

HUMAN HEALTH | ENVIRONMENTAL HEALTH



ICP-OES Applications Overview

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- This multi-element analysis is usually performed by ICP-OES due to its speed and robustness
- National or supra-national regulations are usually applied:
 - EPA 200.7 in USA
 - Determination of Metals and Trace Elements in Water and Wastes by ICP-AES
 - ASTM; APHA /AWWA
 - DIN 38 406 E 22 in Germany
 - EN ISO 11885:2007 Europe and global scale
 - Water quality. Determination of 35 elements by ICP-AES
 - dissolved elements, elements bound to particles ("particulate") and total content of elements in different types of water

Drinking Water - Considerations



- Simple matrix
 - little spectral or no spectral interferences
- Low concentrations
 - detection limits as low as possible
 - high resolution
 - axially viewed plasma
 - ultrasonic nebulizer (USN)
 - processing technique

Drinking Water - Considerations



- Simple matrix
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 - axially viewed plasma
 - ultrasonic nebulizer (USN)
 - processing technique



Element	λ (nm)	found	±	S.D.	certified	±	S.D.
AI	309	125.3	±	4	127.60	±	3.5
As	193	56.6	±	0.9	56.02	±	0.73
Ba	233	506	±	4	506.50	±	8.9
Be	234	12.4	±	0.03	12.53	±	0.28
Cd	214	6.4	±	0.2	6.47	±	0.37
Со	238	24.6	±	0.2	25.00	±	0.59
Mn	257	37.8	±	0.3	37.66	±	0.83
Мо	203	113.	±	0.95	112.90	±	1.7
Ni	231	57.4	±	0.3	58.10	±	2.7
Pb	220	17.8	±	0.15	18.15	±	0.64
Sb	217	52.5	±	1.4	54.10	±	1.1
Se	196	10.9	±	0.8	11.43	±	0.17
V	309	36.4	±	0.4	35.10	±	1.4
Zn	206	72.4	±	0.3	72.48	±	0.65

MiraMist Parallel path nebulizer



- Fits directly into cyclonic spray chamber
- Excellent DL's, especially for water samples





p.n. N0775330.

- Usually increases sensitivity 5-15 fold
- Can have washout problems due to cooled transfer line
- It is expensive

Ultrasonic Nebulizer (Cetac USN-5000AT+)

- Vibrating piezoelectric crystal to generate aerosol
- Would result in introduction of too high amounts of mass into plasma
 - Removal of solvent through vaporization (heating) and condensation (cooling)
 - Cannot tolerate high TDS
 - Not for HF solutions





Ultrasonic Nebulizer DLs (ug/L)



Element Wavelength (nm)		Pneumatic Nebulizer	U-5000AT+
	·····	(GemCone)	
Ag	328.068	1	0.03
AI	396.153	2	0.06
As	188.979	3	0.7
Ba	233.527	0.5	0.01
Be	313.107	0.1	0.009
Bi	233.061	2	0.2
Са	317.933	2	0.03
Cd	228.802	0.1	0.02
Co	228.616	0.2	0.02
Cr	267.716	0.2	0.01
Cu	324.754	0.6	0.02
Fe	238.204	0.1	0.02
Mg	285.213	0.5	0.06
Mn	257.610	0.1	0.03
Mo	202.031	0.6	0.3
Ni	231.604	0.4	0.06
Pb	220.353	2	0.2
Sb	206.836	2	0.3
Se	196.026	3	0.5
Sn	189.927	1	0.4
Ti	334.940	0.2	0.006
TI	190.801	2	0.5
V	290.880	2	0.02
Zn	213.857	0.2	0.03

Detection limits are based on 3 sigma, with a 20 sec. integration time.



