Report No. C029

Survey of Acrylamide and Furan in UK Retail Products - Analysis Phase: Summary Report for Samples Purchased Between November 2010 and April 2011

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1. SUMMARY

This report contains a summary of the results obtained from a one year survey of acrylamide and furan in retail samples purchased over the period November 2010 – April 2011.

- The survey fulfilled the requirement of Commission Recommendation 2010/307/EU concerning the monitoring of acrylamide in food.
- Samples were supplied to Premier Analytical Services[2] by an independent contractor in accordance with a sampling and analysis plan provided by the UK Food Standards Agency. Foods were cooked according to the manufacturers' guidelines prior to testing. A total of 340 analyses were carried out using accredited methods (248 for acrylamide; 92 for furan) on 248 retail products representing 10 food groups specified in the Commission Recommendation.
- Where possible, acrylamide data from the 2010-2011 survey was combined with that obtained from surveys over the period 2007-2009 and the trends analysed.
- Overall, two of five product categories that could be compared showed a significant reduction in acrylaimde over the period 2007-2011.

[2] Formerly known as RHM Technology Ltd

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3. BACKGROUND

Acrylamide is an industrial chemical widely used to make polymers for applications in paper making, soil conditioning, water treatment and sealants. Since the discovery of acrylamide in cooked foods extensive efforts have been made by researchers in the food industry and academia to understand and control its formation. It is now believed that acrylamide is formed as part of the Maillard reaction, a series of reactions that take place when amino acids react with reducing sugars to give much of the colour and flavour characteristics of heated foods. Acrylamide is formed from the reaction of the amino acid asparagine with reducing sugars and other reactive chemicals generated during the course of the Maillard reaction. Asparagine is an essential amino acid and occurs naturally in all plant materials together with naturally occurring reducing sugars. The amounts of acrylamide formed in foods are subject to the laws governing all chemical reactions and depend on e.g. the relative amounts of each reactant, the applied temperature and duration of cooking. Foods naturally high in these precursors can therefore give rise to significant amounts of acrylamide during cooking and processing, e.g. during drying, frying, baking, grilling, roasting, and toasting. Hence significant amounts of acrylamide have been measured in both home cooked and processed foods such as potato crisps, French fries, crispbreads and coffee. Acrylamide has been classified by the International Agency for Research on Cancer as 'probably carcinogenic in humans' and the Joint FAO/WHO Expert Committee on Food Additives (JECFA) concluded that levels of dietary exposure to acrylamide indicate a human health concern. Because of this, experts have advised that dietary exposure to acrylamide should be as low as reasonably practicable.

Commission Recommendation (EC) No. 2007/331 of 3 May 2007, on the monitoring of acrylamide levels in food, set out a three-year monitoring programme (2007 – 2009) for acrylamide in certain foodstuffs. This was followed by Commission Recommendation (EU) No. 2010/307 on the monitoring of acrylamide in food which placed a requirement on the Agency to obtain further monitoring data on acrylamide in UK retail samples. So far, the results for years 2007, 2008 and 2009 have been compiled and reported by the European Food Safety Authority (EFSA).

FoodDrinkEurope[3] maintains and publishes the Toolbox, which provides guidance to industry on intervention steps that may be helpful in reducing acrylamide formation in French fries, crisps, bread, bakery wares, breakfast cereals, and coffee. While efforts by the food industry and member states to reduce acrylamide levels in foodstuffs are ongoing, it has been deemed appropriate to continue the collection of monitoring data to ascertain the efficacy or effects of these measures.

Like acrylamide, furan is also widely used in the chemical industry for the preparation of e.g. resins, lacquers and agricultural and pharmaceutical chemicals. While furan has been known as a flavour volatile for many years, e.g. in coffee, only limited data exist for concentrations in other foods. Various precursors such as reducing sugars, vitamin c, organic acids, carotenes and polyunsaturated fatty acids may lead to furan formation during heating but the underlying mechanism are not fully understood. Furan has been classified by the IARC as a possible human carcinogen. Evidence indicates that the latter may occur via a genotoxic mechanism.

The results from this one year survey support the Agency's current Strategic Plan (to 2015) in ensuring that foods produced or sold in the UK are safe to eat. It also meets the requirements of Commission Recommendation (EU) No. 2010/307 for member states to monitor amounts of acrylamide in retail foods and further supports Agency work to identify and reduce the impact of potential new and re-emerging risks; hence the inclusion of furan testing on selected samples collected for analysis.

[3] Formerly know as CIAA (Confederation of Food and Drink Industries of the EU)

4. METHODS

4.1 SAMPLES

Samples representing the ten food groups specified in Commission Recommendation 2010/307/EU were collected by Ventress Technical Limited and delivered to Premier Analytical Services for analysis. Details of the sampling plan are given in Table 4 of the appendices (see section 7.1).

All samples received at the laboratory were prepared and stored according to the Food Standards Agency "Guidelines for undertaking surveys"[4]. Potato products from Groups 3 and 10 were cooked according to manufacturers' guidelines prior to the determination of acrylamide. Further details of the sample preparation procedures used are given in section 7.2 and Table 5.

4.2 ANALYSIS

Analysis of samples for the 2010-2011 survey was carried out using UKAS accredited methods: the measurements of acrylamide and furan were based on the guidelines laid down in Commission Recommendations 2007/331/EC and 2007/196/EC. All test results were obtained from methods that use internal standards for recovery correction in line with Food Standards Agency Guidelines[18] and EC recommendations[7,8]. Further details of the methods together with the analytical quality assurance procedures and measurement uncertainty can be found in the appendices (see sections 7.2 and 7.3).

[4] Guidelines for undertaking surveys. Available at: http://www.food.gov.uk/science/surveillance/guidefsatechsurv [accessed March 2010]

5. RESULTS AND DISCUSSION

A total of 248 samples were received over the period November 2010 - April 2011 for the analysis of acrylamide and furan in accordance with the test requirements given in Table 1. Potato products for home cooking and microwaveable popcorn were cooked according to manufacturers guidelines prior to analysis (see Table 5 in the Annex). Coffee and coffee substitutes requiring furan analysis were tested as received and as consumed to assess potential losses of volatile furan during preparation.

Table 1. Summary of samples received and tests undertaken

Group	Category	Number of samples received	Analyses			
			acrylamide		furan	
			As received	As consumed	As received	As consumed
1	French fries, sold as ready-to-eat	42	42			
2	Potato crisps	20	20		20	
3	Pre-cooked French fries/potato products for home cooking	16		16		
4	Bread	20	20			
5	Breakfast cereals	20	20			
6	Biscuits / crackers	20	20			
7	Coffee and coffee substitutes	20	20		20	20
8	Baby foods	20	20		20	
9	Processed cereal based foods for infants & young children	20	20			
10	Vegetable crisps	2	2		2	
	Prefabricated potato snacks	2	2		2	
	Other potato products for home cooking	6		6		
	Microwave French fries	2		2		
	Prefabricated protato products for home cooking	4		4		
	Pop corn	2		2		2
	Tortillas	2	2		2	
	Cakes	5	5			
	Pastry	5	5			
	Cereal bars / Granola	4	4			
	Novelty ginger bread	2	2			
	Cocoa powder	2	2			
	Canned prunes	2	2		2	
	Chocolate	1	1			
	Dried fruit (dark)	2	2			
	Canned black olives	1	1			
	Ethnic foods	6	6			

5.1 ACRYLAMIDE

A summary of acrylamide concentrations (mean, range and number of samples) measured in each of the main categories and sub-categories according to Commission Recommendation 2010/307/EU is given in Table 2.

