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REPORT ON THE GUIDELINES FOR

ASSESSMENT OF METHODS FOR ANALYSIS

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ABSTRACT

Background and objectives
Previous investigations have identified that analytical methodology is a key factor in data quality and specific criteria at the component level are needed for comparison of analytical data from different sources, especially by food composition data compilers that are often not experienced analysts. The aim of this work is to describe how the EuroFIR guidelines for assessment of methods of analysis are created and made available to users to support the EuroFIR data quality evaluation system.

Method
Analytical methods used for production of food composition data include methods for analysis of vitamins, minerals and trace elements. Comprehensive information addressing all aspects of analytical procedures was obtained from international standards, quality control procedures and scientific literature. Information on analytical methods was compiled in a confluence WIKI format.

Results
Information provided for each micronutrient included: background information on the component, description of reference methods of analysis and critical steps, available reference materials, proficiency testing schemes, description of other analytical methods available and relevant references. The information for each nutrient was collated, edited and presented with hypertext links to additional pages where more detailed information can be accessed using full text searches. The effectiveness of the analytical method information for selected micronutrients was tested by EuroFIR compilers as part of an exercise to test and improve the EuroFIR system to assess quality of data from scientific publications.

Conclusions
EuroFIR GAMA is a valuable tool for compilers and users to access information on analytical methodology in relation to quality of analytical data. The ‘WIKI’ format is a useful tool for preparing information and disseminating the information to users.
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1 Introduction and context

Previous investigations have identified that analytical methodology is a key factor in data quality. Therefore specific criteria at the component level are needed for comparison of analytical methods which is necessary when data is originated from several sources.

At the global level, a lack of compatibility in methods of analysis for macronutrients, minerals and vitamins was observed and can be a source of error as values may not be comparable.

Analytical methods used for production of food composition data were reviewed by members and method curators. Guidelines were drafted on the basis of international standards, covering all main micronutrients as prioritized by regulations and legislation, with the aim of harmonizing the assessment of analytical data produced by compatible methods.

Individual files have been refined following the decision to base GAMA on WIKI format.

For each micronutrient, selected according to the listed prioritized nutrients in EuroFIR, a substantial e-search was carried out, including their chemical species and forms (if relevant) and a WIKI format was produced.

2 Aim of this work

The work was re-launched from previous EuroFIR NoE. Therefore, the main goal was to refine the existing GAMA in order to improve its usability as a supporting document for the data quality evaluation system (QEscirep). FCDB compiler organizations tested GAMA and feedback was provided to INSA. The review of the document was carried out by method curators having experience in analytical methodologies and compilation. Initial curators were INSA, IFR and EuroFIR AISBL [UHEL]. The chapters and the contents were modified to improve the provision of information to compilers when
assessing the description of analytical method in a scientific paper, or a report to attribute quality index.

It was decided to limit the activities in this task to micronutrients; the main reason being that most compilers expressed their needs to improve their background in micronutrients due to the complexity of analytical procedures. Another practical reason was that GAMA was tested in conjunction with QEScirep with exercises defined for micronutrients.

3 Methodology

3.1 GAMA description

Previous GAMA was refined to be more user friendly and to encompass all comments and suggestions from previous studies. Initially all relevant EuroFIR documents were analyzed in detail. Then comprehensive desk research using the most update literature survey was carried out. This survey covered the literature particularly for the methods of analysis for micronutrients which are presented in Table 1. This allowed us to identify which methods already in place for micronutrients can be designated as ‘standard’. Another very important characteristic of this study was to evaluate method performance according to matrix matching, this allows us to select the methods according with LoQ, LoD, reproducibility, repeatability, working range and, therefore, to decide on the method applicability for each matrix/commodity. Databases such as COMAR, IRMM, NIST IAEA were analyzed in order to identify the most important reference materials matching concentration and food matrix. The EuroFIR thesaurus was visited and analyzed to match the work done previously. To provide the compilers with information about component food content, a link to EuroFIR FoodExplorer platform was carried out. Since external quality assessment is of utmost importance to monitor method robustness, the Eptis Database was linked to GAMA, to allow compilers to check the availability of PT schemes. The aim of this work was to provide compilers with substantial information that allow them to assess method suitability based on scientific evidence. After deciding on the contents of GAMA a teleconference was performed to discuss the GAMA format. In that conference it was decided that the previous information was not sufficient for compilers to make decisions on data quality and that it should be revised. Consequently it was decided that it would be better to provide
compliers with links for information since transposition of information from standards is not allowed due to copyright controls. Another decision was that GAMA will be located at the server of University of Helsinki.

3.2 Wiki format

The documents as presented in annex 2 were sent to University of Helsinki. Information for each micronutrient was revised and tailored to be presented in a WIKI confluence as presented in Fig 2.

Screenshots of EuroFIR GAMA are presented in the Figures 1,2,3,4 and 5.

3.3 QE scirep exercise

It was decided to test GAMA during the QE scirep exercise (Quality Evaluation of analytical data from scientific literature and Laboratory Reports) carried out as part of Workpackage 1. The goal of the collaboration was to refine GAMA and to strengthen the link between analysts and compilers.

The GAMA with hyperlink text in a Word format was sent to the compiler group that agreed to be part of the Data Quality evaluation system (WP1, task 1,4) exercise. Furthermore one of the reasons for GAMA to be used as part of the exercise was to involve the whole compiler network. The exercise implies assessment of analytical methods which is often difficult for compilers since most of them do not have an analytical background. Therefore, the main purpose of GAMA is to assist compilers in assessing quality analytical values obtained from scientific literature or reports and other sources. In the compilation process GAMA has another function which is to assist compliers in data aggregation since single values obtained by methods with similar performance characteristics can be aggregated even if the methods are different.

Summarising, the contribution of GAMA in the Quality Assessment exercise was in:

Criteria of the analytical methodology

Does the analytical method used in the source match the list of appropriate analytical methods given in the guidelines for analytical methods?
Are the key method steps appropriate for the method described?

Criteria of analytical control category

Was the laboratory accredited for this method or was the method validated by performance testing?

If available was an appropriate reference material used?

4 Results

4.1 GAMA contents

Individual files have been refined following the decision to base GAMA on WIKI format.

Files were structured in seven paragraphs as following:

- Introduction with a link to a reference document which could be a peer review paper and EuroFIR e-search platform;

- Method indicator and component EuroFIR thesaurus with a link to EuroFIR website;

- Reference method (CEN Standard) and other methods available with links for corresponding editors (CEN; AOAC; CODEX; Greenfield and Southgate book);

- Reference Materials databanks like COMAR or catalogues from key producers (NIST and IRMM). When available link to certificate was displayed;

- Proficiency testing schemes databanks such as EPTIS, which identify suitable proficiency tests (PT) for laboratories;

- Up to date bibliography

- EuroFIR contact organization.

