

FINAL REPORT

An investigation of perchlorate levels in fruit and vegetables consumed in the UK

Final Report to the UK Food Standards Agency covering perchlorate analysis

FS102077

Fera report Reference FD15/10

1/4/2016

Dr Sadat Nawaz, Fera Science Ltd



© Crown Copyright 2016

This report has been produced by Fera Science Ltd under a contract placed by the Food Standards Agency (the Agency). The views expressed herein are not necessarily those of the Agency. Fera Science Ltd warrants that all reasonable skill and care has been used in preparing this report. Notwithstanding this warranty, Fera Science Ltd shall not be under any liability for loss of profit, business, revenues or any special indirect or consequential damage of any nature whatsoever or loss of anticipated saving or for any increased costs sustained by the client or his or her servants or agents arising in any way whether directly or indirectly as a result of reliance on this report or of any error or defect in this report.

SUMMARY

A total of 342 food samples were tested and 77 samples were found to be contaminated with perchlorate. The highest frequency was in herbs (82%), followed by leafy vegetables & salad crops (50%). Among the countries with a significant number of samples, the highest frequency (%) of perchlorate was found in samples from Spain and Morocco.

The levels of perchlorate in the UK food supply were broadly similar to those found in a BfR study in 2013. One infant food sample and one Tenderheart cabbage sample exceeded the corresponding provisional enforcement levels.

It was not possible to establish a clear relationship between positive findings in the food samples and irrigation methods, fertilisers used or the growing environment. This ambiguity is partly due to limited number of samples with positive findings.

EXECUTIVE SUMMARY

1. Perchlorate is both a naturally occurring and man-made contaminant that can affect the thyroid gland function. An initial risk assessment by the European Food Safety Authority (EFSA) recommended further monitoring.
2. Although perchlorate is not a plant protection product, it has been tentatively regarded and assessed as such in terms of toxicology. This is because its entry pathway into the food-chain has not been clearly identified. Fertilisers, certain man-made industrial products, rainwater and certain mineral deposits are also considered potential sources.
3. This work was commissioned by the Food Standards Agency (FSA) to investigate perchlorate levels in UK food supply (fruit, vegetables, milk and infant foods). Where perchlorate was detected in food samples, water and soil samples associated with these food samples were also tested. Some of the samples collected were also tested to establish relative distribution of perchlorate in edible and inedible parts of the food.
4. This investigation was carried out over a two year period. Fruit, vegetable and milk samples were collected and tested during 2014 and 2015. In addition, infant food samples collected during 2013 for another FSA survey were tested during 2014 and 2015.
5. Perchlorate was detected in 43 of the 192 food samples tested during year 1 and in 34 of the 150 samples tested during year 2. In total, perchlorate was detected in 77 of the 342 food samples.
6. The incidence of perchlorate was more frequent in herbs, leafy vegetables and salad crops.
7. For six samples with peel, the concentration of perchlorate in the peel was higher compared with the edible inner part of the fruit/vegetable.
8. It was not possible to establish correlation between the perchlorate found and
 - (a) specific fertiliser regime
 - (b) growing environment or irrigation methods
 - (c) country of origin.
9. Seven of the soil samples and only one of the water samples, associated with positive food samples were found to contain detectable perchlorate.
10. The levels of perchlorate in the UK food supply were broadly similar to those found in a BfR study in 2013. Only two of the samples contained levels above the provisional enforcement levels.

GLOSSARY

Aq	Aqueous
BfR	Federal Institute for Risk Assessment
CV	Repeatability (Coefficient of Variation)
EC	European Commission
EFSA	European Food Safety Authority
EU	European Union
EURL SRM	European Union Reference Laboratory for Single Residue Methods
F&V	Fruit and Vegetables
Fera	Fera Science Ltd
FSA	Food Standards Agency
IC	Ion Chromatography
KOH	Potassium Hydroxide
LC-MS/MS	Liquid Chromatography – Tandem Mass Spectrometry
LIMS	Laboratory Information Management System
LoD	Limit of Detection
LoQ	Limit of Quantification
mg	Milligram
kg	Kilogram
min	Minute
mL	Millilitre
mM	Millimolar
PT	Proficiency Testing
QC	Quality Control
UK	United Kingdom
µL	Microlitre
US EPA	United States Environment Protection Agency
<	Less than

Contents

SUMMARY	3
EXECUTIVE SUMMARY	4
GLOSSARY	5
SECTION 1. AIMS AND OBJECTIVES OF THE INVESTIGATION	7
SECTION 2. EXPERIMENTAL PROCEDURES	9
2.1 Sample collection	9
2.2 Analytical procedure	9
2.3 Calibrants	9
2.4 Analytical instrumentation and instrument parameters	9
SECTION 3. RESULTS AND DISCUSSION	10
3.1 Method Validation	10
3.2 Sampling	10
3.3 Analytical Results	11
SECTION 4. CONTRACTOR RECOMMENDATIONS FOR FURTHER WORK	14
ACKNOWLEDGEMENTS	14
REFERENCES	15
ANNEX 1. RESULTS TABLES	16
ANNEX 2. SAMPLE PREPARATION PROTOCOL USED FROM PROJECT START TO OCTOBER 2014	32
ANNEX 3. SAMPLE PREPARATION PROTOCOL USED FROM NOVEMBER 2014 ONWARDS	37

SECTION 1. AIMS AND OBJECTIVES OF THE INVESTIGATION

Perchlorate can occur naturally in the environment, in deposits of nitrate and potash. It can also form naturally in atmosphere and therefore lead to contamination of soil and groundwater. Additionally, it can also arise from nitrate fertilisers, ammonium perchlorate used in rocket propellants, explosives, fireworks, flares and air bags. A US EPA study tested a wide variety of fertilisers and found that perchlorate was only detectable in materials derived from Chilean caliche (saltpetre, sodium nitrate) [1]. Perchlorate can also be formed during the degradation of sodium hypochlorite used to disinfect water [2]. Perchlorate can affect the thyroid gland by blocking the uptake of iodide from the blood into the thyroid gland [3].

A high incidence of perchlorate was found in food samples tested in Germany in 2013 [4]. In July 2013 the European Commission (EC) issued a statement regarding the presence of perchlorate in food and introduced levels (not maximum limits) of perchlorate for intra-Union trade [2]. In June 2015, the EC revised the levels of perchlorate for intra-Union trade and updated their previous statement [5]. EFSA has previously concluded that chronic dietary exposure to perchlorate is of potential concern.

The work described in this report was undertaken to investigate perchlorate levels in UK food supply (fruit, vegetables, milk and infant foods), as requested by EFSA. The investigation was spread over a two year period to enable collection of a wide range of samples. In total 280 food samples were to be collected, taking seasonality of the fruit and vegetable into account.

The samples were collected directly from farms (outdoors and protected) and retail outlets. Where different varieties of produce were available, these varieties were sampled.

Where possible, soil and water samples were also be collected. Soil and water samples were tested, if the associated food samples tested positive for perchlorate.

At the commissioning stage, it was agreed that both edible and non-edible parts of food will be tested to establish relative distribution of perchlorate. However, after review of the data generated during first few months of the project, FSA requested that only edible part of samples should be tested, as required by Commission Regulation No (EC) 1881/2006 " [6].

In total, 440 tests were expected over the two-year duration of this project, comprising:

- 280 food samples
- 80 samples of inedible parts of food
- 40 soil samples
- 40 water samples

In addition to sample collection, the sampler also collected relevant information to help identify possible sources of the perchlorate detected (e.g. country of origin, fertilisers used, and growing environment).

The analysis involved extraction of perchlorate using a method developed by the EU reference laboratory [7] and Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) determination using an in-house Ion Chromatography (IC) technique.

SECTION 2. EXPERIMENTAL PROCEDURES

2.1 Sample collection

Samples were collected by HallMark Ltd from farms and retailers in the UK and sent to Fera for analysis. Upon receipt, samples were logged and stored at +4°C prior to sample preparation. Fruit and vegetable samples received from project start to October 2014 were prepared following a protocol agreed with FSA where peel (or inedible portions) and flesh were separated; weights recorded and each given a unique Laboratory Information Management System (LIMS) code ready to be analysed separately (Sample preparation protocol Annex 2). Following review of the data, fruit and vegetable samples received from November 2014 onwards were prepared so that only the edible portion was to be analysed (Sample preparation protocol Annex 3). All fruit and vegetable samples were cryogenically milled to produce representative sub-samples and stored at -20°C prior to analysis.

