LEVELS OF IODINE IN NEW ZEALAND RETAIL SALT

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ACKNOWLEDGEMENTS

The iodine analyses were undertaken by Hill Laboratories, Hamilton.

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SUMMARY

The work described in this report was undertaken to determine the level of iodine in retail salt samples.

A total of 20 different salt products, including six iodised salts, nine sea salts, four rock salts, and one low sodium salt, were purchased from retail outlets between September 2008 and February 2009. Five different batches of each product were purchased in either Christchurch, Auckland or Rotorua with the exception of one salt (Product 19) for which only four batches could be obtained over the time period of the project.

The entire contents of each package were dissolved in deionised water and sub-sampled for analysis. Samples were digested with tetramethylammonium hydroxide (TMAH), filtered and analysed by inductively coupled plasma-mass spectrometry (ICP-MS) by Hill laboratories, Hamilton, in accordance with International Accreditation New Zealand (IANZ) requirements.

Multiple quality control procedures were followed, including repeat analyses and analysis of in-house and certified reference materials, blind duplicates and spiked samples, to ensure robust results.

The mean concentration of iodine in iodised table salts ranged from 32-64 milligrams of iodine per kilogram of salt (mg/kg), within the requirements of Standard 2.10.2 of the Australia New Zealand Food Standards Code (FSANZ, 2009). The 14 sea, rock and "other" salt products contained between 1-5mg/kg.

Variability of iodine concentration, expressed as Co-efficient of Variation (CV) ranged from 1-127% across five batches of the 20 salt products. Variability across the iodised salts ranged from 8-38%.

1 INTRODUCTION

From the 27 September 2009 Standard 2.1.1 – 'Cereals and Cereal Products' of the Australia New Zealand Food Standards Code (the Code) will require the mandatory replacement of non-iodised salt with iodised salt in all New Zealand bread defined for fortification. Iodised salt is defined by Standard 2.10.2 – 'Salt and Salt Products' of the Code. It stipulates that iodised salt must contain 25-65 milligrams of iodine per kilogram of salt (mg/kg) (FSANZ, 2009).

In this context the purpose of this Environmental Science and Research Limited (ESR) project was two-fold. Firstly, any agreement to require mandatory fortification also requires that it be monitored and formally reviewed to assess the effectiveness of, and continuing need for, the mandating of fortification (ANZFRMC 2006). Secondly, the fortification of bread will be inadequate to completely restore iodine status in the population. As such, consumers will be encouraged to choose from a range of other food sources that include iodine - iodised salt is an obvious food choice.

The results of this analytical work will assist the New Zealand Food Safety Authority's (NZFSA) efforts in these areas, allowing better estimation of the population's exposure to iodine through discretionary salt use and providing robust food compositional information appropriate for identifying sources of iodised salt suitable for use by the population.

2 MATERIALS AND METHODS

2.1 Selection of Salts for Inclusion in the Study

A total of 20 different salt products, comprising six iodised salts, nine sea salts, four rock salts and one low sodium salt, were purchased from retail outlets between September 2008 and February 2009.

Five different batches of each product were purchased in either Christchurch, Auckland or Rotorua. For one salt (Product 19) only four batches could be obtained over the agreed time period. For this product, a second sample of one batch was purchased. One salt (Product 6) did not include any batch information. This product was purchased on three occasions from three different outlets.

2.2 Laboratory Analytical Methods

Since the measured iodine concentration in two retail iodised salts has previously been found to be highly variable with Co-efficient of Variation (CV[1]) up to 70% (Thomson, 2009), the methodology was designed to ensure as homogenous a sample as possible prior to sub-sampling for analysis.

The entire contents of each purchase unit were dissolved in deionised water to a salt concentration of 1.5-3.0% and a 50ml subsample of the solution dispatched to Hill Laboratories. Samples were sent in three consignments, on 24 September, 13 October 2008 and 2 March 2009.