A list of micronutrients covered by GAMA is presented in table 1

The document was structured in four types of links as presented in Figures 1,2,3,4
5 Vitamins

5.1 - Vitamin A all-trans-retinol and 13-cis-retinol

Vitamin A comes from two sources of Vitamin A. One group comes from animal sources and is called retinoids, which includes retinol; the other group comes from plants and is called carotenoids, which includes beta-carotene. Carotenoids are present in fruits and vegetables which provide an estimated 2-3 mg/day of pro-vitamin A carotenoids, of which β-carotene is the principal component. The body converts beta-carotene to vitamin A. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Eurreca.org; Nutri-RecQuest; Khokhar S et al., 2012; Santosh Khokhar et al., 2011; Teddy Study, 2011

Vitamin A all-trans-retinol and 13-cis-retinol variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the Vitamin A enters into the human body through food and food products. Data on vitamin A all-trans-retinol and 13-cis-retinol contents of food items is available in FCDBs (VitaminA FCDB).

Vitamin A concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography EuroFIR Method Indicators MI1137 are the reliable methods preceded by saponification.

Method features are described in EN 12823-1:2000 Foodstuffs - Determination of vitamin A by high performance liquid chromatography - Part 1: Measurements of all-trans-retinol and 13-cis-retinol. Other methods are available: AOAC (AOAC 2001.13) and CODEX. Southgate and Greenfield Book; ISO TC34-14

Reference materials in different matrices are available SRM 968B - Fat-Sol. Vitamins and Cholesterol; NIST 1849 - Infant/Adult Nutritional Formula; ERM-BD600 – Whole milk powder, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin A all-trans-retinol and 13-cis-retinol determination in food matrices, launched by providers registered at eptis database. FAPAS

Recently papers were published for vitamin A retinol determination De Vries JW, et al., 2012; Di Stefano V, et al., 2012; Trisconi MJ et al., 2012; Young Ok Lim et al., 2010

EuroFIR contact organization for this document IFR and INSA.
5.2 - Vitamin A Beta-carotene

Carotenoids are present in fruits and vegetables, providing an estimated 2-3 mg/day of pro-vitamin A carotenoids, of which β-carotene is the principal component. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Boon CS, et al., 2010; Eurreca.org; Nutri-RecQuest; Khokhar S, et al., 2012; Santosh Khokhar et al., 2011; Teddy Study, 2011;

Vitamin A Beta-carotene variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Vitamin A enters into the human body through food and food products. Data on Vitamin A Beta-carotene contents of food items is available in all FCDBs (VitaminA FCDB).

Vitamin A Beta-carotene concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography Euro FIR Method Indicators MI1137 are the reliable methods preceded by saponification.

Method features are described in EN 12823-2:2000 Foodstuffs - Determination of vitamin A by high performance liquid chromatography - Part 2: Measurements of Beta-carotene. Other methods are available: AOAC (AOAC 2005.07) and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available; BCR-485 – Mixed vegetables; NIST 2385 - Slurried Spinach; SRM 968e - Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum; NIST 1849 - Infant/Adult Nutritional Formula, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin A all-trans-retinol and 13-cis-retinol determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for vitamin A determination Di Stefano V, ET AL., 2012; Trisconi MJ et al., 2012; Young Ok Lim et al., 2010; Delia B. Rodriguez-Amaya, 2009.

EuroFIR contact organization for this document IFR and INSA.
5.3 - Vitamin B1 Thiamin

Vitamin B1 is found in a large variety of animal and vegetable products but at a relatively low level (<0.5 mg/100 g). Important sources of vitamin B1 are lean pork, legumes and cereal grains (germ fraction). The Nutriscan EC food and nutrition intake study revealed mean daily intake levels of vitamin B1 on an EC average of 1.2 mg per day for women, ranging from 1.0 mg per day (NL) to 1.8 mg per day (Portugal). EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Eurreca.org; Nutri-RecQuest; Khokhar S et al., 2012; Teddy Study, 2011

Vitamin B1 variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential B1 enters into the human body through food and food products. Data on Vitamin B1 contents of food items is available in all FCDBs (VitaminB1 FCDB).

Vitamin B1 concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography EuroFIR Method Indicators MI1137 are the reliable methods preceded by enzymatic method MI1218.

Method features are described in EN 14122:2003 Foodstuffs - Determination of vitamin B1 by HPLC. Other methods are available: AOAC and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; BCR - 121 – Wholemeal flour; BCR - 421 – Milk Powder; BCR - 485 – Mixed Vegetable; BCR - 487 – Pig Liver; NIST 2387 - Peanut Butter; NIST 3244 - Containing Protein Powder; NIST 1849 - Infant/Adult Nutritional Formula; ERM-BD600 – Whole milk powder, are of more popular but others can be found at IRMM, NIST and COMAR databanks

Proficiency testing Schemes can be found for Vitamin B1 determination in food matrices, launched by providers register at eptis database. FAPAS


EuroFIR contact organization for this document IFR and INSA.
5.4 - Vitamin B2 Riboflavin

Vitamin B2 is a precursor of certain essential coenzymes such as flavin mononucleotide (FMN) and flavin-adenine dinucleotide (FAD). The highest mean intake of riboflavin from diet and supplements was reported for males aged 31-50 years: 6.9 mg/day. The highest reported intake at the ninety-fifth percentile was 11 mg/day in females over 70 years. The RDA for riboflavin varies from 0.5-0.9 mg/day for children, 1.3 mg/day for male adults and 1.0-1.1 mg/day for female adults; 0.8-1.6 mg/day children/male, 0.8-1.3 mg/day children/female, 1.3 mg/day male adults and 1.1 mg/day female adults, 0.7-1.6 mg/day children/male, 0.7-1.3 mg/day children/female, 1.2-1.5 mg/day male adults and 1.2 mg/day female adults. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Eurreca.org; Nutri-RecQuest; R. San José Rodriguez et al., 2011; Shailesh Kumar et al., 2004; Konings EJ, 2006; Khokhar S et al., 2012; Teddy Study, 2011

Vitamin B2 variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential B2 enters into the human body through food and food products. Data on Vitamin B2 contents of food items is available in all FCDBs (VitaminB2 FCDB).

Vitamin B2 concentration depends on food: therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography are the reliable methods preceded by enzymatic method.

Method features are described in EN 14152:2003 Foodstuffs - Determination of vitamin B2 by HPLC.
Other methods are available: AOAC and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available: BCR - 421 – Milk Powder; BCR - 487 – Pig Liver; NIST 2384 - Baking Chocolate; NIST 1546 - Meat Homogenate; NIST 2385 - Slurried Spinach; NIST 3244 - Containing Protein Powder; NIST 1849 - Infant/Adult Nutritional Formula; ERM-BD600 – Whole milk powder, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin B2 determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for vitamin B2 determination Di Stefano V, ET AL., 2012; Jette Jakobsen, 2007; Hucker B. et al., 2011; Chen P, Wolf WR, 2006;

EuroFIR contact organization for this document IFR and INSA.
5.5 - Vitamin B5 – Pantothenic Acid

Vitamin B5 plays a specific role in amino acid metabolism. The average dietary requirement for vitamin B5 is 1.0 µg/day, with a population reference intake (PRI) for adults of 1.4 µg/day. This is approximately the amount needed to maintain an adequate vitamin B5 body pool (about 2.5 mg), and to compensate for daily losses (about 0.1% of the total body pool). EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Eurreca.org; Nutri-RecQuest; Konings EJ, 2006; Khokhar S et al., 2012; Teddy Study, 2011

Vitamin B5 variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Vitamin B5 enters into the human body through food and food products. Data on Vitamin B5 contents of food items is available in all FCDBs (VitaminB5 FCDB).