CRM Waste-Water CWW-TM-G Trace Metals in Certified Wastewater (High Purity Standards, Charleston, SC – USA)

Element	Meas.	SD	RSD	Certif.	Accuracy
	[mg/L]	[mg/L]	[%]	[mg/L]	[%]
AI	1.047	0.005	0.51	1.00	4.7
As	0.258	0.006	2.2	0.25	3.2
Ba	0.0241	0.0001	0.52	0.025	-3.6
Cd	0.255	0.004	1.4	0.25	2.0
Со	1.036	0.002	0.23	1.00	3.6
Cr	0.0249	0.00005	0.20	0.025	-0.4
Cu	0.0250	0.0005	1.8	0.025	0.0
Fe	1.019	0.006	0.56	1.00	1.9
Mn	1.035	0.003	0.29	1.00	3.5
Мо	0.980	0.004	0.38	1.00	-2.0
Ni	0.259	0.001	0.29	0.25	3.6
Pb	0.0252	0.0001	0.40	0.025	0.8
V	1.00	0.007	0.71	1.00	0.0
Zn	0.0249	0.0003	1.2	0.025	-0.4



	Diges	tion 1	Digest	tion 2	
	Meas.	SD	Meas.	SD	Certified
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
As	3.5	0.2	3.1	0.2	-
Ва	295	3	332	2	-
Be	0.203	0.001	0.201	0.002	-
Cd	17.7	0.1	17.4	0.1	16.8 ± 1.6
Со	7.7	0.1	7.09	0.02	6.8 ± 1.0
Cr	76.2	0.2	79	1	85.2 ± 16.3
Cu	403	1	401	1	416 ± 23.5
Hg	10.65	0.08	10.43	0.06	8.82 ± 0.88
Ni	38.6	0.1	39.0	0.2	38.5 ± 3.6
Pb	364	0.5	350	1	332 ± 22
V	13.5	0.1	13.5	0.1	-
Zn	2835	5	2799	11	2772 ± 209

(results are "aqua regia" soluble, while Hg is total)

- Sample preparation following DIN S-7 method:
 - 3 g dried sample and 28 mL of aqua regia were heated under reflux for three hours and diluted to 100 mL. Digestions were performed in duplicate.



Element	Filter Digest	SD (mg/L)	Blank Filter Digest	SD (mg/L)	Total Filter	Certified
	(mg/L)		(mg/L)		(ug/Filter) (SD)	(ug/Filter)
Arsenic	0.9648	0.0157	< 0.005	0.0014	48.24 (0.78)	50.48 <u>+</u> 1.16
Arsenic*	0.0536	0.0025	< 0.005	0.0014	2.68 (0.13)	2.69 <u>+</u> 0.065
Barium	0.5126	0.0016	< 0.0003	0.0001	25.63 (0.08)	25.24 <u>+</u> 0.58
Beryllium*	0.0120	0.0001	< 0.0001	0.0000	0.60 (0.05)	0.643 <u>+</u> 0.015
Cadmium	0.2883	0.0040	< 0.0003	0.0000	14.42 (0.02)	15.14 <u>+</u> 0.35
Chromium	0.2056	0.0018	< 0.0005	0.0001	10.28 (0.09)	10.10 <u>+</u> 0.23
Cobalt	<0.001	0.0002	< 0.0005	0.0001	< 0.025	_
Copper	< 0.001	0.0002	< 0.001	0.0003	< 0.050	-
Iron	0.5125	0.0088	< 0.001	0.0003	25.63 (0.44)	25.24 <u>+</u> 0.58
Lead	0.7897	0.0097	< 0.002	0.0006	39.49 (0.49)	40.38 <u>+</u> .92
Magnesium	0.4931	0.0008	0.0052	0.0002	24.66 (0.04)	25.24 <u>+</u> 0.58
Manganese	0.1974	0.0009	0.0002	0.0000	9.87 (0.05)	10.10 <u>+</u> 0.23
Nickel	0.4826	0.0052	0.0006	0.0001	24.13 (0.26)	25.24 <u>+</u> 0.58
Vanadium	0.9593	0.0062	0.0011	0.0001	47.97 (0.31)	50.48 <u>+</u> 1.16
Zinc	1.8733	0.0127	0.0041	0.0002	93.67 (0.63)	100.94 <u>+</u> 2.31





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Food

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Reason for Determining Elements in Foodstuffs



- Nature and concentration of elements in food are related to the biological and physiological role they play in the body
 - Quality and food safety
 - Contamination
 - Toxic levels of trace metals
 - Metal Profiling
 - Labeling Requirements
 - Economic adulteration
 - Geographical origin?
- Factors that influence macro and micro element levels in food materials
 - Natural and environmental processes
 - Inadvertent contamination during growth
 - Manufacturing and preparation processes
- Need information of two classes of trace elements in foodstuffs
 - Toxic and Nutritional

Elements and Matrices



Food products

- Animal
- Vegetable
- Beverage's
- Elements (high/low)
 - Nutritional
 - Mandatory: Fe, Ca, Na
 - Voluntary: K, P, Mg, Zn, Se, Cu, Mn, Cr, Mo, I, Cl, F
 - Toxic (Pb, Cd, Hg...)