2.2.1 Iodine analysis

Samples for iodine analysis were digested with tetramethylammonium hydroxide (TMAH), filtered and analysed by inductively coupled plasma-mass spectrometry (ICP-MS) by Hill laboratories, Hamilton, based on methodology of Fecher *et al*., (1998), in accordance with International Accreditation New Zealand (IANZ) requirements.

2.2.2 Quality control procedures

The following quality assurance procedures were undertaken to ensure robust results:

- The analytical repeatability was determined by undertaking five analyses of the same solution of three of the salts.
- Variability within a batch was assessed by analysing five different packages of the same three salts.

[1] CV = standard deviation/mean expressed as a percentage

- Analytical accuracy was assessed from spike recoveries, blind duplicates (n=5), the analysis of an in-house quality control (QC) milk powder sample and analysis of certified non-fat milk powder (NIST 1549) and bovine muscle powder (NIST 8414) reference materials. The acceptable concentration ranges for iodine in these reference materials were 0.71-0.87, 3.04-3.72 and 0.02-0.06mg/kg for the in-house QC sample, NIST 1549 and NIST 8414 respectively.

3 RESULTS

3.1 Assessment of Data Quality

The repeatability of analysis of iodine in salt, expressed as mean CV of five analyses of three salt solutions was 2.9% (Table 2). The mean CV of nine duplicate analyses was 5.5 (Table 3) and for four of the five blind duplicates was similarly low, at 1.7. One blind duplicate was much more variable with a CV of 59.1%. The iodine level in this product was low where high variability may be apparent but the measured concentration was still low (Table 5). In other words, two times a low number is still a low number. This rationale also applies to two of the duplicates with higher than average CVs. One remaining duplicate sample with a CV of 17% indicates, that although the analytical variability is generally less than 5%, it may be up to 20% for the methodology used in this study.

The mean measured concentration of iodine in the certified reference materials (3.28 ±0.05mg/kg, (n=4) and 0.05 ±0.01mg/kg (n=3)) and the in-house QC sample (0.80 ±0.04mg/kg (n=14)) were within acceptable reference ranges (1.5).

The mean recovery of iodine from eight spiked samples was 105%.

Together these quality assurance data give confidence in the methodologies and analytical results for iodine at salt concentrations above 0.05mg/kg.

3.2 Concentration of Iodine in Retail Salts

The mean concentration of iodine across five batches of each iodised table salt ranged from 32-64mg/kg (Table 1), within the requirements of 25-65mg/kg stipulated in the Code (FSANZ 2009). However, there were seven individual packages that exceeded the upper limit (65mg/kg). None of the individual packages contained less iodine than required by regulation.

The 14 sea, rock and "other" salt products contained iodine between 1-5mg/kg. The elevated mean result obtained for Product 11, a private label sea salt, compared with the other non-iodised salts was due to the relatively high concentration of iodine found in one batch only. This result was confirmed by a repeat analysis and the reason for this elevated result is unclear.

Table 1: Mean concentration of iodine (mg/kg) in retail salts.

Salt type	Iodine concentration mg/kg±1 std. dev.	CV
Iodised salts		
Product 1	57.9 ± 12.9	22
Product 2	54.6 ± 14.2	26
Product 3	32.2 ± 2.7	8
Product 4	64.1 ± 24.6	38
Product 5	52.8 ± 17.9	34
Product 6	38.0 ± 6.9	18
Sea salts		
Product 7	1.0 ± 0.3	35
Product 8	1.5 ± 0.8	57
Product 9	1.3 ± 0.7	54
Product 10	1.0 ± 0.6	60
Product 11	5.0 ± 6.4	127
Product 12	1.7 ± 0.6	36
Product 13	1.7 ± 0.7	44
Product 14	1.6 ± 0.1	6
Product 15	1.1 ± 0.5	42
Rock salts		
Product 16	2.0 ± 1.2	59
Product 17	1.7 ± 0.0	2
Product 18	1.4 ± 0.8	56
Product 19	1.6 ± 1.0	62
Other		
Product 20	1.6 ± 0.8	50

3.3 Batch Variability

A comparison of the results for different packages of the same batch (Table 3) and different batches of the same product (Table 8) showed variability, including both measurement (analytical) uncertainty and batch variability measured as CV, ranged from 1-127% for iodine across the 20 salt products. There was less variability between batches of the iodised salts with CV ranging from 8-38% for the six products.

