

Direct Analysis of Undiluted Soil Digests Using the Agilent High Matrix Introduction Accessory with the 7500cx ICP-MS

When dilution is not the
solution – **Part Two**
Real Life Samples

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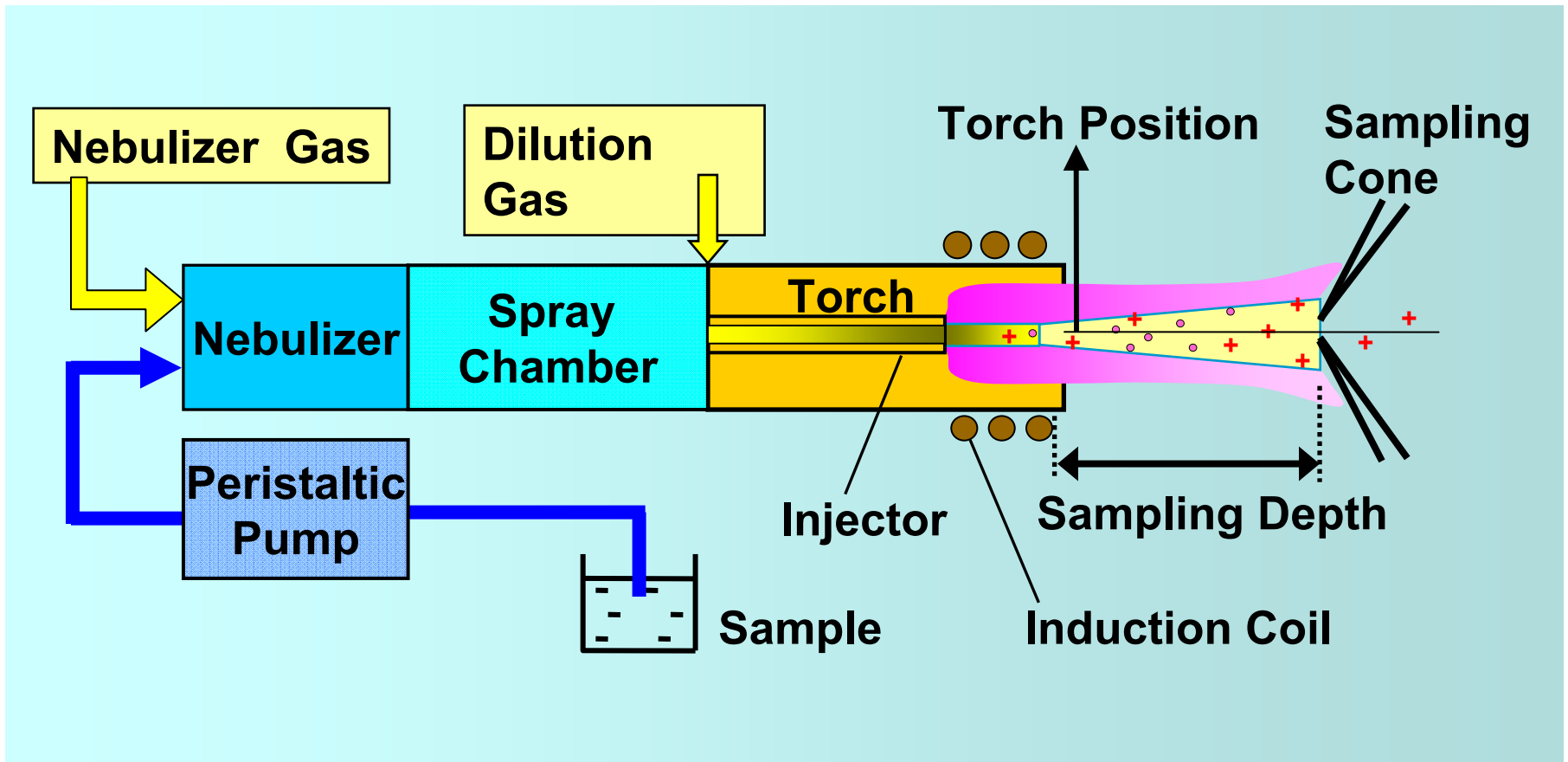
High Matrix Introduction Accessory (HMI)