Complex matrix

Digestion important



- Commission Regulation (EC) No. 333/2007 of 28 March 2007 laying down the methods of sampling and analysis for the official control of the levels of Pb, Cd, Hg, Sn-inorganic, 3-MCPD and benzo(a)pyrene in foodstuffs (Text with EEA relevance)
 - Official Journal L 088 , 29/03/2007 P. 0029 0038
 - ... The methods of sampling and analysis to be used for the official control of levels of Pb, Cd, Hg, 3-MCPD, Sn-inorganic and benzo(a)pyrene in certain foodstuffs are established in
 - Commission Directive 2001/22/EC of 8 March 2001 laying down the sampling methods and the methods of analysis for the official control of the levels of Pb, Cd, Hg and 3-MCPD in foodstuffs [4],
 - Commission Directive 2004/16/EC of 12 February 2004 laying down the sampling methods and the methods of analysis for the official control of the levels of Sn (inorg) in canned foods [5]
 - Commission Directive 2005/10/EC of 4 February 2005 laying down the sampling methods and the methods of analysis for the official control of the levels of benzo(a)pyrene in foodstuffs [6], respectively.



Cd, Pb, Hg e Sn

- Hg: only in fish products;
- Cd and Pb: several foodstuffs;
- Sn: only for canned foods (metal tin)
- Sn organic (TBT): it is still in the "limbo"
 - tributyltin (TBT) and triphenyltin (TPT) are of most concern. EU has banned their usage, they are dangerous molecules with high priority in EU directive on waters
 - there are not yet maximum limits for food, but EU commissions are seriously thinking about
- Cd and Pb (and V)
 - Cd and Pb in food integrators: there are maximum limits as part of food regulations (they are cosidered as food);
 - V: no limits, it is now forbidden its use as "active ingredient" (such as essential elements Fe, Zn, Se)



► As

 It is getting important an understanding of its different forms (inorganic and organic) related to the total, EFSA is now for the first time explicitly interested in this

> Al

- Reduction of maximum dose that can be absorbed via diet, that is tolerable weekly intake (toxicological risk)
- There are no maximum limits for food
- Cr (III)
 - There are no limits for Cr in food, only for drinking waters

> V

- from essential it is now "downgraded" to "not useful"
 - Its "essentiality" has never been proved
 - In the scientific community there has never been a prevailing consensus, while this happened for Cr

FDA Import Alerts – Metal Contaminants Selected Examples PerkinElmer



Import Alert Number	Sample	Country of Origin	Metal Contamination
29-02	Bottled Water	Armenia	Arsenic
33-10	Candy and candy wrappers	Mexico, Philippines	Lead
53-18	Skin whitening cream	Dominican Republic, Mexico	Mercury (up to 8%)
52-08	Ceramic ware	Bolivia, China, France, Germany, Greece, Haiti, Hungary, Indonesia, Italy, Japan, Korea, Mexico, Morocco, Poland, Russia, Slovenia, Spain, Thailand, Tunisia, United Kingdom, Yugoslavia	Lead and/or cadmium
20-05	Grape Juice	Argentina	Lead
29-01	Orange Float drink	South Korea	Lead
20-30	Raisins (sultanas)	Canada, Mexico, Turkey	Lead
99-12	Canned foods	All countries	Examination of can for lead solder
52-11	Enamel ware	China	Cadmium
52-10	Crystal baby bottles, cups, etc	All countries	Lead
15-01	Preserved duck eggs	Hong Kong, Taiwan	Lead



Sample digestion:

- It is a very important part of the analysis.
- It strongly influences the quality of the analysis.
- It has to be optimized with respect to the atomizer and/ or detector.
- The trend in sample digestions is towards automated, closed systems which require minimal amounts of reagents