4 DISCUSSION

The non-iodised sea, rock and low sodium salts contained about 30 times less iodine than the iodised salts. The wide variability found in iodine concentration across the salts presents a challenge when trying to assess exposure to iodine across the diet. This variability may be taken into account in probabilistic modelling approaches to iodine exposure.

5 REFERENCES

ANZFRMC (Australia New Zealand Food Regulation Ministerial Council) (2006). Policy Guideline - Fortification of Food with Vitamins and Minerals. ANZFRMC. Accessed at www.health.gov.au on 5 August 2009.

Fecher PA, Goldman I, Nagenast A. (1998) Determination of iodine in food samples by inductively coupled plasma spectrometry after alkaline extraction. Journal of Analytical Atomic Spectrometry;13: 977-982.

FSANZ (Food Standards Australia New Zealand). Australia New Zealand Food Standards Code. Available at http://www.foodstandards.gov.au/thecode/foodstandardscode/index.cfm. Accessed June 2009.

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APPENDIX 1: QUALITY ASSURANCE DATA

Table 2: Repeatability for iodine in salt solutions of three iodised salts (mg/l).

Sample ID	Analysis					Mean	Std dev.	CV
	1	2	3	4	5			
Product 1/1	18	18	19	18	18	18.2	0.5	2.5
Product 3/1	6.6	6.5	6.5	6.5	6.5	6.5	0.0	0.7
Product 5/1	20	20	22	20	19	20.2	1.1	5.4
							Mean CV	2.9

CV = standard deviation/mean x 100

Table 3: Intra batch variability for iodine in salt solutions – different packages of the same batch (mg/l).

Sample ID	Analysis					Mean	Std dev.	CV
	1	2	3	4	5			
Product 1/1	18.0	18.0	18.0	19.0	18.0	18.2	0.4	2.4
Product 3/1	6.6	6.0	6.1	6.4	6.8	6.4	0.3	5.3
Product 5/1	20.0	8.0	9.4	9.2	14.0	12.1	5.0	40.9
							Mean CV	16.2

Table 4: Duplicates for iodine in salt solutions (mg/l).

Sample ID	Result 1	Result 2	Mean	Std dev.	CV
Product 1/3	20.0	19.0	19.5	0.7	3.6
Product 2/1	11.0	11.0	11.0	0.0	0.0
Product 3/3	7.8	7.9	7.9	0.1	0.9
Product 4/1	12.0	12.0	12.0	0.0	0.0
Product 6/1	14.0	11.0	12.5	2.1	17.0
Product 9/2	<0.025	<0.025	<0.025	0.0	0.0
Product 15/1	0.2	0.2	0.2	0.0	14.9
Product 17/2	0.3	0.3	0.3	0.0	12.9
Product 19/2	<0.025	<0.025	<0.025	0.0	0.0
				Mean CV	5.5

Table 5: Blind duplicates for iodine in salt solutions (mg/l).

Sample ID	Result 1	Result 2	Mean	Std dev.	CV
Product 2/3	19.0	19.0	19.0	0.0	0.0
Product 4/2	9.3	8.9	9.1	0.3	3.1
Product 6/2	9.9	9.8	9.9	0.1	0.7
Product 7/1	0.5	0.5	0.5	0.0	3.1
Product 20/1	0.4	0.2	0.3	0.2	59.1
				Mean CV[1]	1.7

1 = excluding I/5/1

Table 6: Concentration of iodine in certified reference materials and in-house QC sample (mg/kg).

CRM Identification	Description	Acceptable range (mg/kg)	Measured value (mg/kg)
NIST SRM 1549	Non-fat milk powder	3.04-3.72	3.28 ± 0.05 (n=4)
NIST RM 8414	Bovine Muscle Powder	0.02-0.06	0.05 ± 0.01 (n=3)
In-house QC	Milk powder	0.71-0.87	0.80 ±0.04 (n=14)

Table 7: Spike recoveries.

