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Study Director Report for: Cr, Se, and Mo by ICP-MS

Saturday, September 17, 2011

Lei Bao, Shandong Entry-Exit Inspection and Quarantine Bureau, China

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Agenda

- Objectives
- Method Description
- Specific Method Claims
- Timetable
- Questions

Objectives

- To formulate an ICP-MS based method for simultaneous determination of Se, Cr, and Mo, and apply this method to determine the content of Se, Cr, and Mo in infant formula and adult/pediatric nutritional formulas.
- To determine all three elements within 8 hours.
- To achieve quantitative results at ready-to-feed product levels above 20 ng/g for Cr and Mo, and 10 ng/g for Se.

Method Description

- **The method is developed for the determination of elements of Cr、Se and Mo in infant formula and adult/pediatric nutritional formulas.**
- **The sample is first digested in a nitric acid solution using the fully automatic Microwave Digestion System, and the digestion is then measured by ICP-MS.**
- **The analysis time on instrument is less than 3 min.**
- **This method is simple, sensitive, rapid, and accurate.**

Method description: main steps

Weighing
- 0.500 g of sample is accurately weighed

Digestion
- Warm-up Microwave Digestion System
- Digesting using Microwave Digestion System.

Dissolution
- Cool and make up to volume with ultra pure water.
- Preparing the standard solutions with a series of concentration.

Analysis
- warm-up ICPMS
- Determine standard solutions and prepare calibration curve
- Elements analysis by ICP-MS

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Method description: Time to results (30 extracts)

Weighing
- accurately weigh the samples into the Teflon digestion tube prewashed by acid.
- Add 1 mL H_2O_2 and 5 mL HNO_3, and pre-digest for one hour.

Digestion
- Warm up instrument for 10 min.
- Digest the samples using Microwave Digestion System for 45 min.

Dissolution
- Cool and make up to volume with ultra pure water.
- Preparing standard solution with a series of concentrations

Analysis
- Warm up instrument for 1 hour.
- Determine standard solution and prepare standard curve
- Sample analysis by ICP-MS

~5 h

Operating program for microwave digestion system

Stage	Power (W)	Ramping time(min)	Temperature Controlling(℃)	Holding time(min)
1	1600	5	120	5
2	1600	5	160	5
3	1600	3	180	20

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ICP-MS parameters and Operating Conditions

Parameters	Set value
RF power (w)	1500
Sampling depth (mm)	8.0
Flow rate of carrier gas (L/min)	0.9
Flow rate of makeup gas (L/min)	0.3
Integration time (s)	Se2.0,Cr/Mo0.3
Acquisition mode	Full Quant.
Replicates	3
Rate of sample uptake (mL/min)	0.4
Collision mode	He
Flow rate	3.7 ml/min

Interference in ICPMS

- Main interference for ^{53}Cr in ICPMS is $^{37}Cl^{16}O$, and that for ^{78}Se is $^{38}Ar^{40}Ar$.
- Method: using collision/reaction cell with helium as the collision gas to remove interferences.