2.2 Analytical procedure

The analytical procedure was adapted from a method published by the European Union Reference Laboratory for Single Residue Methods (EURL SRM) [7]. The method was validated in a single laboratory to meet the requirements of the EC guidelines [8]. This method involved extraction of highly polar compounds from samples using acidified methanol / water. Any perchlorate present in the extracts was determined using in-house IC LC-MS/MS method developed at Fera. Stable isotope labelled perchlorate was used to compensate for any minor variations in method performance.

2.3 Calibrants

The determination step involved the use of matrix matched, multi-level, bracketed calibration solutions.

2.4 Analytical instrumentation and instrument parameters

Perchlorate analysis was carried out on either a Metrohm Professional IC coupled to an Agilent 6460 mass spectrometer or a Dionex ICS3000 coupled to a Waters Quattro Ultima mass spectrometer. An AG19 (2 x 50 mm) guard column and AS19 (2 x 250 mm) analytical column was used for the separation, with an injection volume of 20 µL (Metrohm-Agilent) or 100 µL (Dionex-Waters) full loop fill injections. The column flow rate was 0.3 mL/min on both systems with a potassium hydroxide (KOH) (aqueous) gradient as detailed below:

Metrohm:		Dionex:	
0 min	20 mM KOH (aq)	0 min	20 mM KOH (aq)
7 min	20 mM KOH (aq)	7 min	20 mM KOH (aq)
8 min	60 mM KOH (aq)	8 min	60 mM KOH (aq)
20 min	60 mM KOH (aq)	18 min	60 mM KOH (aq)
20.1 min	60 mM KOH (aq)	18.1 min	60 mM KOH (aq)
25 min	20 mM KOH (aq)	22 min	20 mM KOH (aq)

Transitions collected (mass/charge ratio):

Perchlorate internal standard (¹⁸O₄): 107>89
Perchlorate: 99>83 (quantification), 99>67 (confirmation), 101>85 (confirmation)

SECTION 3. RESULTS AND DISCUSSION

3.1 Method Validation

All tables referred to are included in Annex 1.

The analytical procedure was adapted from a method published by the EURL [7]. The method was validated in a single laboratory to meet the requirements the EC guidelines [8]. This involved analysis of replicate samples, spiked at known levels of perchlorate. This was done at the start of the study to establish method within-lab repeatability. In addition, replicate samples, spiked at known perchlorate levels were analysed in each analytical run (batch) to establish method reproducibility. The method was successfully validated for food & water samples. The repeatability and reproducibility values were within the 20% criterion set in the guidelines. The method accuracy was also within the acceptable criterion (70-120%) for mean recovery data (see Table 1 & 2). The limit of detection (LoD) and limit of quantification (LoQ) achieved were 0.005 mg/kg and 0.01 mg/kg respectively.

Table 3 shows our proficiency results for EURL PT for analysis of milk. This PT was organised by the EURL SRM during April and May 2013.

It was not possible to successfully meet the acceptance validation criteria for the analysis of soil samples. A quality control (QC) regime using the standard addition technique was employed to generate “fit-for-purpose” data for soil samples.

3.2 Sampling

- 80 infant samples were selected by FSA from an on-going study.
- 262 fruit, vegetable, herb and milk samples were collected during 2014 and 2015.
- 58 of these samples were collected from farms. For these samples, irrigation practices, fertilisers used and growing conditions were recorded. For most of these samples, soil or growth media and water samples were collected.
- 2 retail potted herb samples were collected along with the pots and the compost associated with these samples was available for analysis.
- In total 59 soil / compost / growth media samples and 49 water samples were collected. Only 25 soil / compost and 22 water samples were associated with food samples that were found to contain perchlorate. Only these soil/compost and water samples were tested for perchlorate.
- 23 inedible parts of the food samples were tested for perchlorate.

In total, 412 samples were tested (80+262+25+22+23). The expected number of samples was approximately 440.

3.3 Analytical Results

- 80 samples of Infant foods were tested. One sample (carrot & tomato rice cakes) contained perchlorate at 0.57 mg/kg. This sample exceeds the provisional enforcement level, which is currently set at 0.02 mg/kg [5]. A vegetable infant food sample (sweet potatoes, broccoli and carrot) contained perchlorate at 0.018 mg/kg and a meat & vegetable infant food sample (lamb with winter vegetables) also contained perchlorate at 0.018 mg/kg. In addition, perchlorate was detected in four samples below 0.01 mg/kg (<LoQ) (Table 4a).
- 36 fruit samples were tested. One of the samples (blueberries) was found to contain perchlorate at 0.013 mg/kg. In addition, perchlorate was detected in another four samples below 0.01 mg/kg (<LoQ) (Table 4b).
- 27 samples of fruiting vegetables were tested and four samples (2 peppers and 2 cucumbers) were found to contain perchlorate between 0.012 and 0.034 mg/kg. In addition, perchlorate was detected in one sample below 0.01 mg/kg (Table 4c).
- 33 samples of herbs were tested and 23 samples were found to contain perchlorate between 0.010 and 0.96 mg/kg. In addition, perchlorate was detected in four samples below 0.01 mg/kg (<LoQ) (Table 4d).
- 40 samples of leafy vegetables and salad were tested and 11 samples were found to contain perchlorate between 0.011 and 0.18 mg/kg. In addition, perchlorate was detected in nine samples below 0.01 mg/kg (<LoQ) (Table 4e).
- 32 samples of root vegetables were tested and perchlorate was found in one celeriac sample at 0.011 mg/kg. In addition, perchlorate was detected in three samples below 0.01 mg/kg (<LoQ) (Table 4f).
- 55 samples of miscellaneous vegetables were tested and six samples were found to contain perchlorate between 0.011 and 0.23 mg/kg. The perchlorate level in a Tenderheart cabbage sample was at 0.23 mg/kg, which is just above the provisional enforcement level set at 0.2 mg/kg [5]. In addition, perchlorate was detected in three samples below 0.01 mg/kg (<LoQ) (Table 4g).
- 39 milk samples were tested and none of the samples contained detectable levels of perchlorate (Table 4h).

In total, 342 food samples were tested and 77 samples were contaminated with perchlorate. The highest incidence was associated with herbs (82%), followed by leafy vegetables & salad crops (50%). This is broadly similar to residues found during a study in Germany by BfR in 2013 [4].

The incidence of detectable perchlorate by country of origin was as follows:

- UK: 49 of the 204 samples collected
- Spain: 13 of the 24 samples collected
- Morocco 4 of the 6 samples collected

- Italy 3 of 8 the samples collected
- EU 2 of the 38 samples collected
- Chile 1 of the 1 sample collected
- China 1 of 1 the sample collected
- Kenya: 1 of the 5 samples collected
- The Netherlands: 1 of the 5 samples collected
- South Africa: 1 of the 6 samples collected
- Belgium: 1 of the 8 samples collected

Table 5 shows the results of separate analysis undertaken on edible and inedible parts of the food samples. 23 samples were analysed in this fashion. Only six samples contained perchlorate in one or both parts. For six samples where any perchlorate was detected, the concentrations in the inedible peel were higher compared with edible inner part of fruit and vegetables. However, during project review, FSA requested that only edible portions of the sample were tested *according to the requirements of Commission Regulation No (EC) 1881/2006* [6].

Table 6 shows the results for soil, compost and water samples tested (and corresponding food samples for comparison). There were 58 samples collected from UK farms. Two potted herbs samples were also collected from a retail outlet. Therefore, soil / compost and/or water samples were available for 60 of the 342 food samples collected. Only 25 of these food samples were found to contain detectable perchlorate above the LOD. The soil and/or water samples associated with these positive samples were tested.

- One of the 24 water samples tested was found to be contaminated with perchlorate at 0.041 mg/kg. This sample was collected from a bore hole next to the field.
- Seven of the 25 soil samples were found to be contaminated with perchlorate between approximately 0.005 and 0.039 mg/kg.

No clear relationship was found between positive findings in the food samples and irrigation methods, fertiliser use, the growing environment or country of origin. Some samples contained perchlorate and others with similar treatment/origin showed no detectable levels of perchlorate. There are a number of factors that make it difficult to determine relationship between residues and treatment/origin.

- Limited number of samples with measurable levels of perchlorate
- For values below the LoD, it is not clear when perchlorate is truly absent. In particular, there may be undetectable levels in the soil and water that become more concentrated in the crop
- The degree of uptake of perchlorate is likely to vary between different crop types

- The distribution of perchlorate within a plant is likely to be heterogeneous. The levels in edible parts of plant may vary from crop to crop.

