



**PROCEDURE FOR DETERMINING THE COMPOSITION OF ELEMENTS
IN PARTICULATE MATTER FROM MOTOR VEHICLE EXHAUST BY
ENERGY DISPERSIVE X-RAY FLUORESCENCE (EDXRF)
SPECTROSCOPY**

**SOP MV-AEROSOL-156 Version 2.2
Effective Date: September 1, 2019**

Aerosol Analysis and Methods Evaluation Section
Chemical Analysis and Emissions Research Branch
Mobile Source Laboratory Division

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1. Introduction

This document describes a non-destructive procedure using the Energy Dispersive X-Ray Fluorescence (EDXRF) spectroscopy to determine the composition of elements in particulate matter collected on Polytetrafluoroethylene (PTFE) membrane filters. When samples are irradiated by X-rays within the instrument sampling chamber, they emit X-ray fluorescence that is characteristic for each element. The intensity of emitted fluorescence energy is proportional to the surface concentration of each element (in units of $\mu\text{g}/\text{cm}^2$) and is used for their quantification.

The background X-ray spectrum of a blank filter is subtracted from that of the sample spectrum. The net intensity is proportional to the concentration of the corresponding element collected on the sample filter.

2. Interferences and Limitations

- 2.1. This procedure is capable of quantitative analysis of the non-volatile elements with atomic numbers 11 (sodium) through 92 (uranium). The 44 target elements that are measured in vehicle exhaust samples are listed in Table 1. The vacuum condition in EDXRF during sample analysis causes some loss of elements associated with volatile and semi-volatile compounds. For those elements, the accurate quantification cannot be achieved.
- 2.2. EDXRF method does not distinguish oxidation states; therefore only total elemental concentrations are quantified.
- 2.3. EDXRF spectral interferences cannot be avoided. The Epsilon 5 system uses secondary polarizing targets to minimize the effect of spectral overlapping.
- 2.4. Particulate matter is assumed to be deposited uniformly on the filter. The elements in the particulate matter which are not uniformly deposited over the whole collection area cannot be accurately quantified.
- 2.5. The procedure only applies to PTFE filters. Different type of filter media may exhibit severe background interference.

3. Safety

- 3.1. EDXRF instrument generates X-rays. The instrument has a built-in radiation shielding and safety interlock system with X-ray safety certifications. A Geiger counter is used to monitor for any radiation leaks.

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- 3.2. The EDXRF instrument uses liquid nitrogen to keep the X-ray detector at low temperature. The instrument's liquid nitrogen reservoir is refilled at least once a week. Liquid nitrogen is a cryogenic hazard and causes severe burns on contact with skin, eyes, or lungs. Wear protective gear (cryogenic gloves, safety glasses, lab coat) when handling liquid nitrogen (Reference: Occupational Safety and Health Administration).
- 3.3. Liquid nitrogen vapor displaces air while boiling off the liquid, and is an asphyxiation hazard. Keep the laboratory doors open when filling the liquid nitrogen reservoir.
- 3.4. For general laboratory safety procedures, consult the Chemical Hygiene Plan. Safety Data Sheets (SDS) are available in the laboratory.

4. Equipment and Supplies

- 4.1. The PANalytical Epsilon 5 High Performance EDXRF Spectrometer is used for the quantitative analysis of elements. This consists of the following instrument components:
 - 4.1.1 X-ray Tube: A scandium/tungsten (Sc/W) dual anode X-ray tube, with gadolinium (Gd) side window, with a water cooling system.
 - 4.1.2 HV Generator: 600 Watt maximum power, output voltage range 25-100 kV adjustable with steps of 1 kV, output mA range 0.5-24 mA adjustable by 0.1 mA.
 - 4.1.3 A 3-dimensional polarized optical path with nine automated polarizing secondary targets: These polarizing secondary targets and their corresponding measuring conditions are used for exciting target elements are listed in Table 2.
 - 4.1.4 X-ray Detector: A germanium (Ge) detector with beryllium (Be) window cooled by liquid nitrogen.
 - 4.1.5 Sample chamber equipped with automated controls: The chamber is in a vacuum environment during sample analysis.
 - 4.1.6 Computer system running on Windows 7 with PANalytical Epsilon 5 application software version 2.0N / ICSW 2.11.
- 4.2 Laboratory supplies and other Equipment:
 - 4.2.1 Step up transformer.
 - 4.2.2 Liquid nitrogen source with a pressure regulator at 0-50 psi.