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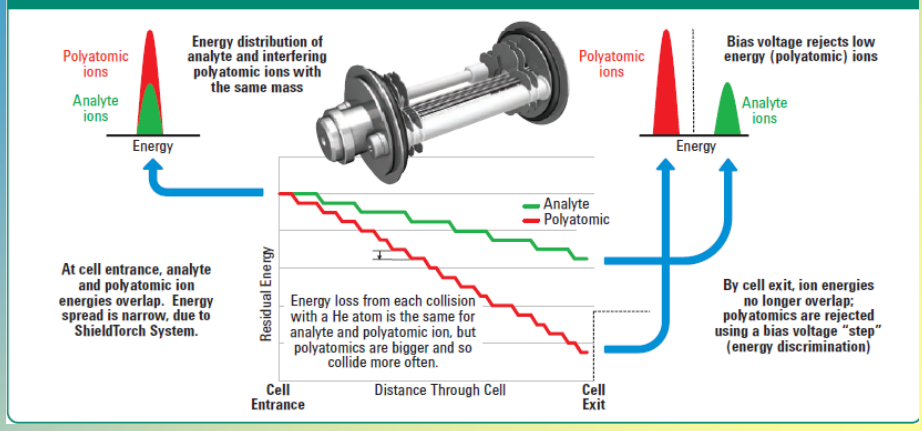
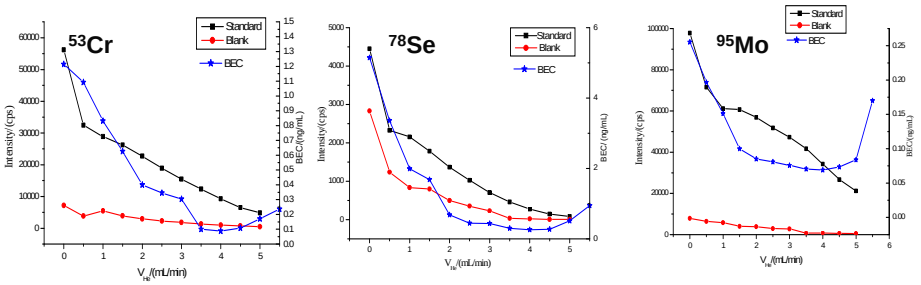
Interference removal using He mode and Kinetic Energy Discrimination (KED)



The flow rate of the collision gas is critical for efficient removal of interference !

Optimization of the flow rate for He gas



Optimized Flow rate: 3.7 ml/min

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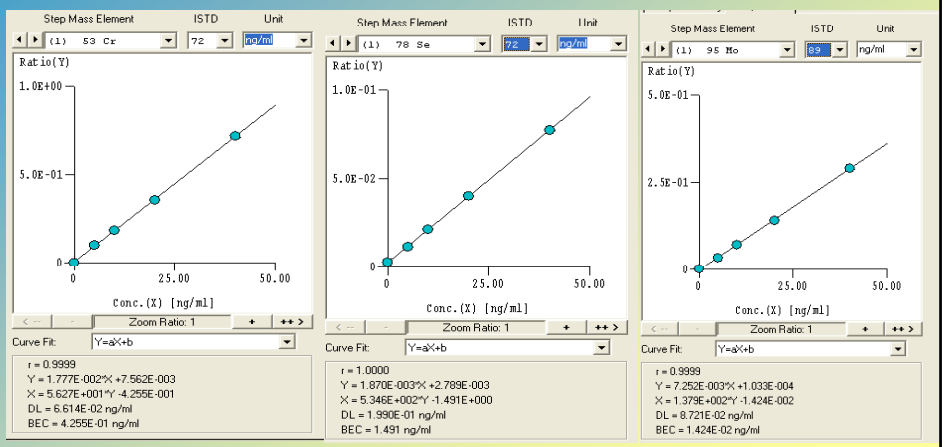
Overview of Performance Data

- Dynamic Linear Range
- Limit of Detection
- Recovery
- Precision
- Reproducibility

Dynamic Linear Range



Concentration: 0, 5, 10, 20, 40 ng/ml for Cr/Se/Mo

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Method Detection Limit

- Determine continuously the blank samples for 11 times.

	Cr	Se	Mo
MDL (ng/g)	1.00	2.00	0.05

Recovery of the Method

Element	Spiked Level (ng)	Measured Value (ng)	Recovery(%)
	0	3.403	——
	5	9.18	115.5
Cr	10	14.39	109.8
	20	23.84	102.2
	40	45.40	105.0
	0	15.25	——
	5	19.40	82.89
Se	10	24.48	92.24
	20	33.93	93.40
	40	55.37	100.3
	0	15.25	——
	5	19.40	82.89
Mo	10	24.48	92.24
	20	33.93	93.40
	40	55.37	100.3