HMI combines the already superior matrix tolerance of the 7500 Series ICP-MS with fully automated *Aerosol Dilution*

Aerosol Dilution

1. *Does not require any special sample preparation*
2. *Does not require complex, expensive hardware*
3. *Does not increase the analysis time*
4. *Reduces the amount of sample and matrix reaching the plasma and MS interface*
5. *Reduces the amount of water or solvent reaching the plasma*

□ □ □ schematic diagram



The Net Effect

1. Plasma robustness is significantly increased
 - Typical CeO^+/Ce^+ in no-gas mode is 0.2%
2. Samples with TDS at 1% or higher can be introduced directly to the ICP-MS with excellent long term stability, reduced suppression and fewer interferences (oxides)
3. No potential contamination from adding the diluent.
4. No potential error from pipetting the diluent.
5. Less time doing sample prep.

HMI is applicable to the following sample types

Neat seawater

Soil digests (undiluted)

High TDS ground waters

TCLP leachates

Geological digestions and fusions

RoHS samples

High purity metals

HPLC-ICPMS applications requiring high salt buffers

Simple to Use – Just Select Pre-set Tuning Condition

	Robust	Very Robust	Ultra Robust
Typical samples	•Any samples <1,000ppm TDS	•1/10 seawater •1,000ppm metal digested •Ground water	•Undiluted seawater •1% metal digested •Soil digested
Matrix tolerance	<1,000 ppm	<3,000 ppm	<3%
Relative reduction factor	1	1/3	1/24
Carrier gas	0.8	0.5	0.2
Make-up gas	0.2	NA	NA
Sheath gas	NA	0.5	0.8

Robust: equivalent to current high sensitivity tune

Very Robust: equivalent to current robust tune (std setting for enviro analysis with 7500cx)

Ultra Robust: new capability for 7500cx with HMI kit – no other ICP-MS has this capability

Analytical Sample Prep for soil samples

- Weigh out 1 gram soil
- Add 8mL Aqua Regia, 3:1 HCl:Nitric Acid
- Microwave 20 minutes
- Dilute to 50mL
- Resulting matrix 4% Nitric 12% HCl

Instrument Tune Conditions

RF Power (W)	1600
Carrier Gas (L/min)	0.28
HMI dilution gas (L/min)	0.67
Aerosol dilution factor	1/12
Sample uptake rate(mL/min)	0.17
ISTD uptake rate (ml/min)	0.17
Total nebulizer flow (mL/min)	0.34
Extract 1 (V)	0
Extract 2 (V)	-160
Helium flow (He mode)	4.0 mL/min
KED (He mode)	2 volts
H2 Flow (H2 mode)	4.0 mL/min
KED (H2 mode)	2 volts

Required detection limits (Dutch regulatory guidelines)

	Dutch Regulatory Limit Soil dry weight (mg/kg)	MDL1	DRL/MDL1	MDL2	DRL/MDL2
Ag	1	0.28	3.6	0.70	1.4
As	4	0.55	7.3	1.08	3.7
Ba	15	3.04	4.9	6.23	2.4
Be	0.1	0.04	2.4	0.05	2.2
Cd	0.17	0.06	2.9	0.07	2.6
Co	1	0.15	6.8	0.35	2.9
Cr	15	2.30	6.5	4.52	3.3
Cu	5	0.50	10.0	1.30	3.8
Hg	0.05	0.01	3.6	0.02	2.0
Mo	1.5	0.20	7.7	0.41	3.6
Ni	3	0.77	3.9	0.92	3.3
Pb	13	1.20	10.9	2.84	4.6
Sb	1	0.33	3.0	0.40	2.5
Se	10	0.83	12.0	2.04	4.9
Sn	6	0.59	10.2	1.35	4.4
Te	10	1.22	8.2	2.11	4.7
Tl	3	0.28	10.5	0.55	5.5
V	1	0.25	3.9	0.48	2.1
Zn	17	1.70	10.0	3.10	5.5

