Title: **Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS**

Quality System Compliance Review and Approval

Approved By:

Tammy L. Blake	**Quality Coordinator, Analytical Sciences**

Signature: Tammy L. Blake
Digitally signed by Tammy L. Blake
DN: cn=Tammy L. Blake, o=Analytical Sciences ID 47286, ou=ALCS/Analytical Sciences, email=Tammy.L.Blake@altria.com, c=US
Date: 2017.02.02 08:18:00 -05'00'

Management Review and Approval

Approved By:

Georgios D. Karles	**Director, Analytical Sciences**

Signature: Georgios D. Karles
Digitally signed by Georgios D. Karles
DN: cn=Georgios D. Karles, o=Analytical Sciences ID 00071009, ou=Analytical Sciences, email=georgios.d.karles@altria.com, c=US
Date: 2017.02.03 17:15:35 -05'00'

Review Team

Hui Liu Hui Liu
Digitally signed by Hui Liu
Date: 2017.02.02 08:08:12 -05'00'

Karl Wagner Karl A Wagner
Digitally signed by Karl A Wagner
DN: cn=Karl A Wagner, o=Analytical Sciences ID 00057372, ou=ALCS/Analytical Sciences, email=karl.a.wagner@altria.com, c=US
Date: 2017.02.02 07:54:10 -05'00'

Title: **Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS**

Release / Revision Record for SOP

Status (Initial/Revision/ Retired)	Document Revision Number	Issue/Revision Date	Revision Identification	Revision Author
Initial Release	1	09/19/2016	Original Issue	Hui Liu
Revision	2	02/03/2017	Inclusion of (b) (4) and FAST autosampler details; reconciliation of reporting units; edit details of run sequence	Marc Krauss & Likun Yang
Revision	3	02/06/2017	Updated scope to include cigars/ cigar filler and pipe tobacco. Added supplemental validation details for (b) (4) products.	Marc Krauss

Table of Contents

Title: **Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS**

A. SCOPE

1. This analytical method describes the procedure for the determination of Cadmium (Cd) and Arsenic (As) in tobacco products by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). Tobacco products include ground tobacco, cigarette filler, cigars/ cigar filler, pipe tobacco, smokeless tobacco (snus, MST, dry snuff) and (b) (4) Results are reported in ng/g on an as-is basis. Portion products may be reported on a per-portion basis.
2. Analytical determinations are performed on a PerkinElmer NexION 350D ICP-MS and ESI SC-4 DX FAST Autosampler.

B. DEFINITIONS

1. **Method Blank (MBK)** - A tobacco-free sample to which all reagents are added in the same volumes or proportions as used in the routine application of the method. The method blank is carried through the complete sample preparation and analytical procedure and is used to assess contamination resulting from the analytical process.
2. **Laboratory Fortified Blank (LFB)** - A tobacco-free sample to which all reagents are added in the same volumes or proportions as used in the routine application of the method, and to which known quantities of target analytes are added. This spike value should be in the bottom quartile of the standard curve range. The LFB is analyzed exactly like a sample, and its purpose is to determine analyte loss during sample preparation.
3. **Laboratory Fortified Matrix (LFM)** - An aliquot of sample to which known quantities of the target analytes are added. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical result.
4. **Calibration Check Standard (CCS)** - A standard, prepared from a source different from the calibration standards, which is used to verify the calibration.
5. **Internal Quality Control Standard (IQCS**) - A method process control sample that is run with the routine application of the method and is control charted to verify that the method is in control.

C. RESPONSIBILITIES

1. The designated, trained analysts are responsible for the operation and maintenance of the ICP-MS.
2. A technical specialist in the experienced with ICP-MS reviews the raw data and quality control practices, and laboratory management approves the final results.

D. VALIDATION

1. This test method was validated for the following sample matrices in August 2016: ground tobacco, cigarette filler, cigar filler, and smokeless tobacco (snus pouch, moist snuff, dry snuff). This validation is documented in the validation report "Determination of Cadmium and Arsenic in Tobacco Products by Inductively Coupled Plasma Mass Spectrometry" August 16, 2016. Although pipe tobacco samples were not specifically included in the supplemental validation, pipe tobacco is considered to be in scope of this test method because the cigars included in this validation contained pipe tobacco.
2. A supplemental validation was completed in December 2016 to include four (b) (4) products including (b) (4) in the scope of the method. This supplemental validation is documented "Test Method Supplemental Validation Report" December, 2016.
3. LOD and LOQ: For the NexION 350D ICP-MS, the LOD can be calculated as (3.3 σ)/S, and the LOQ can be calculated as (10 σ)/S; where σ = the standard deviation of the y-intercepts of the standard curve regression lines, and S = the slope of the calibration curve (Table 1).

Table 1: As and Cd LOD and LOQ

Analyte	AMU	LOD (ng/g)	LOQ (ng/g)
As	75	3.35	10.16
Cd	114	2.33	7.06

E. EQUIPMENT AND APPARATUS

1. Required Equipment and Apparatus: equivalent materials may be used with laboratory management approval.
   a. NexION 350D ICP-MS
   b. Water purification system capable of producing 18.2 MΩ water. PureLab Ultra ELGA.
   c. Designated sample preparation area designed to limit contamination.
   d. Certified volumetric DigiTubes: SCP Science
      1) 15 mL, part number 010-515-607
      2) 50 mL, part number 010-500-263
      3) 100 mL, part number 010-501-263
      4) 17 mm plug stoppers, part number 130-012-017

e. Peristaltic Pump Tubing for ESI pump: SCP Science

1) Orange-green-orange, part number 022-133-004, 0.38 mm I.D.
2) Black-black-black, part number 022-133-007, 0.76 mm I.D.
3) Grey-grey, part number 022-033-411, 1.30 mm I.D.

