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Total Diet Study – Acrylamide Investigation: Phase 1 analysis of all group samples

A report prepared for the Food Standards Agency

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1. **Executive Summary**

Acrylamide is a contaminant that is formed during food processing and was likely therefore to be present in some of the food categories comprising the TDS such as potato products, breakfast cereals and savoury snacks. A previous analysis of UK TDS samples for acrylamide was reported in 2005; the study analysed the main Groups only. This report provides the results from an analytical investigation of samples taken from Groups 1-28 of the 2014 TDS (Phase 1). The results from this study will assist the Agency in calculations of dietary exposure to acrylamide. Recommendations for a further analytical investigation of the Category samples comprising the main Groups of the 2014 TDS (Phase 2) are also given.

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Terms and abbreviations

| | |
|-------|--|
| AA | Acrylamide |
| AQA | Analytical Quality Assurance |
| ALARA | As Low As Reasonably Achievable |
| Asn | Asparagine |
| FERA | Food and Environment Research Agency |
| IV | EC Indicative Value |
| JECFA | Joint Expert Committee on Food Additives |
| PCV | Prescribed Concentration or Value |
| TDS | Total Diet Study |

Introduction

1.1 The Total Diet Study

The Total Diet Study (TDS) is an important part of the UK's surveillance programme for chemicals in food and has been carried out on a continuous annual basis since 1966. Results from the TDS are used to estimate dietary exposures of the general UK population to chemicals in food, such as nutrients, metals and contaminants, to identify changes or trends in exposure and make assessments on the safety and quality of the food supply. The key principle of a TDS is that it is representative of the whole diet. A TDS is different from many surveys as all foods are firstly prepared for consumption, rather than being analysed as sold, before being pooled into Groups for analyses. The design of the TDS typically involves the collection of categories of food which are then combined into Groups of similar foods for analysis. For this survey, the Groups were prepared from 138 categories of foods with each category comprising 24 samples (i.e. sample one from each of 24 local authorities selected by an appointed sampling contractor). The relative proportion of categories within each Group (i.e. the amount of each category making up the pooled Group sample) reflects its importance in the average UK household diet. For this survey, the relative proportions of each category comprising the Groups were derived from three previous years of food purchase data from the Family Food Survey (previously the National Food Survey). In previous TDS, approximately 120 categories were prepared for consumption and combined into Groups 1-20. For this survey, the number of categories was increased to 138 and eight additional Groups were created (Groups 21-28, see Table 1).

Table 1. Groups and categories comprising the 2014 TDS

| Group number | Group name | Category number | Category name | Proportions of each category (g) in the food Groups |
|--------------|-----------------------|-----------------|-----------------------------|---|
| 1 | Bread | 1 | White sliced bread | 394 |
| | | 2 | White unsliced bread | 50 |
| | | 3 | Brown bread | 63 |
| | | 4 | Wholemeal and granary bread | 210 |
| | | 5 | Other bread | 283 |
| 2 | Miscellaneous cereals | 6 | Flour | 76 |
| | | 7 | Buns, cakes and pastries | 187 |
| | | 8 | Savoury biscuits | 21 |
| | | 9 | Sweet biscuits | 104 |
| | | 10 | Chocolate biscuits | 59 |
| | | 11 | Breakfast cereals | 171 |
| | | 12 | Rice | 111 |
| | | 13 | Other cereal products | 61 |
| | | 14 | Pasta | 109 |
| | | 15 | Pizza | 101 |

Table 1 continued...

| | | | | |
|----|----------------------|-----|--|-----|
| 3 | Carcase meat | 16 | Beef | 543 |
| | | 17 | Lamb | 190 |
| | | 18 | Pork | 267 |
| 4 | Offals | 19 | Lambs liver | 203 |
| | | 20 | Pigs liver | 111 |
| | | 21 | Other liver | 151 |
| | | 22 | Kidney | 243 |
| | | 23 | Other offals (excluding kidney and liver) | 291 |
| 5 | Meat products | 24 | Uncooked bacon | 118 |
| | | 25 | Cooked ham and bacon | 72 |
| | | 26 | Corned meat | 14 |
| | | 27 | Other canned or cooked meats | 62 |
| | | 28 | Pork sausages | 101 |
| | | 29 | Beef sausages | 7 |
| | | 30 | Other sausages | 13 |
| | | 31 | Ready to eat meat products | 147 |
| | | 32 | Meat based ready meals | 274 |
| | | 33 | Meat based takeaways | 118 |
| | | 34 | Other meat products | 11 |
| 6 | Poultry | 35 | Burgers | 62 |
| | | 36 | Chicken (RAW) | 704 |
| | | 37 | Other poultry (RAW) | 72 |
| | | 38 | Cooked poultry | 125 |
| 7 | Fish | 39 | Poultry products | 99 |
| | | 40 | White fish | 147 |
| | | 41 | Fatty fish | 92 |
| | | 42 | Shellfish | 78 |
| | | 43 | Canned salmon | 18 |
| | | 44 | Other canned/ bottled fish | 162 |
| | | 45 | Fish based ready meals and fish products | 349 |
| 8 | Oils and fats | 46 | Takeaway fish based meals | 154 |
| | | 47 | Fat spreads or blended spreads | 163 |
| | | 48 | Reduced fat fat spreads or blended spreads | 250 |
| | | 49 | Low fat/light fat spreads or blended spreads | 86 |
| | | 50 | Vegetable oils | 428 |
| | | 51 | Lard | 19 |
| 9 | Eggs | 52 | Other fats | 54 |
| | | 53 | Eggs | 910 |
| 10 | Sugars and preserves | 54 | Egg products | 90 |
| | | 55 | Sugar | 347 |
| | | 56 | Jam and fruit curds | 56 |
| | | 57 | Marmalade | 31 |
| | | 58 | Syrup, Honey, Treacle, Maple Syrup | 41 |
| | | 59 | Jelly | 1 |
| | | 60 | Chocolate confectionery | 354 |
| 61 | Sugar confectionery | 170 | | |

