Fresenius J Anal Chem (1990) 338:473–475

Fresenius' Journal of Analytical Chemistry

Mixed diet reference materials for nutrient analysis of foods: Preparation of SRM-1548 Total Diet

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Summary. Several mixed component diet materials have been available for use as reference materials for determination of elemental contents in food. These included H-9, issued by the IAEA and RM-8431, issued by NIST. Wide acceptance of these materials has exhausted supplies and they are no longer available. A new mixed food Standard Reference Material, SRM-1548 Total Diet, has been prepared and characterized for a number of elemental and organic constituents. This material was prepared from foods obtained from collections of the US FDA's Total Diet Study, which are representative of foods consumed by the US population. SRM-1548 has been composited from these foods, in proportions that are representative of daily intake. The composited foods were blended, freeze-dried, reblended and bottled in portions of 6 g. Homogeneity studies were carried out along with certification analyses for elements, cholesterol proximate and caloric content. This material is available from NIST and will be useful as a multi-purpose SRM for determinations of constituents in the naturally occurring range in foods and food related materials. Since this is a mixture of foods of both good and poorer sources, levels will be at the lower end of the concentration range for any individual constituent. Availability of further SRM's of individual foods with higher levels of specific constituents, such as SRM-1845 Cholesterol in Whole Egg will be required to extend the range of certified concentration values for food reference materials.

Introduction

A previously reported multidimensional evaluation of data for inorganic nutrients in foods has shown that most of the available "certified" reference materials, which are single component matrices, are "not" representative for groups of commonly consumed foods [1]. Several mixed component diet materials have been prepared and characterized for elemental content for use as reference materials for food analysis. These include H-9, prepared by the International Atomic Energy Agency (IAEA), and RM-8431, prepared by the USDA [2] and distributed by the NIST [3]. Wide acceptance and demand for these materials has resulted in exhaustion of supply and neither is still available. A new mixed food component material, has been prepared and is being characterized as a multipurpose material [4] for both inorganic and organic constituents.

This material (Standard Reference Material 1548 Total Diet) was developed in conjunction with an international program for Assessment of Dietary Intakes of Trace Elements, coordinated by the IAEA, Vienna, Austria [5]. The United States effort in this program involves making a composite diet using excess foods collected in the on-going FDA Total Diet Study (TDS) Program [6]. Procedures used in these efforts formed the basis for putting together the material for SRM-1548 Total Diet. This paper will briefly describe the background leading to development of SRM 1548 and its preparation.

US FDA Total Diet Study Program [6]

In this program, 234 individual foods and food items, representative of diets of the United States population, are collected in three cities within each of four geographical regions of the United States. Four to five collections per year are obtained. The individual foods from each collection are sent to the Total Diet Laboratory in Kansas City, MD, where they are prepared ready-to-eat form. Composites are then made of the three like food items collected within each region. Aliquots of each of these 234 food item composites are analyzed for a series of constituents as part of a nationwide monitoring program.

IAEA Coordinated Program On Dietary Intake Assessment [5]

In this program, mixed dietary samples representative of daily dietary intake are collected in each of thirteen participating countries and analysed for trace element content by several reference laboratories. The United States participation in this program is a joint NIST-FDA-USDA effort which includes compositing of representative diet samples utilizing foods from the FDA Total Diet Study Program [7]. After analytical samples were taken from the FDA-TDS individual food item composites, as described above, the excess food material for several regional collections was shipped to NIST in a frozen state. At NIST, the 200 individual foods representing adult intake from each collection were individually proportioned to reflect the

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contribution of each food to the daily intake of a representative 25–30 year old male (3075 g total weight of diet). The beverages were pooled, and the solid foods were blended separately in a commercial food blender equipped with specially fabricated Ti blades and a nylon coated bowl to prevent metal contamination. The pooled beverages were then added to the solids and the entire mixture was blended to produce a ten kilogram USDIET composite. These operations were conducted in the NIST National Environmental Specimen Bank (ESB) Clean Room [8].

Each USDIET composite is representative of a daily intake of foods from each regional FDA-TDS collection sampled. Foods for ten of these USDIET composites have been collected over a three year period. Portions of each USDIET composite from these collections are taken for analyses of trace elemental content within the IAEA Coordinated Program. Additional portions are stored in the NIST ESB for future analyses, with yet an additional portion being taken for nutrient profile analyses (proximates and water soluble vitamins) by the FDA, Nutrient Surveillance Branch, Washington, DC.