Vitamin B5 concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography is a reliable method and by radioimmunoassay or microbiological are used as well as.

Method features are described in AOAC 992.07 - Pantothenic Acid in Milk-Based Infant Formula. Other methods are available: AOAC and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; NIST 2387 - Peanut Butter; NIST 1546 - Meat Homogenate; NIST 3244 - Ephedra-Containing Protein Powder; NIST 1849A - Infant/Adult Nutritional Formula, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin B5 determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for vitamin B5 determination Di Stefano V, ET AL., 2012; Ciulu M, et al., 2011; Chen P, Wolf WR, 2006; Andrieux P et al., 2012

EuroFIR contact organization for this document IFR and INSA.
5.6 - Vitamin C (L (+) ascorbic acid and dehydro L (+) ascorbic acid)

Vitamin C is a permitted anti-oxidant additive in food, with no specified limits on the level of use. Vitamin C is present in numerous dietary supplements with manufacturer recommended daily intakes of 60-3000 mg in single vitamin preparations and 10-1000 mg/day in multi-vitamin preparations. In 1992, the Scientific Committee for Food recommended a Population Reference Intake of 45 mg/day for adults, with an increase to 55 mg/day in pregnancy, and to 70 mg/day during lactation. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Viñas BR et al, 2011; Eurreca.org; Nutri-RecQuest; Konings EJ, 2006; Khokhar S et al., 2012; Teddy Study, 2011; Maria Teresa Tarrago et al., 2012; Katherine M. Phillips et al., 2009; J. Fredriksen et al., 2007; C.M.A. Rodrigues et al., 2008; Jagdish Singh et al., 2005; Ana Valente, et al., 2010

Vitamin C variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Vitamin C enters into the human body through food and food products. Data on Vitamin C contents of food items is available in all FCDBs (VitaminC FCDB).

Vitamin C concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High - performance liquid chromatography EuroFIR Method Indicators Other analytical methods, based on different technologies, are reported in the literature spectrophotometric, colorimetric, fluorometric, voltammetric, potentiometric, titrimetric, and spectrofluorimetric but chromatography due to selectivity and sensitivity is the most reliable Ana Valente, et al., 2010

Method features are described papers cited above other methods are available: AOAC and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available; BCR - 421 – Milk Powder; BCR - 431- Brussels sprouts powder; NIST 3244 - Containing Protein Powder; NIST 1849 - Infant/Adult Nutritional Formula; ERM-BD600 – Whole milk powder, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin C determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for vitamin C determination Di Stefano V, ET AL., 2012; Maria Teresa Tarrago, 2012; Patric Fontannaz, 2005; L. Nováková, 2008; José Fenoll, 2011; Chen P, Wolf WR, 2006

EuroFIR contact organization for this document IFR and INSA.
5.7 - Vitamin D3, D2

Vitamin D comprises two closely related substances of nutritional importance: vitamin D3 (cholecalciferol), which is the physiological form, and the synthetic analogue vitamin D2 (ergocalciferol). The recommended daily amount for vitamin D in the European Union is 5 µg. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Viñas BR et al., 2011; EFSA, 2011; H. Greenfield, et al., 2009; Joanne M ET AL., 2008; Ann Nutr Metab, 2012; Eurreca.org; Nutri-RecQuest; Khokhar S, et al., 2012; Teddy Study, 2011

Vitamin D variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Vitamin D enters into the human body through food and food products. Data on Vitamin D contents of food items is available in all FCDBs (VitaminD FCDB).

Vitamin D3, D2 concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography are the reliable methods preceded by saponification.

Method features are described in EN 12821:2009 - Foodstuffs - Determination of vitamin D by high performance liquid chromatography - Measurement of cholecalciferol (D3) or ergocalciferol (D2).

Other methods are available: AOAC and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available; BCR-421(D3 cholecalciferol) – Milk Powder; BCR-122 (D3 cholecalciferol) – Margarine; NIST 1849A (D3 Cholecalciferol)- Infant/Adult Nutritional Formula; SRM 2972 (25-hydroxyvitamin D2, 25-hydroxyvitamin D3) - Hydroxyvitamin D2 and D3 Calibration Solutions, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Vitamin D determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for Vitamin D determination Di Stefano V, ET AL., 2012; Jette Jakobsen, 2004; L. Bilodeau, 2010; S. Perales, et al., 2005; Chen P, Wolf WR, 2006; S. Perales et al., 2005

EuroFIR contact organization for this document IFR and INSA.
5.8 - Vitamin K1, K2

Vitamin K1, or phylloquinone, is obtained from the diet whereas vitamins K2 are also produced by the intestinal microflora. Reliable measurements of phylloquinone contents in foods are now available, and data from many studies of phylloquinone intake in the United States indicate that the mean intake of younger adults (<45 years) ranges from 60 to 110 µg of phylloquinone/d. In contrast, older adults (>55 years) consume 80 to 210 µg of phylloquinone/d, attributed to their greater vegetable consumption compared to younger age groups. EFSA, 2006; Vitamin Conference, 2012; Southgate and Greenfield Book; Eurreca.org; Nutri-RecQuest; Teddy Study, 2011; Molly Damon et al, 2004

Vitamin K1, K2 variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Vitamin K enters into the human body through food and food products. Data on Vitamin K contents of food items is available in all FCDBs (VitaminK FCDB).

Vitamin K1, K2 concentration depends on food; therefore, accurate determination requires a sensitive analytical method. High-performance liquid chromatography is the reliable methods preceded by enzymatic method.

Method features are described in EN 14148:2003 Foodstuffs - Determination of vitamin K1 by HPLC. Other methods are available: AOAC and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; NIST 1849A (K1 Phylloquinone) - Infant/Adult Nutritional Formula; SRM 968e (K1 Phylloquinone) - Fat-Sol Vitamins, Carotenoids, and Cholesterol in Human Serum, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for vitamin K determination in food matrices, launched by providers register at eptis database. FAPAS

Recently papers were published for vitamin K determination Di Stefano V, ET AL., 2012; Semih Otles, Ozlem Cagindi, 2006; Chen P, Wolf WR,2006

EuroFIR contact organization for this document IFR and INSA.
5.9 - Cobalamin (B12)

Vitamin B12 plays a significant role in amino acid metabolism, i.e. in conversion of homocysteine to methionine in the presence of folate (catalysed by methionine synthase), and in the rearrangement of methylmalonyl CoA into succinyl CoA. The RDA of vitamin B12 for adults is 2.4 µg/day. EFSA, 2006; Elizabeth A Yetley et al., 2011; Sagaya Selva Kumar et al., 2009; Ola Karmi et al., 2010

Food of animal origin is a good source of vitamin B12, however, colon microflora may synthesize vitamin B₁₂ and it appears to be uniquely synthesized by anaerobic bacteria. Data on vitamin B12 contents of food items is available in all FCDBs (VitaminB12 FCDB).