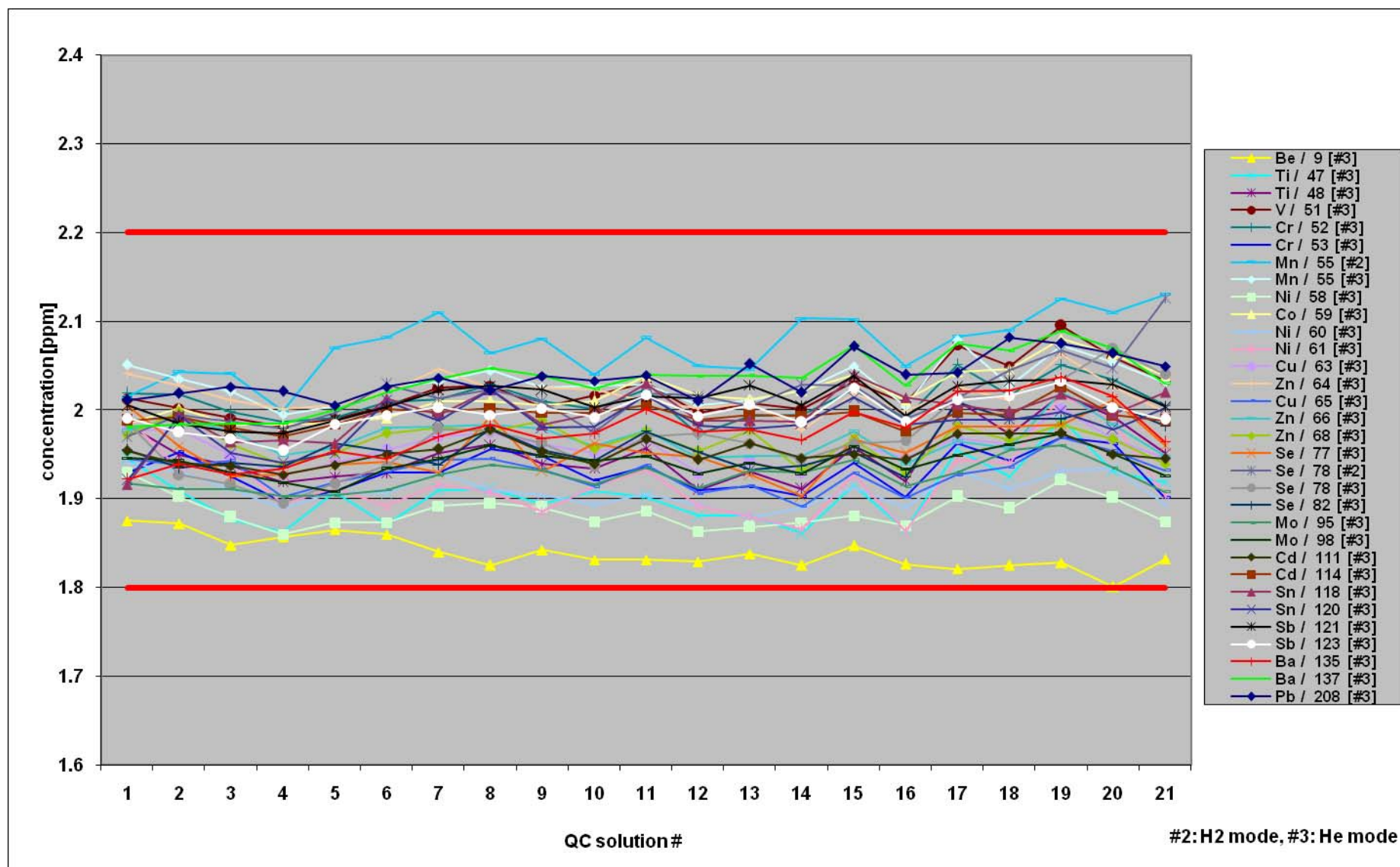
Results of ten replicate analyses of two certified reference materials – over a 30 day period