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- 4.2.3 Cryogenic gloves, lab coat, safety glasses.
- 4.2.4 Latex gloves, disposable, class 100, powder free.
- 4.2.5 Stainless steel forceps with flat wide tips for transferring filters.
- 4.2.6 Solid sample holders (part #9430 050 01221) and sample holder inserts, for 50 mm filters (part #9430 050 11501)
- 4.2.7 Polycarbonate film, 6 microns thickness

5. Standards

5.1. Elemental Calibration standards:

- 5.1.1 These standards are thin vapor-deposited films of ultra-pure non-interfering elemental materials deposited on Nuclepore[®] polycarbonate aerosol membrane.
- 5.1.2 The calibration standard concentrations of the target elements are listed in Table 1.
- 5.1.3 Standards can be purchased from Micromatter, Inc. (4004 Westbrook Mall, Vancouver, BC, V6T 2A3).

5.2. Drift correction standard: FLX S13 (22/07/2011, 10, FLUXANA #D Elektronik).

6. Drift Correction

The X-ray tube and detector can degrade, and the response of the other components can also change over time. The drift correction is conducted once a month using the drift correction standard, FLX S13.

7. Calibration

- 7.1 The target elements are calibrated under their usual measurement conditions and utilize their usual secondary targets. Each element's measurement conditions and corresponding secondary target are listed in Table 2.
- 7.2 Calibration of all the elements is typically valid for one year.

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- 7.3 Recalibration of any elements may need to be performed if the quality control result of a particular element exceeds the limits, when the X-ray tube or the detector is replaced, or when there is a repair that can affect the calibration.

8. Sample Handling and Analysis

8.1 Sample Handling:

- 8.1.1 Sample filters are submitted to the laboratory and stored in the freezer until analyzed.
- 8.1.2 Filter samples are manually inspected before analysis. If irregularities are found, a senior staff shall be consulted. Any irregularities are recorded in the logbook.

8.2 Sample Analysis:

Two methods are used for EDXRF analysis. The primary method is used for the majority of analyses, including all gasoline vehicle exhaust samples. The supplemental method is intended primarily for diesel exhaust and other very high loading samples, and implements a layer of polycarbonate (PC) film over the sample to protect the instrument from possible contamination.

- 8.2.1 The analyst must be trained and familiar with the operation of the EDXRF system.
- 8.2.2 Create a sample list on the logbook and transfer the information to the "Sample Changer Measurement" window. Specify the number of repeats for each sample, and the sample rack position.
- i. For the primary method, select the "Whatman Air Filter" measurement application.
 - ii. For the supplemental method using PC film, select the "Whatman Air Filter – polycarbonate film" measurement application.
- 8.2.3 It is important that the deposit is not scraped, smudged or smeared when loading the filters into the sample holders.
- i. For the primary method, carefully transfer the filter with a clean forceps (with the deposit side up) to the sample holder

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insert. Place the sample holder cup over the filter to complete the sample assembly.



Place the sample assembly in the sample tray with the filter facing down, according to the position on the sample list. Place the tray inside the sampling chamber.

- ii. For the supplemental method using PC film, the filters are first loaded into customized filter pucks, with a sheet of polycarbonate film placed over the top of the filter surface.



With the supplemental method, the inner sample holder ring is not used.

8.2.4 Load sample cups into trays and place into instrument.



Primary method



Supplemental method

- 8.3 A typical analytical sequence includes a blank filter for background correction, a QC standard at the beginning of the sample batch, a sample replicate for every 10 samples or less, and a QC standard at the end of the sample batch.
- 8.4 When the analyses are completed, load the filters back into the sample containers for storage.

