

**FSIS 64/04      September 2004**

## **NUTRIENT ANALYSIS CATCH UP PROJECT**

### **Summary**

The Food Standards Agency has recently carried out a survey to determine the nutrient composition of a diverse range of foods for which a need for new data has been identified. The survey forms part of the Agency's rolling programme of nutrient analysis that provide up-to-date and reliable information on the nutrient content of foods. This data is incorporated into the Agency's nutrient databanks, which support our national dietary surveys.

Thirty-two composite samples were analysed for energy and a range of nutrients including fat, protein, carbohydrate, fibre and a full range of vitamins and minerals. Foods analysed were farmed and wild salmon, quiche, cook-in sauces, stir fry sauces, American muffins, jaffa cakes, coffee, sushi, vegetarian mince, rice cakes, premium chilled carton soups, potato wedges, vegetable curry (ready meal) and cake bars. Nutrient levels were in line with those found previously in other, similar foods.

The results of this survey are being incorporated into the Agency's nutrient databanks used in dietary surveys to monitor the nation's diet and future publications of *McCance and Widdowson's The Composition of Foods* series.

### **Background**

The Food Standards Agency undertakes a rolling programme of nutrient analysis surveys to ensure that reliable, up-to-date information on the nutritional value of foods is available for use in conjunction with food consumption data collected in dietary surveys to monitor the nutritional value of the nation's diet. Therefore, these nutrient surveys need to provide a single, robust set of nutrient values that is indicative of the potentially broad choice available to the consumer when selecting any particular type of food. As a result, composite samples

made up of a number of different brands have been analysed for this survey rather than samples made up of single brands, and a generic name is given to each composite. For this reason, this survey is excluded from the Agency's policy of naming the individual products analysed when the results are published.

The aim of this particular survey was to provide up-to-date nutrient composition data for a diverse range of foods for which the Agency does not have detailed information. This is because they represent a relatively new product type on the market (eg premium chilled soups), have changed significantly since they were last analysed (eg cook-in sauces) or have only recently been widely consumed (eg sushi), hence the title 'Catch Up Project'. For some foods (eg salmon and quiche) this data will update and extend the information currently held (eg providing separate data for farmed and wild salmon, and data for a range of different types of quiche).

## **Methodology**

Two hundred and seventy food samples were purchased from retail outlets across Lancashire, West Midlands, Birmingham, Shropshire, Worcestershire, Gloucestershire, Wiltshire, Somerset, Berkshire and London during October 2002 to March 2003. These retail outlets included supermarkets, wholesalers, fishmongers, market stalls, high street delicatessens, motorway service stations, cafes, restaurants, coffee shops, frozen food centres, sushi bars, health food stores and high street bakeries. The food samples consisted of farmed and wild salmon, quiche, cook-in sauces, stir fry sauces, American muffins, jaffa cakes, coffee, sushi, vegetarian mince, rice cakes, premium chilled carton soups, potato wedges, vegetable curry (ready meal) and cake bars. The wild salmon samples were either caught from one of four UK locations or imported from Canada. These samples were all confirmed as wild.

The food samples were combined into 32 composite samples for analysis. Each composite was made up of ten sub-samples, combined on an equal weight basis. Market share information was used to determine which sub-samples were included in each composite. This process allows a single, robust set of nutrient values to be derived for each product type, covering an appropriate cross-section of retail products available. Samples requiring preparation/cooking were prepared in accordance with manufacturer's

instructions and using normal domestic practices. A full list of the composite food samples analysed is given in Table 1.

The full sampling report is available in hard copy through the Food Standards Agency library (contact details given in Further Information).

The composite samples were analysed for energy and a range of nutrients including fat, protein, carbohydrate, fibre and a full range of vitamins and minerals, depending on the importance of the particular food as a dietary source for each nutrient, and existing compositional data available. New analyses were undertaken to ascertain the lycopene and lutein content of tomato based cook-in sauces and premium tomato soup. These are carotenoids that do not have vitamin A activity, but have a beneficial role as antioxidants in the body. Lycopene, mainly found in tomatoes, is released on cooking and is therefore found in higher amounts in cooked or processed tomatoes than fresh. A full list of nutrients is given in Table 2. The methods used to conduct the analyses are included at Annex One.

## **Results**

As each of the composite samples was analysed for an extensive range of nutrients, this project generated a large number of individual results. The main results are shown in Table 3. The full set of results are provided in the analytical report which is available in hard copy through the Food Standards Agency library (contact details given in Further Information).

The aim of this survey was to provide up-to-date nutrient composition data for a diverse range of foods for which the Agency does not have detailed information. This survey has, therefore, generated data which is not directly comparable to existing analytical data. However, where comparable, nutrient levels were in line with those found previously in similar foods.