Table 1 continued...

| | | | | |
|----|---|-----|-----------------------------------|-----|
| 11 | Green vegetables | 62 | Cabbage | 146 |
| | | 63 | Sprouts | 48 |
| | | 64 | Cauliflower | 251 |
| | | 65 | Lettuce and leafy salads | 262 |
| | | 66 | Peas | 151 |
| | | 67 | Green beans | 103 |
| | | 68 | Other fresh green vegetables | 40 |
| 12 | Potatoes | 69 | Fresh potatoes | 683 |
| | | 70 | Potato products | 317 |
| 13 | Other vegetables | 71 | Onions, leeks | 171 |
| | | 72 | Carrots | 156 |
| | | 73 | Turnips, swedes | 24 |
| | | 74 | Other fresh vegetables | 252 |
| | | 75 | Mushrooms | 55 |
| | | 76 | Tomatoes | 135 |
| | | 77 | Cucumbers | 64 |
| | | 78 | Dried pulses | 13 |
| | | 79 | Herbs, spices | 8 |
| | | 80 | Vegetable based ready meals | 108 |
| | | 81 | Dried soups | 15 |
| 14 | Canned or jarred vegetables | 85 | Canned, carton or jarred soups | 300 |
| | | 86 | Canned or jarred tomatoes | 184 |
| | | 87 | Canned or jarred peas | 64 |
| | | 88 | Canned or jarred beans | 387 |
| | | 89 | Other canned or jarred vegetables | 65 |
| 15 | Fresh fruit | 90 | Oranges | 63 |
| | | 91 | Other citrus fruits | 98 |
| | | 92 | Apples | 193 |
| | | 93 | Pears | 55 |
| | | 94 | Stone fruit | 74 |
| | | 95 | Bananas | 280 |
| | | 96 | Grapes | 70 |
| | | 97 | Other fresh fruit | 166 |
| 16 | Fruit products | 98 | Canned peaches, pears, pineapples | 34 |
| | | 99 | Other canned or frozen fruit | 53 |
| | | 100 | Dried fruit | 55 |
| | | 101 | Fruit juices and vegetable juices | 858 |
| 17 | Non-alcoholic Beverages (Excluding tap water and bottled water) | 102 | Tea | 458 |
| | | 103 | Takeaway Tea | 5 |
| | | 104 | Instant coffee | 247 |
| | | 105 | Ground coffee | 12 |
| | | 106 | Takeaway coffee | 10 |
| | | 107 | Branded food drinks | 4 |
| | | 108 | Cocoa, drinking chocolate | 10 |
| | | 109 | Concentrated soft drinks | 95 |
| | | 110 | Ready to drink soft drinks | 156 |
| | | 113 | Alternatives to milk | 3 |

Table 1 continued...

| | | | | |
|----|--------------------------------|-----|--|------|
| 18 | Milk | 114 | Whole (full fat) milk (cows) | 219 |
| | | 115 | Skimmed/Semi skimmed milks (cows) | 781 |
| 19 | Dairy products | 116 | Condensed milk or Evaporated Milk | 29 |
| | | 117 | Instant milk | 13 |
| | | 118 | Natural cheese | 175 |
| | | 119 | Processed cheese | 18 |
| | | 120 | Butter | 67 |
| | | 121 | Ice-cream | 243 |
| | | 122 | Yoghurt | 297 |
| | | 123 | Other milk products | 101 |
| | | 124 | Cream | 39 |
| | | 125 | Canned milk puddings | 17 |
| 20 | Nuts | 126 | Ground nuts including peanut butter | 470 |
| | | 127 | Tree nuts | 530 |
| 21 | Alcoholic drinks ^a | 128 | Beer | 580 |
| | | 129 | Cider | 101 |
| | | 130 | Wine | 259 |
| | | 131 | Alcopops and cocktails | 13 |
| | | 132 | Spirits | 48 |
| 22 | Meat substitutes ^b | 133 | Soy, mycoprotein or vegetable protein based meat substitutes | 1000 |
| 23 | Snacks ^a | 134 | Potato crisps and potato based snacks | 734 |
| | | 135 | Other snacks (not potato based) | 266 |
| 24 | Desserts ^a | 136 | Desserts (unfrozen) | 739 |
| | | 137 | Desserts (frozen but not ice cream) | 261 |
| 25 | Sandwiches ^b | 138 | Sandwiches | 1000 |
| 26 | Condiments ^a | 82 | Meat or yeast extracts | 30 |
| | | 83 | Spreads, dressings | 245 |
| | | 84 | Pickles, sauces | 726 |
| 27 | Tap water ^b | 111 | Tap water | 1000 |
| 28 | Bottled waters ^{b, c} | 112 | Bottled waters | 1000 |