Standard Reference Material 1548 Total Diet

Significant knowledge and experience had been gained both in the production and characterization of the RM 8431-Mixed Diet and in the compositing and analyses of the diet materials for the IAEA program. Upon the wide acceptance and quick exhaustion of supply of RM 8431, it was decided to produce a replacement material based upon the FDA-TDS materials. This SRM is then representative of the US dietary intake. In addition, it can also be directly linked to the extensive information and data obtained from the materials collected in both the FDA-TDS and the IAEA Programs.

To obtain sufficient material for SRM-1548, we took excess food items from USDIET II–IV collections for the IAEA study. These food items were portioned and composited as previously described except that extra drinking water was omitted to produce six ten-kilogram batches of material. Each of the six batches was freeze-dried separately and the dried batches were reblended under liquid nitrogen. The six batches were then combined and reblended. The combined material was radiation sterilized in bulk (^{60}Co, 3mrad). Prior to bottling, the sterilized bulk material was passed through a 30 mesh polyethylene sieve to remove any coarse material. Sieving was carried out in a "Class 100" clean air hood; the material was kept frozen and handled in small batches to prevent contamination and excess exposure. The resulting material was then placed in glass bottles (2 oz), in a clean air hood; each bottle contained about 6.5 g of the final material.

Characterization and homogeneity of SRM-1548

During the bottling process, every 20th bottle was removed for characterization analyses and homogeneity testing. SRM 1548 was analyzed by a number of individuals and groups, analysts within NIST and outside collaborators, using a variety of different analytical techniques. This material has been characterized for a number of inorganic and organic constitutents. Detailed information on certified values, analytical methodology, and participating analysts is given in the Certificate of Analysis for SRM-1548 available from NIST [9]. Certified information is given for the elements N, S, P, Cl, Na, K, Ca, Mg, Fe, Zn, Mn, Cu, Se, and Cd, values for which are given in another paper in these proceedings [10]. Certified content of fat, protein (Kjeldahl nitrogen), ash, cholesterol, fiber and caloric (bomb calorimetry) content are given in the certificate for SRM-1548. Additional information values are given for the elements Al, Sn, B, Ni, Mo, Pb, Cs, Rb, and Eu. The content of water soluble vitamins in this material can be estimated from published information on analyses of the IAEA USDIET samples [11].

The homogeneity of SRM-1548 was determined by repeat analysis of 10 bottles of the packaged material for a number of trace elements by instrumental neutron activation analysis. Acceptable reproducibility was shown for the elements characterized for certification as reported in the certificate of analysis for SRM-1548 [10]. One element of nutritional concern, chromium, was shown to be significantly inhomogeneous, and several other trace elements showed some evidence of inhomogeneity. While additional analyses by stable isotope dilution mass spectrometry verified the inhomogeneity for chromium in the bottled material, they were not conclusive in showing inhomogeneity for a portion of the unbottled final material [12]. Based on these considerations, SRM-1548 is not certified for chromium content and no use of this material should be made to determine Cr content.

Since the fat content of SRM-1548 is high relative to most other available certified reference materials, it is important to demonstrate homogeneity of the lipid soluble components. Measurement of the cholesterol content, by a highly precise and accurate isotope dilution mass spectrometry technique [13] showed a coefficient of variation of less than 1% for multiple analyses of SRM-1548 [10]. This high fat content also leads to recommendations for extra precautions in handling and use of this material. The material should be kept refrigerated and not exposed to intense direct light, UV radiation, nor excessive atmospheric exposure. Drying studies showed that freeze drying of the material is recommended to remove residual moisture and that elevated temperatures or extended drying times will lead to loss of non-water volatiles, most probably lipid components.

Future programs for food related RM's

It is hoped that SRM-1548 will serve as a multi-purpose material for determinations in the naturally occurring range for the certified constituents in food and food related materials. This material is a complex mixture of foods representative of a total diet and includes both good and poorer sources of any individual constituent. Thus, values for most certified constituents would be expected to be in the lower end of the natural concentration range for foods. To extend the concentration range for a variety of individual constituents, it will be necessary to identify individual foods with specific constituents at higher concentrations. An example of this is the recently available SRM-1845, Cholesterol in Whole Egg Power [14].

SRM-1548 is a freeze-dried material as are most presently available food reference materials. Therefore, it may be of limited use for analyses which are usually carried out on fresh, non-dried food materials or diets. Thus, it is necessary to begin efforts to produce undried, frozen (not freeze-dried)

reference materials, which are especially needed to determination of a number of the less stable organic constituents of interest.

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Received July 17, 1990