Vitamin B12 concentration varies depending on food. Food rich in vitamin B12 includes liver, milk, meat, eggs, fish, oysters, and clams. Accurate determination of vitamin B12 in food requires a sensitive analytical method. For this purpose, High-performance liquid chromatography method seems to be better than the enzymatic method.

Methods are available: AOAC and CODEX, Southgate and Greenfield Book

Standard Reference materials for vitamin B12 are available in different matrices, however BCR 487 – Pig liver; ERM - BD600 – whole milk powder, are preferred than others that can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes report the vitamin B12 contents in various foods which was launched by providers registered at eptis database. FAPAS

Recently determination of vitamin B12 has been reported in the following papers; Di Stefano V, ET AL., 2012; Szterk A et al., 2012; Guggisberg D, et al., 2012; Xubiao Luo et al., 2005; Heudi O, et al., 2005;

EuroFIR contact organization for this document IFR and INSA.
Folates is a generic term encompassing naturally occurring folates and folic acid. The chemical structure of folate is composed of pterin ring, para amino benzoic acid and glutamic acid, the latter can be found in monoglutamate and polyglutamate residues. Folates in foods are present in a reduced state, mostly in polyglutamate forms - Folic acid refers to the oxidized, stable, and easily absorbable synthetic form (as monoglutamate). The most common dietary folates are 5-methyltetrahydrofolate, tetrahydrofolate, 5-formyltetrahydrofolate, 10-formylfolic acid, including folic acid that is present in a very low concentration. Folates can be sourced from a wide variety of foods, including vegetables (especially dark green leafy vegetables), fruits and fruit juices, nuts, beans, peas, dairy products, poultry and meat, eggs, seafood, and grains. Spinach, liver, yeast, asparagus, and Brussels sprouts are among the foods with the highest levels of folate; (www.eurofir.org)

A recent study carried out by the EURRECA project reveals that dietary intake of folates was inadequate among European populations (Vinãs 2011). Also, another study revealed that in developing countries dietary intake of folates during pregnancy was (Blumfield 2013)

The term "folic acid" (i.e. pteroylmonoglutamic acid) is compatible with the standard mode of expression, but it is specific to synthetic form and used to fortify foods and for dietary supplements. This form is considered to be more bioavailable than intrinsic (i.e. naturally occurring) folate. Thus its compositional value in fortified foods is necessary for calculating the total folate value.(EuroFIR). Data on folates contents in many food types is available in all FCDBs (folates FCDB). A critical evaluation of folate in European and international databases was recently reviewed by Bouckaert et al, 2011

Because of the health benefits of folates, there is a trend to increase the dietary intake by supplementation, fortification with synthetic folic acid, or bio-fortification Vishnumohan, 2011 Therefore, accurate determination requires a sensitive analytical method which is able to differentiate the various folate forms including the fortificant. Liquid chromatography mass spectrometry appears to be the most promising method, although. However, microbiological assay measures the total folate, while HPLC is recommended by EuroFIR to quantify synthetic folic acid Bouckaert KP et al 2011 Other Methods available are: AOAC (AOAC 2011.05; AOAC 2004.05) and CODEX. Southgate and Greenfield Book

Standard Reference materials are available in different food matrices, such BCR 121 – whole meal flour; BCR 421 – milk powder; BCR 485 - mixed vegetables; BCR 487– Pig Liver; NIST 3244– Ephedra - Containing Protein Powder, are more commonly used but others can be found at IRMM, NIST and COMAR databanks. Recently a new SRM was presented by NIST Camara JE, 2013.

Proficiency testing schemes can be found for folates determination in food matrices, launched by providers register at eptis database. FAPAS

The following papers were published to quantify folates Camara JE, Lowenthal MS, et al. 2013; Di Stefano V et al., 2012; Wabaidur et al., 2012; Chandra-Hioe MV et al., 2011; De Brouwer V et al., 2010; S. Ndawa, et al. 2001; Konings, E.J.M et al., 2001; EuroFIR contact organization for this document IFR.
6 Minerals

6.1 - Calcium

Calcium (Ca) was the nutrients showing a higher prevalence of inadequate intakes in Europe. The best sources are milk (120 mg/100 g) and milk products (up to 1100 mg/100 g), from which about 32% is absorbable. In European diets about 45 to 70% of the dietary calcium intake is provided by dairy products. Drinking water and mineral waters (>150 mg calcium/L) can also be good sources of absorbable calcium. Joannie Dobbs, 2006; EFSA 2006, Viñas BR et al., 2011; Southgate and Greenfield Book.

Calcium variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential calcium enters into the human body through food and food products. Data on copper contents of food items is available in all FCDBs (CalciumFCDB).

Calcium concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES).


Reference materials in different matrices are available; LGC 7104 - Sterilized cream, NIST 2384 - Baking chocolate, NIST 2387 - Peanut Butter, NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR-383- Freeze Dried Green Beans, NIST 1567a - Wheat Flour, NIST 1568a - Rice Flour, NIST 8432 - Corn Starch, NIST 8433 - Corn Bran, NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding, NIST 1548A - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non- Fat Milk Powder, NIST 1566B - Freeze Dried Oyster Tissue, NIST 2385 - Slurried Spinach, NIST 1946 - Fish Tissue, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder, NIST 8418 - wheat gluten, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Calcium determination in food matrices, launched by providers register at eptis database


EuroFIR contact organization for this document INSA.
6.2 - Copper

Copper (Cu) is an essential trace metal found in all living organisms in the oxidized Cu(II) and reduced Cu(I) states. It is required for survival and serves as an important catalytic cofactor in redox chemistry for proteins that carry out fundamental biological functions that are required for growth and development. The average intakes of copper by human adults, vary from 0.6 to 1.6 mg/d and the main sources are seeds, grains, nuts, and beans (concentrated in the germ and bran), shellfish and liver. 

Copper variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential copper enters into the human body through food and food products. Data on copper contents of food items is available in all FCDBs (CopperFCDB).

Copper concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) proceeded by wet digestion or pressure digestion.