SECTION 4. CONTRACTOR RECOMMENDATIONS FOR FURTHER WORK

If establishing the possible sources of perchlorate is a priority, a more targeted sampling approach is recommended as detailed below:

1. Further samples should be collected directly on-farm to enable additional sample information and data to be obtained with regard to irrigation methods, fertilisers and growing environment.
2. Further samples of infant food, herbs, salads and leafy vegetables should be collected to provide further information to help establish which sub-categories of these foods have the highest levels of perchlorate.
3. Additional samples of produce from Spain and North Africa should be collected and analysed. This will help broaden the range of produce sampled so far from these countries and provide further information and data on the levels of perchlorate in other crops.

ACKNOWLEDGEMENTS

The author is grateful to the Food Standards Agency for funding this work.

REFERENCES

[1] Survey of Fertilizers and Related Materials for Perchlorate (ClO_4^-); EPA/600/R-01/049, May 2001

[2] http://ec.europa.eu/food/food/chemicalsafety/contaminants/statement-perchlorate_en.pdf

Statement as regards the presence of perchlorate in food agreed by the Standing Committee of the Food Chain and Animal Health on 16 July 2013

[3] A. Srinivasan and T. Viraraghavan, Perchlorate: health effects and technologies for its removal from water resources, *Int J Environ Res Public Health* (2009) 6(4): 1418–42

[4] <http://www.bfr.bund.de/cm/349/health-assessment-of-perchlorate-residues-in-foods.pdf>

Health assessment of perchlorate residues in foods BfR Opinion No. 022/2013, 28 June 2013

[5]

http://ec.europa.eu/food/safety/docs/cs_contaminants_catalogue_perchlorate_statement_food_update_en.pdf

Statement as regards the presence of perchlorate in food endorsed by the Standing Committee on Plants, Animals, Food and Feed on 10 March 2015, updated on 23 June 2015

[6] <http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32006R1881&from=EN>

COMMISSION REGULATION (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs.

[7] http://www.eurl-pesticides.eu/library/docs/srm/meth_QuPPE.pdf

Quick Method for the Analysis of Residues of numerous Highly Polar Pesticides in Foods of Plant Origin involving Simultaneous Extraction with Methanol and LC-MS/MS Determination (QuPPE-Method) - Version 7 (Dec 2012); M. Anastassiades; D. I. Kolberg; D. Mack; Chr. Wildgrube; I. Sigalova; D. Roux; D. Fügler

[8] http://www.eurl-pesticides.eu/library/docs/allcrl/AqcGuidance_Sanco_2013_12571.pdf

Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed. SANCO/12571/2013

ANNEX 1. RESULTS TABLES

Table 1: Repeatability validation data for perchlorate

Job: 38055	Worksheet: 21221	Potatoes
spike level (mg/kg)	mean recovery n=5	CV
0.05	116%	2%
0.25	99%	4%

Job: 37604	Worksheet: 21203	Oats
spike level (mg/kg)	mean recovery n=5	CV
0.05	105%	7%
0.25	111%	7%

Job: 40043_c	Worksheet: 21312	Infant Food
spike level (mg/kg)	mean recovery n=5	CV
0.01	111%	6%
0.02	109%	7%
0.05	91%	2%

Job: 44447	Worksheet: 21353	Water
spike level (mg/kg)	mean recovery n=5	CV
0.01	103%	3%
0.02	104%	2%

Table 2: Routine QC data (% Recovery) from individual batches

Worksheet	Job	Matrix	Spike level		
			0.01 mg/kg	0.02 mg/kg	0.05 mg/kg
PRA14-00935	40848	Infant Food	112%	114%	107%
PRA14-00936	40847.2	Infant Food	98%	92%	94%
PRA14-00937	40843	Infant Food	115%	111%	102%
PRA14-00938	40846	Infant Food	106%	101%	93%
PRA14-02333	41782.2	Milk	92%	102%	118%
PRA14-02334	47181	F&V	101%	117%	121%
PRA14-02335	41784	F&V	98%	118%	120%
PRA14-02336	41783	F&V	116%	119%	115%
PRA14-02337	41786	F&V	94%	101%	99%
PRA14-02795	47004	Water	101%	102%	
PRA14-03618	42884	Milk	117%	102%	108%
PRA14-03621	42882	F&V	140%	131%	112%
PRA14-03622	42883	F&V	90%	110%	108%
PRA14-03623	42879	F&V	114%	117%	116%
PRA14-03624	42885	F&V	106%	118%	120%
PRA14-06342	47350	F&V	84%	95%	93%
PRA14-06343	47314	F&V	95%	102%	95%
PRA14-06344	47318	Milk		126%	125%
PRA15-00113	47485	Water	95%	90%	
PRA15-00124	47481	Infant Food	111%		101%
PRA15-00125	47482	Infant Food	113%	98%	96%
PRA15-00324	47645	Infant Food	125%	107%	107%
PRA15-00496	47797	F&V	133%	116%	111%
PRA15-00497	47794	F&V	119%	110%	101%
PRA15-02043	49294	F&V	107%	115%	105%
PRA15-02044	49102	F&V	109%	112%	105%
PRA15-02046	49265	Milk	119%	111%	104%
PRA15-05141	52196	F&V	120%	104%	
PRA15-05155	52197	F&V	88%	94%	
PRA15-05157	52335	Milk	111%	113%	
PRA15-05158	52252	F&V	103%	96%	
PRA15-05159	52264	F&V	110%	108%	
PRA15-05160	52295	F&V	121%	101%	
PRA15-05561	53465	F&V	96%	98%	
PRA15-05563	53467	Water	100%	105%	
average			104%	109%	108%
CV			12.8%	10.5%	10.0%

Table 3: Proficiency data for chlorate and perchlorate

Compound	PT round	Lab number	Sample	Result reported (mg/kg)	z-score
chlorate	EUPT SRM 09	53	Milk	0.152	-0.7
perchlorate	EUPT SRM 09	53	Milk	0.156	-0.6

Table 4 (parts 4a – 4h). Measured concentrations in Infant Food, Fruit, Vegetables and Milk (mg/kg)

~ indicates the measured value is between LoD (0.005 mg/kg) and LoQ (0.01 mg/kg)

Table 4a. Infant Food

Fera LIMS code	Sample ID	Description/ingredient	Perchlorate (mg/kg)	Country of Origin
S13-057159	1397457	Infant food; Apple, Strawberry, Banana	<0.005	United Kingdom
S13-057160	1397462	Infant food; Peaches, Rice	<0.005	Belgium
S13-057163	1397464	Infant food; Mediterranean Vegetable, Rice	<0.005	United Kingdom
S13-057167	1397495	Infant food; Apple, Bananas	<0.005	European Union
S13-057169	1397544	Infant food; Milk (powder)	<0.005	European Union
S13-057172	1397620	Infant food; Broccoli, Pears, Peas	<0.005	Spain
S13-057174	1397521	Infant food; Peaches, Bananas	<0.005	European Union
S13-057176	1397524	Infant food; Carrots, Cauliflower, Peas	<0.005	France
S13-057188	1398074	Infant food; Pears	<0.005	European Union
S13-057189	1398072	Infant food; Banana	<0.005	European Union
S13-057354	1397572	Infant food; Milk (ready to feed)	<0.005	European Union
S13-057358	1397575	Infant food; Apple, Blueberry	<0.005	France
S13-057363	1397571	Infant food; Plums, Pears, Parsnips, Swedes	<0.005	European Union
S13-057371	1397488	Infant food; Sweet potatoes, Broccoli, Carrots	0.018	European Union
S13-057656	1397504	Infant food; Sweet Potato, Carrot	<0.005	Italy
S13-057658	1397599	Infant food; Apples, Rice	<0.005	Belgium
S13-058737	1397437	Infant food; Carrot, Potato	<0.005	European Union
S13-058738	1397432	Infant food; Plum, Prune, Peach, Rice	<0.005	United Kingdom
S13-058745	1397588	Infant food; Mangoes	<0.005	European Union
S13-058747	1397568	Infant food; Sweet potato	<0.005	European Union
S13-058753	1397627	Infant food; Butternut Squash, Carrot, Apple	<0.005	Unknown
S13-058757	13987548	Infant food; Milk (powder)	<0.005	United Kingdom
S13-058760	1397501/02	Infant food; Cauliflower, Broccoli, Cheese	<0.005	United Kingdom
S13-058763	1397498	Infant food; Carrot, Potato, Lamb	<0.005	European Union
S13-058766	1397607	Infant food; Apple, Pear	<0.005	European Union
S13-058769	1397608	Infant food; Red Fruit, Apple	<0.005	Germany
S13-058815	1397375	Infant food; Apple, Banana	<0.005	European Union
S13-058821	1397387	Infant food; Banana, Peach, Strawberry	<0.005	European Union