Table 2. Summary of acrylamide concentrations[a] by product category

Product category	n	mean (µg/kg)	min (µg/kg)	max (µg/kg)
Group 1 - French fries sold as ready to eat	42	239	41	1285
Nov-10 delivery	21	255	41	660
Mar-11 delivery	21	223	53	1285
Group 2 - Potato Crisps	20	835	220	2061
Nov-10 delivery	10	738	220	1859
Mar-11 delivery	10	933	444	2061
Group 3 - Pre-cooked French fries for home cooking	16	194	21	1155
Nov-10 delivery	8	206	21	1155
Mar-11 delivery	8	183	39	491
Group 4 - Soft bread	20	16	3	51
Plant white	8	15	7	37
Plant wholemeal	4	24	15	33
Others (e.g. in-store bakery, rye, linseed, rolls etc)	8	14	3	51
Group 5 - Breakfast Cereals	20	149	35	325
Group 6 - Biscuits & crackers	20	380	27	1573
Crackers	9	275	48	473
Crispbread	3	197	120	326
Wafers	1	154	-	-
Other (sweet)	7	625	27	1573
Group 7 - Coffee	20	501	49	1009
Roast coffee	8	212	172	243
Instant coffee	6	865	724	997
Coffee subs	6	521	49	1009
Group 8 - Baby Food other than processed cereal based	20	13	3	27
Group 9 - Processed Cereal Baby Food	20	65	3	598
Biscuits and rusks for infants and young children	10	110	3	598
Other processed cereal based foods for infants and young children	10	18	6	68
Group 10 - Others	50	311	5	3972
Vegetable crisps	2	2651	1330	3972
Canned black olives	1	884	884	884
Other potato products for home cooking	6	579	44	1604
Cocoa	2	442	176	707
Prefabricated crisps	2	364	285	443
Popcorn	2	328	205	451
Microwave French Fries	2	327	327	328
Canned prunes	2	305	247	362
Novelty gingerbread	2	247	51	443
Cereal bars & granola	4	135	82	259
Tortila / corn chips	2	103	79	127
Prefabricated potato products for home cooking	4	68	18	108
Ethnic foods	6	64	25	120
Dried fruit	2	59	49	68
Cakes	5	33	12	86
Pastries	5	29	5	57
Chocolate	1	24	24	24

[a] N.D. <0.5 (LOD); relative uncertainty of measurement 14% (expanded uncertainty for a single determination at 49 µg/kg, coverage factor = 2)

Figure 1, which has been ordered by increasing mean concentrations, shows that categories / sub-categories with the lowest mean amounts of acrylamide (<100 µg/kg) were: prefabricated potato products for home cooking (Gp 10); other processed cereal based foods for infants & young children

(Gp 9); ethnic foods (Gp 10); dried fruit (Gp 10); cakes and pastries (Gp 10); chocolate (Gp 10); soft bread (Gp 4); and baby foods other than processed cereal based foods (Gp 8).

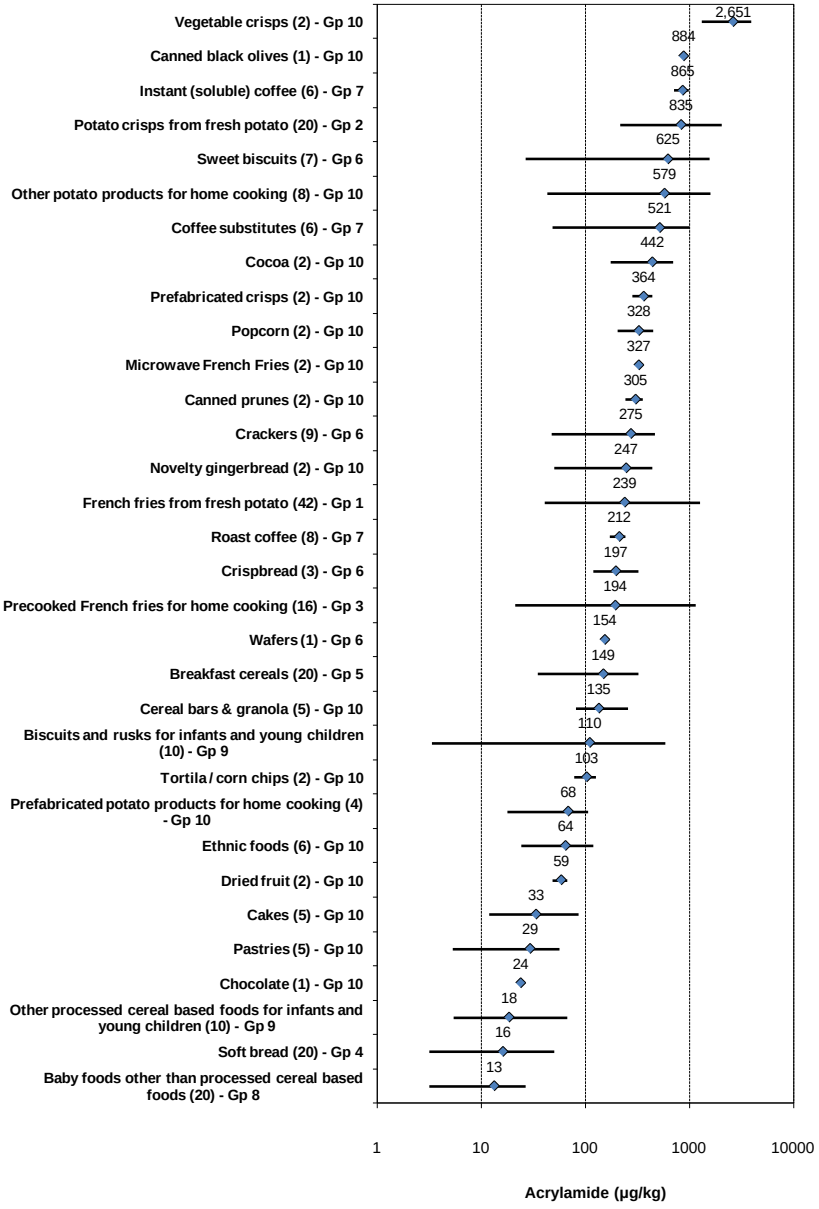

Figure 1. Individual product categories in order of increasing acrylamide concentrations (logarithmic scale): mean, range and number of samples (in parentheses).

Products with the highest mean concentrations of acrylamide (>500 µg/kg) included (see Figure 1): the vegetable crisps (Gp 10); canned black olives (Gp 10); instant (soluble) coffee (Gp 7); potato crisps from fresh potato (Gp 2); sweet biscuits (Gp 6); other potato products for home cooking (Gp 10); and the coffee substitutes (Gp 7).

5.1.1 Products prepared from fresh potato (Groups 1, 2, 3 and 10).

Products prepared from fresh potato were sampled biannually to assess the effects of seasonal variation on acrylamide generated during cooking (see Table 4). The approximate cook times and pre-processing conditions for French fries sold as ready to eat (Group 1) were supplied by each outlet. French fries for home cooking (chilled and frozen) were cooked using domestic appliances according to manufacturers guidelines.