The recoveries of samples spiked with 1.25ml of 2mg/l iodine solution:

	Spike 1	Spike 2	Spike 3	Spike 4	Mean
Batch 1	100	95	110	110	104
Batch 2	115	100	110	NA	108
Batch 3	100	NA	NA	NA	100

NA = not applicable

APPENDIX 2: INDIVIDUAL SALT SAMPLES

Table 8: Iodine content (mg/kg) of individual salt samples.

Product	Batch	Product type	Unit Quantity of package	Total weight g	Total volume L	Measured iodine mg/l	Iodine in salt mg/kg	Std dev.	%CV
Product 1	1	Iodised salt	1kg	5283	20000	18.2	69.1		
	2		1kg	1051	4000	11.0	41.9		
	3		1kg	1073	4000	19.5	72.7		
	4		1kg	1044	4000	13.0	49.8		
	5		1kg	1070	4000	15.0	56.1		
						mean	**57.9**	**12.9**	**22**
Product 2	1	Iodised salt	1kg	1051	4000	11.0	41.9		
	2		1kg	1063	4000	17.0	64.0		
	3		1kg	1027	4000	19.0	74.0		
	4		1kg	1087	4000	14.0	51.5		
	5		1kg	1056	4000	11.0	41.7		
						mean	**54.6**	**14.2**	**26**
Product 3	1	Iodised salt	100g	570	2500	6.4	28.0		
	2		100g	113	500	7.5	33.1		
	3		100g	116	500	7.9	33.9		
	4		100g	127	500	8.8	34.8		
	5		100g	118	500	7.4	31.3		
						mean	**32.2**	**2.7**	**8**
Product 4	1	Iodised salt	300g	321	2000	12.0	75.0		
	2		300g	314	2000	8.9	56.6		
	3		300g	318	2000	7.8	49.0		
	4		300g	311	2000	6.0	38.6		
	5		300g	316	2000	16.0	101.2		
						mean	**64.1**	**24.6**	**38**
Product 5	1	Iodised salt	1kg	5314	20000	12.1	45.6		
	2		1kg	1073	4000	15.0	55.9		
	3		1kg	1039	4000	18.0	69.3		
	4		1kg	1067	4000	18.0	67.5		
	5		1kg	1039	4000	6.7	25.8		
						mean	**52.8**	**17.9**	**34**
Product 6	1	Iodised salt	1kg	1021	4000	12.5	49.0		
	2		1kg	1031	4000	9.8	38.0		
	3		1kg	1050	4000	10.0	38.1		
	4		1kg	1000	4000	8.6	34.4		
	5		1kg	1034	4000	7.9	30.6		
						mean	**38.0**	**6.9**	**18**
Product 7	1	Sea salt	2kg	2106	8000	0.3	1.1		
	2		2kg	2040	8000	0.1	0.4		
	3		2kg	2055	8000	0.3	1.0		
	4		2kg	2064	8000	0.3	1.2		
	5		2kg	2119	8000	0.3	1.2		
						mean	**1.0**	**0.3**	**35**
Product 8	1	Sea salt	100g	109	500	0.3	1.2		
	2		100g	129	500	0.2	0.7		
	3		100g	129	500	0.2	0.7		
	4		100g	110	500	0.5	2.4		
	5		100g	108	500	0.5	2.3		
						mean	**1.5**	**0.8**	**57**

