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Analysis of 20 elements in Platform 1 aerosol

PRODUCT TESTING LABORATORY AND GOVERNANCE

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1. DETERMINATION OF 20 ELEMENTS IN PLATFORM 1 AEROSOL

1.1. Abstract

The aerosol samples are generated with rotary smoking machine. The collection of aerosol particles is performed in a quartz electrostatic precipitation tube place in electrostatic precipitation unit, followed by a micro-impinger filled with a 65% nitric acid solution to collect the gas phase fraction. At the end of the smoking process, the impinger solution is transferred in the electrostatic precipitation unit and used to extract the precipitated aerosol particles. The extracts are then digested by microwave treatment with addition of hydrogen peroxide.

The resulting solutions are analyzed by Inductively Coupled Plasma (ICP) Mass Spectrometry (MS).

Results are expressed as ng/item for P1.

1.2. Applicability

The method described is used for the determination of 20 elements in Platform 1 (P1) aerosols generated under ISO or Health Canada (HC) conditions, or under alternative regimens. The elements investigated are: aluminum, arsenic, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, selenium, strontium, tin, titanium, tungsten and zinc.

1.3. Reagents

- Nitric acid
- Hydrogen peroxide
- Sodium bicarbonate
- Argon
- Helium
- Hydrogen
- Ethanol
- Ultrapure water
- Multi-element ICP standard solution
- Aluminum ICP standard solution
- Arsenic ICP standard solution
- Beryllium ICP standard solution
- Cadmium ICP standard solution
- Calcium ICP standard solution
- Chromium ICP standard solution
- Cobalt ICP standard solution
- Copper ICP standard solution
- Indium ICP standard solution
- Iron ICP standard solution
- Lead ICP standard solution

- Magnesium ICP standard solution
- Manganese ICP standard solution
- Molybdenum ICP standard solution
- Nickel ICP standard solution
- Selenium ICP standard solution
- Strontium ICP standard solution
- Tin ICP standard solution
- Titanium ICP standard solution
- Tungsten ICP standard solution
- Zinc ICP standard solution

1.4. Aerosol generation

P1 items are conditioned in opened packs for at least 48 hours at target conditions of 22 ± 1°C and relative humidity of 60 ± 3% before used for aerosol generation.

The aerosol samples are generated on a linear or rotary smoking machine under ISO or HC smoking regimens. The collection conditions for the different smoking regimes are summarized in Table **1**.

Table 1: Aerosol Collection Condition

Regimen	Accumulation number	Puff number	Regimen Condition [puff volume/Puff duration/Puff Interval] [ml/s/s]
ISO	10	6	35/2/60
HC	10	12	55/2/30

The collection of aerosol particles is performed in a quartz electrostatic precipitation (EP) tube placed in EP unit connected to the smoking machine. An electrostatic potential applied between the gold cathode and the external anodic cage determines the deviation and the precipitation of the aerosol particles in the EP tube.

The EP tube is followed by a micro-impinger filled with 5 mL of a 65% nitric acid solution, to collect the gas phase fraction of the aerosol. In order to protect the pump of the machine from the nitric vapours, this micro-impinger is followed by a second one, filled with 10 mL of a saturated sodium bicarbonate solution, and a Cambridge filter pad.

At the end of the smoking process, the EP tube is removed from the smoking machine and weighed; the difference between the weights after and before the smoking step correspond to the total particulate matter (TPM) of the tested items. Then, the EP tube is closed with two stoppers and transferred to the analytical laboratory for analysis with the corresponding micro-impinger.

Four replicates for each sample are generated. With each series of aerosol collections, three blanks are performed and analyzed to ensure that no contamination is carried out. These blanks are generating by performing the entire procedure described above while any item is charged on the smoking machine.

1.5. Samples preparation

The following sample preparation steps and the ICP-MS analysis are performed in a clean room under the ISO 7 classification.

The content of the micro-impinger is transferred into the EP tube. The micro-impinger is then rinsed with 1.5 mL of 65% nitric acid solution to collect possible residues. After this, the rinsing solution is combined with the content of the EP tube and shaked, in order to remove the aerosol condensate from the sides. Hence, 5 mL of the aerosol exctract are transferred into a digestion vessel liner and mixed with 2 mL of a 30% hydrogen peroxide solution. The digestion vessel is then closed and positioned in the microwave oven.