	FeNelab River Clay			BCR144R Sewage Sludge		
	Average Conc (mg/kg)	Certified amount	% Recovery	Average Conc (mg/kg)	Certified amount	% Recovery
Ag	2.9			8.2		
As	44.7	44	102	3.2		
Ba	828	817	101	319	367	87
Be	1.6			0.2		
Cd	8.5	8	105	1.6	1.84	90
Co	19.8	19	106	13.6	13.3	102
Cr	192	187	103	88.8	90	99
Cu	154	156	99	270	300	90
Hg	4.1	4	107	3.2	3.11	102
Mo	1.3			6.9		
Ni	55.7	53	105	40.7	44.9	91
Pb	297	274	108	94.9	96	99
Sb	1.5			2.8	3.05	92
Se	2.0			1.7		
Sn	0.0			36.0	40.8	88
Te	0.3			0.1		
Tl	1.1			0.1	0.1	102
V	59.6			13.9		
Zn	1032	970	106	825	919	90

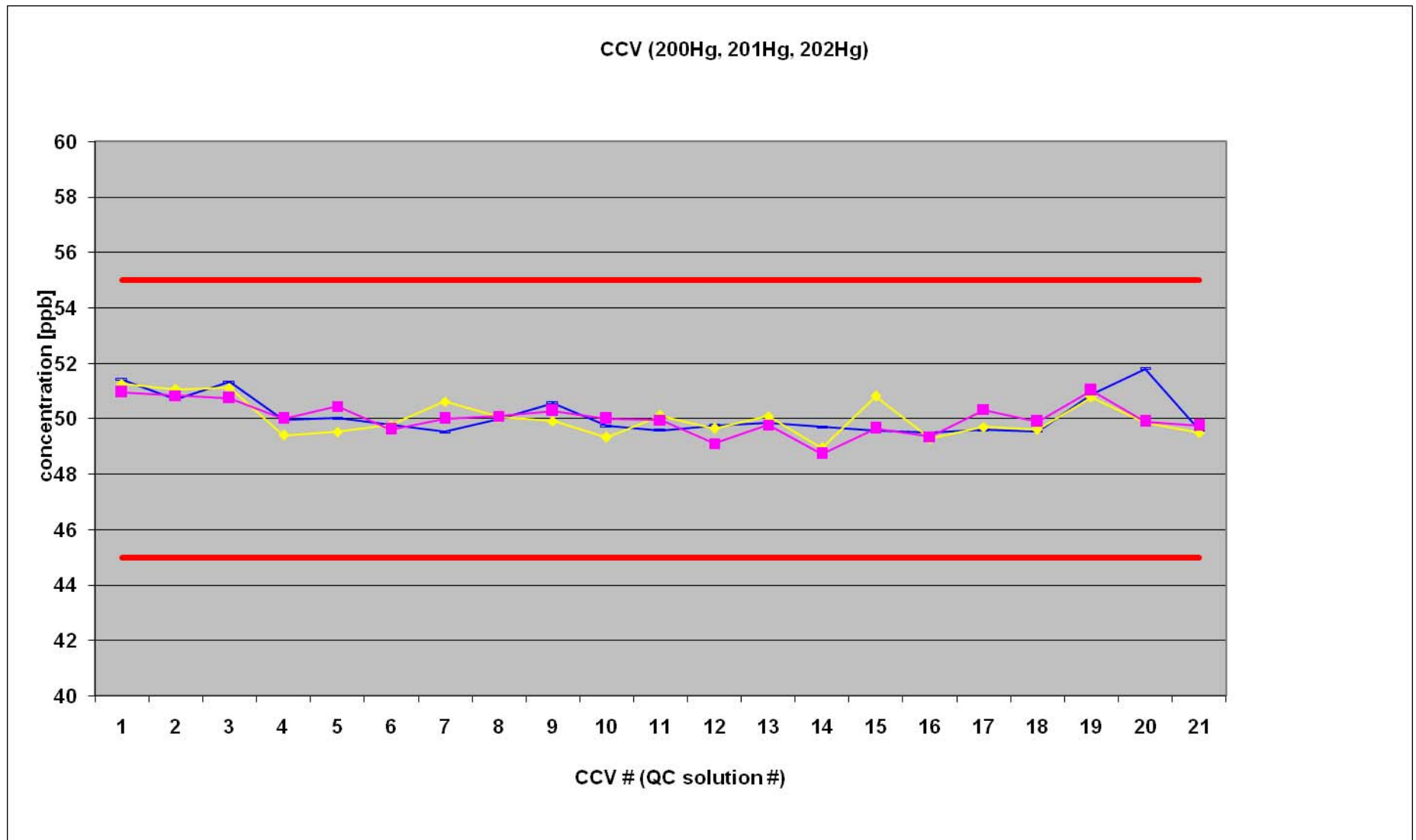
Results of ten replicates on ten non-consecutive days of a low-level spike and a high-level spike – over a 30 day period