f. 2 mL Sample Loop for FAST Autosampler multi-valve, 1.0 mm I.D. (grey Marker), ¼ - 28 fittings, ESI, part number N0777479 REV A.

g. Pipettes and tips: Eppendorf Research Plus pipettes (10 mL, 1000 µL, and 100 µL). Eppendorf pipette tips: epT.I.P.S. standard bulk packaging (1 - 10 mL, 50 - 1000 µL, and 20 - 300 µL).

h. Internal Standard Addition Tee, Perkin Elmer, part number N0777294

2. Instrument Setup

a. ICP-MS Method

1) The Software Manual for Syngistix™ for ICP-MS is recommended as a reference for the execution of this procedure. Setup the instrument as shown in the nine screen shots in Appendix 1 and in the table below. While most of these parameters do not change during daily operation, the nebulizer gas and helium flow rates are frequently adjusted during the tuning operation of the ICP-MS.

Table 2: ICP-MS Method Parameters

Parameter	Setting
Power	1600 W
Nebulizer gas flow	0.94 L/min
Sample Flow Rate (analysis)	3 rpm
Sample Flow Rate (wash)	3 rpm
Dilution Gas (He) Flow Rate (high)	5.0 L/min
Dilution Gas (He) Flow Rate (low)	4.1 L/min

2) Peristaltic pump: A tee is used to add the internal standard to all standards and samples just prior to the nebulizer. The peristaltic pump is plumbed as described below:

a) Orange-green-orange (0.38 mm I.D.): used for delivering internal standard directly to the mixing tee.
b) Black-black-black (0.76 mm I.D.): used for delivering the carrier solution to the valve.
c) Grey-grey (1.30 mm I.D.): used for removing liquid from the bottom of the nebulizer spray chamber.

The three-stop tubing is designed to provide two fresh areas of tubing for exposure to the pump rollers before having to install a new piece of tubing. If the tubing appears or feels flattened, switch to an unused region of the tube or replace it with a new one. Check the pressure of the pump plate by observing continuous flow of liquid in the tubing. To extend the useful life of the tubing, release the pressure on the tubing roller when not in use.

3) The FAST Autosampler multi-valve is plumbed as described below:

Table 3: FAST Autosampler Connection

Valve Port Number	Connection
#1	To Sample Loop
#2	Carrier Solution from Black-Black-Black tubing
#3	To the Mixing tee
#4	From Sample Loop
#5	Sample Solution from autosampler probe
#6	Vacuum

4) Position the Carrier Solution and Internal Standards next to the autosampler. Insert the Carrier solution tube into the Carrier solution flask and insert the ISTD tube into the ISTD flask. Lead the waste line to the waste container.

b. Instrument Operating Conditions and Performance Check: Daily maintenance, performance checks and optimization procedures are described in Work Instruction 097-6041 ICP-MS Daily Instrument Tuning. The analyst is responsible for verifying that the instrument configuration and operating conditions satisfy acceptable operating parameters as described in the work instructions.

3. Instrument Maintenance

Instrument maintenance checks are performed according to daily, weekly, monthly and annual schedules; the logbooks for which are stored in the Metals laboratory, CRT LL0362. The analyst is responsible for verifying that the instrument maintenance is performed and results are recorded in the appropriate logbooks.

F. CHEMICALS AND REAGENTS

1. Chemicals Required: Equivalent substitutions may be made based on certified purity with laboratory management approval.
   a. Argon: refrigerated liquid from Airgas, part number UN1951. The required minimum instrument pressure is 85-90 psi. The current instrument setting is 105 psi.
   b. Helium: compressed gas from Airgas, part number UN1046, ultra high purity UHP300.
   c. Nitric Acid (HNO_3): Optima grade, Fisher Scientific, Cat. #A467-1.
   d. Methanol: Optima grade, Fisher Scientific, Cat. #A456-1.
   e. Cd: 1000 mg/L, in 2% HNO_3, SPEX-Certiprep, Cat. #PLCD2-2Y.
   f. As: 1000 mg/L, in 2% HNO_3, SPEX-Certiprep, Cat. #PLAS2-2Y.
   g. Germanium (Ge): 10000 mg/L, in 1.6% HF, SCP Science, Cat. #140-060-321.
   h. Rhodium (Rh): 1000 mg/L, in 2.5% HNO_3, Inorganic Ventures, Cat. # CGRHN1.
   i. Quality Control Standard 21 (QCS-21): 100 mg/L, in 5% HNO_3, SPEX-Certiprep, Cat. #QC-21-500.
2. Reagent Preparation: Note, volumes can be adjusted proportionally.
   a. CCS: The CCS is prepared from a source different from that used to prepare the calibration standards. For example, SPEX CertiPREP QCS-21 may be used.
      1) CCS Intermediate Stock Solution, 1000 μg/L Cd and As:

         To a 100-mL DigiTube, add approximately 50 mL of reagent water. Pipette 5 mL of HNO_3 and then 1 mL of 100 mg/L QCS-21 into the tube. Dilute to volume with reagent water and mix well. This solution is stable up to 6 months when stored at ambient conditions.