- > Add 5 mL HNO₃ conc. (and H_2O_2), wait 15', close vessels
- Digest in m/w oven

	-		
Phase	Power (W)	Time	Power (W)
1	100	5:00	600
2	600	5:00	600
3	1000	10:00	1000
4	0	15	0

A Streamlined Approach to the Determination

of Trace Elements in Foods

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tomic

Vol. 19(2), Mar./Apr. 1998

Cool, open, dilute to final volume



100 % fat

PerkinElmer

For the Better



	crm (069R	DDP 1		DDP 2	
	found (SD)	certified	found (SD)	certified	found (SD)	certified
elem.	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Ca	12220 (212)	12400	2002 (20)	2000	5506 (22)	5750
Ca	13339 (213)	13490	2093 (20)	2020	- <u>3390 (23)</u>	5750
Fe	2.43 (0.66)	2.32	59.6 (0.2)	64.3	78.3 (1.1)	93.4
К	16674 (235)	17680	5839 (33)	6250	9051 (29)	8860
Mg	1195 (20)	1263	306 (1)	342	532 (9)	552
Na	4109 (81)	4370	1273 (18)	1290	2319 (40)	2650
Р	10636 (74)	11100	1507 (13)	1570	4506 (44)	4655
Zn	44.8 (0.3)	49.0	36 (<0.1)	36.6	55.1 (0.4)	64.1

	DDP 5		DD	MDL	
	found (SD)	certified	found (SD)	certified	(3xSD blk)
elem.	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Ca	5576 (20)	5750	2869 (38)	3070	0.84
Fe	90.2 (0.4)	93.5	62.0 (0.49)	64.3	0.30
К	8773 (131)	8800	6110 (5)	6210	8.88
Mg	551 (7)	552	330 (1)	339	0.12
Na	2570 (47)	2640	1223 (30)	1300	1.98
Р	4561 (47)	4680	1519 (25)	1585	1.62
Zn	64 (0.3)	64.9	36.7 (0.7)	36.9	0.12



	crm (069R	DDP 1		DDP 2	
	found (SD)	certified	found (SD)	certified	found (SD)	certified
elem.	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
AI	2.19 (0.45)	n.a.	0.97 (0.21)	0.55	1.81 (0.37)	0.41
Cu	0.59 (0.02)	0.60	3.40 (0.04)	3.15	6.00 (0.19)	6.65
Fe	2.51 (0.08)	2.32	61.8 (0.2)	64.3	79.9 (0.3)	93.4
Mn	0.22 (<0.01)	n.a.	0.20 (<0.01)	0.24	0.25 (0.02)	0.26
Zn	47.4 (0.4)	49.0	37.6 (0.7)	36.6	59.2 (0.4)	64.1
Мо	0.15 (0.07)	(0.33)	< MDL	n.a.	< MDL	n.a.
Pb	< MDL	0.018	< MDL	n.a.	< MDL	n.a.
Se	< MDL	(0.13)	< MDL	n.a.	< MDL	n.a.

	DDP 5		DD	MDL	
	found (SD)	certified	found (SD)	certified	(3xSD blk)
elem.	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
AI	1.60 (0.41)	0.37	1.43 (0.45)	0.53	0.086
Cu	7.02 (0.09)	6.65	3.23 (0.11)	3.20	0.047
Fe	94.0 (2.1)	93.5	63.6 (0.5)	64.3	0.066
Mn	0.24 (<0.01)	0.27	0.22 (0.02)	0.25	0.007
Zn	67.0 (1.8)	64.9	38.2 (0.3)	36.9	0.039
Мо	< MDL	n.a.	< MDL	n.a.	0.064
Pb	< MDL	n.a.	< MDL	n.a.	0.104
Se	< MDL	n.a.	< MDL	n.a.	0.570

Food Testing (Pb, Sn and Fe in canned vegetables)

Italy DMS 18 february 1984

"... containers ... soldered with Sn-Pb alloy ..."