	Low level spike sample			High level spike sample		
	Spike amount (mg/kg)	Measured amount (mg/kg)	% Recovery	Spike amount (mg/kg)	Measured amount (mg/kg)	% Recovery
Ag	8	8.3	103	40	41	102
As	40	39.0	97	400	395	99
Ba	120	121.1	101	1100	1102	100
Be	1	0.8	81	800	774	97
Cd	1.2	1.1	94	1100	1063	97
Co	10	10.4	104	800	766	96
Cr	150	149.6	100	1100	1063	97
Cu	40	40.0	100	1100	1058	96
Hg	0.4	0.4	106	13	13	104
Mo	13	12.2	93	1300	1235	95
Ni	25	25.3	101	1100	1074	98
Pb	100	101.9	102	800	778	97
Sb	10	9.7	97	1300	1188	91
Se	100	102.7	103	1300	1336	103
Sn	40	41.7	104	1300	1263	97
Te	80	82.1	103	750	807	108
Tl	23	24.0	104	40	41	103
V	50	50.9	102	800	771	96
Zn	150	151.6	101	1100	1094	99

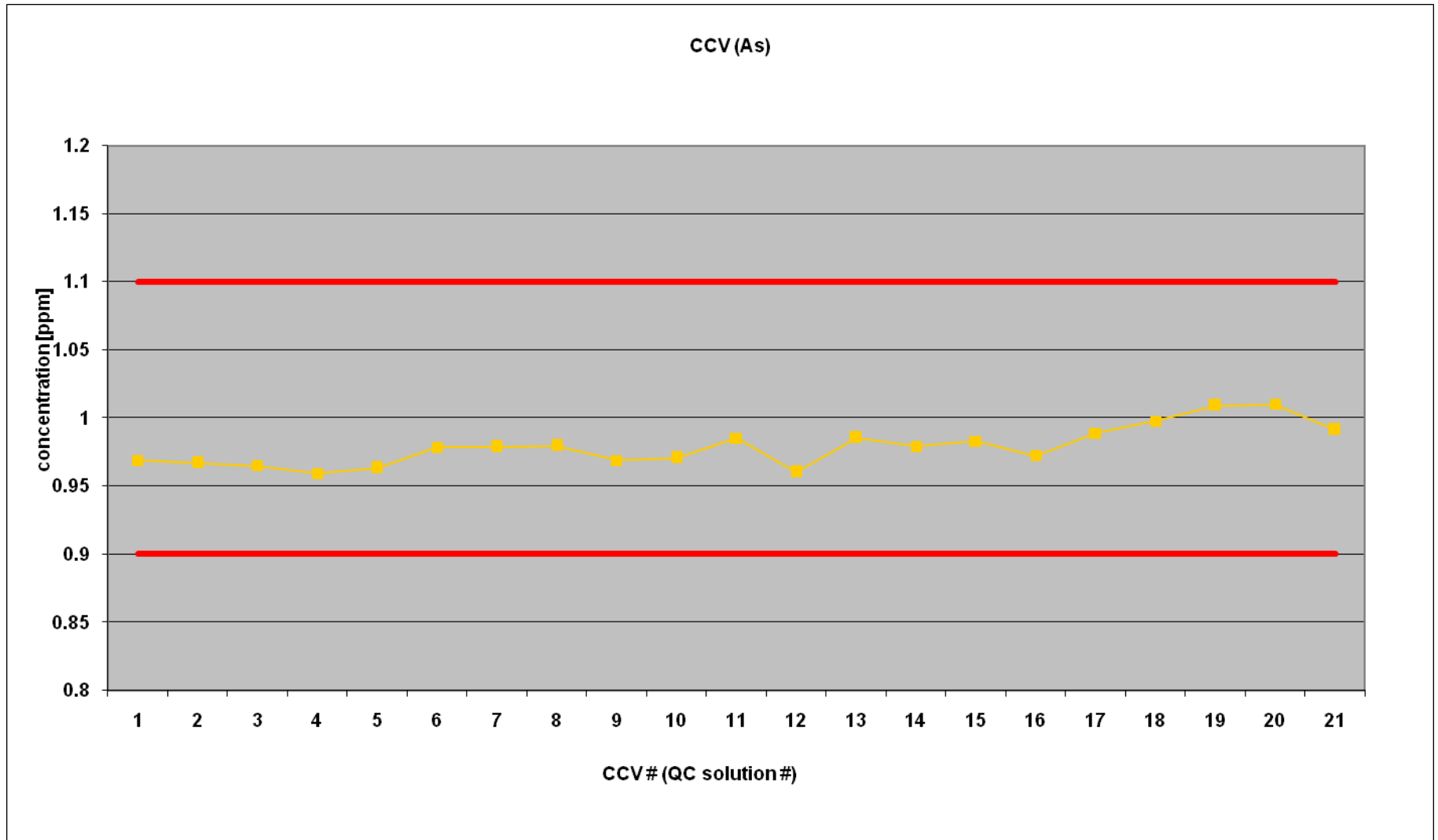
CCV stability during the run of 235 samples