## **Interpretation**

This survey has generated much new data where none were previously available, updated and extended existing data, and has provided information on new products that have become more popular and widely available in recent years. The data from this survey will

enable us to more accurately monitor the nutritional value of the nation's diet. The results of this survey will be incorporated into the Agency's nutrient databanks for current and future surveys, together with future publications in the *McCance and Widdowson's The Composition of Foods* series.

### **Further Information**

The report of this survey (entitled Nutrient Analysis Catch Up Project) is held in the Dr Elsie Widdowson Library and Information Service at the Food Standards Agency headquarters in London. If you would like to consult or receive a copy, please contact:

Dr Elsie Widdowson Library and Information Service

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A small charge for photocopying will be made.

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**Table 1: Details of Composite Samples Analysed**

<b>Sample</b>	<b>Food group</b>
1	Salmon, raw, farmed
2	Salmon, grilled, farmed
3	Salmon, steamed, farmed
4	Salmon, baked, farmed
5	Salmon, raw, wild
6	Quiche, meat, retail
7	Quiche, fish, retail
8	Quiche, vegetable, retail
9	Indian cook-in sauces, korma/tikka masala
10	Indian cook-in sauces, other
11	Chinese cook-in sauces, sweet & sour
12	Chinese stir fry sauces
13	Traditional cook-in sauces, tomato based
14	Traditional cook-in sauces, white sauce based
15	American muffins, chocolate
16	American muffins, not chocolate
17	Jaffa cakes
18	Cappuccino/latte
19	Sushi, salmon nigiri
20	Sushi, tuna nigiri
21	Sushi, vegetable
22	Frozen vegetarian mince
23	Rice cakes
24	Premium chilled carton soup, carrot and coriander
25	Premium chilled carton soup, mushroom
26	Premium chilled carton soup, tomato
27	Premium chilled carton soup, broccoli and stilton
28	Potato wedges, raw
29	Potato wedges, cooked (oven baked)
30	Vegetable curry, no rice (ready meal) cooked
31	Cake bars, chocolate
32	Cake bars, not chocolate

**Table 2: List of Nutrients Analysed**

Proximates	Water Protein (nitrogen and nitrogen factor) Fat Dry Ash content
Fatty acids	Individual fatty acids (cis & trans isomers, positional isomers, branched chain) (Expressed as percentage total fatty acids and per 100g food)
Sterols	Cholesterol and other main sterols (brassicasterol, campesterol, stigmasterol, beta-sitosterol, fucostanol, d5-avenasterol, d7-stigmastenol, d7-avenasterol)
Carbohydrate	(All expressed as monosaccharide equivalents) Starch, total sugars, total carbohydrate, glucose, fructose, sucrose, maltose, lactose, galactose, oligosaccharides, maltodextrins
Fibre	As non-starch polysaccharide i.e. Englyst method, and AOAC method
Inorganics	Sodium, potassium, calcium, magnesium, manganese, phosphorus, iron, zinc, copper, iodide, selenium, chloride
Water soluble vitamins	Thiamin, vitamin B <sub>6</sub> , niacin, folate, riboflavin, vitamin B <sub>12</sub> , biotin, pantothenic acid, tryptophan (to calculate niacin equivalent), vitamin C
Vitamin A	13-cis retinol, all trans retinol, carotenoids (alpha and beta-carotene, beta-cryptoxanthin)
Other carotenoids	Lutein, cis and trans lycopene
Vitamin D	Vitamin D <sub>3</sub>
Vitamin E	All tocopherol fractions

**Note:** Each of the samples was analysed for a range of nutrients in the above list, depending on existing compositional data available and the importance of the particular food as a dietary source of each nutrient.

## **Annex 1: Analytical Methods Used**

### ***Moisture:***

Homogenised samples mixed with sand were oven dried at 102°C plus or minus 2°C for three hours and the moisture loss determined gravimetrically. Analysis by Direct Laboratories. In house method Q/005 - UKAS accredited.

### ***Protein:***

Nitrogen was determined using the Kjeldahl technique. The nitrogen was converted to protein using the conversion factors detailed in Appendix1 of the Analytical Report. Analysis by Direct Laboratories. In house method Z/002 - UKAS accredited.

### ***Fat:***

Samples were heated with hydrochloric acid, cooled and filtered. The residue was then washed, dried and subjected to a petroleum spirit extraction. Analysis by Direct Laboratories. In house method Q/002 – UKAS accredited.

### ***Ash:***

The homogenised samples were ashed in a muffle furnace by heating at 550°C plus or minus 20°C and the residue (ash) determined gravimetrically. Analysis by Direct Laboratories. In house method Q/001 – UKAS accredited.