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9. Quality Control

9.1 Quality Control (QC) Standard: FLX S13

9.1.1 FLX S13 is analyzed before and after each set of samples. The limits are defined as follows:

Lower Control Limit (LCL) = mean value - 3 times the standard deviation
Lower Warning Limit (LWL) = mean value - 2 times the standard deviation
Upper Warning Limit (UWL) = mean value + 2 times the standard deviation
Upper Control Limit (UCL) = mean value + 3 times the standard deviation

9.1.2 The values for the control limits were calculated from the results of 20 analyses of the FLX S13, shown in Table 3.

9.1.3 QC values outside the control limit are considered as a “QC failure.” If any of the QC values fall between the control and warning limits, it is considered a QC “warning”. If the second QC value falls within the warning limits, it is considered a QC failure. QC failure requires corrective actions such as recalibration of the elements.

9.2 Replicate sample analysis

9.2.1 At least ten percent of the samples are randomly selected for replicate analysis. The relative percent difference (RPD) between the pair of analyses is calculated for each element above the reporting limit as follows:

$$RPD = \frac{|\text{Sample Conc.} - \text{Replicate Conc.}|}{\text{Average Conc. of Both Analyses}} \times 100$$

9.2.2 The limit on the allowable RPD is established based on the average concentration of the replicate runs, as shown in the following table:

Average Measurement for Replicate Runs	Allowable RPD (%)
1 to <3 times RL	= or <60%
3 to 5 times RL	= or <30%
Greater than 5 times RL	= or <10%

9.2.3 If the RPD of any of the target elements is greater than the allowable limit, the samples within the batch are re-analyzed.

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10 Reporting Limit

- 10.1 Data reporting for the elements is based on the significant figures of the calibration standards listed in Table 1.
- 10.2 The reporting limit (RL) is based on at least 30 blank filters analyzed from 2012 to 2014. RL is equal to the mean plus 3 times the standard deviation.
- 10.3 Sampling area of 11.34 cm² is used to convert the reporting limit from surface concentration to mass for each element for a 47mm PTFE filter. The reporting limits are listed in Table 4.
- 10.4 Any element with mass detected below the reporting limit will be reported as <RL.

11 Data Handling

- 11.1 Confirm that all QC criteria are met before reporting data. Otherwise, make corrections and re-analyze samples.
- 11.2 Data analysis is processed by the Epsilon 5 software.
- 11.3 Export the report generated by the Epsilon 5 software to an Excel file. Back up data periodically.
- 11.4 Raw data in µg/cm² from Epsilon 5 is transferred to the SLB LIMS, where it is converted to µg/filter (by multiplying the sample deposit area of 11.34 cm²) before reporting.

12 Reference standard

Standard Reference Material (SRM) 2783 from National Institute of Standards and Technology (NIST) is analyzed once a quarter. SRM 2783 is deposited on a polycarbonate membrane filter. A blank polycarbonate membrane filter (from NIST) is analyzed for background correction. Table 5 shows the NIST certified concentrations values and ARB values.

13 Maintenance

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- 13.1 It is important that the Epsilon 5 system stays ON (Epsilon 5, computer and Epsilon 5 software). If the system is shut down for more than 2 hours, a maintenance call might be needed.
- 13.2 The XRF detector requires routine calibration and continuous supply of liquid nitrogen. Fill the liquid nitrogen reservoir once a week and recalibrate the detector after every liquid nitrogen refill. Allow the detector temperature to stabilize for at least 30 minutes after the liquid nitrogen refill before initiating detector calibration.
- 13.3 Check the instrument status on the software display screen and alert lights on the instrument often. If the X-ray alert lights (yellow light on top of the Epsilon 5 EDXRF, light inside of the instrument loading bay, or LEDs on the front of the instrument housing) are OFF, it indicates that the X-ray tube is not on. Check for alert messages in the software, and call for service if necessary.