Reference materials in different matrices are available; NIST 2381 - Morphine and Codeine in Urine, NIST 2387 - Peanut Butter, NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR 679 – White Cabbage, NIST 1567a - Wheat Flour, NIST 1568 a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8432 - Corn Starch, NIST 8433 - Corn Bran, NIST 8436 - Durum Wheat Flour, NIST 1548a - Typical Diet, NIST 8435 - Whole Milk Powder, NIST 1846 - Infant Formula (milk-based), NIST 1549 - Non-Fat Milk Powder, NIST 1566b - Oyster Tissue, NIST 1946 - Lake Superior Fish Tiss, BCR 422 -Trace elements in cod muscle, BCR 679 – White Cabbage, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Copper determination in food matrices, launched by providers register at eptis database.


Euro FIR contact organization for this document INSA.
6.3 - Iodine

Iodine was the nutrient showing a higher prevalence of inadequate intakes in Europe. The major natural food sources are marine fish (mean 1220 µg/kg, up to 2.5 mg I/kg), shellfish (mean 798 µg/kg, up to 1.6 mg I/kg), marine algae, seaweed (1000-2000 µg/kg) and sea salt (up to 1.4 mg I/kg). In industrialised countries the most important sources of iodides are dairy products, e.g. whole cow’s milk (mean 27-47 µg/kg), UK winter milk (mean 210 µg/kg), UK summer milk (90 µg/kg), eggs (mean 93 µg/kg), and grain and cereal products (mean 47 µg/kg depending on the soil). Other food sources are freshwater fish (mean 30 µg/kg), poultry and meat (mean 50 µg/kg), fruits (mean 18 µg/kg), legumes (mean 30 µg/kg) and vegetables (mean 29 µg/kg). EFSA, 2006; Iodine, 2001; Angermann L, Clar C., 2004; Viñas BR et al., 2011; Southgate and Greenfield Book.

Iodine variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Iodine enters into the human body through food and food products. Data on Iodine contents of food items is available in FCDBs (IodineFCDB).

Iodine concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS). Method features are described in EN 15111:2007. Other methods are available: AOAC and CODEX. Southgate and Greenfield Book.

Reference materials in different matrices are available; BCR 63R - skim milk powder (ICP_MS), is of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Iodine determination in food matrices, launched by providers register at eptis database.

Recently papers were published for Iodine determination Pacquette et al,2012; Mesko et al,2010; Sullivan et al, 2012; Lin L. et al, 2011.

Euro FIR contact organization for this document INSA.
6.4 - Iron

Iron is a metal with an atomic mass of 55.8. It is present in biological systems in one of two oxidation states, and redox inter-conversions of the ferrous (Fe2+) and ferric (Fe3+) forms are central to the biological properties of this mineral. Groups vulnerable to iron deficiency are infants over 6 months, toddlers, adolescents and pregnant women (due to high requirements), older people and people consuming foods high in iron absorption inhibitors (see below) (due to poor absorption) and menstruating women or individuals with pathological blood loss (due to high blood losses). Total daily iron absorption is about 0.9 mg in males and 0.5 and 0.6 mg higher in menstruating women and blood donors. EFSA 2006; EFSA, 2004; Iron, 2001; John Beard, 2008; Christine A. Swanson, 2003; Southgate and Greenfield Book

Iron variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential iron enters into the human body through food and food products. Data on iron contents of food items is available in all FCDBs (IronFCDB).

Iron concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) are the reliable methods preceded by wet digestion or pressure digestion.

Method features are described in ISO 11885: 2007 Water quality -- Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES), ISO 17294:2003: Water quality -- Application of inductively coupled plasma mass spectrometry (ICP-MS) -- Part 2: Determination of 62 elements, EN 14084:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion, EN 14082:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing. Other methods are available: AOAC (AOAC 984.27) and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available; NIST 2384 - Baking chocolate, NIST 2387 - Peanut Butter, NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR 679 - Freeze-Dried White Cabbage, NIST 1567a - Wheat Flour, NIST 1568a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8433 -Corn Bran, NIST 8436 - Durum Wheat Flour, NIST 1548a - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non-Fat Milk Powder, NIST 1566b - Freeze Dried Oyster Tissue, NIST 1946 - Fish Tissue, BCR 422 - Freeze dried Cod Muscle, NIST 1577B - Freeze Dried Bovine liver, NIST 8418 - wheat gluten, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Iron determination in food matrices, launched by providers register at eptis database. Recently papers were published for iron determination Poitevin, 2012, Jennifer S., et al., 2001 EuroFIR contact organization for this document INSA.
6.5 - Magnesium

Magnesium (Mg) belongs to group II of the third period of the Periodic Table of Elements, a group that includes two other physiologically important elements: calcium and zinc. Mg has an atomic weight of 24.312; its atomic number is 12; its valency 2.

The adult healthy body contains approximately 21-28 g (about 1 mole) of Mg; related to an average body weight of 70 kg, this corresponds to circa 14.3 mmol/ kg, or to 0.034% of body weight.

Based on a NOAEL of 250 mg Mg per day and an uncertainty factor of 1.0 an UL of 250 mg Mg per day can be established for readily dissociable magnesium salts (e.g., chloride, sulphate, aspartate, and lactate) and compounds like MgO in nutritional supplements, water, or added to food and beverages. EFSA, 2006; Eurreca; Southgate and Greenfield Book;

Magnesium variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential magnesium enters into the human body through food and food products. Data on magnesium contents of food items is available in all FCDBs (MagnesiumFCDB).

Magnesium concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) are the reliable methods preceded by wet digestion or pressure digestion.

Method features are described in ISO 11885: 2007 Water quality -- Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES), ISO 17294:2003: Water quality -- Application of inductively coupled plasma mass spectrometry (ICP-MS) -- Part 2: Determination of 62 elements, EN 15505 Foodstuffs -- Determination of trace elements - Determination of sodium and magnesium by flame atomic absorption spectrometry (AAS) after microwave digestion. Other methods are available: AOAC (AOAC 984.27) and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; LGC 7104 - Sterilized cream, NIST 2384 - Baking chocolate, NIST 2387 - Peanut Butter , NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR 381- Rye Flour, NIST 1567a - Wheat Flour, NIST 1568 a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8432 - Corn Starch, NIST 8433 -Corn Bran , NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding, NIST 1548a - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non- Fat Milk Powder, NIST 1566b - Freeze Dried Oyster Tissue, NIST 2385 - Slurried Spinach, NIST 1946 - Fish Tissue, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder, NIST 8418 - wheat gluten, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Magnesium determination in food matrices, launched by providers register at eptis database

EuroFIR contact organization for this document INSA.
6.6 - Phosphorus

Phosphorus is an element of group 5 of the periodic table and has an atomic weight of 30.97. The average intake from foods in adults is usually between 1000-2000 mg/day. EFSA, 2006; Ray J.Winger, 2012; Southgate and Greenfield Book

Phosphorus variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential phosphorus enters into the human body through food and food products. Data on phosphorus contents of food items is available in all FCDs (PhosphorusFCDB).

Phosphorus concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) are the reliable methods preceded by wet digestion or pressure digestion.