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Precision of the Method

	Cr(ng/g)	Se(ng/g)	Mo(ng/g)
1	30	103	166
2	32	100	160
3	31	104	153
4	31	100	153
5	31	103	144
6	33	106	145
7	32	109	148
Ave	31	104	153
Dev	1	3	8
RSD	3.1%	3.1%	5.3%

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Cat.	Sample	Cr (ng/g)		Se (ng/g)		Mo (ng/g)		Exp. Time
		Exp.	Avg.	Exp.	Avg.	Exp.	Avg.	
Infant Formula	S1	22.8	22.5 (0.5)	200	202.7 (6.4)	187	190 (12.8)	2011-7-7
		21.9		198		204		
		22.7		210		179		
	S2	20.7	20.3 (0.4)	173	175.0 (7.2)	200	209 (8.5)	2011-7-7
		20.3		169		210		
		19.9		183		217		
	S3	23.0	23.1 (0.4)	182	180.0 (2.6)	201	195 (7.2)	2011-7-7
		23.5		177		197		
		22.8		181		187		
	S4	13.3	13.0 (0.3)	114	120.0 (6.6)	330	337.3 (6.4)	2011-7-23
		13.0		119		340		
		12.8		127		342		
Milk Powder	S5	183	172 (30)	125	124 (13.5)	305	279 (39)	2011-8-3
		197		110		298		
		138		137		234		
	S6	12.8	14.3 (2.3)	130	144 (12.5)	210	237 (35)	2011-7-23 211-8-3
		13		148		225		
		17		154		277		
Liquid milk	S7	15.7	18.2 (2.8)	142	151.5 (13.4)	263	258 (6.4)	2011-8-10
		17.5		161		254		
		21.3		210		264		

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Method Claim

- **Linear range: 0-40 ng/ml for ^{53}Cr, ^{78}Se, and ^{95}Mo**
- **Method detection limit**

  1.0 ng/g for ^{53}Cr, 2.0 ng/g for ^{78}Se, and 0.05 ng/g for ^{95}Mo
- **Recovery**

  Within the range of 82.9%～115% at various spiked levels for the three elements.
- **Evaluation of Precision**

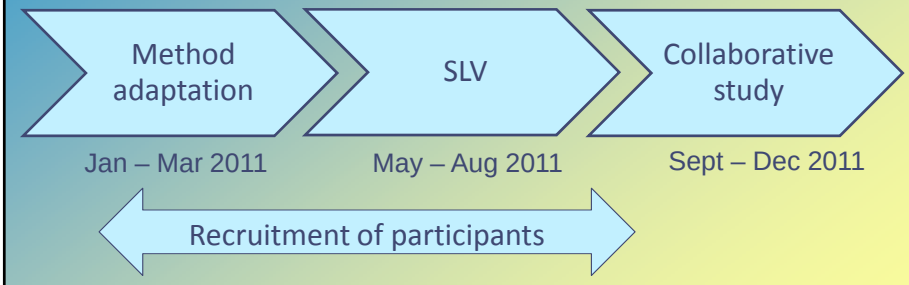
  < 6%

- **Advantages**
  - Faster than other methods, simultaneous analysis of multiple elements
  - Lower detection limit, lower interference.
  - Low contamination and loss.
- **Weaknesses / Limitations**
  - Weighted samples limited by the quality
  - High concentrated HNO_3 introduced to ICPMS
  .

Specific method claims

- Determination of elements Cr、Se、Mo in all forms of infant, adult, and/or pediatric formula (powders, ready-to-feed liquids, and liquid concentrates.
- Cr isotope: 53
- Se isotope：78
- Mo isotope：95

Proposed Timetable



Validation protocol

- The same protocol for other elements: Na, K, Cu, Mg, Fe, Zn, Mn , Ca, P, Pb.
- Materials availability
- Timeline for SLV data back to AOAC

Questions?