^a New category; ^b New single category; ^c previously a category in the non-alcoholic beverages group

The purpose of this total diet study (TDS) was to calculate up to date background exposure to inorganic contaminants, acrylamide (AA) and mycotoxins from the whole diet.

1.2 Acrylamide

Acrylamide is a chemical substance that can form naturally when some foods are subjected to high temperatures during cooking (including home-cooking) and processing. Acrylamide is formed from the naturally occurring amino acid asparagine when foods are heated at temperatures greater than 120°C in the presence of sugars and other amino acids (Stadler and Studer 2016; Stadler 2005). Although AA does not occur in such foods subjected to lower temperatures and relatively short process times e.g. boiled potatoes (Ahn et al 2002), it has been found

in a wide range of home-cooked and processed foods, including potato crisps, French fries, bread, crispbreads and coffee.

The potential release of AA into potable water from the use of polyacrylamide based coagulants and flocculants for drinking water purification has also been considered although the amounts of residual AA (monomer) in these materials are very low and subject to controls.

Regular and prolonged exposure over a lifetime to foods containing AA has the potential to increase the risk of developing cancer. Experts, including the international Joint Food and Agriculture Organisation and the World Health Organisation Expert Committee on Food Additives (JECFA), have concluded that current global levels of dietary exposure to AA indicate a human health concern. In the UK, the Food Standards Agency (FSA) has concluded that exposure to AA should be as low as reasonably achievable (ALARA).

There are no statutory maximum levels for AA in foods although a maximum limit of 0.1 µg/l has been set for drinking water in the EU¹ (Council Directive 98/83/EC) and the UK (SI 3184, as amended; SI 2785). The latest European Commission Recommendation² on investigations into the levels of AA in food specifies 'indicative values' (IV) for AA for certain categories of food. While these IV are not intended as maximum limits, they are intended to initiate investigations by enforcement authorities into food business operators' understanding of AA and actions taken to mitigate its production.

An AA "toolbox" is available to industry to provide guidance on measures to reduce AA levels in various categories of foods³.

1.3 Project brief and lines of approach

Acrylamide is a contaminant that is formed primarily during the thermal processing of food and was likely therefore to be present in some of the categories comprising the 2014 TDS such as potato products, breakfast cereals and savoury snacks. The most recent analysis of UK TDS samples for AA was carried out using the 20 Group samples from the 2003 TDS (FSA 2005): no further analysis of individual foods (categories) comprising these Groups had been reported. The analytical investigation of the 2014 TDS samples was proposed in two phases: phase 1 (this report) comprised the analysis of all 28 Groups for AA using an accredited method of analysis; the analytical results were then used to inform the selection (together with other relevant information) of category samples for further investigation / analyses (phase 2). The results from this project would assist the Agency in calculations of dietary exposure to AA.

¹ Council Directive 98/83/EC of 3 November 1998 on the quality of water intended for human consumption, oj L 330, 32-54, 5.12.98

² Commission Recommendation of 8 November 2013 on investigations into the levels of acrylamide in food (2013/647/EU), oj L 301, 15-17, 12.11.13

³ Available at: <http://www.fooddrinkeurope.eu/publications/category/toolkits/>

2. Experimental

2.1 Sample collections and preparation

Homogeneous sub-samples (100 g), taken from Groups 1-28 of the 2014 TDS, were delivered to PAS in polystyrene containers packed in ice, directly from FERA (York). Sub-samples from Groups 27 and 28 (bottled/drinking waters), together with laboratory control water samples, were transferred to 500 ml amber glass bottles on receipt and transported immediately to a specialist laboratory accredited (UKAS/ISO17025) for the analysis of AA in drinking water. All other samples were stored at -18°C until required for analysis.

2.2 Analysis of acrylamide

2.2.1 Analysis of foods

Defrosted samples were stirred thoroughly with a glass rod prior to analysis. Acrylamide was determined as the brominated derivative, 2-bromopropenamide using GC/MS/MS, according to the method of Hamlet, Sadd & Liang (2008). 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).