Table 4a. Infant Food, continued

Fera LIMS code	Sample ID	Description/ingredient	Perchlorate (mg/kg)	Country of Origin
S13-058823	1397392	Infant food; Mango, Apple, Peach	<0.005	Belgium
S13-059369	1397419	Infant food; Banana, Rice	<0.005	Germany
S13-059377	1397389	Infant food; Fruit	<0.005	European Union
S13-059383	1397381	Infant food; Apple, Rice	<0.005	Belgium
S13-059384	1397383	Infant food; Rice	<0.005	European Union
S13-059387	1397360	Infant food; Fruit	<0.005	Italy
S13-059392	1398058	Infant food; Apple, Orange, Banana	<0.005	European Union
S13-059400	1397382	Infant food; Apple	~0.005	China
S13-059407	34216	Infant food; Rice	<0.005	United Kingdom
S13-059410	1398055	Infant food; Pears	<0.005	Germany
S13-059416	1397342	Infant food; Apple, Cranberry	<0.005	Germany
S13-059421	1397345	Infant food; Milk (ready to drink)	<0.005	Unknown
S13-059425	1397514	Infant food; Apple, Mango	<0.005	European Union
S13-059427	1397516	Infant food; Apple, Pear	<0.005	European Union
S13-059428	1397691	Infant food; Carrot, Sweetcorn, Rice	~0.008	United Kingdom
S13-059431	1397694	Infant food; Milk (ready to feed)	<0.005	European Union
S13-059435	1397558	Infant food; Plum, Banana, Strawberry	~0.006	United Kingdom
S13-059439	1397618	Infant food; Parsnip, Potato	<0.005	Germany
S13-059442	F605010	Infant food; Banana, Strawberry	~0.009	European Union
S13-059448	1397373	Infant food; Strawberry, Rice	<0.005	Belgium
S13-059451	1397372	Infant food; Apple, Pear	<0.005	Germany
S13-060112	1397509	Infant food; Banana, Pear, Mango	<0.005	Belgium
S13-060115	1397420	Infant food; Vegetable, Rice	<0.005	Germany
S13-060118	1397429	Infant food; Mangoes, Apples, Rice	<0.005	European Union
S13-060119	1397430	Infant food; Bananas, Blueberries, Rice	<0.005	European Union
S13-060129	1397596	Infant food; Fruit Cocktail, Rice	<0.005	European Union
S13-060145	1397619	Infant food; Mangoes, Bananas, Apples, Rice	<0.005	European Union
S13-060150	F605011	Infant food; Raspberry, Blueberry, Rice	<0.005	Belgium
S13-060206	1398172	Infant food; Fruit	<0.005	European Union
S13-060208	1398165	Infant food; Plum, Banana, Apricot, oats	<0.005	United Kingdom
S13-060210	1398167	Infant food; Rice	<0.005	Croatia
S13-061738	1397335	Infant food; Milk (ready to feed)	<0.005	Unknown
S13-057157	1397461	Infant food; Veggie chicken casserole	<0.005	European Union
S13-057162	1397463	Infant food; Pear & pineapple	<0.005	European Union
S13-057173	1397520	Infant food; Pumpkin & carrot with chicken	<0.005	European Union
S13-057177	1397525	Infant food; Parsnip, potato & turkey Casserole	<0.005	Germany

Table 4a. Infant Food, continued

Fera LIMS code	Sample ID	Description/ingredient	Perchlorate (mg/kg)	Country of Origin
S13-057370	1397487	Infant food; Vegetable risotto with white fish	<0.005	Austria
S13-057655	1397500	Infant food; Vegetable risotto with chicken	<0.005	European Union
S13-058751	1397552	Infant food; Growing up milk	<0.005	European Union
S13-058811	1397408	Infant food; Parsnip & potato	<0.005	European Union
S13-058820	1397385	Infant food; Strawberry, raspberry & banana	<0.005	Unknown
S13-059372	1397438	Infant food; Mango, pear & papaya	<0.005	European Union
S13-059397	1397361	Infant food; Apricot, apple & banana Yogurt	<0.005	European Union
S13-059437	1397556	Infant food; Lamb with winter vegetables	0.018	Italy
S13-059444	1398162	Infant food; Fruity yoghurt	<0.005	European Union
S13-059452	1397370	Infant food; Veggie lasagne with cheese	<0.005	European Union
S13-059454	1397384	Infant food; Sweet potato & lamb hotpot	<0.005	European Union
S13-060121	1397685	Infant food; Apple & pear with yogurt	<0.005	Germany
S13-060127	1397593	Infant food; Carrot & tomato rice cakes	0.57	Belgium
S13-060132	1397591	Infant food; Banana & peach dessert	<0.005	Germany
S13-060146	1397613	Infant food; Vegetable & beef casserole	<0.005	Austria
S13-060207	1398168	Infant food; Mango, banana & peach	<0.005	Italy

Table 4b. Fruit

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012894	1363679	Unwaxed Lemons (flesh)	<0.005	Italy
S14-023925	1363723	Tangerines (flesh)	~0.007	Spain
S14-041902	1614551	Clementines	<0.005	South Africa
S14-052491	1418267	Red Grapefruit	<0.005	South Africa
S14-012518	1363663	Cox's Apples (flesh)	<0.005	United Kingdom
S14-012898	1363681	Braeburn Apples (flesh)	<0.005	United Kingdom
S14-022421	1363678	Cantaloupe (flesh)	<0.005	Honduras
S14-023926	1363721	Sharon Fruit (flesh)	<0.005	South Africa
S14-023927	1363722	Black Grapes	<0.005	South Africa
S14-041234	1614562	Mango (flesh)	<0.005	Dominican Republic
S14-041900	1614553	Red Seedless Grapes	<0.005	Brazil
S14-012896	1363651	Blueberries	<0.005	United Kingdom
S14-032339	1363705	Blackberry	<0.005	United Kingdom
S14-041200	1364352	Strawberry	<0.005	United Kingdom
S14-041291	1614571	Strawberries	<0.005	United Kingdom
S14-041318	1363690	Strawberries	<0.005	United Kingdom
S14-052475	1614540	Blueberries	<0.005	Argentina
S15-006755	1613211	Lemon	<0.005	Italy
S15-006756	1613210	Galia Melon	<0.005	Honduras
S15-006795	1613205	Blueberries	0.013	Chile
S15-050612	1613194	Strawberries	<0.005	Spain
S15-050626	1613197	Lemon	<0.005	Spain
S15-051732	1613929	Strawberries	<0.005	United Kingdom
S15-090476	1613132	Strawberries	<0.005	United Kingdom
S15-090490	1613169	Strawberries	<0.005	United Kingdom
S15-091026	1614958	Blueberries	~0.008	The Netherlands
S15-091027	1614959	Tangerines	<0.005	South Africa
S15-091439	1614652	Strawberries	<0.005	United Kingdom
S15-091443	1397612	Blueberries	~0.008	United Kingdom
S15-093485	1613123	Strawberries	<0.005	United Kingdom
S15-093488	1613125	Apple	<0.005	United Kingdom
S15-100141	1563661	Blackberries	<0.005	United Kingdom
S15-100144	1563666	Apple	<0.005	United Kingdom
S15-100408	1614964	Strawberries	<0.005	United Kingdom
S15-100409	1614965	Oranges	~0.005	South Africa
S15-100414	1363662	Apple	<0.005	United Kingdom

Table 4c. Fruiting Vegetables

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012895	1363675	Sweet Pointed Peppers	0.017	Spain
S14-023615	1363671	Tomatoes	<0.005	United Kingdom
S14-023908	1363741	Vine Tomatoes	<0.005	United Kingdom
S14-024856	1363724	Peppers (flesh)	<0.005	Spain
S14-030029	1363688	Mini Plum Vine Tomatoes	<0.005	United Kingdom
S14-032110	1363686	Tomatoes	<0.005	United Kingdom
S14-041207	1614560	Pepper	0.012	United Kingdom
S14-041901	1614545	Baby Plum Tomatoes	<0.005	The Netherlands
S14-042108	1614566	Tomatoes	<0.005	United Kingdom
S14-052476	1614537	Tomatoes	<0.005	United Kingdom
S14-052478	1614538	Apple	<0.005	United Kingdom
S14-052485	1418271	Cucumber	0.013	Spain
S15-000400	1614645	Tomato	<0.005	United Kingdom
S15-006759	1613208	Tomato	<0.005	Morocco
S15-023700	1614971	Pineapple	<0.005	Unknown
S15-050622	1613189	Tomato	<0.005	Spain
S15-050627	1613195	Pineapple	<0.005	Costa Rica
S15-050629	1613196	Pineapple	<0.005	Unknown
S15-051736	1613927	Tomato	<0.005	United Kingdom
S15-051781	1613930	Pepper	<0.005	United Kingdom
S15-051785	1613919	Tomato	<0.005	United Kingdom
S15-091028	1614960	Pepper	<0.005	The Netherlands
S15-091141	1615109	Cucumber	0.034	United Kingdom
S15-091746	1563997	Cucumber	<0.005	United Kingdom
S15-091747	1563800	Tomato	<0.005	United Kingdom
S15-093486	1613175	Tomato	~0.006	United Kingdom
S15-100359	1613913	Tomato	<0.005	United Kingdom