.40

- AAS suggested after dry ashing sample prep
- Pb limit 1.25 1.50 mg/kg

Fe limit 50 mg/kg

Sn limit 150 mg/kg

elemento	Pb	Fe	Sn
Conc. Attesa			
mg/kg	10.75	47.09	86.00
Conc. Media			
Trovata mg/Kg	10.01	45.17	85.49
Recupero			
%	93.1	96.0	99.4

(data courtesy ARPA – Parabiago)



kg (tomatoes)
" (canned vegetables)



- Plasma is running and the green bullet is at or just above the top of the load coil.
- Bottom of the plasma is 1-2mm above the injector
- Samples Diluted 1+1 in Kerosene (IPA)







	Plant Feed		Degummed Oil			Crude Oil			
Feed Stock	Ρ	Са	Mg	Р	Са	Mg	Р	Са	Mg
Batch 1	7.07	0.83	0.79	30.00	3.63	3.30	448.5	57.9	54.7
Batch 2	11.63	1.48	1.45	7.89	0.72	0.72	550.7	68.3	66.1
Batch 3	8.64	1.10	1.10						
Batch 4	8.97	1.00	1.01	6.36	0.67	0.79	521.0	60.7	61.5
Batch 5	6.32	0.86	0.89	6.87	0.79	0.82	625.7	73.3	67.0
Batch 6	6.01	0.84	0.86	10.04	1.11	1.04	447.4	55.7	51.5

Sample Preparation

- 1 g of sample is added to a 25 mL volumetric flask
- The sample is diluted to volume with kerosene
 - kerosene must be very high quality and purity

US Regulations



- Food and Drug Administration (FDA)
 - Analysis of ceramics for leachable Pb and Cd
 - Refusal because it appears to contain a poisonous or deleterious substance, which may render it injurious to health (Adulteration, 402 (a) (1))

Category	Criteria	Guidelines micrograms/ mL
Flatware	average of 6 units	3.0
Small Hollowware other than cups and mugs	any one of 6 units	2.0
Cups/mugs	any one of 6 units	0.5
Large Hollowware other than pitchers	any one of 6 units	1.0
Pitchers	any one of 6 units	0.5

Ceramic Leachable Lead Limits

Ceramic Leachable Cadmium Limits

Category	Criteria	Guidelines micrograms/mL		
Flatware	Average of 6 units	0.5		
Small Hollowware	any one of 6 units	0.5		
Large Hollowware	any one of 6 units	0.25		



THIRTEENTH SCHEDULE (Regulation 28) TEST FOR PACKAGES

A. TEST FOR PACKAGES, APPLIANCES, CONTAINERS AND VESSELS USED FOR STORAGE OF FOOD

1. Preparation:

The surface of the ware to be tested shall be washed in water containing detergent and rinsed with clean water. The surface to be tested shall not be handled thereafter.

All remnants of water shall be removed from the washed ware by rinsing it with leaching solution that comprises 4 per cent of acetic acid in water v/v.

2. Test:

The ware shall then be filled with the leaching solution at room temperature to the maximum capacity of the ware.

The ware shall be covered to minimise contamination and shall be left at room temperature for 24 hours.

After the period of 24 hours, the leaching solution shall be thoroughly stirred and a portion shall be removed for analysis.

The leachate shall not contain antimony, arsenic, cadmium or lead above the following limits, expressed in ppm:

$$\frac{Sb}{0.2} \qquad \frac{As}{0.2} \qquad \frac{Cd}{0.2} \qquad \frac{Pb}{2.0}$$





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Pharma

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- This General Chapter describes procedures for the evaluation of elemental impurities ... [per] <232> and <2232>
- Two compendial procedures and criteria for the acceptability of alternative procedures are described (... ICP-OES and ICP-MS).
- Alternative procedures
 - require complete validation for each element of interest.
 - meeting the validation requirements demonstrates equivalence to Compendial Procedures.
- System suitability evaluation should be performed on the day of analysis.

Sample Preparation



- Direct aqueous solution
- Direct organic solvent solution
- Indirect aqueous solution
- Closed-vessel digestion
 - For Hg determination, it is recommended to add Au (100 ug/L as a stabilizer).
- Neat only applicable to alternative procedures
- Note: Samples and Blanks may be spiked with Target Elements where an analyte has limited solubility to the solvent system of choice.]