CCV for three isotopes of HG



CCV for Arsenic



Analytico Concluded:

- The HMI easily met the Dutch Regulation AS3000
- HMI saved time – no need to dilute samples manually.
- HMI is low maintenance – no additional tubing or moving parts.
- HMI is simple – no critical timing or plumbing issues.
- HMI is flexible – there is no hardware changes from HMI to conventional sample input.

Comparison of Standard Robust Conditions versus HMI Conditions

	Method 1	Method 2
	Standard Robust	HMI
RF	1600 W	1600 W
Carrier gas	0.7 L/min	0.3 L/min
Dilution gas	0.3 L/min	0.7 L/min
CeO+/Ce+	1	0.3
Extract 2	-150 V	-150 V
H2 flow	3 mL/min	3m L/min
Helium flow	4.5 mL/min	4.5m L/min
Relative dilution	1	1/10
Sample prep	10: 1 dilution	straight

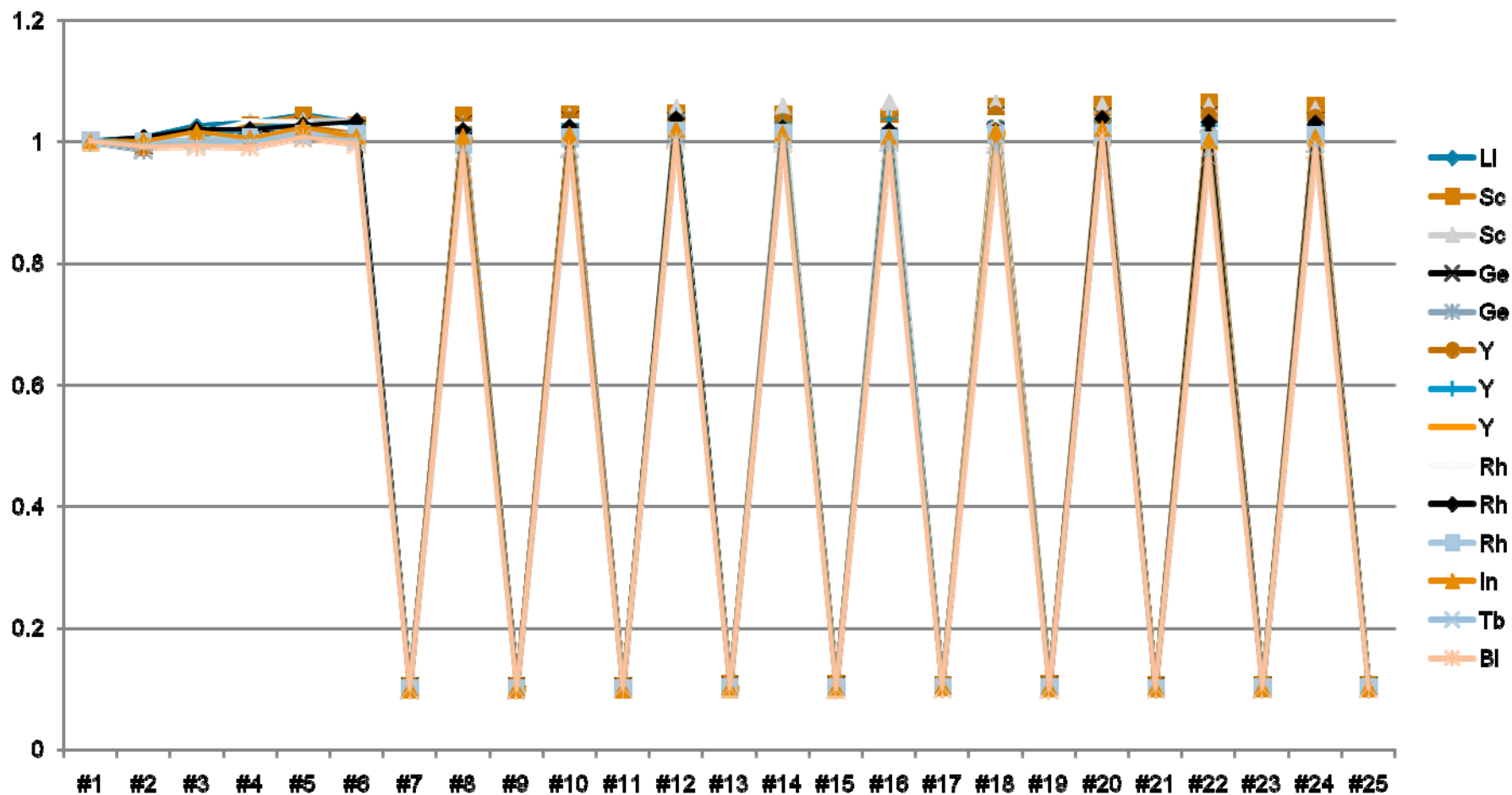
Sample Sequence (each solution spiked with ISTD's)