The mineralization process lasts for 70 minutes. After the mineralization, the digested aerosol solution is transferred into a 15 mL volumetric polypropylene tube and combined with about 2.5 mL of ultrapure water used to rinse the vessel. The tube is filled up to 15 mL with ultrapure water. This solution is called sample treated solution.

The preparation of dilution is detailed in Table **2** and are prepared in a 15 mL volumetric polypropylene tube. Blanks are diluted as the samples.

Table 2: Preparation of dilution for sample treated solutions

	Dilution
Sample treated solution	2000 µL
Internal standard solution	30 µL
Ultrapure water	12.97mL

1.6. Internal standard solution

The internal standard solution is prepared by dilution of an indium ICP standard solution with a 2% nitric acid solution.

1.7. Calibration solutions preparation

1.7.1. Elements stock solution

An intermediary elements stock solution is prepared by dilution of the single element ICP standard solutions of Co, Cd, Cr, Ni, Sn, As, Se, Sr and W with a 2% nitric acid solution. This solution expires the day of the preparation.

The elements stock solution is prepared by dilution of the intermediary elements stock solution with a 2% nitric acid solution and addition of precise amounts of Cu, Pb, Mn, Ti, Al, Fe, Mg, Zn and Ca single element ICP standard solutions.

1.7.2. Calibration standards

Seven standard (STD) solutions are prepared by dilution of the elements stock solution with a 2% nitric acid solution and addition of internal standard solution. The range of concentrations covers the ranges relevant for analysis and are provided in Table 3.

Table 3: Typical standard solution concentrations for the 20 elements of interest

Standard level	Conc. of Be, Mo [pg/mL]	Conc. of Co, Cd, Cr, Ni, Sn [pg/mL]	Conc. of As, Se, Sr, W [pg/mL]	Conc. of Cu, Pb, Mn, Zn [pg/mL]	Conc. of Al, Fe, Mg, Zn [pg/mL]	Conc. of Ca [pg/mL]
1	10	30	50	300	1000	10000
2	40	120	200	1200	4000	40000
3	75	225	375	2250	7500	75000
4	125	375	625	3750	12500	125000
5	250	750	1250	7500	25000	250000
6	375	1125	1875	11250	37500	375000
7	500	1500	2500	15000	50000	500000

1.8. Instrumental Conditions

The samples are analyzed by Inductively Coupled Plasma (ICP) mass spectrometry (MS) following tables below:

Table 4: Sample introduction settings

Spray chamber temperature	2°C
Carrier gas flow	~ 1.05 L/min, optimized daily with auto-tune
Nebulizer pump speed	0.1 rps

Table 5: Octopole Reaction System (ORS) conditions

Helium gas flow	~ 4.5 mL/min
Hydrogen gas flow	~ 4.5 mL/min

Table 6: Plasma conditions

ICP RF Power	~ 1550 Watts
Plasma gas flow	~ 15 L/min
Cones material and dimensions	Platinum, Ø 0.9 and 1.1 mm

1.9. Testing procedure

The following typical analytical sequence is used for the determination of 20 elements:

- 1 calibration blank (2% nitric acid solution with internal standard)
- Calibration curve (STD 1 to 7)
- Monitors, blanks and samples
- After every 15-20 samples, inject a quality check (STD level 4)
- Sequence ends with a quality check (STD level 4)

1.10. Verification fo results

1.10.1. Calibration curve

A calibration curve is used to quantify the unknown samples using the response ratio of analyte to the internal standard. The peak intensity ratio is applied to generate the curve. The linear regression with a weighting factor 1/x is calculated automatically by MassHunter software.

1.10.2. Quality Check

The validity of the calibration is continuously verified during the batch analysis by analysing the calibration control standard injections. All quality checks must be within ±15% of its theoretical value.

1.11. Example Spectra

N/A

1.12. Limit of Detection (LOD) / Lower Limit of Quantitation (LLOQ)

The LOD and LLOQ were both calculated in terms of the standard deviation of five different measurements of the lowest calibration standard for all other analytes produced under intermediate precision conditions (five different preparations from at least two different operators and analyzed on five different days).

$$LOD = 3 \times SD_{level1}$$

$$LLOQ = 10 \times SD_{level1}$$

Level 1 is the lowest calibration level.

LOQ is calculated based on aerosol collection blank variability to include the potential environmental contamination coming from the overall process (especially aerosol collection) for some elements.