14. Registration of the X-Ray Tube Radiation

The X-Ray radiation registration renewal is required every two years. The Radiation Machine Registration renewal invoices are issued by the California Department of Public Health Radiologic Health Branch, MS 7610, Registration Unit P.O. Box 997414 Sacramento, CA 95899-7414.

15. References

- 15.1 U.S. Environmental Protection Agency, June 1999. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air , Compendium Method IO-3.3, *Determination of Metals in Ambient Particulate Matter using X-ray Fluorescence (XRF) Spectroscopy*, EPA/625/R-96/010a, Office of Research and Development
- 15.2 ARB SOP MLD 034, January 2006: *Standard Operating Procedure for the Determination of Elemental Concentrations in Ambient Air by Energy-Dispersive X-ray Fluorescence (XRF) Spectroscopy*
- 15.3 Epsilon 5 software Help manual: PANalytical, Incorporated, 117 Flanders Road, Westborough, MA 01581
- 15.4 California Code of Regulations (CCR), title 17, section 30108 <http://www.cdph.ca.gov/certlic/radquip/Pages/RadiologicEquipment.aspx>
- 15.5 Occupational Safety and Health Guidelines for Nitrogen <http://www.osha.gov/SLTC/healthguidelines/nitrogen/recognition.html>

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15.6 SLB Chemical Hygiene Plan, in draft, submitted to ARB safety officer for review and approval, December 2012.

16. Revision Record

Revision	Date	Responsible Person	Description of Change	Status
	January 2013	Luzviminda Salazar	Initial release	Version 1.0
1	August 2014	Erin Shields	Updated division and branch names	Version 1.1
2	January 2016	Erin Shields	Updated reporting limits, added supplemental method for diesel exhaust samples, and editorial changes	Version 2.0
3	December 2016	Erin Shields	Updated reporting limits, minor editorial changes	Version 2.1
4	September 2019	Inna Dzhema	Updated division name	Version 2.2

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**Table 1 Target elements and the corresponding calibration standard
concentration (Conc. uncertainty \pm 5%)**

Target Element 1	Conc. ($\mu\text{g}/\text{cm}^2$)	Target Element 2	Conc. ($\mu\text{g}/\text{cm}^2$)	Serial Number of Standards
sodium	6.02	chlorine	9.28	34063 NaCl
magnesium	7.26	fluorine	11.3	34064 MgF ₂
aluminum	22.2			34065 Al metal
aluminum	51.8			12906 Al metal
silicon	11.0	oxide	6.28	34066 SiO
phosphorous	6.80	gallium	15.3	34067 GaP
sulfur	5.94	copper	11.8	34068 CuS _x
potassium	6.90	chlorine	7.61	34069 KCl
calcium	8.11	fluorine	7.69	34070 CaF ₂
scandium	8.20	fluorine	10.4	34071 ScF ₃
titanium	17.7			34072 Ti metal
vanadium	20.8			34073 V metal
chromium	17.6			34074 Cr metal
manganese	18.7			34075 Mn metal
iron	14.8			34076 Fe metal
iron	49.4			12907 Fe metal
cobalt	15.7			34077 Co metal
nickel	17.0			34078 Ni metal
copper	16.9			34079 Cu metal
zinc	5.93	tellurium	11.6	34080 ZnTe
germanium	17.3			34081 Ge metal
selenium	25.6			30482 Se metal
cesium	12.2	bromine	7.36	34083 CsBr
rubidium	7.69	iodine	11.4	34084 RbI
strontium	11.9	fluorine	5.14	34085 SrF ₂
yttrium	13.5	fluorine	8.67	34086 YF ₃
molybdenum	11.5	oxide	5.74	34087 MoO ₃
rhodium	14.9			34088 Rh metal
palladium	17.6			34089 Pd metal
silver	19.6			34090 Ag metal
cadmium	10.7	selenium	7.55	34091 CdSe
indium	18.7			34092 In metal
tin	15.5			34093 Sn metal
antimony	16.4			34094 Sb metal
tellurium	18.0			34095 Te metal
barium	13.0	fluorine	1.80	34096 BaF ₂
lanthanum	15.2	fluorine	6.23	34097 LaF ₃
cerium	13.9	fluorine	5.67	34098 CeF ₃
thallium	16.4	chlorine	2.85	34099 TlCl
lead	19.5			34100 Pb metal
bismuth	16.5			34101 Bi metal
platinum	17.4			12908 Pt metal
zirconium	11.5	fluorine	9.57	12909 ZrF ₄