2.2.2 Analysis of drinking waters

Samples were submitted to a specialist laboratory accredited (UKAS/ISO17025) for the analysis of AA in drinking waters. Acrylamide was measured by a direct aqueous injection procedure using LC/MS/MS. Quantification was based on an internal standardisation procedure. Method performance (typical): the limit of detection was 0.008 µg/l; the estimated value for the method uncertainty was ±15.3% at the PCV (0.1 µg/l).

2.2.3 Analytical quality assurance

Foods. The analysis of all food samples was performed by trained staff in a UKAS accredited laboratory operating an internal audit and review process. The test method for AA was validated in-house and accredited by UKAS (ISO 17025).

Each batch of samples (1-20 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 charts⁴ (this data is summarised in Table 3). The laboratory also participated in the FAPAS proficiency testing schemes available for AA and furan (see Table 5). In addition, 10% of all samples were subjected to a repeat analysis: the acceptance criteria for the duplicate was for the result to be within ±10% of the first measurement.

Data from all analytical quality assurance (AQA) test samples can be found in Tables 4-6 of the Appendices.

⁴ BS 7785:1994, ISO 8258:1991, Shewhart control charts, British Standards Institution. Available at: <http://www.bsonline.bsi-global.com/server/index.jsp>

3. Results and discussion

3.1 2014 TDS results

Table 1 shows the results in order of decreasing AA concentrations for each Group from the 2014 TDS. As expected, the highest concentrations of AA were found in the Snacks, Potatoes and Miscellaneous cereals Groups. While these Groups comprised foods items known to contain relatively high levels of AA⁵ (e.g. crisps and potato / cereal snacks, French fries, biscuits, breakfast cereals, etc), the contribution from other categories was less clear. For example, the ‘Fresh potato’ category accounted for nearly 70% of the composition of the Potatoes group (UK FSA, personal communication) and further comprised a significant proportion of food items labelled as ‘other fresh potatoes’ for which cooking details, and hence AA contributions, were unclear (e.g. frying, roasting, boiling).

Table 2. Amounts of acrylamide measured in the 2014 TDS Group samples

| Sample Code | Group No | Group description | Acrylamide (µg/kg) |
|-------------|----------|--|---------------------|
| 15C-02065 | 23 | Snacks | 360 ^{a,b} |
| 15C-02054 | 12 | Potatoes | 181 ^{a,b} |
| 15C-02044 | 2 | Miscellaneous Cereals | 65 ^{a,b} |
| 15C-02062 | 20 | Nuts | 25 ^b |
| 15C-02068 | 26 | Condiments | 22 ^b |
| 15C-02055 | 13 | Other Vegetables | 21 ^b |
| 15C-02052 | 1 | Sugars and Preserves | 20 ^b |
| 15C-02066 | 24 | Desserts | 20 ^b |
| 15C-02047 | 5 | Meat Products | 17 ^b |
| 15C-02046 | 4 | Offals | 16 ^b |
| 15C-02043 | 1 | Bread | 16 ^b |
| 15C-02064 | 22 | Meat substitutes (single category group) | 14 ^b |
| 15C-02056 | 14 | Canned Vegetables | 12 ^b |
| 15C-02067 | 25 | Sandwiches (single category group) | 12 ^b |
| 15C-02061 | 19 | Dairy Products | 9 ^b |
| 15C-02049 | 7 | Fish | 9 ^b |
| 15C-02053 | 11 | Green Vegetables | 8 ^b |
| 15C-02048 | 6 | Poultry | 7 ^b |
| 15C-02058 | 16 | Fruit Products | 7 ^b |
| 15C-02063 | 21 | Alcoholic drinks | 6 ^b |
| 15C-02059 | 17 | Non-alcoholic Beverages | 6 ^b |
| 15C-02057 | 15 | Fresh Fruit | 6 ^b |
| 15C-02060 | 18 | Milk | 6 ^b |
| 15C-02051 | 9 | Eggs | 3 ^b |
| 15C-02045 | 3 | Carcase Meat | 3 ^b |
| 15C-02050 | 8 | Oils and Fats | 1 ^b |
| 15C-02069 | 27 | Tap Water (single category group) | 0.008 ^c |
| 15C-02070 | 28 | Bottled Waters (single category group) | <0.008 ^c |

^a result is mean of duplicate analyses; ^b analysis by GC/MS/MS (LOD 0.5 µg/kg, LOQ 3 µg/kg); ^c analysis by LC/MS/MS (LOD 0.008 µg/kg)

⁵ Available at: <http://www.food.gov.uk/science/research/surveillance/food-surveys/food-survey-information-sheets-2014/acrylamide-and-furan>

EC Indicative values did not apply to the individual Groups as tested although they may apply to the Categories comprising each Group such as biscuits, bread, coffee and etc. Amounts of AA measured in the new single category Groups comprising tap and bottled waters were below the parametric value of 0.1 µg/l specified in the EU Drinking water standard (Council Directive 98/83/EC) and their equivalent UK Statutory instruments (SI 3184, as amended; SI 2785), and the WHO guideline (WHO 2011) of 0.5 µg/l for drinking water.