      2) CCS, 10 μg/L Standard Solution:

         To a 50-mL DigiTube, add approximately 25 mL of reagent water. Add 2.5 mL of HNO_3 and then 500 μL of CCS Intermediate Stock (1000 μg/L) into the tube. Dilute to volume with reagent water and mix well. Although not explicitly stated in the validation report, this solution is stable for three weeks when stored at ambient conditions.

b. Internal Standard (ISTD), 2 mg/L Ge and 40 µg/L Rh:

To a 1-L volumetric flask, add approximately 400 mL of reagent water, exactly 400 mL (use 100 mL DigiTube to measure) Methanol and 50 mL (use 50 mL DigiTube to measure) of HNO_3. Add to the flask: 200 µL of 10,000 mg/L Ge and 40 µL of 1,000 mg/L Rh. Dilute to volume with reagent water and mix well. This solution is stable up to 3 months when stored at room temperature.

c. Probe Rinse Solution and Carrier Solution, 5% HNO_3:

To a 2-L volumetric flask, add approximately 400 mL of reagent water and 100 mL (use 100 mL DigiTube to measure) of concentrated HNO_3. Dilute to volume with reagent water and mix well. Although not tested during validation, this solution is considered stable for 3 months, when stored at room temperature.

3. Standard Preparation

a. Calibration Intermediate Stock Standards:

1) Calibration Stock Standard I, 10 mg/L Cd and As:

To a 100-mL DigiTube containing approximately 50 mL of reagent water, pipette 2 mL of HNO_3 and then add 1 mL each of the 1000 mg/L of Cd and As purchased standards. Bring to volume with reagent water and mix well. This solution is stable under ambient conditions for 6 months.

2) Calibration Stock Standard II, 100 µg/L Cd and As:

To a 100-mL DigiTube containing approximately 50 mL of reagent water pipette 2 mL of HNO_3, and then add 1 mL of Calibration Stock Standard I. Bring to volume with reagent water and mix well. This solution is stable under ambient conditions for 6 months.

b. Working Standards:

Prepare the calibration blank and the calibration standards #1 - 9 by filling 50-mL Digitubes approximately half full with reagent water and then adding the specified amounts of HNO_3 and stock standards given in Table 4 to each tube. Make to volume with reagent water and mix well. Calibration standards are stable for three weeks when stored at ambient conditions.

Table 4: Preparation of Working Calibration Standards

Calibration Standard	Concentration (µg/L)	HNO_3 (mL)	Calibration Stock Std I (µL)	Calibration Stock Std II (µL)	Final Volume (mL)
Cal. Blank	0.0	2.5	---	---	50
#1	0.1	2.5	---	50	50
#2	0.2	2.5	---	100	50
#3	0.5	2.5	---	250	50
#4	1	2.5	---	500	50
#5	5	2.5	25	---	50
#6	10	2.5	50	---	50
#7	25	2.5	125	---	50
#8	50	2.5	250	---	50
#9	100	2.5	500	---	50

G. SAMPLE REQUIREMENTS

1. Samples that require grinding and/or removal of filler from cigarettes or cigars should be prepared following SOP 095-1105 “Preparing and Processing Cigarette Samples” and WI 097-1108 “Sample Preparation”. Refer to WI 097-5503 “Sample Digestion for Elemental Analysis” for specific instructions for digesting the various types of tobacco products.

H. PROCEDURE

1. Sample Handling
   a. Prepare tobacco samples for metals analysis as described in WI 097-5503 “Sample Digestion for Elemental Analysis.” The exact weight of each sample, to the nearest 0.0001 g will be entered manually into the instrument software, or captured directly from the balance by the LIMS system.
   b. IQCS: Prepare at least three IQCSs with every batch of samples. Include at least one IQCS with each turntable of 40 samples.
   c. MBK and LFB: Prepare one Method Blank and one Laboratory Fortified Blank with every batch of samples.
   d. LFM: Laboratory Fortified Matrix samples should be prepared using samples that are thought to have a significantly different matrix from those samples evaluated during method validation. The unfortified samples should be analyzed to determine the endogenous concentrations of Cd and As. Representative samples from each matrix should then be prepared with a fortification at 1x - 2x addition of Cd and As and re-analyzed to determine the spike recovery. The fortification shall not change the sample volume by more

than 5%. Deviations greater than 20% from the fortified value suggest a matrix bias and should be investigated and discussed with management.

2. Calibration
   a. Prior to calibration, perform the following procedures. Do not proceed to calibration until acceptable results are acquired:
      1) Perform the instrument optimization process according to the procedure described in ICP-MS Daily Instrument Tuning WI 097-6041.
      2) Ensure that the instrument has passed all performance criteria for standard and KED modes.
      3) Check the sample information file for correct sample sequence and inclusion of all applicable CCS samples.
      4) Select all lines of the sample file list and generate a run list. This will include all CCS samples inserted at appropriate places in the run sequence.
   b. Generation of Calibration curves
      1) Consult the appropriate data acquisition software manuals for information on using the system software for generating calibration curves.
      2) Setup the instrument to perform a standard calibration using a weighted linear regression ($1/x^2$). The origin is not included in the calibration.
      3) The calibration standards are used to generate the calibration curves. The exact concentration of each standard, based upon the information provided in the COA, should be entered into the instrument software.
      4) Calibrate the instrument before sample analysis using the prepared standards and calibration blank. Calibration curves are saved as a data file named for the current sample batch. Calibration curves must meet the criteria specified in the Quality Control and Acceptance Criteria section.
3. Analysis
   a. Use the instrument software to build a run sequence. Enter the sample names, autosampler location and method name for each. If LIMS is not to be used to capture and process the data, the following information should also be added: sample volume, dilution factor, and sample mass.
   b. A typical run sequence is as follows:
      - Calibration blank
      - Calibration standards
      - CCS

- System Suitability Check (triplicate measurements of Calibration Standard 1)
- MBK and LFB associated with the batch digestion
- Three IQCS samples
- CCS
- Samples in batches of 7 followed by a CCS
- If needed, add LFMs at the end of the run in batches of 7 followed by a CCS.