### ***Fatty Acids:***

Fatty acids were determined by capillary gas liquid chromatography. Analysis by Direct Laboratories. In house method CHROM/215 – UKAS accredited.

### ***Sterols:***

Sterols were determined by capillary-column chromatography. Analysis by Direct Laboratories. In house method CHROM/200 – UKAS accredited for stigmasterol and beta-sitosterol in butter and butteroil.

### ***Total and Individual Sugars:***

Sugars were analysed using ion chromatography on a Dionex IC system using a CarboPac IC column. Results expressed as monosaccharide equivalents. Sub-contracted to Eclipse Scientific, method AM/C/405.

***Maltodextrins:***

Maltodextrins were determined by high performance liquid chromatography (HPLC) using a Dionex system with a CarboPac PA-100 anion exchange column. Results expressed as monosaccharide equivalents. Sub-contracted to CCFRA Technology Ltd, method TES-AC-270.

***Starch:***

Samples were treated with 40% ethanol to remove free sugars and the residue heated with de-ionised water to gel the starch. Amyloglucosidase enzyme is used to hydrolyse starch to glucose. Glucose is measured colorimetrically using a 'God Perid' test kit. Results expressed as monosaccharide equivalents. Analysis by Direct Laboratories. In house method H/042.

***Total Non-Starch Polysaccharide:***

Total non-starch polysaccharide was determined using the Englyst Fiberzym kit. Analysis by Direct Laboratories. In house method Q/004 – UKAS accredited.

***AOAC Fibre:***

Soluble dietary fibre was precipitated from samples with ethanol. The residue was then filtered, dried and weighed. The total dietary fibre was corrected for protein and ash. Analysis by Direct Laboratories. In house method Q/026 – UKAS accredited.

***Inorganics:***

Sodium, potassium, calcium, magnesium, manganese, phosphorus, iron, zinc and copper were determined by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES). Analysis by Direct Laboratories. In house methods I/046, ICP/003 and GFAA/010 – UKAS accredited. Low level manganese samples were re-analysed by graphite furnace atomic absorption spectrophotometer.

***Iodine:***

Samples (0.3-0.5g) were placed in a vial and 5ml of a 5% solution of tetramethyl ammonium hydroxide (TMAH) added. The vials were shaken thoroughly and placed in an oven at 90°C for three hours. After cooling, solutions were made up to 50ml with 1%

solution of TMAH in a volumetric flask. This solution was then analysed using ICP-MS. Sub-contracted to LGC.

**Selenium:**

Selenium was determined by cold vapour generation and atomic fluorescence. Analysis by Direct Laboratories. In house method I/030 – UKAS accredited.

**Chloride:**

Organic matter is destroyed by wet digestion with potassium permanganate and nitric acid. In the presence of excess silver nitrate, chloride is precipitated as silver chloride. Urea was added to decompose the nitrates and the excess silver nitrate titrated with potassium thiocyanate in the presence of acetone using ferric iron as the indicator. Analysis by Direct Laboratories. In house method Q/012 – UKAS accredited.

**Thiamin (Vitamin B<sub>1</sub>):**

Thiamin was determined by normal phase HPLC using fluorescence detection. Sub-contracted to Eclipse Scientific, method AM/C/703.

**Vitamin B<sub>6</sub>:**

Vitamin B<sub>6</sub> was extracted from the samples using dilute hydrochloric acid and assayed microbiologically using *Saccharomyces cerevisiae*. Bacterial growth was determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/102.

**Vitamin B<sub>12</sub>:**

The sample was extracted using sodium acetate buffer. All liberated vitamin B<sub>12</sub> was converted to cyanocobalamin with potassium cyanide and assayed microbiologically using *Lactobacillus leichmannii*. Bacterial growth was determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/103.

**Niacin:**

The sample was extracted with dilute hydrochloric acid. All liberated niacin was assayed microbiologically using *Lactobacillus leichmannii*. Bacterial growth was determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/101.

**Tryptophan (to calculate niacin equivalent):**

Tryptophan was determined after hydrolysis with lithium hydroxide by reverse phase HPLC with UV detection. Niacin equivalent is calculated as the sum of tryptophan/60 and niacin. Analysis by Direct Laboratories. In house method CHROM/314.

**Riboflavin (Vitamin B<sub>2</sub>):**

Vitamin B<sub>2</sub> was extracted from the samples by digestion with dilute hydrochloric acid followed by enzyme digestion with clara-diastrase. After filtration the vitamin B<sub>2</sub> was determined using reverse phase HPLC using fluorescence detection. Sub-contracted to Eclipse Scientific, method AM/C/703.