Table 4d. Herbs

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012599	1004	Curly Leaf Parsley	0.020	United Kingdom
S14-012887	1448	Thyme	0.099	United Kingdom
S14-012899	1363674	Flat Leaf Parsley	0.011	Spain
S14-012900	1363677	Rosemary (leaves)	0.044	United Kingdom
S14-023918	1466	Curly Parsley	0.010	United Kingdom
S14-023928	1363719	Curly Parsley	0.010	Morocco
S14-024908	1363735	Mint	<0.005	United Kingdom
S14-030028	1363689	Parsley	0.033	United Kingdom
S14-032348	1363709	Parsley (fresh)	~0.005	United Kingdom
S14-041205	1614561	Curly Parsley	0.034	United Kingdom
S14-042106	1614547	Mint	0.098	Morocco
S14-042310	1363698	Lovage	0.024	United Kingdom
S14-042513	1363693	Mint	0.012	United Kingdom
S14-042515	1363699	Mint	<0.005	United Kingdom
S14-042517	1363697	Lovage	0.032	United Kingdom
S14-052477	1614541	Fresh Dill	0.11	Morocco
S14-052499	1438	Greek Basil	0.022	United Kingdom
S14-052538	1614656	Tarragon	~0.008	United Kingdom
S15-000425	1784	Parsley	<0.005	United Kingdom
S15-006718	1779	Basil	<0.005	United Kingdom
S15-006760	1613214	Parsley	0.96	Spain
S15-050615	1613192	Coriander	<0.005	Spain
S15-050620	1914	Parsley	0.029	United Kingdom
S15-051618	1916	Basil	0.018	United Kingdom
S15-090469	1614649	Thyme	0.072	United Kingdom
S15-090472	1614651	Tarragon	0.053	United Kingdom
S15-090477	1613134	Parsley	0.44	United Kingdom
S15-091435	1563804	Rosemary	~0.009	United Kingdom
S15-091748	163124	Mint	0.084	Spain
S15-100109	1563676	Rosemary	0.015	United Kingdom
S15-100112	1563797	Flat Leaf Parsley	<0.005	United Kingdom
S15-100115	1615135	Coriander	0.013	United Kingdom
S15-100116	1615134	Mint	~0.007	United Kingdom

Table 4e. Leafy Vegetables and Salad

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012488	1363661	Spinach	0.050	United Kingdom
S14-012871	1363655	Kale	0.013	United Kingdom
S14-012892	1397830	Spring Greens	<0.005	United Kingdom
S14-024887	1363725	Broccoli	~0.009	Spain
S14-025320	1363714	True Spinach	<0.005	United Kingdom
S14-025359	1363728	Spinach	<0.005	United Kingdom
S14-041409	1418103	Spreen Green	<0.005	United Kingdom
S14-052479	1614542	Broccoli	~0.006	Spain
S14-012611	1363669	Iceberg Lettuce	<0.005	Spain
S14-022609	1397832	Lettuce	~0.006	United Kingdom
S14-023917	1363737	Gem Lettuce	<0.005	United Kingdom
S14-023924	1363668	Gem Lettuce	~0.007	Spain
S14-041292	1614568	Wild Rocket	0.011	United Kingdom
S14-041319	1363683	Lettuce	<0.005	United Kingdom
S14-041899	1461	Wild Rocket	0.10	Italy
S14-052469	1613116	Gem Lettuce	<0.005	United Kingdom
S14-052493	1613119	Apollo Lettuce	~0.009	United Kingdom
S14-052520	1614662	Gem Lettuce	<0.005	United Kingdom
S14-052533	1418262	Crispy Salad	0.014	United Kingdom
S15-000383	1614638	Butterhead Lettuce	<0.005	United Kingdom
S15-000404	1614639	Kale	0.013	United Kingdom
S15-006688	1982	Spinach	0.050	Italy
S15-006689	1444	Broccoli	0.016	Spain
S15-006794	1613213	Iceberg Lettuce	<0.005	Spain
S15-050613	1921	Spinach	0.18	Spain
S15-050621	1905	Iceberg Lettuce	<0.005	Spain
S15-050783	1613120	Broccoli	~0.008	United Kingdom
S15-050890	1613924	Iceberg Lettuce	<0.005	United Kingdom
S15-051616	1918	Curly Leaf Lettuce	~0.005	United Kingdom
S15-051794	1613911	Broccoli	0.023	United Kingdom
S15-090489	1563863	Little Gem lettuce	<0.005	United Kingdom
S15-091031	1934	Spinach	0.019	United Kingdom
S15-091447	1563803	Gem Lettuce	<0.005	United Kingdom
S15-091448	1563810	Iceberg Lettuce	<0.005	United Kingdom
S15-091745	1563998	Iceberg Lettuce	<0.005	United Kingdom
S15-100320	1615150	Iceberg Lettuce	<0.005	United Kingdom
S15-100411	1941	Spinach	~0.007	United Kingdom
S15-100413	1614968	Broccoli	~0.008	Kenya
S15-100415	1614932	Iceberg Lettuce	<0.005	United Kingdom
S15-100498	1903	Iceberg Lettuce	<0.005	United Kingdom

Table 4f. Root Vegetables

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012515	1363650	Beetroot (flesh)	<0.005	United Kingdom
S14-012532	1363670	Sweet Potato (flesh)	<0.005	United States
S14-032376	1363701	New Potatoes (flesh)	<0.005	United Kingdom
S14-025354	1363715	Carrot (flesh)	<0.005	United Kingdom
S14-024883	1363720	Salad Potatoes	<0.005	Israel
S14-023907	1363729	Desiree potatoes (flesh)	<0.005	United Kingdom
S14-025284	1363733	Celeriac (flesh)	0.011	United Kingdom
S14-023916	1363736	Parsnips (flesh)	<0.005	United Kingdom
S14-012529	1398173	Carrots (flesh)	~0.006	United Kingdom
S14-052492	1418264	Carrots	<0.005	United Kingdom
S14-052486	1418269	Carrot	<0.005	Unknown
S14-052480	1614539	Sweet Potatoes	<0.005	United States
S14-041904	1614552	Celeriac	<0.005	The Netherlands
S14-041247	1614557	Baking Potatoes (flesh)	<0.005	United Kingdom
S14-052530	1614653	Potatoes	<0.005	United Kingdom
S14-052535	1614668	White Potatoes	<0.005	United Kingdom
S15-000403	1614644	Swede	<0.005	United Kingdom
S15-000428	1614648	Carrot	<0.005	United Kingdom
S15-000431	1614650	Parsnip	~0.005	United Kingdom
S15-009526	1613206	Potatoes	<0.005	United States
S15-023699	1614970	Beetroot	<0.005	United Kingdom
S15-050614	1613193	Carrot	<0.005	United Kingdom
S15-050624	1613180	Beetroot	<0.005	Spain
S15-050887	1613922	Parsnip	<0.005	United Kingdom
S15-051786	1613921	Celeriac	~0.006	United Kingdom
S15-091024	1613135	Carrot	<0.005	United Kingdom
S15-091030	1613129	Potatoes	<0.005	Spain
S15-091145	1615105	Parsnip	<0.005	United Kingdom
S15-100146	1563665	Potatoes	<0.005	United Kingdom
S15-100309	1615148	Carrot	<0.005	United Kingdom
S15-100321	1615151	Carrot	<0.005	United Kingdom
S15-100412	1614967	Parsnip	<0.005	United Kingdom