3.2 Comparison of results with previous TDS

In the 2003 TDS, approximately 120 categories were prepared for consumption and combined into Groups 1-20. For the 2014 TDS, the number of categories was increased to 138 and eight additional Groups were created (Groups 21-28, see Table 1) which included Alcoholic beverages, Bottled Waters, Condiments, Desserts, Meat Substitutes, Snacks, Sandwiches and Tap Water. Some of the food categories comprising the Groups also differed in their composition and cooking / preparation compared to the 2003 TDS. For example, the 2014 TDS potato Group included French fries whereas 2003 TDS potato Group did not (53 µg/kg) and this may account for the significantly higher AA measured in this study (181 µg/kg). While a like for like comparison of AA in food Groups between survey years should be treated with caution, some of the 2014 TDS Groups contained unexpected amounts of AA. These included Groups such as green vegetables, fresh fruit and milk which might not be expected to contain measurable amounts of AA. For example, the green vegetable Group comprised fresh and frozen green vegetables (see Table 1), which had been boiled in salted water where appropriate (Brereton 2016 personal communication). As far as the authors are aware, AA has not been detected in uncooked or boiled fresh green vegetables (FDA 2016a; 2016b; FSA 2005): in general, the domestic preparation of vegetables by boiling in water, e.g. potatoes, has not resulted in the generation of measurable amounts of AA (Ahn et al 2002; *ibid*); formation of AA occurs mainly via the Maillard reaction pathway at temperatures typically above 120°C (Stadler and Studer 2016). Similarly, detectable amounts of AA do not appear to have been reported for fresh fruits (FSA 2005; FDA 2016a). In a study of AA in feed for dairy cows and carry-over into milk, Pabst et al (2005) showed that a maximum concentration of AA of 0.2 µg/kg might be expected in the milk from cows fed diets containing AA at concentrations of up to 180 µg/kg. As far as the authors are aware, detectable amounts of AA have not been found in fresh milk (FDA 2016a; 2016b; FSA 2005).

3.2.1 Repeat analysis investigations

To confirm the unexpected results for green vegetables, fresh fruit and milk Groups, a series of additional analysis was undertaken at the request of the Agency on samples retained at PAS and additional sub-samples requested from FERA. These repeat analysis, carried out 12-15 mths after the analysis of the original samples, were all positive for AA but at reduced concentrations relative to the initial results (see Table 6). The reduction in AA was greatest for samples retained at PAS (n=6; mean 74%; range 45-85%) compared to samples obtained directly from FERA (n=3; mean = 37%; range 30-43%). Although all samples had been retained at -18°C between shipping and analyses, it is known that AA can reduce significantly in stored foods as a consequence of chemical / microbial degradation (Andrzejewski et al 2004; Brown et al 1980; EU 2000; Hoenicke & Gatermann 2005; Moens & Smats 1957; Stadler 2005; Zamora et al 2016; Zamora et al 2010). It is interesting to note that the apparent reduction in AA of the retained samples was greatest for those subject to the most

freeze / thaw cycles (see Table 6). Despite these complications, it was concluded that the repeat analyses and adherence to AQA protocols supported the original test results.

3.2.2 Investigation of milk

While the presence of AA in the 2014 TDS milk samples had been confirmed, its presence in fresh dairy milk was contrary to literature values (*ibid*). To confirm the expected absence of AA in milk, samples of whole, semi-skimmed and fat free milk were purchased from three retail outlets in Berkshire and Buckinghamshire and prepared in proportions according to Group 18 of the 2014 TDS. A blank sample was also prepared under the same conditions using deionised water instead of milk.

Amounts of AA measured in the “blank” preparation sample and the composite milk sample were below the method detection limit of 0.5 µg/kg. These results suggest that the amount of AA measured in the 2014 TDS milk sample from Group 18 was not indicative of AA in retail milk. Details of the preparation and analysis of these samples can be found in Table 7 of the Appendices.

3.2.3 Analytical quality assurance

Throughout the study all AQA protocols had been followed, control limits had not been exceeded and no issues were identified that could affect the validity of the test results. A summary of all AQA data is provided in Tables 4-6 of the Appendices.

4. Conclusions and recommendations

- Highest concentrations of AA were found in the Snacks, Potatoes and Miscellaneous cereals Groups.
 - These Groups comprised foods items known to contain relatively high levels of AA (e.g. crisps and potato / cereal snacks, French fries, biscuits, breakfast cereals)
- Lowest concentrations of AA were found in the Tap and bottled water Groups
 - AA was below EU / UK limits of 0.1 µg/l for drinking water

4.1 Recommendations for Phase 2

Measure amounts of AA in selected categories according to the following criteria:

- Groups with highest amounts of AA
- Dietary staples e.g. bread
- Categories comprising Groups with significant contributions to AA intakes
- Categories that may contain AA as a consequence of processing conditions e.g. temperature / time / moisture
- Categories with little or no data on AA

The final selection of samples for analysis may also require access to the 2014 TDS cooking methods e.g. food items in Groups: 12 (potatoes), 13 (other vegetables) etc. The proposed categories for analyses are given in Table 8 of the Appendices.