Note: there is not a requirement for the number of times the CCS is analyzed in a batch except that all samples must be bracketed by passing CCSs. The analyst should determine if it is appropriate to add CCSs in the middle of a sample batch based upon batch size and the length of a run.

c. Quantify the resulting data against the internal standard calibration curves using the instrument software.

d. If the concentration of a sample is outside the calibration range, dilute the sample with 5% HNO_3 to bring the analytical concentration within the calibration range.

4. Calculations and Reporting

a. Syngistix™ Software allows for input of sample volume, dilution factor, and sample mass. This permits automatic reporting of data on a per-mass basis. Further data analysis may be required if final units need to be on a DWB or on a per-portion basis.

b. Report data in memo format or in LIMS, according to sample/customer reporting requirements.

c. Percent relative concentration residual (%RCR) is calculated to show the degree of deviation of individual concentration points from the established calibration equation. It is calculated using the following equation:

$$\%RCR = \frac{RC\text{-}NC}{NC} \times 100$$

RC = The concentration calculated from the calibration curve
NC = The nominal or theoretical concentration

d. The concentration of the analyte is calculated with the formula below:

$$\text{Analyte Concentration } \left(\frac{ng}{g}\right) = \frac{Ca \times V \times D}{W} \times 1000$$

Ca = Concentration from calibration (µg/L)

V = Final Volume of prepared sample (L)

D = Dilution factor (if necessary)

W = Weight of sample (g)

e. Percent Spike Recovery for laboratory fortified matrix spikes (LFM) is calculated with the formula below:

$$\% \text{ Spike Recovery} = \frac{Ca - Cs}{Ce} \times 100$$

Ca = Concentration of fortified sample from calibration curve (µg/L)

Cs = Concentration of un-fortified sample from calibration curve (µg/L)

Ce = Theoretical concentration of target (spike) analyte (µg/L)

5. Quality Control and Acceptance Criteria

a. Calibration: A calibration curve is considered valid if the following conditions are met:

1) Coefficient of determination (R^2): Calibration curves for each analyte should have R^2 values of 0.995 or higher. Recalibrate if R^2 falls below 0.995.

2) %RCR: All calibration curves should have %RCR values not exceeding ±20% for CAL 1 and ±15% for the remaining standards.

b. System Suitability Test: System suitability should be evaluated after calibration by running a sequence that includes triplicate measurements of Cal Standard 1. The concentrations for each analyte are evaluated. The data should be within 20% of the established mean value. If not, the instrument should be re-tuned and management informed.

c. Calibration Check Standard (CCS): The validity of the calibration curves must be checked after calibration and during an analysis batch by injecting the CCS. All samples must be bracketed by passing CCSs. The %RCR for the CCSs should be within ±15% of the nominal concentration for each analyte. If %RCR results for a CCS fall outside of ±15%, the samples bracketed by that CCS must be reanalyzed.

d. Evaluate the calibration standards for the ISTD responses. If there is significant instrument drift in the ISTD data, e.g. >20%, stop and then restart

the analysis. This will reset the reference values for the drifted ISTD. Repeat this step as needed until ISTD response is stable throughout the measurement of blanks and standards.

e. IQCS Samples: A suitable reference tobacco (e.g. 3R4F) is used as a method process control sample. It is prepared and analyzed with every batch. Analytical results for the IQCS samples must be plotted on control charts for each analyte. If any results fall outside of established control limits the batch must be discarded and re-prepared and analyzed. Consult laboratory management.

f. Calibration Blank: Analytical results for the calibration blank shall be below the LOD Analyzing the calibration blank can be used to check for contamination and for carryover originating from samples with excessively high analyte concentrations. If the calculated concentrations of an analyte in the blank samples exceed half of the concentration of standard 1, appropriate corrective measures should be conducted.

   1) The MBK should be below the lowest calibration standard. Sample data with MBK above quantitation limits must include an assessment of the impact of these blanks on the results.

g. Laboratory Fortified Blank should be within ±15% of the nominal concentration for each analyte (10 μg/L).

h. Laboratory Fortified Matrix Samples (LFM): An LFM is prepared as a method control with a batch of samples to monitor the performance of the method when the sample matrix is suspected to be significantly different from validated matrices. The recoveries for the LFM should be within ±20% of the theoretical value. Results outside the recovery limits should be investigated and may serve as justification for rejecting the associated sample(s).

i. Using the current methods, data for the cadmium 111 isotope are collected simultaneously with cadmium 114. The Cd-114 data are reported, while the Cd-111 data remain available for secondary reference if a problem is suspected with cadmium sensitivity or interference.

j. In the event that any of the quality control measures outlined in this section pass acceptance criteria for some but not all of the analytes in this method, data for analytes with acceptable quality controls can be accepted, but analytes which fail quality control measures must be re-analyzed before reporting. Consult laboratory management for assistance.