Group 1. Acrylamide amounts measured in 42 samples of French fries sold as ready to eat (Group 1) showed considerable variation, consistent with the wide range of pre-processing (e.g. chipping/soaking/blanching) and cooking conditions (time/temperature) employed. The ratio of thin to thick cut chips was approximately 1:3. The overall mean acrylamide concentration in the November 2010 fries (255 µg/kg) was slightly higher than that in March 2011 (223 µg/kg) although this difference was more apparent in the thin cut samples. Although no sampling was undertaken during March 2010, this apparent decrease in mean acrylamide values over the period November to March did not follow the expected trend observed for previous years. The seasonal trends for both thin and thick cut French fries over the period March 2007 – March 2011 are given in Figure 2.

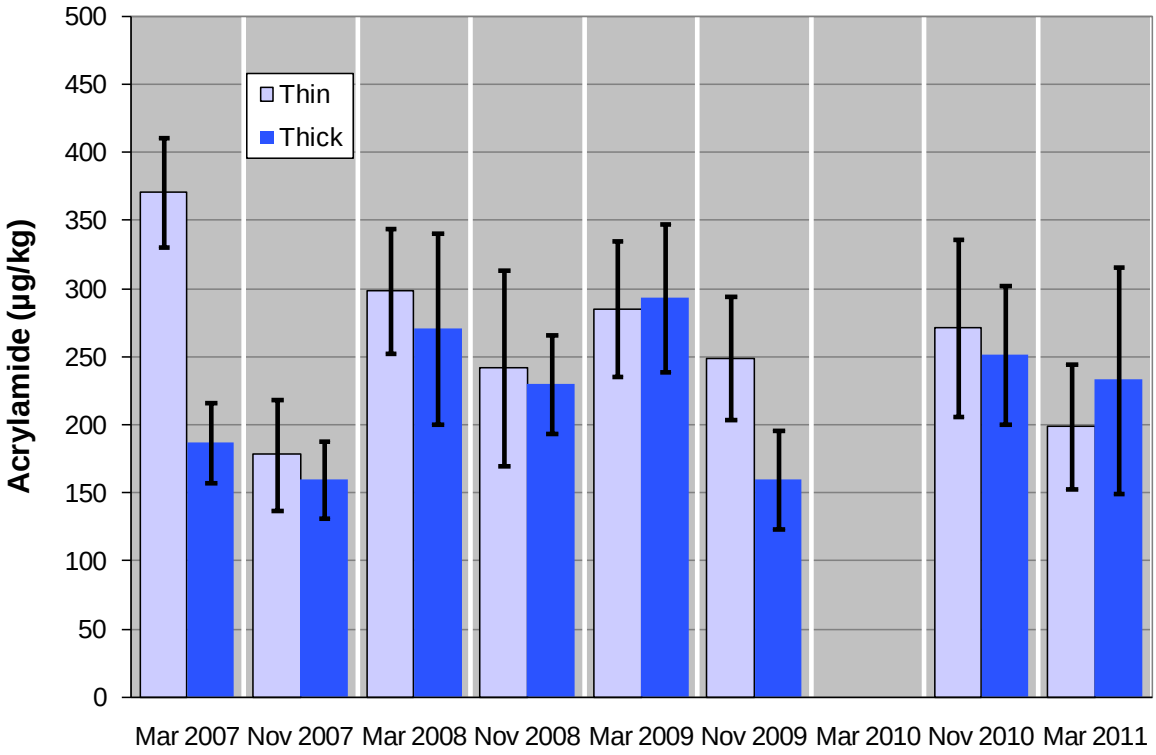

Figure 2. Comparison of seasonal means for acrylamide in thin and thick cut French fries sold as ready to eat (vertical bars are 2 standard errors). No sampling was undertaken during March 2010.

Group 3. A total of 16 samples of pre-cooked French fries for home cooking were cooked using either a domestic oven or a deep fat fryer according to manufacturers guidelines (see Table 5). Amounts of acrylamide in these samples (mean 194 µg/kg) were only marginally less than the French fries sold as ready to eat (Group 1; 239 µg/kg): French fries prepared by frying (4 samples) produced circa three times less acrylamide (78 µg/kg) than products cooked in a domestic oven (12 samples; 233 µg/kg). In keeping with the Group 1 samples, these French fries also showed an apparent decrease in mean acrylamide concentrations over the period November to March. A comparison with data from previous years could not be made since previous surveys of the Group 3 products included data for other potato products e.g. roast potatoes.

Group 10. Additional potato products for domestic preparation (8) not covered by the EFSA Group 3 classification were included in the survey under Group 10. These products were also sampled in March and November and included roast potatoes and potato chunks for oven cooking, and microwaveable French fries. The mean acrylamide concentration of 516 µg/kg for all the potato products in this category was significantly higher than that of the Group 3 pre-cooked French fries for home cooking (mean 194 µg/kg) although this relatively small dataset appeared skewed by a single

sample taken in November which gave an acrylamide concentration of 1604 µg/kg when cooked (see Figure 3).

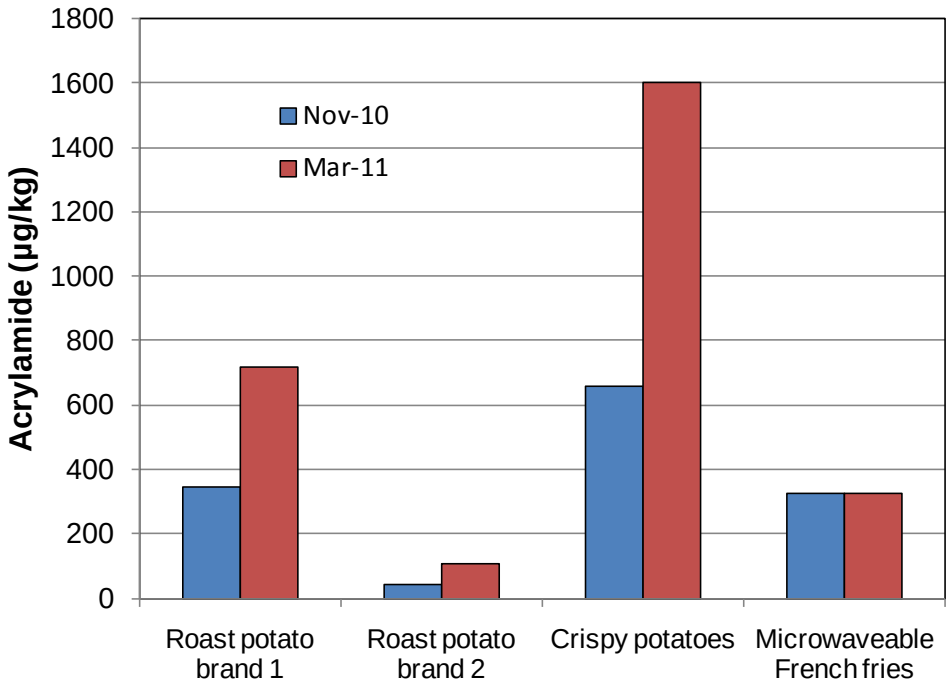

Figure 3. Acrylamide in microwave French fries and other potato products for home cooking (Group 10): data relate to individual samples

Group 2. Figure 4 shows the seasonal trends for acrylamide in potato crisps over the period 2008 – 2011 (data from the 2007 survey could not be included as these samples had been collected in April and December). The annual mean concentration of acrylamide for 20 samples of potato crisps sampled in November 2010 and March 2011 (835 µg/kg) was lower than the annual mean value obtained for November 2008 and March 2009 (1343 µg/kg) although, in the absence of the March 2010 data set, a clear trend could not be determined. Amounts of acrylamide in the 2010-2011 survey crisps ranged from 220 to 2061 µg/kg and did not appear to be associated with the cooking method, i.e. mean acrylamide levels in hand cooked crisps (839 µg/kg) were not significantly different from amounts in regular crisps (834 µg/kg). The mean acrylamide values for samples taken in November 2010 (738 µg/kg) were less than those taken in March 2011 (933 µg/kg).