1	Method 1	Calibration Blank		
2	Method 1	Calibration Standard	0.1ppb Trace	10ppb Majors
3	Method 1	Calibration Standard	1ppb	100ppb
4	Method 1	Calibration Standard	10ppb	1000ppb
5	Method 1	Calibration Standard	100ppb	10000ppb
6	Method 1	NIST 1640	10:1	
7	Method 2	NIST 1640	neat	
8	Method 1	NIST 1640	10:1	
9	Method 2	NIST 1640	neat	
	.			
	.			
	.			
24	Method 1	NIST 1640	10:1	
25	Method 2	NIST 1640	neat	

Average of 10 samples 10:1 and 10 samples HMI 10:1

	Expected Values ppb	Method 1		Method 2	
		Measured Amount ppb	%Recovery	Measured Amount ppb	%Recovery
Al	5.2	5.52	98	5.24	101
As	2.667	2.55	99	2.42	91
B	30.1	33.0	100	31.4	104
Ba	14.8	13.8	96	13.1	88
Be	3.494	3.3	101	3.14	90
Ca	705	699	97	664	94
Cd	2.279	2.08	97	1.98	87
Co	2.028	1.88	98	1.79	88
Cr	3.86	3.67	93	3.49	90
Cu	8.52	9.63	102	9.15	107
Fe	3.43	3.34	106	3.17	93
Hg	0.762	0.76	100	0.72	95
K	99.4	108	91	103	103
Li	5.07	5.2	100	4.94	97
Mg	582	601	95	571.0	98
Mn	12.15	11.87	97	11.3	93
Mo	4.675	4.36	93	4.14	89
Na	2935	3112	95	2956	101
Ni	2.74	2.66	99	2.53	92
Pb	2.789	2.54	93	2.41	87
Sb	1.379	1.41	92	1.34	97
Se	2.196	2.23	100	2.12	96
Si	473	510	108	485	102
Sr	12.42	12.56	100	11.9	96
V	1.299	1.34	97	1.27	98
Zn	5.32	5.23	98	4.97	93

Internal Standard Performance (normalized CPS)



Results

- Reproducibility of EPC modules are excellent
- Reproducibility of switching between HMI and conventional mode is excellent
- Once a system is plumbed for HMI there is no need to change.