision
	Whole milk	4.7	8.8	
	Partially skimmed milk	3.6	6.2	
Estimation error (%)	Sunflower oil	4.5	2.1	Acceptable accuracy
	Whole milk	3.0	5.8	
	Partially skimmed milk	2.0	3.6	

**Table 3**  
Milk Composition.

Milk type	Whole (n = 9)					Partially skimmed (n = 11)					
	Brand (n)	Sancor <sup>*</sup> (4)	La Suipa chense <sup>*</sup> (4)	Milkaut <sup>*</sup> (1)	Mean <sup>**</sup> (9)	CV <sup>**</sup> (%)	Milkaut <sup>*</sup> (-1)	Coto <sup>*</sup> (-5)	Las tres Niñas <sup>*</sup> (5)	Mean <sup>**</sup> (11)	CV <sup>**</sup> (%)
Moisture (g/100 g)		88.4	88.7	88.2	88.5	0.23	90	90.9	89.9	90.3	0.62
Total solids (g/100 g)		11.6	11.3	11.8	11.5	1.78	10	9.1	10.2	9.66	5.75
Protein (g/100 g)		3.23	3.13	3.35	3.2	3.28	3.3	3.13	3.4	3.27	7.7
Ash (g/100 g)		0.66	0.68	0.7	0.67	2.57	0.68	0.65	0.7	0.67	5.53
Lipids (g/100 g)		3.05	3	3.14	3.04	1.62	1.88	1.58	1.66	1.6	6.88
Ca (mg/100 g)		120	132	137	127	8.8	119	115	131	123	6.2
SAFA (g/100 g lipid)		66.7	66.6	67.2	66.7	0.8	72.4	70.8	69.8	70.5	1.3
MUFA (g/100 g lipid)		29.7	29.8	29.7	29.7	1.4	25	26.3	26.9	26.4	2.8
PUFA (g/100 g lipid)		3.65	3.61	3.16	3.58	5.6	3.16	2.92	3.33	3.12	7.15

n: number of samples; CV: coefficient of variation.

<sup>\*</sup> Mean value corresponding to samples of each commercial brand.

<sup>\*\*</sup> Mean value and CV corresponding to all samples of milk.

calculating process of the number of samples ( $n$ ) are shown in Table 2. Number of samples calculated by Cochran's formula was 9 and 11 for whole and partially skimmed milk respectively. Calcium was used as the nutrient with greatest variability. The number of samples was 21 for sunflower oil. The nutrient considered of greater variability was the content of unsaturated fatty acids.

In developing the sampling plan, enough observations are desired to obtain reasonably representative estimates of parameters of interest. However, this must be compatible with the available budget for sampling and analysis, which usually is limited. In the analyzed cases the low variability and standard deviations of the samples found in previous data for both foods helped to define a small sample number ( $n$ ) to be representative of the nutritional components of selected foods, both of high consumption.

### 3.2. Analytical results

The proximal composition, calcium content and fatty acid composition of whole and partially skimmed milk are shown in Table 3. The protein and lipids contents are within the range established by the Código Alimentario Argentino (ANMAT, 2013). Calcium concentration had the greatest variability as it was expected. Values for these nutrients as well as the profile of fatty acids are consistent with those reported by several FCDB and TCA (Agricultural Research Service, 2013; National Food Institute, 2013; INCAP, 2012).

The analytical results of fatty acid composition of sunflower oil are shown in Table 4. They are within the range established by the Código Alimentario Argentino (ANMAT, 2013) and are consistent with those informed by FCDB of USDA (Agricultural Research Service, 2013); Denmark (National Food Institute, 2013) and TCA of Central America (INCAP, 2012).

The results indicate that samples of both whole and skimmed milk are homogeneous and that the nutrient content did not vary significantly between brands, confirming the hypothesis that the variables selected for the development of the sampling frame are the most changeable in the composition. For sunflower oil it was assumed that the greater variability correspond to PUFA fraction, however the results indicate greater variability for MUFA content. Significant variability between brands was not observed.