Reference materials in different matrices are available; LGC 7104 - Sterilized cream, NIST 2381 - Baking chocolate; NIST 2387 - Peanut Butter; NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder; NIST 1567a - Wheat Flour, NIST 1568a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8432 - Corn Starch, NIST 8433 -Corn Bran , NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding, NIST 1548a - Typical Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non- Fat Milk Powder, NIST 1570a - Freeze Dried Spinach Leaves, NIST 2385 - Slurried Spinach, NIST 1946 - Fish Tissue, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder, NIST 8418 - wheat gluten, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Phosphorus determination in food matrices, launched by providers register at eptis database
Recently papers were published for phosphorus determination Poitevin, 2012; Dolan S.P., Capar S.G, 2002.

Euro FIR contact organization for this document INSA.
6.7 - Potassium

Potassium in foods is associated with salts of weak organic acids. Various potassium salts, e.g. KCl, are used in many applications, amongst others as ingredients in foods (e.g. additives), food supplements and drugs, household chemicals etc. In this opinion, the term potassium refers to ionic potassium, except where specific potassium compounds are stated. One mmol potassium is equivalent to 39.1 mg. The average dietary intake of potassium according to European food consumption studies is in the range of 3000 to 4000 mg/day. The 95th to 97th percentile intake is in the range of 4000-5500 mg/day. Chekri, et al, 2010; EFSA, 2006; Southgate and Greenfield Book

Potassium variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential potassium enters into the human body through food and food products. Data on potassium contents of food items is available in all FCDBs (PotassiumFCDB).

Potassium concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP-OES) are the reliable methods preceded by wet digestion or pressure digestion.

Method features are described in ISO 11885: 2007 Water quality -- Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES), ISO 17294:2003: Water quality -- Application of inductively coupled plasma mass spectrometry (ICP-MS) -- Part 2: Determination of 62 element. Other methods are available: AOAC (AOAC 984.27; AOAC 985.35 ) and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; LGC 7104 - Sterilized cream, NIST 2384 - Baking chocolate, NIST 2387 - Peanut Butter , NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR 383 - Freeze Dried Green Beans, NIST 1567a - Wheat Flour, NIST 1568 a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8432 - Corn Starch, NIST 8433 -Corn Bran , NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding, NIST 1548a - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non- Fat Milk Powder, NIST 1566b - Freeze Dried Oyster Tissue, NIST 1570a - Freeze Dried Spinach Leaves, NIST 2385 - Slurried Spinach, NIST 1946 - Fish Tissue, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder, NIST 8418 - wheat gluten, BCR 381 - Rye Flour, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Potassium determination in food matrices, launched by providers register at eptis database

Recently papers were published for potassium determination Poitevin, 2012, Chekri R, et al, 2010

EuroFIR contact organization for this document INSA.
6.8 - Selenium

Selenium is an important micronutrient element, with a higher prevalence of inadequate intakes in Europe. The total body pool of selenium has been estimated to be 5-15 mg in adults. For a 65 kg reference man the average normative requirement of individuals for selenium was estimated to be 26µg/day, and from this value the lower limit of the need of population mean intakes was estimated to be 40µg/day. Viñas BR, et al, 2011; Mistry HD, Et al., 2012; Berr C, et al, 2012; Huawei et al, 2007; EFSA, 2006; Southgate and Greenfield Book

Selenium variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential selenium enters into the human body through food and food products. Data on selenium contents of food items are relatively scarce (seleniumFCDB)

The concentration of Selenium in most foods is low; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) is the most reliable method preceded by wet digestion or pressure digestion.

Method feature is described in EN 14627:2005 - Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion. Other methods are available: AOAC (AOAC 986.15) and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; NIST 8415 - Whole Egg Powder, BCR 381 - Rye Flour, NIST 1567a - wheat Flour, NIST 1568a - Rice Flour, NIST 2383 - Baby Food, NIST 8432 - Corn Starch, NIST 8433 - Corn Barn, NIST 8436 - Durum Wheat Flour, NIST 1548a - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non- Fat Milk Powder, NIST 1566b - Freeze Dried Oyster Tissue, NIST 1570a - Freeze Dried Spinach Leaves, NIST 1946 - Fish Tissue, BCR 422 - Freeze Dried Cod Muscle, NIST 1577B - Freeze Dried Bovine liver, NIST 8418 - wheat gluten are more popular but others can be found at IRMM, NIST and COMAR databanks.

Quite a few Proficiency testing Schemes can be found for selenium determination in food matrices, Diet Food; Iodized salt; Premix launched by providers register at eptis database.

Recently papers were published for selenium determination Elene P. Et al, 2008; Govasmark, et al., 2007, Pacquette et al., 2011;

Euro FIR contact organization for this document INSA.
6.9 - Sodium

Sodium (Na) is a metal with an atomic mass of 23. It is found widely in nature and as a normal constituent of foods. It is added to foods, most frequently as sodium chloride (NaCl), common known as salt (1 mmol equivalent to 23 mg sodium and approximately 58 mg sodium chloride).

The sodium content of natural foods varies from around 0.1 to 3 mmol/100g, with fruit containing 0.1 mmol/100g, vegetables 0.3 mmol/100g, and meat, fish or eggs 3.0 mmol/100g. Corina M. Et al., 2010; EFSA, 2006, Southgate and Greenfield Book

Sodium variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential Sodium enters into the human body through food and food products. Data on Sodium contents of food items is available in all FCDBs (SodiumFCDB).

Sodium concentration depends on food; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma optical emission spectrometry (ICP-OES) are the reliable methods preceded by wet digestion or pressure digestion.

Method features are described in EN 15505:2008 Foodstuffs - Determination of trace elements - Determination of sodium, magnesium and calcium by flame atomic absorption spectrometry (AAS) after microwave digestion. Other methods are available: AOAC (AOAC 984.27) and CODEX, Southgate and Greenfield Book

Reference materials in different matrices are available: LGC 7104 - Sterilized cream, NIST 2384 - Baking chocolate, NIST 2387 - Peanut Butter, NIST 1546 - Meat Homogenate, NIST 8415 - Whole Egg Powder, BCR 381 - Rye Flour; BCR 383 - Freeze Dried Green Beans, NIST 1567a - Wheat Flour, NIST 1568 a - Rice Flour, NIST 2383 - Baby Food Composite, NIST 8432 - Corn Starch, NIST 8433 - Corn Bran, NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding, NIST 1548a - Freeze Dried Mixed Diet, NIST 1846 - Infant Formula, NIST 8435 - Whole Milk Powder, NIST 1549 - Non-Fat Milk Powder, NIST 1566b - Freeze Dried Oyster Tissue, NIST 1570a - Freeze Dried Spinach Leaves, NIST 2385 - Slurried Spinach, NIST 1946 - Fish Tissue, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder, NIST 8418 - wheat gluten, are of more popular but others can be found at IRMM, NIST and COMAR databanks.