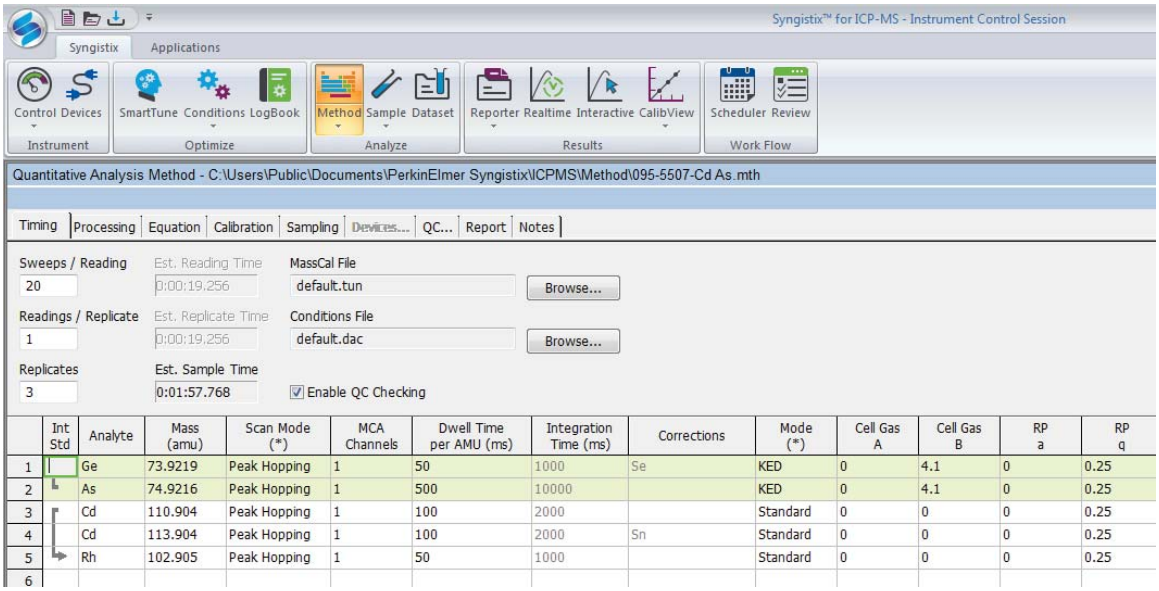
Title: **Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS**

I. REFERENCES

1. SOP 095-1105, Preparing and Processing Cigarette Samples, September 21, 2016
2. WI 097-1108, Sample Preparation, September 23, 2016
3. WI 097-6041, ICP-MS Daily Instrument Tuning, September 19, 2016
4. WI 097-5503 Sample Digestion for Elemental Analysis, September 19, 2016
5. Software Manual for Syngistix™ for ICP-MS (digital help files)
6. Test Method Validation Report 095-5507: Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS, August 16, 2016.
7. Test Method Supplemental Validation Report 095-5507: Analysis of Cadmium and Arsenic in Tobacco Products by ICP-MS, December 1, 2016.

J. APPENDIX

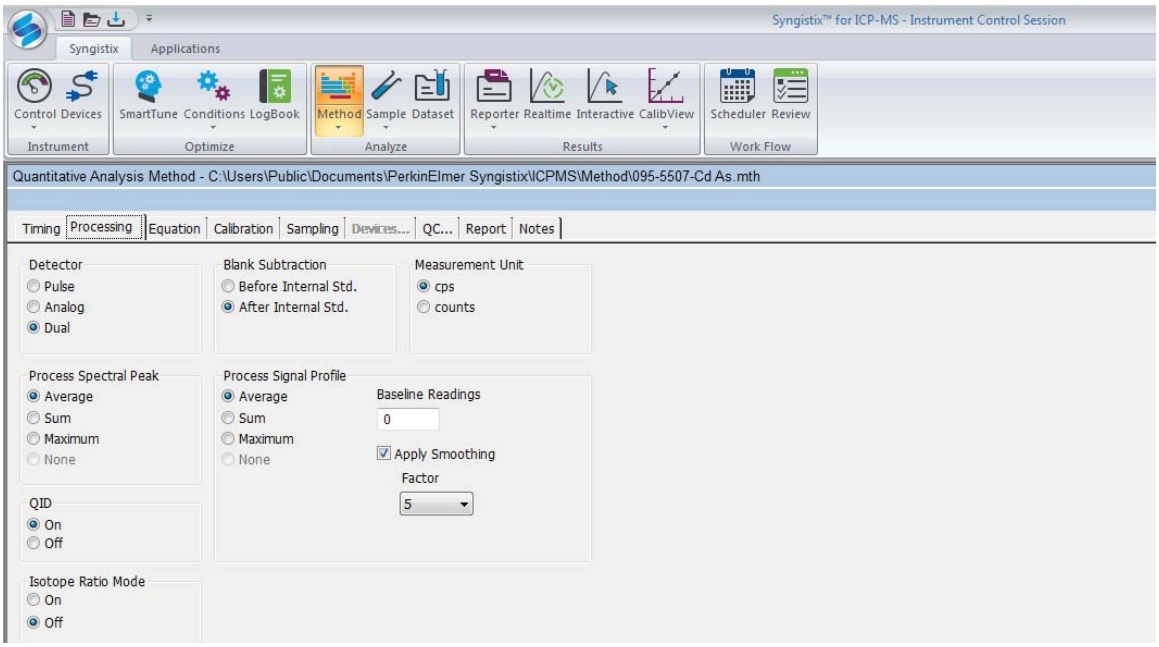
Figure 1: Screenshot showing Method parameters for Timing



	Int Std	Analyte	Mass (amu)	Scan Mode (*)	MCA Channels	Dwell Time per AMU (ms)	Integration Time (ms)	Corrections	Mode (*)	Cell Gas A	Cell Gas B	RP a	RP q
1		Ge	73.9219	Peak Hopping	1	50	1000	Se	KED	0	4.1	0	0.25
2		As	74.9216	Peak Hopping	1	500	10000		KED	0	4.1	0	0.25
3		Cd	110.904	Peak Hopping	1	100	2000		Standard	0	0	0	0.25
4		Cd	113.904	Peak Hopping	1	100	2000	Sn	Standard	0	0	0	0.25
5		Rh	102.905	Peak Hopping	1	50	1000		Standard	0	0	0	0.25
6													