Product	Batch	Product type	Unit Quantity of package	Total weight g	Total volume L	Measured iodine mg/l	Iodine in salt mg/kg	Std dev.	%CV
Product 9	1	Sea salt	500g	497	2000	<0.5	2.0		
	2		500g	505	2000	<0.5	2.0		
	3		500g	504	2000	0.1	0.6		
	4		500g	506	2000	0.2	0.7		
	5		500g	502	2000	0.3	1.2		
						mean	**1.3**	**0.7**	**54**
Product 10	1	Sea salt	500g	506	2000	0.5	1.8		
	2		500g	494	2000	0.1	0.4		
	3		500g	502	2000	0.1	0.4		
	4		500g	505	2000	0.3	1.1		
	5		500g	501	2000	0.3	1.2		
						mean	**1.0**	**0.6**	**60**
Product 11	1	Sea salt	95g	94	500	0.8	4.2		
	2		95g	86	500	0.3	1.6		
	3		95g	92	500	0.3	1.6		
	4		95g	98	500	0.3	1.4		
	5		95g	94	500	3.1	16.2		
						mean	**5.0**	**6.4**	**127**
Product 12	1	Sea salt	85g	91	500	0.1	0.6		
	2		85g	90	500	0.3	2.0		
	3		85g	88	500	0.3	2.0		
	4		85g	85	500	0.3	2.0		
	5		85g	88	500	0.3	2.0		
						mean	**1.7**	**0.6**	**36**
Product 13	1	Sea salt	72g	76	500	0.1	0.7		
	2		72g	79	500	0.2	1.0		
	3		72g	81	500	0.3	2.2		
	4		72g	78	500	0.3	2.2		
	5		72g	80	500	0.3	2.2		
						mean	**1.7**	**0.7**	**44**
Product 14	1	Sea salt	450g	450	2000	0.4	1.7		
	2		450g	451	2000	0.3	1.6		
	3		450g	453	2000	0.3	1.5		
	4		450g	452	2000	0.3	1.5		
	5		450g	451	2000	0.3	1.6		
						mean	**1.6**	**0.1**	**6**
Product 15	1	Sea salt	1kg	1031	4000	0.2	0.7		
	2		1kg	1050	4000	0.4	1.4		
	3		1kg	1050	4000	0.2	0.6		
	4		1kg	1062	4000	0.5	1.7		
	5		1kg	1059	4000	0.3	1.1		
						mean	**1.1**	**0.5**	**42**
Product 16	1	Rock salt	1kg	799	4000	0.1	0.5		
	2		1kg	711	4000	0.7	3.6		
	3		1kg	509	4000	0.2	1.3		
	4		1kg	527	4000	0.3	2.1		
	5		1kg	545	4000	0.4	2.6		
						mean	**2.0**	**1.2**	**59**
Product 17	1	Rock salt	100g	104	500	0.3	1.6		
	2		100g	102	500	0.3	1.7		
	3		100g	101	500	0.3	1.7		
	4		100g	102	500	0.3	1.7		
	5		100g	104	500	0.3	1.7		

Product	Batch	Product type	Unit Quantity of package	Total weight g	Total volume L	Measured iodine mg/l	Iodine in salt mg/kg	Std dev.	%CV
						mean	**1.7**	**0.0**	**2**
Product 18	1	Rock salt	500g	508	2000	0.1	0.4		
	2		500g	503	2000	0.3	1.2		
	3		500g	502	2000	0.3	1.2		
	4		500g	254	2000	0.3	2.2		
	5		500g	252	2000	0.3	2.3		
						mean	**1.4**	**0.8**	**56**
Product 19	1	Rock salt	95g	97	500	<0.5	2.6		
	2		95g	103	500	<0.5	2.4		
	3[1]		95g	108	500	<0.05	0.1		
	4		95g	102	500	0.3	1.3		
	5[1]		95g	103	500	0.3	1.6		
						mean	**1.6**	**1.0**	**62**
Product 20	1	Other	350g	351	2000	0.5	2.7		
	2	Low sodium	350g	348	2000	0.2	0.9		
	3		350g	350	2000	0.1	0.7		
	4		350g	351	2000	0.3	1.7		
	5		350g	352	2000	0.3	1.7		
						mean	**1.6**	**0.8**	**50**

Std dev. = Standard deviation
%CV= standard deviation/mean x 100
< = less than the stated value.
Limit of quantitation (LOQ) = 0.050mg/l, except for two non-iodised samples in one run where the sensitivity was reduced and the LOQ was 0.5mg/l.
1 = same batch purchased in different locations