5. Appendices

Table 3. Summary of data obtained from the analysis of IHRMs and spiked samples during this survey

(A) Acrylamide IHRM data

| | Accepted value ($\mu\text{g}/\text{kg}$) | | | | Measured values ($\mu\text{g}/\text{kg}$) | | | |
|------------------------|--|------|--------------------|------|---|------|-----------|------|
| | n | mean | range ^a | SE | n | mean | range | SE |
| IHRM 2013 ^b | 12 | 73 | 68.5-77.5 | 0.43 | 9 | 73.4 | 70.0-76.6 | 0.84 |
| IHRM 2014 ^b | 12 | 70 | 60.8-79.8 | 0.91 | 11 | 67.2 | 62.1-73.0 | 1.20 |

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

(B) Acrylamide spike / recovery data

| Matrix | Nominal spike level ($\mu\text{g}/\text{kg}$) | n | Mean recovery (%) ^a |
|-------------------------|---|---|--------------------------------|
| Alcoholic beverages | 10 | 1 | 106 |
| Green vegetables | 10 | 1 | 83 |
| Milk | 10 | 3 | 87 |
| Non-alcoholic beverages | 10 | 1 | 93 |

^a acceptable recovery range 80-120%

Drinking waters. Samples of ultrapure laboratory water, non-spiked and spiked with AA, were submitted as blind samples to the specialist sub-contract laboratory to assess the validity of the method. The recoveries of AA from these blind samples are given in Table 4.

Table 4 Recoveries of acrylamide from blind water samples

| | Acrylamide added ($\mu\text{g}/\text{l}$) | Acrylamide measured ($\mu\text{g}/\text{kg}$) |
|-----------------|---|---|
| Ultrapure water | 0.0 | <0.008 |
| Ultrapure water | 2.0 | 2.46 |

Table 5. FAPAS acrylamide z-scores (Jan 2014 – Jan 2015)

| Series | n | z-score | |
|--------|---|---------|-------------|
| | | mean | range |
| 30 | 3 | 0.0 | -0.4 to 0.3 |

Table 6. 2014 TDS: Repeat analyses

(A) Samples retained at PAS

| Sample code | Sample description | Date of analysis | Freeze / thaw cycles | Acrylamide added ($\mu\text{g}/\text{kg}$) | Acrylamide recovered ($\mu\text{g}/\text{kg}$) | Spike recovery (%) | Comments | Reduction in acrylamide relative to original result (%) |
|-----------------|------------------------------------|------------------|----------------------|--|--|--------------------|--|---|
| 15C-02059 | Group 17 - Non-alcoholic Beverages | 23/02/2015 | 1 | - | 6 | - | Initial result | - |
| 15C-02059R | Group 17 - Non-alcoholic Beverages | 07/03/2016 | 2 | - | 3 | - | Repeat analysis after 13 mths | 45 |
| 15C-02059R SPK | Group 17 - Non-alcoholic Beverages | 07/03/2016 | 2 | 10 | 13 | 92.9 | Repeat analysis after 13 mths | - |
| 15C-02060 | Group 18 - Milk | 23/02/2015 | 1 | - | 6 | - | Initial result | - |
| 15C-02060R1 | Group 18 - Milk | 26/02/2016 | 2 | - | 1 | - | Repeat analysis after 12 mths | 85 |
| 15C-02060R2 | Group 18 - Milk | 26/02/2016 | 2 | - | 1 | - | Repeat analysis after 12 mths | 84 |
| 15C-02060R3 | Group 18 - Milk | 26/02/2016 | 2 | - | 1 | - | Repeat analysis after 12 mths | 80 |
| 15C-02060R4 | Group 18 - Milk | 07/03/2016 | 2 | - | 2 | - | Repeat analysis after 12 mths | 71 |
| 15C-02060R4 SPK | Group 18 - Milk | 07/03/2016 | 2 | 10 | 11 | 91.2 | Repeat analysis after 13 mths, spiked sample | - |
| 15C-02063 | Group 21 - Alcoholic drinks | 23/02/2015 | 1 | - | 6 | - | Initial result | - |
| 15C-02063R | Group 21 - Alcoholic drinks | 07/03/2016 | 2 | - | 1 | - | Repeat analysis after 13 mths | 78 |
| 15C-02063R SPK | Group 21 - Alcoholic drinks | 07/03/2016 | 2 | 10 | 12 | 106 | Repeat analysis after 13 mths, spiked sample | - |

Table 6 continued...