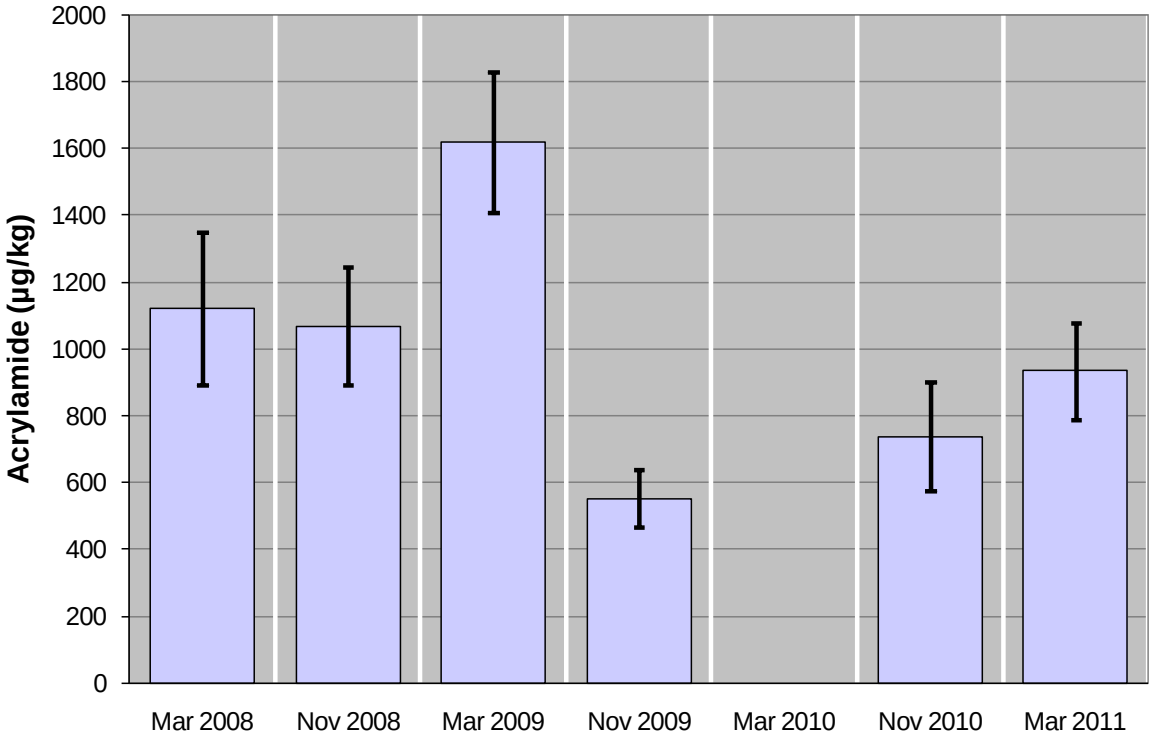

Figure 4. Comparison of seasonal means for acrylamide in crisps prepared from fresh potato slices (vertical bars are 2 standard errors). No sampling was undertaken during March 2010.

5.1.2 Soft bread (Group 4 – 20 samples).

Retail bread products representing plant produced white and wholemeal loaves, rolls and baguettes and speciality products such as soya and linseed, sunflower and barley, olive ciabatta etc, were included in the survey (samples taken in December 2010).

Overall, the amounts of acrylamide in the 2010 breads ranged from 3 – 51 µg/kg and the mean concentration of 16 µg/kg was slightly lower than previous years (see Figure 5 and Table 2). As expected, white bread had a lower mean acrylamide content (15 µg/kg) compared to wholemeal bread (24 µg/kg) presumably due to the lower asparagine content of white flour.

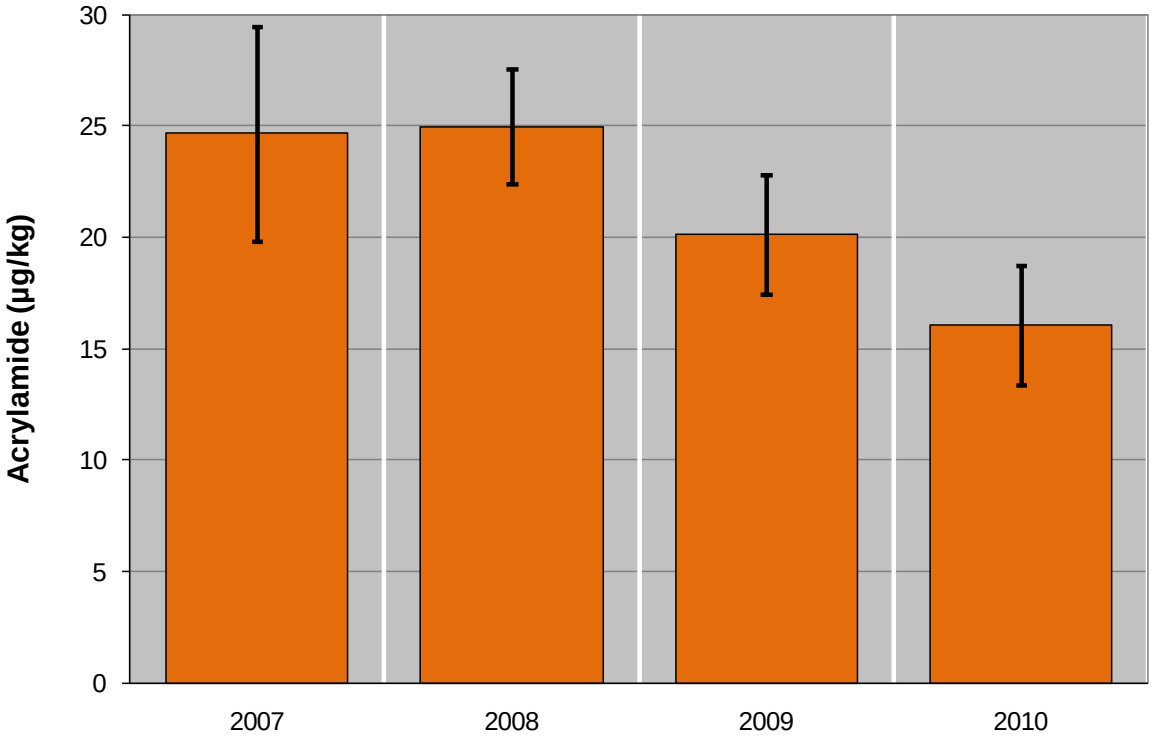

Figure 5. Comparison of annual mean acrylamide concentrations in retail soft bread (vertical bars are 2 standard errors).