- The choice of a strong acid is highly dependent upon the sample matrix.
- Weights and volumes provided may be adjusted to meet the requirements of the microwave digestion apparatus used
- An example procedure that has been shown to have broad applicability provided.
 - Dehydrate and predigest 0.5 g of primary sample in 5 mL of *Strong Acid*.
 - Allow to sit loosely covered for 30 min in a fume hood.
 - Add 10 mL more of *Strong Acid*.
 - Digest, using a closed vessel technique.
 - Repeat if necessary by adding 5 mL more of *Strong Acid*.



- Procedure 1: Can be used for elemental impurities generally amendable to detection by ICP-OES.
- Procedure 2: Can be used for elemental impurities generally amendable to detection by ICP-MS.
- Verification: Meet Procedure Validation Requirements.

Reagents and Reference Standards



- Reagents should be free of elemental impurities
- Appropriate reference materials =
 - Certified Reference Material (CRM) from a National Metrology Institute (NMI) (e.g. NIST)
 - traceable to the CRM of the NMI.
- USP is not intending to supply individual element standards. USP may supply mixtures as convenience standards at a later date.

Procedure 1: ICP-OES



Sample solution

- Use appropriate sample preparation
- Dilute to about J
- Standardization solution 1:
 - 2J of the element(s) of interest matched matrix
- Standardization solution 2:
 - 0.5J of the element(s) of interest matched matrix
- Blank
 - Matched matrix

- 1 gram sample
- 5 mL HNO3 +2 mL HCI
- Heat 90 °C Water Bath for > 2 hours
- Filter 0.45 um
- QS to 50 mL with DI Water







Element	Wavelength (nm)	View Mode	Upper Calib. Std. (mg/L)	
Calcium	317.933	Radial	400	
Phosphorus	213.617	Radial	200	
Magnesium	285.213	Radial	200	
Potassium	766.491	Radial	200	
Iron	259.393	Radial	40	
Zinc	206.200	Radial	40	
Copper	327.393	Radial	5	
Manganese	257.610	Radial	5	
Boron	249.677	Axial	0.2	
Chromium	267.716	Axial	0.2	
Molybdenum	202.031	Axial	0.2	
Selenium	196.026	Axial	0.2	

ICP-OES Multi-Mineral Results and Label Claims



Element	Found MM-1	Label MM-1	Found MM-2	Label MM-2	Units
Calcium	166.2	160	99.32	100	mg/Tablet
Phosphorus	109.3	110	49.61	48	mg/Tablet
Magnesium	107.2	100	101.4	100	mg/Tablet
Potassium	85.04	80	42.35	40	mg/Tablet
Iron	18.69	18	7.47	9	mg/Tablet
Zinc	15.98	15	16.51	15	mg/Tablet
Copper	2.09	2	1.93	2	mg/Tablet
Manganese	2.16	2	2.44	2.5	mg/Tablet
Boron	148	150	177	150	ug/Tablet
Chromium	136	120	83.5	100	ug/Tablet
Molybdenum	77	75	32	25	ug/Tablet
Selenium	25	20	18	20	ug/Tablet
Tin	12	10	-	-	ug/Tablet
Vanadium	11.5	10	13.5	10	ug/Tablet
Nickel	5.5	5	3.5	5	ug/Tablet



Found Tab 1 Element Label Tab 1 Found Tab 2 Label Tab 2 Units Calcium 166.2 160 99.32 100 mg/tablet 109.3 110 49.61 48 Phosphorus mg/tablet 107.2 100 101.4 Magnesium 100 mg/tablet 85.04 80 42.35 40 Potassium mg/tablet 18.69 18 7.47 9 Iron mg/tablet 15.98 15 16.51 Zinc 15 mg/tablet Copper 2.09 2 1.93 2 mg/tablet 2.16 2 2.44 2.5 Manganese mg/tablet Boron 148 150 177 150 µg/tablet 100 Chromium 136 120 83.5 µg/tablet Molybdenum 77 75 32 25 µg/tablet Selenium 25 20 18 20 µg/tablet Tin 12 10 µg/tablet -Vanadium 11.5 10 13.5 10 µg/tablet 5 5 Nicke1 5.5 3.5 µg/tablet

Table 3. ICP-OES Sample Results and Label Claims



Questions?