Table 4g. Miscellaneous Vegetables

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012403	1363646	White Cabbage (loose)	<0.005	United Kingdom
S14-012613	1363676	Tenderheart Cabbage	0.23	Spain
S14-012616	1363665	Garden Peas	<0.005	United Kingdom
S14-012617	1363648	Broad Beans	<0.005	United Kingdom
S14-012890	1363666	Cauliflower	<0.005	United Kingdom
S14-012897	1363680	Runner Beans	<0.005	Kenya
S14-023909	1363739	Cabbage	<0.005	United Kingdom
S14-024884	1363726	Sugar Snap Peas	<0.005	Guatemala
S14-025306	1363713	Cauliflower (flesh)	<0.005	United Kingdom
S14-030023	1363710	Hispie Cabbage	<0.005	United Kingdom
S14-032347	1363707	Broad Beans (fresh)	<0.005	United Kingdom
S14-041245	1614558	Brussels Sprouts	<0.005	Morocco
S14-041246	1614559	Pod Peas	<0.005	United Kingdom
S14-041305	1614569	Pea Shoots	0.015	United Kingdom
S14-041411	1363711	French Beans	0.015	United Kingdom
S14-041634	1614564	Peas (in Pod)	<0.005	United Kingdom
S14-041642	1363731	Broad Beans	<0.005	United Kingdom
S14-041895	1363685	Runner Beans	<0.005	United Kingdom
S14-041977	1614546	Sugar Snap Peas	<0.005	Namibia
S14-042105	1614567	Runner Beans	<0.005	United Kingdom
S14-052470	1613118	Cauliflower	<0.005	United Kingdom
S14-052484	1418273	Green Beans	0.011	Morocco
S14-052488	1613115	Runner Beans	0.041	United Kingdom
S14-052496	1614549	French Beans	<0.005	United Kingdom
S14-052528	1614661	Tenderheart Cabbage	<0.005	United Kingdom
S14-052531	1614654	Cauliflower	<0.005	United Kingdom
S14-052534	1614667	Pointed Cabbage	~0.005	United Kingdom
S14-052536	1614671	Pointed Cabbage	<0.005	United Kingdom
S15-000379	1614633	Brussel Sprouts	<0.005	United Kingdom
S15-000396	1614640	Cauliflower	~0.006	United Kingdom
S15-000397	1614646	Pak Choi	0.057	United Kingdom
S15-000402	1614543	Savoy Cabbage	<0.005	United Kingdom
S15-006758	1613212	Fine Beans	<0.005	Kenya
S15-009524	1613207	Peas	<0.005	United Kingdom
S15-009525	1613200	Broad Beans (frozen)	<0.005	United Kingdom
S15-050623	1613188	Cabbage	~0.007	Spain
S15-050625	1613190	Peas	<0.005	United Kingdom
S15-050628	1613198	Dwarf Bean	<0.005	Egypt
S15-050630	1613186	Beansprouts	<0.005	United Kingdom
S15-051617	1613178	Broad Beans (frozen)	<0.005	United Kingdom
S15-051735	1613925	Red Cabbage	<0.005	United Kingdom
S15-051796	1613912	Savoy Cabbage	<0.005	United Kingdom
S15-090485	1613131	Broad Beans	<0.005	United Kingdom
S15-090491	1613170	Peas	<0.005	United Kingdom
S15-091025	1614957	Cauliflower	<0.005	The Netherlands
S15-091029	1613130	Fine Beans	<0.005	Egypt
S15-091437	1563808	Broad Beans	<0.005	United Kingdom
S15-091438	1563807	Peas	<0.005	United Kingdom

Table 4g. Miscellaneous Vegetables, continued

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S15-091743	1615106	Peas	<0.005	United Kingdom
S15-093461	1613174	Dwarf French Beans	<0.005	United Kingdom
S15-093464	1613173	Runner Beans	<0.005	United Kingdom
S15-100365	1613914	Brussel Sprouts	<0.005	United Kingdom
S15-100416	1614931	Bean Sprouts	<0.005	United Kingdom
S15-100474	1614930	Peas	<0.005	Kenya
S15-100475	1614929	Runner beans	<0.005	Kenya

Table 4h. Milk

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S14-012503	1363642	Semi-Skimmed Milk	<0.005	United Kingdom
S14-012489	1363643	Semi-Skimmed Milk	<0.005	United Kingdom
S14-012872	1363644	Milk	<0.005	United Kingdom
S14-012514	1363649	Organic Whole Milk	<0.005	United Kingdom
S14-012400	1363657	Semi-Skimmed Milk	<0.005	United Kingdom
S14-012402	1363659	Semi-Skimmed Milk	<0.005	United Kingdom
S14-012517	1363664	Semi-Skimmed Milk	<0.005	United Kingdom
S14-023347	1363667	Semi-Skimmed Milk	<0.005	United Kingdom
S14-023346	1363672	Whole Milk	<0.005	United Kingdom
S14-041290	1363682	Full Cream milk	<0.005	United Kingdom
S14-041320	1363691	Milk	<0.005	United Kingdom
S14-032375	1363700	Whole Milk	<0.005	United Kingdom
S14-041412	1363712	Whole Milk	<0.005	United Kingdom
S14-025318	1363727	Milk	<0.005	United Kingdom
S14-025317	1363730	Milk	<0.005	United Kingdom
S14-052498	1364353	Pasteurised Whole Organic Milk	<0.005	United Kingdom
S14-023623	1418105	Semi-Skimmed Milk	<0.005	United Kingdom
S14-042104	1614563	Milk	<0.005	United Kingdom
S14-042103	1614565	Milk	<0.005	United Kingdom
S14-052532	1614655	Milk	<0.005	United Kingdom
S14-052541	1614658	Whole Milk	<0.005	United Kingdom
S14-052521	1614663	Fresh Pasteurised Milk	<0.005	United Kingdom
S15-000380	1614634	Full Fat Milk	<0.005	United Kingdom
S15-050784	1613122	Semi-skimmed milk	<0.005	United Kingdom
S15-050889	1613923	Milk	<0.005	United Kingdom
S15-051797	1613177	Semi skimmed Milk	<0.005	United Kingdom
S15-090475	1398175	Milk	<0.005	United Kingdom
S15-090478	1613133	Whole Milk	<0.005	United Kingdom
S15-090492	1613171	Milk	<0.005	United Kingdom
S15-091144	1615104	Semi skimmed Milk	<0.005	United Kingdom
S15-091436	1563805	Milk	<0.005	United Kingdom
S15-091442	1398174	Milk	<0.005	United Kingdom
S15-093487	1613176	Whole Milk	<0.005	United Kingdom

Table 4h. Milk, continued

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)	Country of Origin
S15-093489	1613128	Semi Skimmed Milk	<0.005	United Kingdom
S15-100117	1563843	Semi Skimmed Milk	<0.005	United Kingdom
S15-100145	1563667	Milk	<0.005	United Kingdom
S15-100319	1615149	Milk	<0.005	United Kingdom
S15-100410	1614966	Milk	<0.005	United Kingdom
S15-100764	1614928	Milk	<0.005	United Kingdom

Table 5. Measured concentrations in edible and inedible parts of food

~ indicates the measured value is between LOD (0.005 mg/kg) and LOQ (0.01 mg/kg)

Data for inedible parts highlighted in yellow

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)
S14-012515	1363650	Beetroot (flesh)	<0.005
S14-023630		Beetroot (peel)	<0.005
S14-012518	1363663	Cox's Apples (flesh)	<0.005
S14-022423		Cox's Apples (peel)	<0.005
S14-012890	1363666	Cauliflower (heart)	<0.005
S14-022425		Cauliflower (leaves)	<0.005
S14-012532	1363670	Baking Sweet Potato (flesh)	<0.005
S14-022426		Baking Sweet Potato (peel)	<0.005
S14-022421	1363678	Cantaloupe (flesh)	<0.005
S14-022422		Cantaloupe (peel)	<0.005
S14-012894	1363679	Unwaxed Lemons (flesh)	<0.005
S14-023629		Unwaxed Lemons (peel)	<0.005
S14-012898	1363681	Braeburn Apples (flesh)	<0.005
S14-022424		Braeburn Apples (peel)	<0.005
S14-032376	1363701	New Potatoes (flesh)	<0.005
S14-032377		New Potatoes (peel)	<0.005
S14-025306	1363713	Cauliflower (flesh)	<0.005
S14-025308		Cauliflower (leaves)	<0.005
S14-025354	1363715	Carrot (flesh)	<0.005
S14-025355		Carrot (peel)	~0.005
S14-023926	1363721	Sharon Fruit (flesh)	<0.005
S14-024818		Sharon Fruit (peel)	~0.005
S14-023925	1363723	Tangerine (flesh)	~0.007
S14-024815		Tangerine (peel)	~0.008
S14-024856	1363724	Pepper (flesh)	<0.005
S14-024857		Pepper (seeds)	<0.005
S14-023907	1363729	Desiree Potato (flesh)	<0.005
S14-024817		Desiree Potato (peel)	<0.005
S14-041642	1363731	Broad Beans (beans)	<0.005
S14-041637		Broad Beans (pods)	<0.005
S14-025284	1363733	Celeriac (flesh)	0.011
S14-025285		Celeriac (peel)	0.042
S14-023916	1363736	Parsnips (flesh)	<0.005
S14-024814		Parsnips (peel)	<0.005
S14-012529	1398173	Carrots (flesh)	~0.006
S14-023628		Carrots (peel)	~0.008