5.1.3 Breakfast Cereals (Group 5 – 20 samples).

A total of 20 samples of breakfast cereal were purchased in December 2010 for the 2010-2011 survey compared to 10 samples annually over the period 2007 - 2009. The mean acrylamide concentration for the 2010 samples was 149 µg/kg (range 35-325 µg/kg) and this appeared to continue the trend towards lower acrylamide values over the period 2007 – 2010 (see Figure 6).

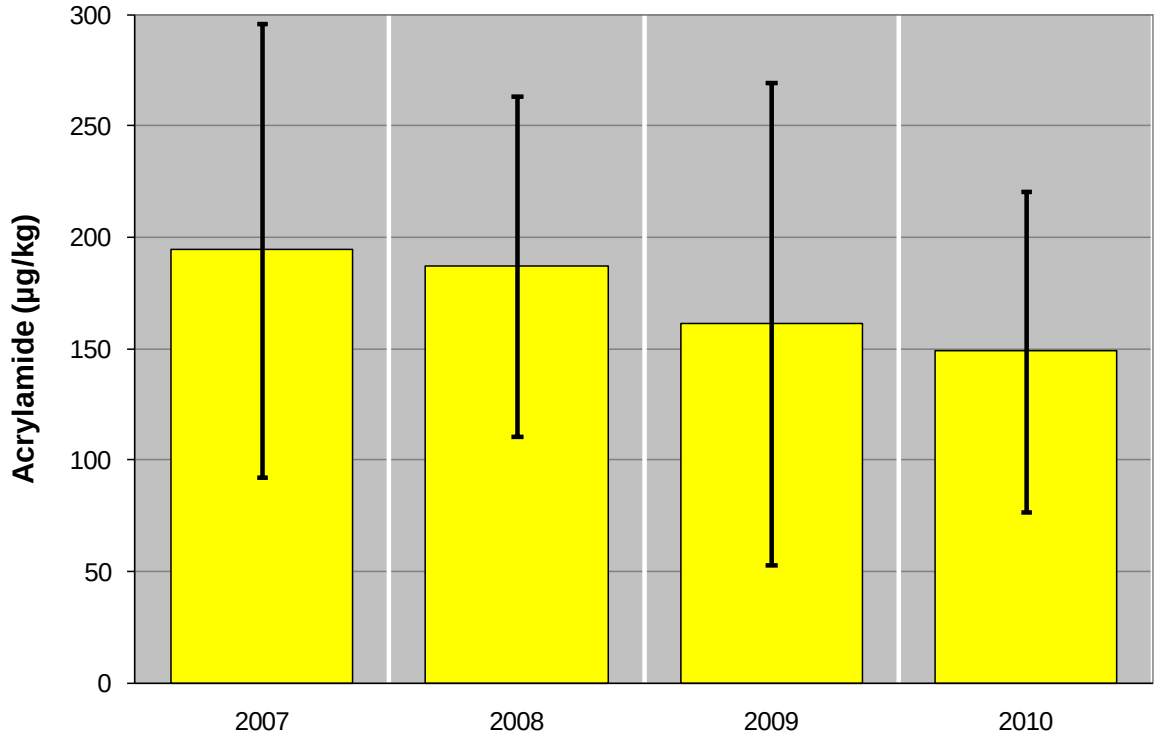

Figure 6. Comparison of annual mean acrylamide concentrations in breakfast cereals (vertical bars are 2 standard errors).

5.1.4 Biscuits and crackers (Group 6 – 20 samples).

This Group comprised four of the five sub-categories according to the Commission Recommendation (2010/307/EU), i.e. crackers (9), crispbreads (3), a wafer and other (7 sweet) biscuits (the Gingerbread category was placed in Group 10 as the UK product / recipe was different to that consumed in Europe. The mean acrylamide concentration of 380 µg/kg (range 27-1573 µg/kg) for the 20 samples purchased in January 2011 was slightly higher than the value of 329 µg/kg (range 30-902 µg/kg) obtained in the previous (2009) survey, but still significantly lower than the period 2007-2008 (mean values 465 – 456 µg/kg). Within this category, the sweet biscuits sub-category had the highest mean acrylamide concentration (625 µg/kg, range 27-1573 µg/kg) with the ginger biscuits accounting for the highest individual amounts (range 817-1573 µg/kg, n=3). Products declaring yeast in the recipe accounted for some of the lowest values of acrylamide (range 48 – 120 µg/kg, n=3).

5.1.5 Coffee (Group 7 – 20 samples).

This category purchased in January 2011 comprised roast and instant coffees and the coffee substitutes. Measurement of acrylamide was carried out on the coffee as received in order to assess mitigation measures on an annual basis. Hence the amounts of acrylamide do not represent values as consumed in the brew, which will be much reduced by dilution with water. The instant coffees and coffee substitutes had the highest mean acrylamide contents at 865 µg/kg and 521 µg/kg respectively. Both the instant coffees and coffee substitutes were not included in the Group 7 category of earlier surveys making comparisons with previous data difficult. However, roast coffee had been included in previous surveys and the mean concentration of acrylamide in the 2011 samples (212 µg/kg) showed little change from the means obtained in 2009 (199 µg/kg), 2008 (245 µg/kg) and 2007 (229 µg/kg).

5.1.6 Baby foods other than processed cereal based foods (Group 8 – 20 samples)

This category accounted for the lowest mean acrylamide concentration (13 µg/kg) in the survey and included infant foods packaged in jars and pouches. The annual mean result was within the range of means measured in previous UK surveys (jarred baby foods 12 – 29 µg/kg, 2007 – 2009).

5.1.7 Processed cereal based foods for infants & young children (Group 9 – 20 samples)

A total of 20 samples were purchased in February 2011 comprising the two categories specified in the Commission Recommendation, i.e. infant biscuits & rusks and infant cereals (e.g. baby rice and porridge). The mean acrylamide concentration in the infant cereals (i.e. baby rice / porridge) was relatively low (18 µg/kg, n=10) and within the range of means obtained in previous UK surveys (6-30 µg/kg, 2007-2009). Acrylamide levels were significantly higher in the infant biscuits and rusks (mean 110 µg/kg , range 3-598 µg/kg , n=10) with the sweet biscuits accounting for some of the highest concentrations: The highest amount of acrylamide was found in an Italian Biscotti. Biscotti are traditionally subjected to a heavy bake giving a very dry product. The latter, together with the declared ammonium based raising agent and ingredients relatively high in reducing sugars (e.g. apple puree and banana[5]), are all significant factors for increased acrylamide formation.

5.1.8 Other products (Group 10 – 50 samples).

This Group included a range of foodstuffs not defined by Groups 1-9 and for which little or no data was available from previous surveys of acrylamide in UK retail products. All samples were analysed for their acrylamide content and the results are summarised in Table 2; the results for the potato products for home cooking in this Group are discussed in Section 5.1.1.