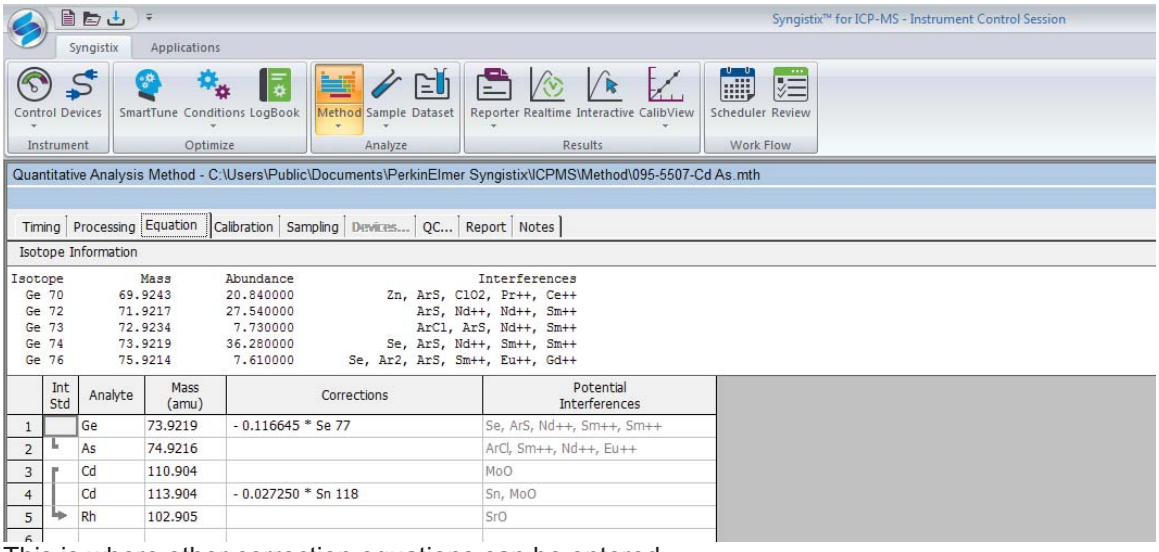
This is where the KED gas flow rates need to be changed if they are adjusted during tuning and optimization.

Figure 2: Screenshot showing Method parameters for Processing



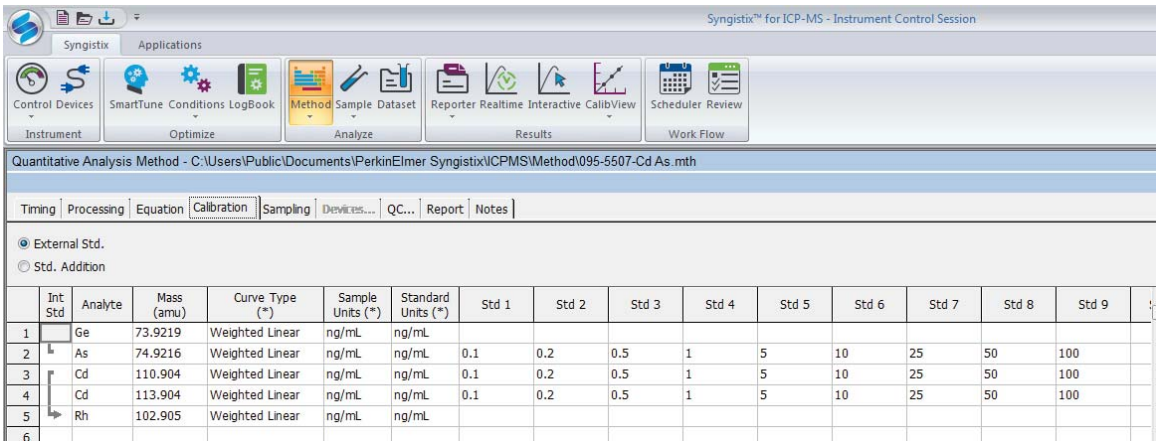
These parameters are not adjusted.

Figure 3: Screenshot showing Method parameters for Equation



This is where other correction equations can be entered.

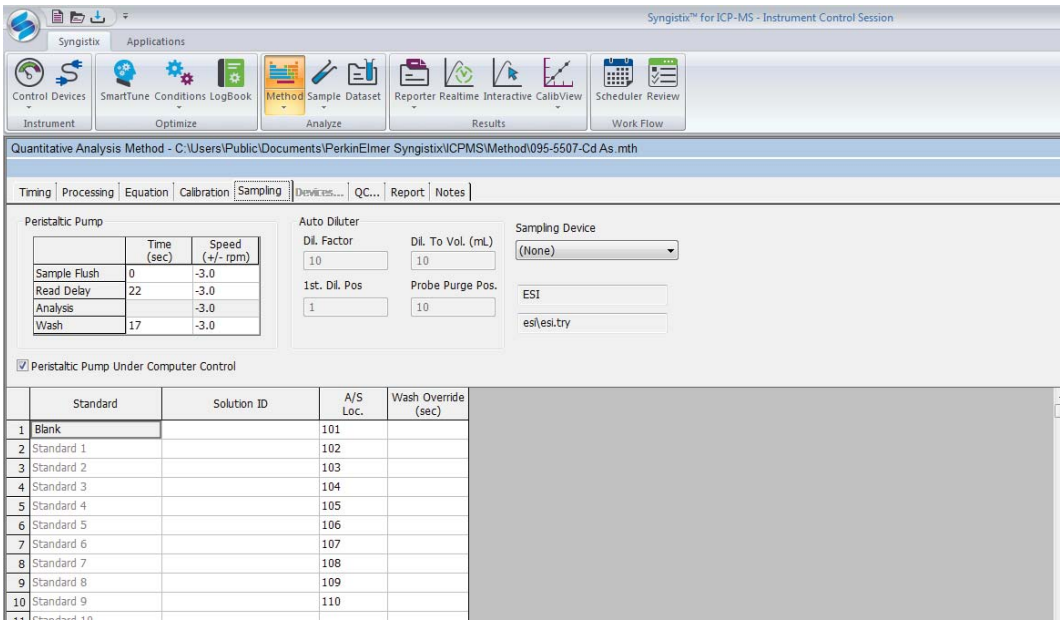
Figure 4: Screenshot showing Method parameters for Calibration