Table 5. Measured concentrations in edible and inedible parts of food, continued

Fera LIMS code	Sample ID	Description	Perchlorate (mg/kg)
S14-041902	1614551	Clementine (flesh)	<0.005
S14-041903		Clementine (peel)	0.047
S14-041904	1614552	Celeriac	<0.005
S14-041905		Celeriac (peel)	<0.005
S14-041247	1614557	Baking potatoes (flesh)	<0.005
S14-041248		Potatoes (peel)	<0.005
S14-041234	1614562	Mango (flesh)	<0.005
S14-041244		Mango (peel)	<0.005
S14-041634	1614564	Fresh peas in a pod (peas)	<0.005
S14-041635		Fresh peas in pods (pods)	<0.005

Table 6. Measured concentrations in Soil and Water samples (& associated food samples)

~ ' indicates the measured value is between LOD (0.005 mg/kg) and LOQ (0.01 mg/kg)

Fera LIMS number	Sample ID	Sample Description	Perchlorate (mg/kg)	Growing environment
S14-012599	1004	Curly leaf parsley	0.020	Potted herbs
S14-012600		Soil	<0.005	
S14-052499	1438	Greek Basil	0.022	Glasshouse
S14-052513		Water	<0.005	
S14-052500		Soil	~0.005	
S14-012887	1448	Thyme	0.099	Glasshouse
S14-012889		Water	<0.005	
S14-012888		Soil	0.016	
S14-023918	1466	Curly parsley	0.010	Glasshouse - cool area
S14-023920		Water	<0.005	
S14-023921		Water	<0.005	
S14-023922		Water	<0.005	
S14-023923		Water	<0.005	
S14-023919		Soil	<0.005	
S14-042513	1363693	Mint	0.012	Base dressed soil
S14-042514		Soil	<0.005	
S14-042109		Water	<0.005	
S14-042517	1363697	Lovage	0.032	Base dressed soil
S14-042518		Soil	<0.005	
S14-042109		Water	<0.005	
S14-042310	1363698	Lovage	0.024	Base dressed soil
S14-042311		Soil	<0.005	
S14-042109		Water	<0.005	
S14-025354	1363715	Carrot (flesh)	<0.005	Field
S14-025355		Carrot (peel)	~0.005	
S14-025357		Water	<0.005	
S14-025358		Water	<0.005	
S14-025356		Soil	<0.005	

Table 6. Measured concentrations in Soil and Water samples (& associated food samples), continued

Fera LIMS number	Sample ID	Sample Description	Perchlorate (mg/kg)	Growing environment
S14-025284	1363733	Celeriac (flesh)	0.011	Field
S14-025285		Celeriac (peel)	0.042	
S14-025286		Soil	<0.005	
		no water collected		
S14-022609	1397832	Lettuce	~0.006	Tunnel/Greenhouse
S14-022611		Water	<0.005	
S14-022610		Soil	<0.005	
S14-012529	1398173	Carrots (flesh)	~0.006	Field
S14-023628		Carrots (peel)	~0.008	
S14-012531		Water	<0.005	
S14-012530		Soil	0.023	
S14-052488	1613115	Runner Beans	0.041	Field
S14-052490		Water	0.041	
S14-052489		Soil	0.010	
S14-052493	1613119	Apollo Lettuce	~0.009	Cloche
S14-052495		Water	<0.005	
S14-052494		Soil	~0.008	
S14-041292	1614568	Wild Rocket	0.011	Field (uncovered)
S14-041294		Water	<0.005	
S14-041293		Soil	<0.005	
S14-041305	1614569	Pea Shoots	0.015	Field (uncovered)
S14-041312		Water	<0.005	
S14-041309		Soil	<0.005	
S14-052538	1614656	Tarragon	~0.008	Poly tunnel
S14-052540		Water	<0.005	
S14-052539		Soil	~0.005	
S15-051618	1916	Basil	0.018	Potted Herbs
S15-051619		Soil	<0.005	
S15-100109	1563676	Rosemary	0.015	Field
S15-100110		Water	<0.005	
S15-100111		Soil	<0.005	
S15-051794	1613911	Broccoli	0.023	Field
		No water collected		
S15-051795		Soil	<0.005	
S15-000404	1614639	kale	0.013	Field
		No water collected		
S15-000405		Soil	<0.005	
S15-000397	1614646	Pak Choi	0.057	Greenhouse
S15-000399		Water	<0.005	
S15-000398		Soil	<0.005	
S15-000428	1614648	Carrot	Trace	Field
S15-000429		Water	<0.005	
S15-000430		Soil	<0.005	

Table 6. Measured concentrations in Soil and Water samples (& associated food samples), continued

Fera LIMS number	Unique Reference	Sample Description	Perchlorate (mg/kg)	Growing environment
S15-090469	1614649	Thyme	0.072	Polytunnel
S15-090471		Water	<0.005	
S15-090470		Soil	<0.005	
S15-090472	1614651	Tarragon	0.053	Polytunnel
S15-090474		Water	<0.005	
S15-090473		Soil	<0.005	
S15-091141	1615109	Cucumber	0.034	Glasshouse
S15-091143		Water	<0.005	
S15-091142		Soil	0.039	

ANNEX 2. SAMPLE PREPARATION PROTOCOL USED FROM PROJECT START TO OCTOBER 2014

Summary

Sample size of <400g & <4 units is not acceptable.

HallMark will send each sample with a unique sample number. The HallMark number will be used to label related samples that represent same source (i.e. vegetable samples, soil sample from the field where it was grown and sample of the water used to irrigate the field).

Record the HallMark sample number as the sample ID on LIMS. Label the packaging clearly with the LIMS number and store for future reference.

All samples are to be sub-sampled for perchlorate and metals analysis.

Samples are to be treated individually and prepared one at a time by one member of staff to avoid any sample mix up.

Samples should be rinsed with deionized water if the samples are contaminated with soil.

Each type of sample will require specific sample preparation. In essence the edible part of the sample will be separated from rest before analysis. In addition to the edible part, sample peeling will be analyzed separately. Out of the inedible parts of the sample, only peeling will be analyzed and all other inedible parts will be discarded.

All samples that may be peeled by some consumers will be peeled. For the samples that are peeled, the edible part and the peelings will be logged individually into LIMS. Reference is to be made to the customer sample ID (HallMark number). Both parts of the sample are to be sub-sampled for perchlorate and metal analysis.

All equipment used for sample preparation and extraction should be cleaned using deionized water. Tap water should be avoided throughout.

All sub-samples of edible portion of the samples taken for perchlorate analysis will be homogenized using cryogenically milling. No milling is required for liquid samples, soil samples or peelings. These will be homogenized using appropriate equipment depending on the type and size of the sample.

The examples below illustrate some possible scenarios: If a sample does not fit one of the criteria below, please consult Fera project manager.

Soil

- Login the sample (sample type: soil, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- Soil samples are to be sub-sampled for perchlorate and metals analysis.
- The soil sample for perchlorate is to be mixed thoroughly and stored in a freezer.
- The soil sample to be analyzed if the corresponding food sample is found to contain residues of perchlorate.

Water

- Login the sample (sample type: water, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- Water samples are to be sub-sampled for perchlorate and metals analysis.
- Transfer a sub-sample for metal analysis into an acid cleaned pot.
- Store the remainder for perchlorate analysis into clear plastic beakers and store in a freezer.
- The water sample to be analyzed if the corresponding food sample is found to contain residues of perchlorate.

Milk

- Login the sample and create two aliquots (metals & perchlorate).
- Transfer a sub-sample for metal analysis into an acid cleaned pot.
- Store the remainder for perchlorate analysis into 3 clear plastic beakers and store in a freezer.