Products with the lowest mean acrylamide levels (<100 µg/kg) were: prefabricated potato products for home cooking, ethnic foods (papadum, onion bhaji, pancake rolls), dried fruit, cakes, pastries and chocolate. Vegetable crisps, canned black olives and the potato products for home cooking (other than French fries) all had mean acrylamide concentrations of >500 µg/kg; the vegetable crisps accounted for the highest acrylamide concentrations (1330-3972 µg/kg) presumably due to their relatively high content of the acrylamide precursor asparagine. While occurrence data for some of these products was lacking in the UK the amounts of acrylamide found were consistent with those reported elsewhere (e.g. US FDA surveys).

[5] Daniali, G. 2010. Determination of acrylamide in banana-based snacks and effect of different maturity stages on formation of acrylamide in banana fritters. Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in fulfilment of the requirements for the Master of Science, July 2010

5.2 FURAN

The concentrations of furan in samples of potato crisps (Group 2), coffee (Group 7), jarred baby foods (Group 8) and other products (Group 10) are given in Figure 7 and Table 3.

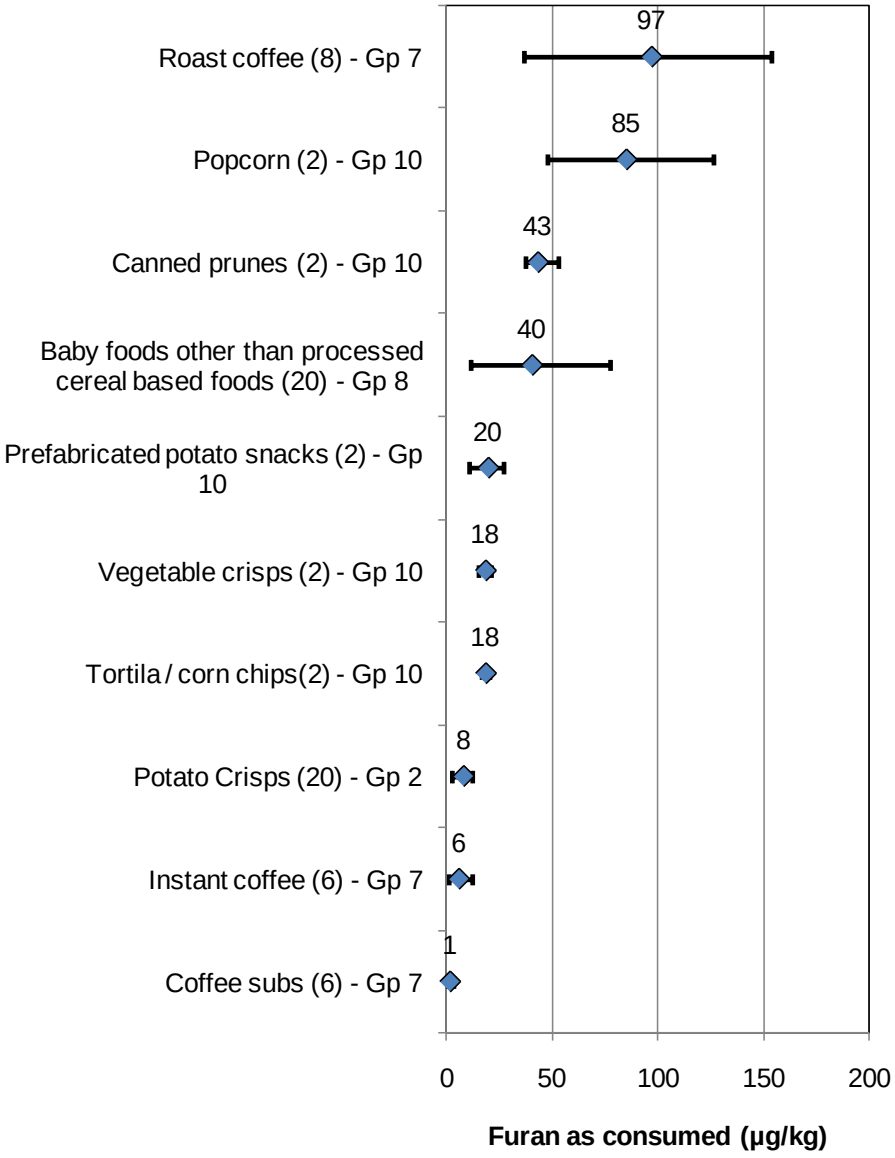

Figure 7. Individual product categories in order of increasing furan concentrations: mean, range and number of samples (in parentheses).

Figure 7, which has been ordered by increasing mean concentrations, shows that categories / sub-categories with the lowest mean amounts of furan (<10 µg/kg) were: potato crisps (Gp 2); instant coffee (Gp 7); and coffee substitutes (Gp 7). Categories with significant amounts of furan (>50 µg/kg) were popcorn and roast coffee (as consumed). Of the categories falling between the range 10-50 µg/kg furan, baby foods in jars and pouches (mean = 40 µg/kg, n=20) purchased in February 2011 showed a slight decrease in furan compared to the mean furan level that was previously measured in 2009 (47 µg/kg, n=10).

Table 3. Furan concentrations[a] (mean and range) as consumed in each product category

Product category	n	mean (µg/kg)	min (µg/kg)	max (µg/kg)
Group 2 - Potato crisps	20	8	3	13
Group 7 - Coffee and coffee substitutes	20	41	0.3	154
Roast coffee	8	97	37	154
Instant coffee	6	6	1	13
Coffee subs	6	1	0.3	4
Group 8 - Baby foods other than processed cereal based foods	20	40	12	78
Group 10 - other products	10	38	11	127
Vegetable crisps	2	18	15	22
Prefabricated potato snacks	2	20	11	28
Popcorn	2	85	49	127
Tortila / corn chips	2	18	17	21
Canned prunes	2	43	38	54

[a] N.D. = <0.07 µg/kg (LOD); relative measurement uncertainty = 20% in concentration range 20-100 µg/kg (expanded uncertainty for a single determination, coverage factor = 2)

All samples from Group 7 were analysed as received and as consumed to determine potential furan losses during preparation. Figure 8 shows that the greatest losses of furan (approximately 50%) occurred during the preparation of the roast and ground coffees (data expressed on solids basis). The preparation of instant coffee on the other hand appeared to result in much less furan loss (data expressed on solids basis) although it is not known if this effect was due to an enhanced "extraction" of furan during the addition of hot water or in situ formation of furan at elevated temperature.