	Int Std	Analyte	Mass (amu)	Curve Type (*)	Sample Units (*)	Standard Units (*)	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	Std 7	Std 8	Std 9
1		Ge	73.9219	Weighted Linear	ng/mL	ng/mL									
2		As	74.9216	Weighted Linear	ng/mL	ng/mL	0.1	0.2	0.5	1	5	10	25	50	100
3		Cd	110.904	Weighted Linear	ng/mL	ng/mL	0.1	0.2	0.5	1	5	10	25	50	100
4		Cd	113.904	Weighted Linear	ng/mL	ng/mL	0.1	0.2	0.5	1	5	10	25	50	100
5		Rh	102.905	Weighted Linear	ng/mL	ng/mL									
6															

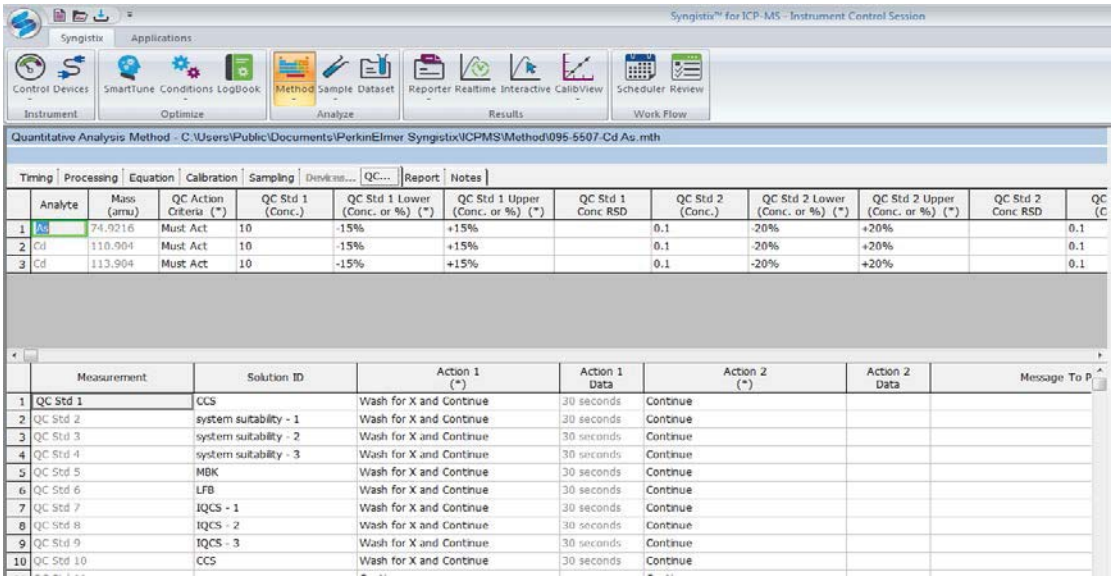
This is where the concentrations of the calibration standards are entered. There are no entries for the internal standard analytes.

Figure 5: Screenshot showing Method parameters for Sampling



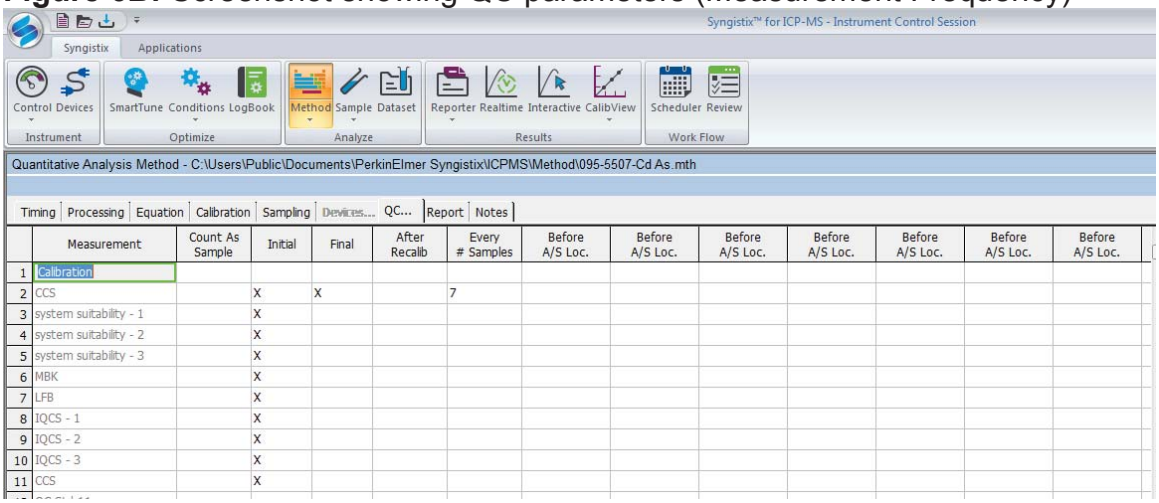
This is where alternate autosampler (A/S) locations can be specified.