Cabbage, lettuce

- Rinse with deionized water if the sample is contaminated with soil.
- Login the sample and create two aliquots (metals & perchlorate).
- Chop each head in half using a clean stainless steel knife and split 50/50 for metal and perchlorate analysis.
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

Beetroot

- Rinse with deionized water if the sample is contaminated with soil.
- Remove and discard any 'tops' if present.
- Peel all units using a clean stainless steel knife and split the peeling 50/50 for metal and perchlorate analysis.
- Chop the inner edible portion(s) in half using a clean stainless steel knife and split 50/50 for metal and perchlorate analysis.
- Login the peeling (sample type: beetroot, sample description: peeling, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the inner edible part (sample type: beetroot, sample description: inner edible part, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The peeling and inner sub-samples for perchlorate are to be homogenized and stored in a freezer.

Sweet potato

- Rinse with deionized water if the sample is contaminated with soil.
- Peel all units using a clean stainless steel knife and split the peeling 50/50 for metal and perchlorate analysis.
- Chop the inner edible portion(s) in half using a clean stainless steel knife and split 50/50 for metal and perchlorate analysis.
- Login the peeling (sample type: sweet potato, sample description: peeling, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the inner edible part (sample type: sweet potato, sample description: inner edible part, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The peeling and inner sub-samples for perchlorate are to be homogenized and stored in a freezer.

Apple

- Peel all units using a clean stainless steel knife and split the peeling 50/50 for metal and perchlorate analysis.
- Chop the inner edible portion(s) in half using a clean stainless steel knife
- Remove inedible core and pips and discard.
- Split the edible part 50/50 for metal and perchlorate analysis.
- Login the peeling (sample type: apple, sample description: peeling, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the inner edible part (sample type: apple, sample description: inner edible part, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The peeling and inner sub-samples for perchlorate are to be homogenized and stored in a freezer.

Potted Parsley

- Carefully remove parsley from the pots and rinse with deionized water if the sample is contaminated with soil.
- Split parsley 50/50 for metal and perchlorate analysis.
- Carefully separate the soil/compost from the roots.
- Split soil 50/50 for metal and perchlorate analysis.
- Discard pots and roots.
- Rinse with deionized water if the sample is contaminated with soil.
- Login the parsley sample (sample type: parsley, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the soil/compost sample (sample type: soil, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The parsley sample for perchlorate is to be homogenized and stored in a freezer.
- The soil/compost sample for perchlorate is to be mixes thoroughly stored in a freezer.

Melon

- Peel all units using a clean stainless steel knife and split the peeling 50/50 for metal and perchlorate analysis.

- Chop the inner edible portion(s) in half using a clean stainless steel knife
- Remove inedible seeds and discard.
- Split the edible part 50/50 for metal and perchlorate analysis.
- Login the peeling (sample type: melon, sample description: peeling, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the inner edible part (sample type: melon, sample description: inner edible part, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The peeling and inner sub-samples for perchlorate are to be homogenized and stored in a freezer.

Kale

- Split the leaves 50/50 for metal and perchlorate analysis.
- Login the sample and create two aliquots (metals & perchlorate).
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

Lemons

- Peel all units using a clean stainless steel knife and split the peeling 50/50 for metal and perchlorate analysis.
- Chop the inner edible portion(s) in half using a clean stainless steel knife
- Remove inedible seeds and discard.
- Split the edible part 50/50 for metal and perchlorate analysis.
- Login the peeling (sample type: lemons, sample description: peeling, customer ID: HallMark reference) and create two aliquots (metals & perchlorate)
- Login the inner edible part (sample type: lemons, sample description: inner edible part, customer ID: HallMark reference) and create two aliquots (metals & perchlorate).
- The peeling and inner sub-samples for perchlorate are to be homogenized and stored in a freezer.

Peppers

- Destalk the peppers.
- Chop each unit in half using a clean stainless steel knife
- Remove seed and inedible inner core and discard
- Split 50/50 for metal and perchlorate analysis.
- Login the sample and create two aliquots (metals & perchlorate).
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

Blueberries:

- Split the berries 50/50 for metal and perchlorate analysis.
- Login the sample and create two aliquots (metals & perchlorate).
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

Runner beans

- De-stalk if necessary and discard.
- Split the beans 50/50 for metal and perchlorate analysis.
- Login the sample and create two aliquots (metals & perchlorate).
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

Rosemary

- Split the sample 50/50 for metal and perchlorate analysis.
- Login the sample and create two aliquots (metals & perchlorate).
- The perchlorate sub-sample is to be homogenized and stored in a freezer.

ANNEX 3. SAMPLE PREPARATION PROTOCOL USED FROM NOVEMBER 2014 ONWARDS

Contaminants in foodstuffs Regulation No 1881/2006:

The maximum level applies after washing the fruit and vegetables and separating the edible part.

For potatoes, the maximum level applies to peeled potatoes.

1. SAMPLE RECEIPT & LOGGING

Samples will be analysed by FESA (perchlorate/chlorate) and FESB (Cd and Pb)

Samples will be received from Hallmark and logged into Nautilus by FESA

2 x LIMS labels will be printed for each sample (1 label for each team)

FESA will split each sample approximately into 2 and deliver one third to 14G04

FESB and the remainder will stay with FESA. The sample will be split without chopping (i.e. pass whole units to FESB)

The sample delivery to FESB will include the corresponding LIMS labels and paperwork.

2. SAMPLE PREPARATION PRIOR TO HOMOGENISATION

(Carried out in FESA and FESB):

General rule - analyse the edible portions of the fruit and vegetables.

Equipment:

Stainless steel knives, vegetable peelers and plastic/glass chopping boards.

Washing samples:

Tap water can be used initially, followed by a final rinse with deionised water.

Herbs (e.g. tarragon, basil, rosemary)

Cut plant away from the pot (if appropriate).

Remove and discard any woody stems or roots.

Wash and shake to remove excess water (blot dry with paper towel if necessary).

Leafy vegetables (e.g. cabbage, lettuce, spinach)

Remove and discard inedible leaves and/or end of stems (e.g. discoloured/dried out).

Open out the leaves, wash and shake to remove excess water (blot dry with paper towel if necessary).

“Ready to eat” Salad leaves, spinach etc.

received as a retail “ready to eat” product, won’t need any preparation or washing.

Beans (e.g. runner beans, green beans)

Remove and discard stalks.

Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Tomatoes

Remove and discard green tops and vine (if present).

Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Root vegetables (Carrots, parsnips and beetroot)

Remove and discard top and root (if present).

Wash vegetable (scrub if necessary to remove soil) and shake to remove excess water (blot dry with paper towel if necessary)

[Do not peel].

Celeriac

Remove and discard skin.

Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Brassicas (e.g. broccoli, cauliflower)

Remove and discard outer leaves and end of stalk.

Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Potatoes peeled (excluding new potatoes, baking potatoes and sweet potatoes)

Remove and discard skin.

Wash vegetable (scrub if necessary to remove soil) and shake to remove excess water (blot dry with paper towel if necessary).

New/salad potatoes

Wash vegetable (scrub if necessary to remove soil) and shake to remove excess water (blot dry with paper towel if necessary)

[Do not peel].

Baking potatoes

Wash vegetable (scrub if necessary to remove soil) and shake to remove excess water (blot dry with paper towel if necessary)

[Do not peel].

Sweet potatoes

Wash vegetable (scrub if necessary to remove soil) and shake to remove excess water (blot dry with paper towel if necessary)

[Do not peel].

Leeks

Remove and discard roots (if present).

Cut vegetable open to wash between the layers where soil can collect

Onions

Remove and discard skin, top portion and roots.
Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Peas

Shell peas and discard pods (if present).
Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Peppers

Remove and discard stalk and seeds.
Wash vegetable and shake to remove excess water (blot dry with paper towel if necessary).

Berries (e.g. strawberries, blueberries, raspberries)

Remove and discard green tops.
Wash fruit and shake to remove excess water (blot dry with paper towel if necessary).

Grapes

Remove and discard stalks.
Wash fruit and shake to remove excess water (blot dry with paper towel if necessary).

Stone fruit (e.g. plums, peaches)

Remove and discard stalks and stones.
Wash fruit and shake to remove excess water (blot dry with paper towel if necessary).

Mangos

Wash fruit and shake to remove excess water (blot dry with paper towel if necessary).
Hold fruit upright and cut along the sides, separating the flesh from the stone/pit.
Discard the central portion containing the pit. Cut the flesh away from the skin and discard the skin.

Apples & pears

Wash fruit and shake to remove excess water (blot dry with paper towel if necessary).
Discard stalk, core and pips.

Citrus fruit (e.g. oranges, lemons)

Remove and discard outer skin and pips.