(B) Samples retained at FERA

| Sample code | Sample description | Date of analysis | Freeze / thaw cycles | Acrylamide added ($\mu\text{g}/\text{kg}$) | Acrylamide recovered ($\mu\text{g}/\text{kg}$) | Spike recovery (%) | Comments | Reduction in acrylamide relative to original result (%) |
|---------------|-----------------------------|------------------|----------------------|--|--|--------------------|---|---|
| 15C-02053 | Group 11 - Green Vegetables | 23/02/2015 | 1 | - | 8 | - | Initial result on sample ex FERA Jan 2015 | - |
| 16C-08221 | Group 11 - Green Vegetables | 20/05/2016 | 1 | - | 5 | - | Result on retained duplicate ex FERA May 2016 | 30 |
| 16C-08221 SPK | Group 11 - Green Vegetables | 07/03/2016 | 1 | 10 | 14 | 83 | Spiked sample | - |
| 15C-02057 | Group 15 - Fresh Fruit | 23/02/2015 | 1 | - | 6 | - | Initial result on sample ex FERA Jan 2015 | - |
| 16C-08222 | Group 15 - Fresh Fruit | 20/05/2016 | 1 | - | 3 | - | Result on retained duplicate ex FERA May 2016 | 37 |
| 16C-08222 | Group 15 - Fresh Fruit | 20/05/2016 | 1 | 10 | 13 | 94 | Spiked sample | - |
| 15C-02060 | Group 18 - Milk | 23/02/2015 | 1 | - | 6 | - | Initial result on sample ex FERA Jan 2015 | - |
| 16C-08223 | Group 18 - Milk | 20/05/2016 | 1 | - | 3 | - | Result on retained duplicate ex FERA May 2016 | 43 |
| 16C-08223 SPK | Group 18 - Milk | 20/05/2016 | 1 | 10 | 12 | 88 | Spiked sample | - |

Table 7. Preparation details and acrylamide results from the 2016 retail milk investigation

| Milk Category / Group | Sample Code | Description of unit purchased | BBE / Code | Date of purchase | Place of purchase | Proportion of each unit in category (l) | Total for category (l) | Proportion of category in Group (l) | Date of preparation | Date of analysis | Acrylamide measured (µg/kg) |
|-----------------------|-------------|---|------------------------------|------------------|-------------------|---|------------------------|-------------------------------------|---------------------|------------------|-----------------------------|
| Whole | 16C-05844 | Asda Fresh Whole Milk 1136 ml | 22 APR AY023 B6 07:21 | 11/04/2016 | High Wycombe | 0.833 | | | | | |
| | 16C-05845 | Sainsbury's Whole British Milk, 1.136 l | 19 APR EN006 A5 07:07 | 11/04/2016 | Maidenhead | 0.833 | 2.500 | 0.547 | 12/04/2016 | | |
| | 16C-05846 | Tesco Whole Milk, 1.13 litres | 18 APR EN006 A4 18:30 | 11/04/2016 | Maidenhead | 0.833 | | | | | |
| Semi-skimmed | 16C-05847 | Asda Fresh Milk, Semi Skimmed, 1136 ml | 22 APR EN006 BB2 07:24 | 11/04/2016 | High Wycombe | 0.833 | | | | | |
| | 16C-05848 | Sainsbury's Semi Skimmed British Milk, 1.136 L | 20 APR EN006 A6 08:39 | 11/04/2016 | Maidenhead | 0.833 | 2.500 | 0.276 | 12/04/2016 | | |
| | 16C-05849 | Tesco Semi Skimmed Milk, 1.13 l | 20 APR EN006 A6 14:18 | 11/04/2016 | Maidenhead | 0.833 | | | | | |
| Skimmed | 16C-05850 | Asda Fresh Milk, Skimmed, 1136 ml | 22 APR AY023 B6 03:11 | 11/04/2016 | High Wycombe | 0.833 | | | | | |
| | 16C-05851 | Sainsbury's Fat Free Skimmed British Milk, 1.136 l | 18 APR EN006 A4 11:14 | 11/04/2016 | Maidenhead | 0.833 | 2.500 | 1.677 | 12/04/2016 | | |
| | 16C-05852 | Tesco Skimmed Milk, 1.13 l | 19 APR EN006 A5 12:28 | 11/04/2016 | Maidenhead | 0.833 | | | | | |
| Group composite | 16C-05923 | Group composite blank (deionised water) prep 12/04/16 | | | | | | | 12/04/2016 | 15/04/2016 | <0.5 |
| | 16C-05924 | Group composite milk prep 12/04/16 | | | | | | | 12/04/2016 | 15/04/2016 | <0.5 |