Table 2 shows the results obtained for the sampling variability indicators. In the case of sunflower oil, the estimation error and the coefficient of variation calculated with the experimental data were less than the used for the development of the sampling plan. This shows that the composition obtained is within the range of variability expected, regardless of the nutrient preselected as the most variable. In milk case, both indicators were higher than those used

**Table 4**  
Fatty acids content in sunflower oil (g/100 g methyl ester).

Brands	Cocinero <sup>a</sup>	Natura <sup>a</sup>	Legítimo <sup>a</sup>	Cañuelas <sup>a</sup>	Vicentin <sup>a</sup>	Mean <sup>**</sup>	SD <sup>**</sup>	CV (%)
n	8	4	4	3	2	21		
C 16:0	6.06	5.92	6.37	6.13	5.68	6.07	0.20	3.25
C 18:0	3.52	3.53	3.37	3.15	3.41	3.43	0.14	4.17
C 20:0	0.26	0.26	0.25	0.23	0.25	0.25	0.01	4.54
C 22:0	0.68	0.73	0.67	0.68	0.72	0.69	0.04	5.51
C 18:1	29.2	30.2	27.5	27.4	36.0	29.5	2.46	8.36
C 20:1	0.24	0.23	0.23	0.25	0.27	0.24	0.03	12.8
C 18:2	60.1	59.1	61.6	62.2	53.7	59.9	3.4	5.7
SAFA	10.72	10.44	10.66	10.19	10.06	10.37	0.24	2.36
MUFA	29.44	30.52	27.73	27.65	36.27	30.30	3.52	11.62
PUFA	60.10	59.10	61.60	62.20	53.70	59.33	3.37	5.69

SD: standard deviation, CV: coefficient of variation.

<sup>a</sup> Mean value corresponding to samples of each commercial brand.

<sup>\*\*</sup> Mean value, SD and CV corresponding to all oil samples.

for development the sampling plans. However, in all cases *r* values used for the calculation of *n* were very low (3.0% and 2.0%); normally the accepted values of *r* are <10% and CV >12,5% (Holden and Davis, 1997, chap 12) indicating that values obtained are acceptable.

The results would indicate high accuracy and reliability in the overall process of sampling and statistical treatment of the data obtained.

We can affirm that the mean analytical values obtained represent the composition of whole and partially skimmed milk, pasteurized by UHT system, fortified with A and D vitamins and pure sunflower oil which is mostly consumed by the Argentinean population.

#### 4. Conclusions

A methodology that could be useful for those who wish to generate food composition data was proposed. The data obtained confirmed the initial hypotheses and allows concluding that the proposed methodology is adequate to develop food sampling plans for composition analysis purposes.

The sampling design and the results show that it is possible to take samples at the places of production and achieve representative data of the composition of food consumed/sold in a country. This methodology could be adopted by the LATINFOODS network for their regional FCDB, and also by other countries.

It is necessary to continue studying sampling methods to be applied when required to generate/update food composition data, especially for foods whose production is widespread throughout the country.

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#### References

AENOR. (1991). Spanish association for standardization. UNE 55037-73. UNE Standards Catalogue. Madrid.  
Agricultural Research Service. SR25 – Reports by single nutrient. USDA. URL <<http://www.ars.usda.gov/Services/docs.htm?docid=22114>> (Accessed 15.12.13).  
ANMAT. Código Alimentario Argentino. Administración Nacional de Medicamentos, Alimentos y Tecnología Médica. Argentina. <[http://www.anmat.gov.ar/alimentos/normativas\\_alimentos\\_caa.asp](http://www.anmat.gov.ar/alimentos/normativas_alimentos_caa.asp)> (Accessed 15.12.13).