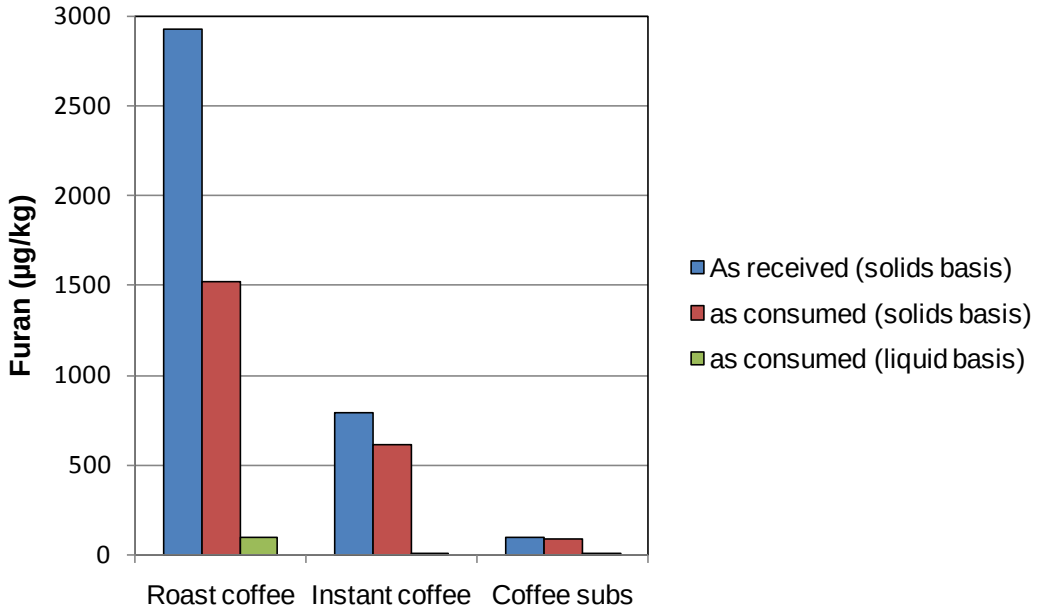

Figure 8. Amounts of furan measured in roast and instant coffees and coffee substitutes expressed on an as received and as consumed basis

6. CONCLUSIONS

- Acrylamide
  - Categories / sub-categories with the lowest mean amounts of acrylamide, i.e. <100 µg/kg), included:
    - prefabricated potato products for home cooking
    - baby rice / porridge
    - ethnic foods e.g. onion Bhaji, papadam
    - dried fruit
    - cakes and pastries
    - chocolate
    - soft bread
    - baby foods in jars and pouches.
  - The highest mean concentrations of acrylamide, i.e. >500 µg/kg were found in:
    - vegetable crisps
    - canned black olives
    - instant (soluble) coffee
    - potato crisps from fresh potato
    - sweet biscuit
    - potato products for home cooking (excluding French fries)
    - coffee substitutes
  - Acrylamide data from five categories / sub-categories purchased in 2010 could be combined with data from previous surveys over the period 2007-2009 and an annual trend determined:
    - Categories showing a significant decrease (R^2>0.9) in annual means included:
      - soft bread
      - breakfast cereals
    - Categories showing no significant change in annual means included:
      - roast coffee
      - baby foods in jars / pouches
      - baby cereals (e.g. rice / porridge)

- Furan
  - Mean amounts of furan in all product categories as consumed were relatively low (< 100 µg/kg).
  - Categories with the lowest mean furan concentration (< 10 µg/kg) as consumed were:
    - potato crisps
    - instant coffee
    - coffee substitutes
  - The highest amounts of furan (as consumed) were found in sweet popcorn and roast coffee
  - When coffee samples were prepared for consumption the most significant losses of furan appeared to occur for the roast coffees

7. APPENDICES

7.1 SAMPLING PLAN

Table 4. Sampling plan 2010 - 2011

	Sample numbers per product category																	
Month	French fries sold as ready to eat (1)			Potato crisps (2)	Pre-cooked French fries for home cooking (3)		Bread (4)	Breakfast cereals (5)	Biscuits, crackers, crispbread (6)	Coffee and coffee substitutes (7)				Baby foods (8) [a]	Processed cereal based foods for infants & young children (9)		Other products (10)[b]	Total
	From fresh potato			From fresh potato	From fresh potato					Roast	Instant		Substitutes		Biscuits / rusks	Other		
	restaurants	fast food	chip shops		fresh	frozen					normal	decaf						
Nov	8	4	8	10	2	8											7	47
Dec							20	20										40
Jan									20	8	3	3	6					40
Feb														20	10	10		40
March	8	4	8	10	2	8											7	47
April																	36	36
TOTALS	16	8	16	20	4	16	20	20	20	8	3	3	6	20	10	10	50	**250**

[a] excluding processed cereals; [b] Vegetable crisps, Canned black olives, Potato products for home cooking other than French fries, Cocoa, Prefabricated crisps, Popcorn, Canned prunes, Novelty gingerbread, Cereal bars & granola, Tortila / corn chips, Prefabricated potato products for home cooking, Ethnic foods, Dried fruit, Cakes, Pastries, Chocolate

7.2 SAMPLE PREPARATION DETAILS AND ANALYTICAL METHODS

7.2.1 Sample preparation

All samples were prepared according to the Food Standards Agency "Guidelines for undertaking surveys"[4]. Where practical, digital images (samples and packaging) were recorded and all packaging retained.

Potato products for home cooking from Group 3 and 10 were cooked according to the manufacturers' guidelines using domestic appliances (oven, grill, microwave, deep fat fryer – see Table 5). The grill and oven were preheated for 5-10 minutes and a single layer of the product was applied to a baking try (placed in middle of oven) or mesh grill pan respectively; samples were typically turned at half of the recommended cooking time. Pure sunflower oil was used for frying.

Coffee samples were prepared according to the manufacturers guidelines given on the packaging: retail domestic coffee preparation equipment was used as required, e.g. a cafetiere or espresso coffee maker.

7.2.1.1 Homogenisation

Samples requiring furan analysis, e.g. jars and packets were separated and retained for individual processing; coffee and coffee substitutes prepared for consumption were analysed immediately after the preparation cycle. These samples were not subjected to bulk homogenisation as this would have resulted in significant losses of volatile furan.

Where practical, entire samples (approximately 1kg) were homogenised for analysis:

- Groups 2, 5, 6, 8 and 10 samples (excluding group 10 potato products for home cooking – see below) were homogenized directly using a food processor.
    - A laboratory mill was used for the additional processing of samples containing seeds.
    - A domestic cheese grater was used for the chocolate.
    - Stones were removed from canned prunes prior to homogenization.
- Group 4 samples were air-dried (weight loss recorded) prior to homogenising.
- Potato products from Groups 1, 3 and 10 were homogenized with water (1:2 sample/water w/w) using a high speed blender or food processor.
- Group 7 and 9 samples (ground/milled products) were mixed thoroughly prior to analysis
    - A coffee grinder was used for the whole coffee beans.

An aliquot of the homogenised sample was taken for analysis while the remainder was divided into four pots and stored at -18°C.

7.2.1.2 Homogeneity testing

Representative samples from Groups 1-10 were selected at random and the homogeneity determined by replicate (6) sodium analyses. This was based on the assumption that both sodium and the analytes of interest had an equivalent distribution in these samples. Samples were confirmed as homogeneous by comparing the relative standard deviation (RSD) of the replicate measurements with that obtained from method control samples using a statistical F-test.

Samples (2-3 g) were treated with hot concentrated nitric acid prior to determination by atomic absorption spectroscopy on a PE 2100 (Perkin Elmer, USA) according to the procedure of Gorsuch[6]. Each batch was analysed with a blank and an in-house reference material.