Proficiency testing Schemes can be found for Sodium determination in food matrices, launched by providers register at eptis database

Recently papers were published for sodium determination Julshamn, Kaare1; Lea, Per2; Norli, Hilde Skaar, 2005; Ehling, Stefan; Tefera, Sebhat; Earl, Robert; Cole, Shannon, 2010; Poitevin, 2012.

EuroFIR contact organization for this document INSA.
6.10 - Zinc

Zinc has an atomic weight of 65.37 and is classified as a group IIB post-transition metal. Estimated Average Requirements (EAR) are 7.3 mg/day and 5.5 mg/day for males and females, respectively. In the US, new guidelines recommend daily intakes of 11 mg/day and 8 mg/day for men and women respectively. \( \text{Zinc, 2001; Heimo Scherz, Eva Kirchhoff, 2006; EFSA, 2006, Southgate and Greenfield Book} \)

Zinc variability is of practical significance when it occurs in foods that are normally relied on as important sources. The major part of the essential zinc enters into the human body through food and food products. Data on zinc contents of food items are relatively scarce (ZincFCDB).

The concentration of zinc in most foods is low; therefore, accurate determination requires a sensitive analytical method and freedom from contamination. Inductively coupled plasma mass spectrometry (ICP-MS) is the most reliable method preceded by wet digestion or pressure digestion.

Method feature is described ISO 11885: 2007 Water quality -- Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES); ISO 17294:2003 Water quality -- Application of inductively coupled plasma mass spectrometry (ICP-MS) -- Part 2: Determination of 62 elements; EN 14084:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion; EN 14082:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing. Other methods are available: AOAC (AOAC 984.27) and CODEX. Southgate and Greenfield Book

Reference materials in different matrices are available; LGC 7104 - Sterilized cream; NIST 2384 - Baking chocolate; NIST 2387 - Peanut Butter; NIST 1546 - Meat Homogenate; NIST 8415 - Whole Egg Powder; BCR 679 - Freeze Dried White Cabbage; BCR 381 - Rye Flour; NIST 1567a - wheat Flour, NIST 1568a - Rice Flour, NIST 2383 - Baby Food, NIST 8432 - Corn Starch, NIST 8433 - Corn Barn, NIST 8436 - Durum Wheat Flour, LGC 7105 - Rice Pudding; NIST 1549 - Non- Fat Milk Powder; NIST 1566b - Freeze Dried Oyster Tissue, NIST 1570a - Freeze Dried Spinach Leaves, NIST 1946 - Fish Tissue, BCR 422 - Freeze Dried Cod Muscle, NIST 1577B - Freeze Dried Bovine liver, NIST 3244 - Protein Powder; NIST 8418 - wheat gluten are more popular but others can be found at IRMM, NIST and COMAR databanks.

Quite a few Proficiency testing Schemes can be found for zinc determination in food matrices, Diet Food; Iodized salt; Premix launched by providers register at eptis database.

Recently papers were published for zinc determination Elene P.,Et al, 2008; C.V.S. et al, 2010

Euro FIR contact organization for this document INSA.
7 Conclusions

EuroFIR GAMA was developed to provide compilers with background support to decide on appropriateness of analytical methods used. It was difficult to define the design of GAMA content due to variability of compilers’ analytical background. Therefore it was necessary to refine the document and its contents several times to improve the usability of the document.

Vitamin analyses were basically supported in CEN Methods in which HPLC is commonly used for the detection and quantification according their specific wavelength. After several discussions it was decided that sample preparation is crucial however the procedure is not described in this report.

Golden standards method for mineral are based on Atomic Absorption Spectroscopy or ICP-OES assisted by microwave digestion for which CEN and AOAC methods are available. Trace elements are determined by ICP-MS. Iodine determination is described in ISO standard and Selenium in several review papers.

Initially it was decided to include key method steps in GAMA, but many compilers thought the level of information was too complex and preferred simpler summaries of available methods. It was agreed that it would be better to provide compilers with information linking to the original documentation where the standard method is described (ISO/CEN standard; AOAC), although in many cases access to full standard documents required payment.

GAMA was part of the exercise on the Guidelines for Quality Index attribution to original data (QE scirep). All the information and remarks obtained during the exercise launched for quality index attribution were incorporated in the final document.

The information compiled for each micronutrient served as support for the confluence WIKI GAMA application. This allowed an interactive document to be made available. The initial document structure to be used in Word version gives access to documents and reports that support GAMA. The Word version was initially thought to be directly used by compilers because it was not yet available in the application created by the University of Helsinki. That version was successfully used by compilers during QE scirep exercise. The responses of the compilers did not show any difficulty in handling the Word version.

However, during the project timeline it was possible to create a specific application for EuroFIR as well as to extend access to this application beyond EuroFIR Nexus. This WIKI application whose screenshots are shown in Figures 3 and 4 allows users to get all information for each nutrient but also to be filtered by compilers interest. The application now available allows the following questions to be answered.

a) What are the most appropriate methods for determination of mineral content in foods?
b) What are the reference materials available for simultaneous analysis of several micronutrients?

c) What are the standards published for the analysis of water-soluble and fat-soluble vitamins in food?

The application built during the project by the University of Helsinki allows this work to continue beyond the EuroFIR NEXUS project and to be developed further. Since GAMA is a document that is supported by the latest data published on methods of analysis it must be updated regularly otherwise it becomes obsolete and serves no useful purpose. Thus, the committee of curators for the methods of analysis recommended by EuroFIR will continue beyond the project allowing updating the information within an appropriate timeframe.

This update will be made according to the decisions and following the rules set by EuroFIR AISBL executive board. The platform already considered this update; therefore, various categories of access were created. Figure 5 presents a screenshot of the types of access created. The aim is to update and disseminate GAMA with other EuroFIR members with expertise in each component area.

Also, beyond the end of EuroFIR Nexus, GAMA will be extended for macronutrients, amino acids and fatty acids with texts prepared in line with the format previously presented. GAMA will cover forty nutrients that have been defined as priority nutrients by the network of excellence EuroFIR. These groups of nutrients encompass all the components referenced by European directives and by the majority of national food composition data banks. Expanding the WIKI GAMA to include the contaminants as well is under discussion.

The access to the application was also designed to include food components of interest for the project Total Diet Studies Exposure of which EuroFIR AISBL is a partner.

**Improvement of GAMA and recommendations for future work**

Substantial improvements on methods of analysis occurred in the last decades. The present work is based on updated information.

- This information needs to be revised periodically in order to keep the compilers well informed.
- Extend GAMA contents to other components of interest.
- Create a EuroFIR forum where compilers, analysts and users of FCDB can change ideas and suggest improvements.
- Disseminate GAMA to those involved in food analysis like in Total Diet Studies
- Distribute the information to other users and producers of food composition data beyond Europe who are interested in the methods.
8 References

AOAC 984.27 Foodstuffs determination of nine nutritional elements in food products by Inductively coupled plasma-atomic emission spectroscopy after microwave digestion:

AOAC 992.07 - Pantothenic Acid in Milk-Based Infant Formula

AOAC 2011.05 Folate in Infant Formula and Adult/Pediatric by Optical Biosensor Assay

EN 12821:2009 - Foodstuffs - Determination of vitamin D by high performance liquid chromatography - Measurement of cholecalciferol (D3) or ergocalciferol (D2).