AOAC (1995). *Official methods of analysis of the Association of Official Analytical Chemists* (16th ed.). Washington, DC: Association of Official Analytical Chemists.  
Chu, D. H., Lee, M. S., Hsu, Y. H., Yu, H. L., Wu, T. Y., Chang, S. C., et al. (2009). Quality assurance with an informatics auditing process for food composition tables. *Journal of Food Composition and Analysis*, 22(7–8), 718–727.  
CIL. *Estadísticas*. Centro de la Industria Lechera Argentina. Argentina. <<http://www.cil.org.ar/indices.html>> (Accessed 28.11.13).  
Cochran, W. G. (1977). *Sampling techniques* (3rd ed.). New York: Wiley.  
Davey, M. W., Stals, E., Ngoh-Newilah, G., Tomepke, K., Lusty, C., Markham, R., et al. (2007). Sampling strategies and variability in fruit pulp micronutrient contents of West and Central African bananas and plantains (*Musa* species). *Journal of Agricultural and Food Chemistry*, 55, 2633–2644.  
Franco, D. (2010). *Girasol y soja*. Argentina: Alimentos Argentinos, Secretaría de Agricultura, Ganadería y Pesca. Ministerio de Agricultura. 38–43.  
Galeazzi, M. A. M., Lima, D. M., Colugnati, F. A. B., Padovani, R. M., & Rodriguez-Amaya, D. B. (2002). Sampling plan for the Brazilian TACO project. *Journal of Food Composition and Analysis*, 15, 499–505.  
Greenfield, H., & Southgate, D. A. T. (2003). Sampling. In B. A. Burlingame & U. R. Charrondiere (Eds.), *Food composition data: Production, management and use*. Rome: FAO.  
Greenfield, H., Arcot, J., Barnes, J. A., Cunningham, J., Adorno, P., Stobaus, T., et al. (2009). Nutrient composition of Australian retail pork cuts 2005/2006. *Food Chemistry*, 117, 721–730.  
Haytowitz, D. B., Pehrsson, P. R., & Holden, J. M. (2008). The national food and nutrient analysis program: A decade of progress. *Journal of Food Composition and Analysis*, 21, S94–S102.  
Haytowitz, D. B., Pehrsson, P. R., & Holden, J. M. (2000). Setting priorities for nutrient analysis in diverse populations. *Journal of Food Composition and Analysis*, 13, 425–433.  
Holden, J., & Davis, C. (1997). Estrategias para muestreo: el aseguramiento de valores representativos. In *Producción y Manejo de Datos representativos de alimentos en nutrición*. Rome: FAO y Universidad de Chile.  
Holden, J. M., Bhagwat, S. A., Haytowitz, D., Gebhardt, S., Dwyer, J., Peterson, J., et al. (2005). Development of a database of critically evaluated flavonoid data: Application of USDA's data quality evaluation system. *Journal of Food Composition and Analysis*, 18, 829–844.  
Holden, J. M., Pehrsson, P., Perry, C., & Greenfield, H. (2012). *Sampling workbook for Latin American countries*. Rome: FAO (Unpublished results).  
Horwitz, W. (1990). Nomenclature for sampling in analytical chemistry (Recommendations 1990). *Pure and Applied Chemistry*, 62(6), 1193–1208.  
INCAP (2012). *Tabla de Composición de Alimentos de Centroamérica* (2nd ed.). Instituto de Nutrición de Centroamérica y Panamá Organización Panamericana de la Salud.  
LATINFOODS. *Tabla de Composición de Alimentos de América Latina*. Red Latinoamericana de Composición de Alimentos. <<http://www.inta.cl/latinfoods/>> (Accessed 11.07.2012).  
National Food Institute. *The official Danish Food Composition Database*. Technical University of Denmark. <[http://www.foodcomp.dk/v7/fcdb\\_default.asp](http://www.foodcomp.dk/v7/fcdb_default.asp)> (Accessed 11.11.2013).  
Nickle, M., & Pehrsson, P. (2013). USDA updates nutrient values for fast food pizza. *Procedia Food Science*, 2, 87–92.  
Noncioli, A. (2013). La importancia del muestreo en la cadena agroalimentaria. *Alimentos Argentinos*, 60, 70–77.  
Pehrsson, P. R., Haytowitz, D. B., Holden, J. M., Perry, C. R., & Beckler, D. G. (2000). USDA's National Food and Nutrient Analysis Program: Food sampling. *Journal of Food Composition and Analysis*, 12, 379–389.  
Pehrsson, P., Perry, C., & Daniel, M. (2013). Nutrient ARS, USDA updates food sampling strategies to keep pace with demographic shifts. *Procedia Food Science*, 2, 52–59.  
Perry, C.R., Pehrsson, P.R., & Holden, J. (2003). A revised sampling plan for obtaining food products for nutrient analysis for the USDA National Nutrient Database. In: *Proceedings of the American Statistical Association, Section on Survey Research*

- Methods [CD-ROM]*, Alexandria, VA, San Francisco, CA: American Statistical Association.
- Samman, N., & Kleiman, E. (2009). *Directrices para el Muestreo y Remisión de Muestras*. Muestreo de Alimentos Prioritarios, Red Nacional de Protección de Alimentos (RENAPRA). 40p.
- Samman, N., Masson, L., de Pablo, S., & Ovelar, E. (2011). Food composition activities in Argentina, Chile and Paraguay. *Journal of Food Composition and Analysis*, 24(4–6), 716–719.
- Tarley, C. R. T., Visentainer, J. V., Matsushita, M., & de Souza, N. E. (2004). Proximate composition, cholesterol and fatty acids profile of canned sardines (*Sardinella brasiliensis*) in soybean oil and tomato sauce. *Food Chemistry*, 88, 1–6.
- Third International Conference Food Data. (1999). Final report – FAO. Rome, Italy (5–7 July).
- Trainer, D., Pehrsson, P. R., Haytowitz, D. B., Holden, J. M., Phillips, K. M., Rasor, A. S., et al. (2010). Development of sample handling procedures for foods under USDA's National Food and Nutrient Analysis Program. *Journal of Food Composition and Analysis*, 23, 843–851.
- Westenbrink, S., Oseredczuk, M., Castanheira, I., & Roe, M. (2009). Food composition databases: The EuroFIR approach to develop tools to assure the quality of the data compilation process. *Food Chemistry*, 113(3), 759–767.