Figure 6A: Screenshot showing Method parameters for QC (Stds)



This is where the “QC” samples are specified. These are automatically inserted into the batch run list when it is populated with samples.

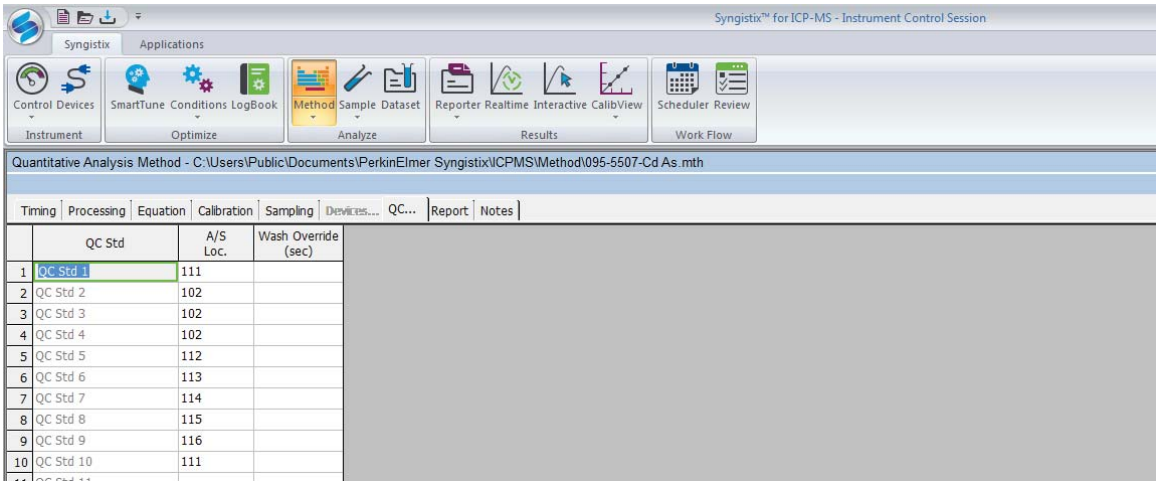
Figure 6B: Screenshot showing QC parameters (Measurement Frequency)



	Measurement	Count As Sample	Initial	Final	After Recalib	Every # Samples	Before A/S Loc.	Before A/S Loc.	Before A/S Loc.	Before A/S Loc.	Before A/S Loc.	Before A/S Loc.	Before A/S Loc.
1	Calibration												
2	CCS		X	X		7							
3	system suitability - 1		X										
4	system suitability - 2		X										
5	system suitability - 3		X										
6	MBK		X										
7	LFB		X										
8	IQCS - 1		X										
9	IQCS - 2		X										
10	IQCS - 3		X										
11	CCS		X										

This is where the frequency of CCS measurements is specified.

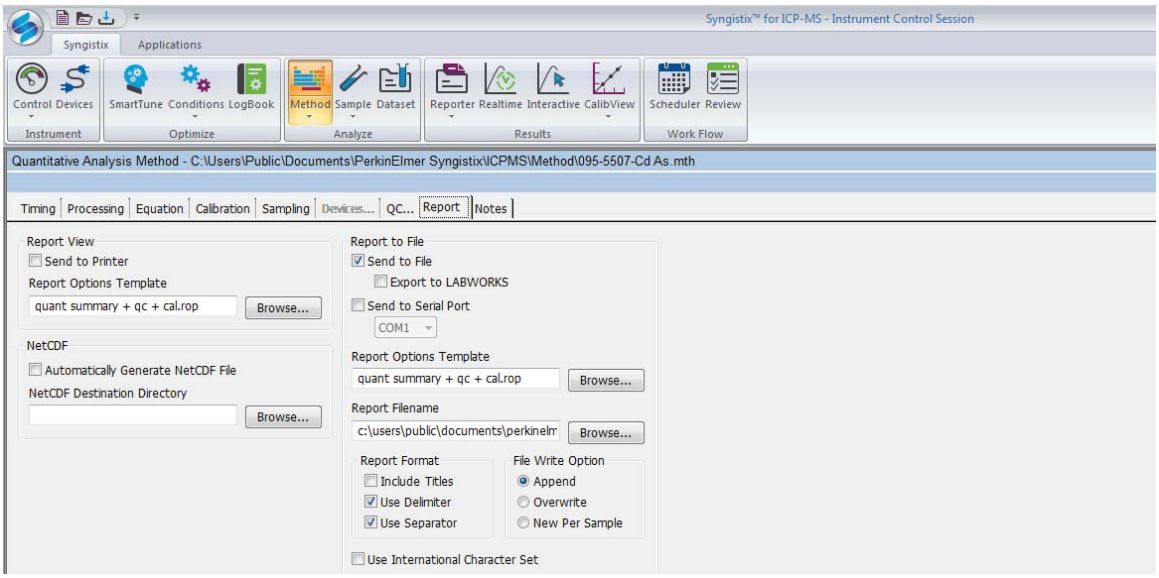
Figure 6C: Screenshot showing QC parameters (Autosampler)



	QC Std	A/S Loc.	Wash Override (sec)
1	QC Std 1	111	
2	QC Std 2	102	
3	QC Std 3	102	
4	QC Std 4	102	
5	QC Std 5	112	
6	QC Std 6	113	
7	QC Std 7	114	
8	QC Std 8	115	
9	QC Std 9	116	
10	QC Std 10	111	

These are the autosampler locations of the “QC” samples.

Figure 7: Screenshot showing Method parameters for Report



This is where the path to an alternate reporting template and storage location can be specified.