EN12823-1:2000 Foodstuffs - Determination of vitamin A by high performance liquid chromatography - Part 1: Measurements of all-trans-retinol and 13-cis-retinol


EN 14084:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion

EN 14082:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing

EN 14122:2003 Foodstuffs Determination of vitamin B1 by HPLC.

EN 14152:2003 Foodstuffs - Determination of vitamin B2 by HPLC.

EN14627:2005- Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion.

EN 15505:2008 Foodstuffs - Determination of trace elements - Determination of sodium, magnesium and calcium by flame atomic absorption spectrometry (AAS) after microwave digestion. Other methods are available

EN 15111:2007 Foodstuffs Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)


EuroFIR Network of Excellence, 2007, Value documentation and quality assessment testing, Work package 1.8, internal report, Mark Roe

EuroFIR Network of Excellence, 2008a, Work package 1.3, Task group 4, Deliverable 1.3.21, Guidelines for Quality Index attribution to original data from Scientific literature or reports for EuroFIR data interchange published on website, June 30th, 2008,

http://www.eurofir.net/eurofir_partner/integration_platform_deliverables

EuroFIR Network of Excellence, 2008b, Work package 1.3, Task group 4, Deliverable 1.3.14, Report comparing USDA data quality assessment system(s) to existing European systems, second draft July 22th, 2008,

http://www.eurofir.net/eurofir_partner/integration_platform_deliverables


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http://www.eurofir.net/eurofir_partner/integration_platform_deliverables
Westenbrink S, Oseredczuk, M., Castanheira I., Roe M., 2009, The EuroFIR approach to develop tools to assure the quality of the data compilation process, Food Chemistry, 113 (3), 759-767

# 9 Tables & Figures

Table 1 - List of micronutrients for which GAMA was created

<table>
<thead>
<tr>
<th>Component</th>
<th>Analytical Method</th>
<th>Official method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vitamin A Vitamin A all-trans-retinol and 13-cis-retinol</td>
<td>HPLC</td>
<td>EN12823-1:2000 Foodstuffs - Determination of vitamin A by high performance liquid chromatography - Part 1: Measurements of all-trans-retinol and 13-cis-retinol</td>
</tr>
<tr>
<td>Vitamin B1 Thiamin</td>
<td>HPLC</td>
<td>EN 14122:2003 Foodstuffs Determination of vitamin B1 by HPLC</td>
</tr>
<tr>
<td>Vitamin B2 Riboflavin</td>
<td>HPLC</td>
<td>EN 14152:2003 Foodstuffs - Determination of vitamin B2 by HPLC</td>
</tr>
<tr>
<td>Vitamin B5 – Pantothenic Acid</td>
<td>HPLC</td>
<td>AOAC 992.07 - Pantothenic Acid in Milk-Based Infant Formula</td>
</tr>
<tr>
<td>Component</td>
<td>Analytical Method</td>
<td>Official Method</td>
</tr>
<tr>
<td>-------------------------------------------------------------------------</td>
<td>------------------</td>
<td>---------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Vitamin C (L (+) ascorbic acid and dehydro L (+) ascorbic acid)</td>
<td>HPLC</td>
<td></td>
</tr>
<tr>
<td>Vitamin D3, D2</td>
<td>HPLC</td>
<td>EN 12821:2009 - Foodstuffs - Determination of vitamin D by high performance liquid chromatography - Measurement of cholecalciferol (D3) or ergocalciferol (D2).</td>
</tr>
<tr>
<td>Vitamin K1, K2</td>
<td>HPLC</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td>HPLC</td>
<td>AOAC</td>
</tr>
<tr>
<td>Folic acid and Folates</td>
<td>ICP-OES; AAS</td>
<td>AOAC 2011.05: Chandra-Hioe MV et al., 2012</td>
</tr>
<tr>
<td>Calcium</td>
<td>ICP-OES; AAS</td>
<td>AOAC</td>
</tr>
<tr>
<td>Copper</td>
<td>ICP-OES; AAS</td>
<td>EN 14084:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion EN 14082:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing</td>
</tr>
<tr>
<td>Iodine</td>
<td>ICP-MS</td>
<td>EN 15111:2007 Foodstuffs - Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)</td>
</tr>
<tr>
<td>Component</td>
<td>Analytical Method</td>
<td>Official Method</td>
</tr>
<tr>
<td>---------------</td>
<td>-------------------</td>
<td>---------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Iron</td>
<td>ICP-OES; AAS</td>
<td>EN 14084:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion. EN 14082:2003 Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing.</td>
</tr>
<tr>
<td>Magnesium</td>
<td>ICP-OES; AAS</td>
<td>EN 15505:2008 Foodstuffs -- Determination of trace elements - Determination of sodium and magnesium by flame atomic absorption spectrometry (AAS) after microwave digestion. AOAC 984.27</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>ICP-OES; AAS</td>
<td>ISO 9874:2006: Milk -- Determination of total phosphorus content -- Method using molecular absorption spectrometry. Other methods are available: AOAC (AOAC 984.27)</td>
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<tr>
<td>Potassium</td>
<td>ICP-OES; AAS</td>
<td>ISO 11885: 2007 Water quality -- Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) AOAC (AOAC 984.27; AOAC 985.35)</td>
</tr>
<tr>
<td>Selenium</td>
<td>ICP-MS (HGAAS)</td>
<td>EN14627:2005- Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion.</td>
</tr>
<tr>
<td>Component</td>
<td>Analytical Method</td>
<td>Official Method</td>
</tr>
<tr>
<td>-----------</td>
<td>------------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>Sodium</td>
<td>AAS; ICP-OES</td>
<td><strong>EN 15505:2008</strong> Foodstuffs - Determination of trace elements - Determination of sodium, magnesium and calcium by flame atomic absorption spectrometry (AAS) after microwave digestion. Other methods are available: <strong>AOAC (AOAC 984.27)</strong></td>
</tr>
<tr>
<td>Zinc</td>
<td>ICP-OES; ICP-MS</td>
<td><strong>EN 14082:2003</strong> Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper, iron and chromium by atomic absorption spectrometry (AAS) after dry ashing. Other methods are available: <strong>AOAC (AOAC 984.27)</strong></td>
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</tbody>
</table>
Figure 1: Linkage between GAMA and other tasks of WP1 EuroFIR Nexus

Figure 2: Testing GAMA at Data Quality Evaluation System
Figure 3: General overview of WIKI GAMA – list of nutrients so far
Figure 4: Main WIKI page of folate
Figure 5: List of EuroFIR members with expertise in each component area and their responsibilities to updating and disseminating GAMA beyond the EuroFIR Nexus.