Detailed values are provided in Table 7.

Table 7: LOD and LLOQ for ISO and HC smoking regimens

Matrix	Compound	LOD [ng/item]	LLOQ [ng/item]	STD1 [ng/item]	LOQ [ng/item]
P1	Beryllium	0.04	0.12	0.15	0.15
	Magnesium	1.08	3.59	14.63	14.63
	Aluminum	1.68	5.59	14.63	87.32
	Calcium	14.69	48.96	146.25	258.07
	Titanium	0.51	1.72	4.39	21.10
	Chromium	0.10	0.33	0.44	2.99
	Manganese	0.26	0.87	4.39	4.39
	Iron	0.97	3.24	14.63	17.10
	Cobalt	0.04	0.14	0.44	0.44
	Nickel	0.07	0.23	0.44	7.28
	Copper	0.36	1.19	4.39	4.39
	Zinc	1.07	3.55	14.63	42.79
	Arsenic	0.07	0.24	0.73	0.73
	Selenium	0.18	0.60	0.73	0.73
	Strontium	0.16	0.53	0.73	0.73
	Molybdenum	0.02	0.08	0.15	0.26
	Cadmium	0.04	0.15	0.44	0.44
	Tin	0.05	0.15	0.44	0.44
	Tungsten	0.14	0.47	0.73	0.81
	Lead	0.30	0.99	4.39	4.39

1.13. Repeatability limit (r) and Intermediate precision limit (IP)

$$r = 2 \cdot \sqrt{2} \cdot s_r$$

$$IP = 2 \cdot \sqrt{2} \cdot s_{IP}$$

s_r is the standard deviation of repeatability.

s_{IP} is the standard deviation of intermedidate precision.

Repeatability limit and intermediate precision limit are determined during four different days using different standard solutions preparation. Different operators are involved in the analysis.

Table 8: Repeatability (r) and intermediate precision (IP) for ISO smoking regimen

Matrix	Compound	r [ng/item]	IP [ng/item]	Mean [ng/item]
P1	Beryllium	NA	NA	<LOQ
	Magnesium	NA	NA	<LOQ
	Aluminum	NA	NA	<LOQ
	Calcium	NA	NA	<LOQ
	Titanium	NA	NA	<LOQ
	Chromium	NA	NA	<LOQ
	Manganese	NA	NA	<LOQ
	Iron	NA	NA	<LOQ
	Cobalt	NA	NA	<LOQ
	Nickel	NA	NA	<LOQ
	Copper	NA	NA	<LOQ
	Zinc	NA	NA	<LOQ
	Arsenic	NA	NA	<LOQ
	Selenium	0.479	0.479	0.92
	Strontium	NA	NA	<LOQ
	Molybdenum	NA	NA	<LOQ
	Cadmium	NA	NA	<LOQ
	Tin	NA	NA	<LOQ
	Tungsten	NA	NA	<LOQ
	Lead	NA	NA	<LOQ

Table 9: Repeatability (r) and intermediate precision (IP) for Health Canada smoking regimen

Matrix	Compound	r [ng/item]	IP [ng/item]	Mean [ng/item]
P1	Beryllium	NA	NA	<LOQ
	Magnesium	NA	NA	<LOQ
	Aluminum	NA	NA	<LOQ
	Calcium	NA	NA	<LOQ
	Titanium	NA	NA	<LOQ
	Chromium	NA	NA	<LOQ
	Manganese	NA	NA	<LOQ
	Iron	NA	NA	<LOQ
	Cobalt	NA	NA	<LOQ
	Nickel	NA	NA	<LOQ
	Copper	NA	NA	<LOQ
	Zinc	NA	NA	<LOQ
	Arsenic	NA	NA	<LOQ
	Selenium	0.527	0.685	1.31
	Strontium	NA	NA	<LOQ
	Molybdenum	NA	NA	<LOQ
	Cadmium	NA	NA	<LOQ
	Tin	NA	NA	<LOQ
	Tungsten	NA	NA	<LOQ
	Lead	NA	NA	<LOQ

1.14. NORMATIVE REFERENCES

- ISO 3308:2000 – Routine analytical cigarette smoking machine – definitions and standard conditions
- ISO 3402:1999 – Tobacco and tobacco products – atmospheres for conditioning and testing
- ISO 14644-1:2015 – Classification of air cleanliness