**Folate:**

Folic acid and conjugated forms of folic acid were extracted using potassium phosphate buffer. All conjugated forms were converted to folic acid by enzyme digestion. All liberated folic acid was determined microbiologically using *Lactobacillus rhamnosus*. Bacterial growth was determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/105.

**Pantothenic Acid:**

All available pantothenic acid was liberated from the sample using trisbuffer and an enzyme system. Pantothenic acid was determined microbiologically using *Lactobacillus plantarum*. Bacterial growth is determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/104.

**Biotin:**

The sample was extracted using dilute sulphuric acid. The biotin was determined microbiologically using *Lactobacillus plantarum*. Bacterial growth is determined by turbidmetry. Sub-contracted to Eclipse Scientific, method AM/M/106.

**Vitamin C:**

Vitamin C is extracted from the sample using metaphosphoric acid and EDTA. All the ascorbic acid is enzymatically oxidised to dehydro ascorbic acid, which is then converted with O-phenylenediamine to the quinoxaline derivative. Determination is by reverse phase HPLC with fluorescence detection. Sub-contracted to Eclipse Scientific, method AM/C.710.

**Vitamin A: retinol fractions (especially all-trans and 13-cis), carotenoids (alpha and beta-carotene, cryptoxanthins). Non pro-vitamin A carotenoids e.g. lycopene, lutein**

Vitamin A and carotenoids determined using an in-house procedure involving saponification of the sample, solvent extraction and determination by HPLC. Total vitamin A is expressed as micrograms/100g all-trans retinol equivalents (ATRE) calculated as follows:

All-trans retinol + (0.749 X 13 cis-retinol) + (trans beta-carotene ÷ 6) + (other active carotenoids ÷ 12)

Sub-contracted to LGC, method FFF/B1-5007 determination of tocopherol, retinol and carotene isomers in foods and FFF/B1-5008 determination of carotene isomers in foods – UKAS accredited except for beta-cryptoxanthin.

**Vitamin D<sub>3</sub>:**

Vitamin D<sub>3</sub> is extracted from the samples by saponification with alcoholic potassium hydroxide containing pyrogallol. The unsaponified fraction is separated by extraction into hexane. Following solid phase clean up the sample is concentrated and injected onto a semi-preparative HPLC column. The D<sub>3</sub> containing fraction of the eluent is evaporated and dissolved in methanol. The vitamin D<sub>3</sub> is assayed by reverse phase HPLC with UV detection, using vitamin D<sub>2</sub> as an internal standard. Sub-contracted to Eclipse Scientific, method AM/C/723.

NOTE: Results reported for samples of Stir-fry sauce, tomato soup, and broccoli & cheese soup were re-analysed by Direct Laboratories using a UKAS accredited method. Vitamin D<sub>2</sub> is added as an internal standard to the sample prior to extraction. The sample is hydrolysed with ethanolic potassium hydroxide solution and unsaponifiable matter extracted into petroleum spirit. The petroleum spirit is removed by evaporation and the

residue dissolved in hexane. Interfering components are removed by solid phase clean up followed by reverse phase HPLC using an UV detector.

***Vitamin E (all tocopherol fractions):***

The samples are saponified with alcoholic potassium hydroxide and the unsaponified portion (containing vitamin E) is extracted into petroleum ether. After evaporation the residue is dissolved in propan-2-ol. The tocopherols are assayed by reverse phase HPLC using fluorescence detection. Sub-contracted to Eclipse Scientific, method AM/C/733.

Note: The analysis undertaken by Eclipse Scientific separated alpha and delta tocopherol fractions but beta and gamma tocopherols co-eluted. In most cases the total for these two fractions is below the reporting limit. In cases where levels above the reporting limit were detected by Eclipse, Direct Laboratories undertook confirmatory analysis using a method capable of separating the beta and gamma tocopherol.

Methodology: The samples are hydrolysed with ethanolic potassium hydroxide solution and the unsaponifiable matter, containing the vitamins, are extracted into petroleum spirit. The petroleum spirit is removed by evaporation and the residue dissolved in hexane. The concentration of the tocopherols is determined by normal-phase liquid chromatography using fluorescence detection. British Standard BS EN ISO 6867:2001 BS 5766-24:2001. Animal Feeding Stuffs - Determination of Vitamin E Content - Method Using High Performance Liquid Chromatography. Agilent Technologies, Publication Number 5966-0641E. Normal Phase Analysis of Tocopherols in Margarine using HPLC.

***Specific gravity:***

A specific gravity bottle, at 20 °C, is filled with the sample and the weight of the liquid contained compared with the weight of the same volume of water at 20°C. The ratio of the two weights is the specific gravity.

Details of the quality control measures employed are given in the analytical report, available in hard copy through the Food Standards Agency library (contact details given in Further Information).