Table 8. Proposed list of category samples for acrylamide analyses

| Group No / name to which category belongs | Category number | Category name | Proportions of each category (g) in the food Groups | Measure acrylamide? |
|---|---------------------------|--|---|---------------------|
| 1-Bread | 1 | White sliced bread | 394 | Y |
| | 2 | White unsliced bread | 50 | Y |
| | 3 | Brown bread | 63 | Y |
| | 4 | Wholemeal and granary bread | 210 | Y |
| | 5 | Other bread | 283 | Y |
| 2-Miscellaneous cereals | 6 | Flour | 76 | |
| | 7 | Buns, cakes and pastries | 187 | Y |
| | 8 | Savoury biscuits | 21 | Y |
| | 9 | Sweet biscuits | 104 | Y |
| | 10 | Chocolate biscuits | 59 | Y |
| | 11 | Breakfast cereals | 171 | Y |
| | 12 | Rice | 111 | |
| | 13 | Other cereal products | 61 | Y |
| | 14 | Pasta | 109 | |
| | 15 | Pizza | 101 | Y |
| 5-Meat products | 24 | Uncooked bacon | 118 | |
| | 25 | Cooked ham and bacon | 72 | |
| | 26 | Corned meat | 14 | |
| | 27 | Other canned or cooked meats | 62 | |
| | 28 | Pork sausages | 101 | |
| | 29 | Beef sausages | 7 | |
| | 30 | Other sausages | 13 | |
| | 31 | Ready to eat meat products | 147 | Y |
| | 32 | Meat based ready meals | 274 | Y |
| | 33 | Meat based takeaways | 118 | Y |
| | 34 | Other meat products | 11 | |
| 7-Fish | 35 | Burgers | 62 | |
| | 40 | White fish | 147 | |
| | 41 | Fatty fish | 92 | |
| | 42 | Shellfish | 78 | |
| | 43 | Canned salmon | 18 | |
| | 44 | Other canned/ bottled fish | 162 | |
| | 45 | Fish based ready meals and fish products | 349 | Y |
| 46 | Takeaway fish based meals | 154 | Y | |
| 10-Sugars and preserves | 55 | Sugar | 347 | |
| | 56 | Jam and fruit curds | 56 | |
| | 57 | Marmalade | 31 | |
| | 58 | Syrup, Honey, Treacle, Maple Syrup | 41 | |
| | 59 | Jelly | 1 | |
| | 60 | Chocolate confectionery | 354 | Y |
| 12-Potatoes | 61 | Sugar confectionery | 170 | |
| | 69 | Fresh potatoes | 683 | Y |
| | 70 | Potato products | 317 | Y |

Table 8 continued...

| | | | | |
|--|-----|---------------------------------------|-----|---|
| 13-Other vegetables | 71 | Onions, leeks | 171 | Y |
| | 72 | Carrots | 156 | |
| | 73 | Turnips, Swedes | 24 | Y |
| | 74 | Other fresh vegetables | 252 | |
| | 75 | Mushrooms | 55 | Y |
| | 76 | Tomatoes | 135 | |
| | 77 | Cucumbers | 64 | |
| | 78 | Dried pulses | 13 | |
| | 79 | Herbs, spices | 8 | |
| | 80 | Vegetable based ready meals | 108 | Y |
| | 81 | Dried soups | 15 | |
| 14-Canned or jarred vegetables | 85 | Canned, carton or jarred soups | 300 | |
| | 86 | Canned or jarred tomatoes | 184 | Y |
| | 87 | Canned or jarred peas | 64 | |
| | 88 | Canned or jarred beans | 387 | Y |
| | 89 | Other canned or jarred vegetables | 65 | |
| 16-Fruit products | 98 | Canned peaches, pears, pineapples | 34 | Y |
| | 99 | Other canned or frozen fruit | 53 | Y |
| | 100 | Dried fruit | 55 | Y |
| | 101 | Fruit juices and vegetable juices | 858 | |
| 17-Non-alcoholic Beverages (Excluding tap water and bottled water) | 102 | Tea | 458 | |
| | 103 | Takeaway Tea | 5 | |
| | 104 | Instant coffee | 247 | Y |
| | 105 | Ground coffee | 12 | Y |
| | 106 | Takeaway coffee | 10 | Y |
| | 107 | Branded food drinks | 4 | Y |
| | 108 | Cocoa, drinking chocolate | 10 | Y |
| | 109 | Concentrated soft drinks | 95 | |
| | 110 | Ready to drink soft drinks | 156 | |
| | 113 | Alternatives to milk | 3 | |
| 20-Nuts | 126 | Ground nuts including peanut butter | 470 | Y |
| | 127 | Tree nuts | 530 | Y |
| 23-Snacks | 134 | Potato crisps and potato based snacks | 734 | Y |
| | 135 | Other snacks (not potato based) | 266 | Y |
| 24-Desserts ^a | 136 | Desserts (unfrozen) | 739 | Y |
| | 137 | Desserts (frozen but not ice cream) | 261 | |
| 26-Condiments | 82 | Meat or yeast extracts | 30 | Y |
| | 83 | Spreads, dressings | 245 | Y |
| | 84 | Pickles, sauces | 726 | Y |

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