[6] Gorsuch, T., T., 1970. The Destruction of Organic Matter. Pergamon Press. p21

Table 5. Details of the domestic cooking conditions used for Group 3 and Group 10 products

Sample No.	*Group*	*Description*	*Cooking method*				*Time (min)*	*Pre-cook weight (g)*	*Cooking Procedure*
			Oven (°C)[a]	*Grill*[a]	*Microwave (W)*[b]	*Fryer (°C)*[c]			
10C-11928	3	Oven chips	210				20	1000	cooked on baking tray, turned @ 10 min
10C-11929	3	Thin & crispy oven chips	210				19	800	cooked on baking tray, turned @ 10 min
10C-11930	3	Crispy French fries	220				16	800	cooked on baking tray, turned @ 8 min
10C-11931	3	Straight cut frying chips				190	10	670	Tesco pure vegetable oil, 2.2 l
10C-11932	3	Fresh chunky chips	200				25	900	cooked on baking tray, turned @ 10 & 20 min
10C-11934	3	Organic oven chips				175	4	670	Tesco pure vegetable oil, 2.2 l
10C-11935	3	Homestyle chips	210				23	1000	cooked on baking tray, turned @ 10 min
10C-11936	3	Chunky oven chips	210				23	1000	cooked on baking tray, turned @ 10 min
10C-12109	3	Oven chips	210				20	1000	cooked on baking tray, turned @ 10 min
10C-12110	3	Thin & crispy oven chips	210				19	800	cooked on baking tray, turned @ 10 min
10C-12111	3	Crispy French fries	220				16	800	cooked on baking tray, turned @ 8 min
10C-12112	3	Straight cut frying chips				190	10	670	Tesco pure vegetable oil, 2.2 l
10C-12113	3	Fresh chunky chips	200				25	900	cooked on baking tray, turned @ 10 & 20 min
10C-12115	3	Organic oven chips				175	4	670	Tesco pure vegetable oil, 2.2 l
10C-12116	3	Homestyle chips	210				23	1000	cooked on baking tray, turned @ 10 min
10C-12117	3	Chunky oven chips	210				23	1000	cooked on baking tray, turned @ 10 min

Table 5. Continued…

Sample No.	*Group*	*Description*	*Cooking method*				*Time (min)*	*Pre-cook weight (g)*	*Cooking Procedure*
			Oven (°C)[a]	*Grill*[a]	*Microwave (W)*[b]	*Fryer (°C)*[c]			
10C-11933	10	Roast potatoes	200				45	900	cooked in tray supplied, turned @ 20 min
10C-11937	10	Crispy potatoes	230/200				30	750	Turned @ 20 min & reduced temp to 200
10C-11939	10	Roast potatoes	200				30	1000	Turned at 15 min
10C-11944	10	Microwave chips			750		2.5	600	individual 100 g boxes, 1 min @ 750 W, shake, 1.5 min @ 750 W
10C-12114	10	Roast potatoes	200				45	900	cooked in tray supplied, turned @ 20 min
10C-12118	10	Crispy potatoes	230/200				30	750	Turned @ 20 min & reduced temp to 200
10C-12130	10	Roast potatoes	200				30	1000	Turned at 15 min
10C-12135	10	Microwave chips			750		2.5	600	individual 100 g boxes, 1 min @ 750 W, shake, 1.5 min @ 750 W
10C-11938	10	Waffles		med			8.5	680	grill mesh, turned at 3 min
10C-11940	10	Smiles	200				15	820	cooked on tray, turned @ 6 min
10C-12129	10	Waffles		med			8.5	680	grill mesh, turned at 3 min
10C-12131	10	Smiles	200				15	820	cooked on tray, turned @ 6 min
10C-11988	10	Microwave popcorn			600		5	700	7 x 100 g bags individually microwaved, 2 bags removed for immediate analysis of furan

[a] Fan assisted Tricity Bendix; [b] Panasonic microwave (600 or 750 W/Cat D); [c] Tefal Visialis 1 kg.

7.2.2 Acrylamide analysis by GC/MS/MS

Acrylamide was determined as the brominated derivative, 2-bromopropenamide, according to the method of Hamlet and Sadd[7].

Method performance (typical): limits of detection and quantification were 0.5 and 3.0 µg/kg respectively; the estimated value for the method uncertainty (single determination) was: ±14% at 49 µg/kg (expanded relative uncertainty with a coverage factor of 2). A summary of the method performance data from this survey can be found in Table 6.

7.2.3 Analysis of furan

Furan was measured in samples using a headspace method adapted from the procedure of Becalski *et al*[8]. Pre-chilled samples were homogenised at 3°C with deionised water and an aliquot was contained in a headspace. Deuterium labelled furan (internal standard) was added and the samples incubated at 35°C prior to sampling the headspace for GC/MS analysis.

Method performance (typical): The limits of detection and quantification were 0.07 µg/kg and 0.2 µg/kg respectively; the estimated uncertainty for a single determination over the concentration range 20-100 µg/kg furan was ±20% (expanded relative uncertainty with a coverage factor of 2). A summary of the method performance data from this survey can be found in Table 6.

7.3 ANALYTICAL QUALITY ASSURANCE

All analyses were performed by trained staff in a UKAS accredited laboratory operating an internal audit and review process. All the test methods were all validated in-house and accredited by UKAS (ISO 17025).

Each batch of samples (1-10 samples) included a procedural blank and an In-House Reference Material (IHRM) and / or spiked sample. Method performance was monitored by assessing the data from IHRMs and / or spiked reference materials in accordance with the rules governing Shewhart control charts[9] (this data is summarised in Table 6). The laboratory also participated in the FAPAS proficiency testing scheme available for acrylamide and furan (see Table 7). In addition, 10% of all samples were subjected to a repeat analysis and the repeat measurement was ±10% of the first measurement or the two measurements ±10% of the mean (see Table 8).

Table 6. Summary of data obtained from the analysis of IHRMs and spiked samples during this survey

(A) IHRM data

	Accepted value (µg/kg)		Measured values (µg/kg)			
	mean	Range[a]	n	mean	range	95%CI
Acrylamide (IHRM 1[b])	22	14-38	9	23	21-26	1.3
Acrylamide (IHRM 2[b])	42	37-48	11	43	39-45	1.3
Acrylamide (IHRM 3[b])	47	38-56	4	45	43-49	4.0

[a] upper and lower action limits; [b] dried cereal based materials

[7] Hamlet, C. G., & Sadd, P. A. (2004) Rapid, sensitive and selective analysis of acrylamide in cereal products using bromination and GC/MS/MS. *Czech Journal of Food Sciences*. 22, 290-293.

[8] Becalski, A., Forsyth, D., Casey, V., Lau, B.P.-Y., Pepper, K., Seaman, S. (2005), Development and validation of a headspace method for determination of furan in food. *Food Additives and Contaminants*, 22 (6), 535-540.

[9] BS 7785:1994, ISO 8258:1991, Shewhart control charts, British Standards Institution. Available at: http://www.bsonline.bsi-global.com/server/index.jsp

(B) Spiked recovery data

	nominal spike level (μg/kg)	Recovery (%)[a]			
		n	mean	range	95%CI
Acrylamide	500	26	102	93-113	2.1
Furan	15-30	8	94	81-111	7.4
	200	8	99	93-107	3.0

[a] the acceptable recovery range was 80-120%

Table 7. FAPAS z-scores (2009-2011)

Contaminant	Series	z-score		
		n	mean	range
Acrylamide	30	6	0.3	0-0.7
Furan	30	1	0.4	-

Table 8. Percentage of Samples Reanalysed

Contaminant	No. of samples for testing	No. of samples reanalysed	%
Acrylamide	250	26	10
Furan	92	9	10