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Table 2 Nine polarizing secondary targets, the measuring conditions and the corresponding elements of interest

No.	Polarizing Secondary Target (Elemental composition)	Measuring Conditions				Element of interest
		kV	mA	Measured time (seconds)	Condition Name	
1	aluminum (Al)	25	24	500	Mg	Mg
2	calcium fluoride (CaF ₂)	40	15	600	Si-K	Al, Si, P, S, Cl, K
3	iron (Fe)	75	8	400	Ti-Cr	Ca, Sc, Ti, V, Cr
4	germanium (Ge)	75	8	400	Cu-Zn	Mn, Fe, Co, Ni, Cu, Zn
5	zirconium (Zr)	100	6	400	Rb_Re-Tl	Ga, Ge, Se, Br, Rb, Pt, Tl, Pb
6	molybdenum (Mo)	100	6	400	Sr-Y_Pb-U	Bi
7	silver (Ag)	100	6	400	Mo-Tc	Zr
8	cerium oxide (Ce ₂ O ₃)	100	6	400	I	Mo, Sn, Sb, Te, I
9	Barkla target aluminum oxide (Al ₂ O ₃)	100	6	500	Xe-La	Rh, Pd, Ag, Cd, In, Cs, Ba, La and Ce

Table 3 Quality control limits for selected elements

Element	Mean (ug/cm ²)	One standard deviation (ug/cm ²)	LCL (ug/cm ²)	LWL (ug/cm ²)	UWL (ug/cm ²)	UCL (ug/cm ²)
sulfur	1.28	0.03	1.19	1.22	1.34	1.37
chromium	8.77	0.05	8.62	8.67	8.87	8.92
iron	13.4	0.1	13.1	13.2	13.6	13.7
copper	27.1	0.1	26.8	26.9	27.3	27.4
lead	279	2	273	275	283	285

Lower Control Limit (LCL) = mean value - 3 times the standard deviation

Lower Warning Limit (LWL) = mean value - 2 times the standard deviation

Upper Warning Limit (UWL) = mean value + 2 times the standard deviation

Upper Control Limit (UCL) = mean value + 3 times the standard deviation

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Table 4 Reporting limit for the elements analyzed by EDXRF

Primary Method				PC film method			
Element	Abrv	RL (ug/sample)	RL (ug/cm2)	Element	Abrv	RL (ug/sample)	RL (ug/cm2)
magnesium	Mg	0.660	0.0582	magnesium	Mg	N/R	N/R
aluminum	Al	3.35	0.295	aluminum	Al	N/R	N/R
silicon	Si	0.500	0.0441	silicon	Si	0.900	0.0790
phosphorus	P	0.0900	0.00794	phosphorus	P	0.120	0.0106
sulfur	S	0.210	0.0185	sulfur	S	0.210	0.0185
chlorine	Cl	1.16	0.102	chlorine	Cl	1.16	0.102
potassium	K	0.330	0.0291	potassium	K	0.850	0.0750
calcium	Ca	0.700	0.0617	calcium	Ca	0.700	0.0617
scandium	Sc	0.450	0.0397	scandium	Sc	0.450	0.0397
titanium	Ti	0.150	0.0132	titanium	Ti	0.150	0.0132
vanadium	V	0.0100	0.000880	vanadium	V	0.0200	0.00176
chromium	Cr	0.100	0.00882	chromium	Cr	0.130	0.0115
manganese	Mn	0.0760	0.00670	manganese	Mn	0.180	0.0159
iron	Fe	0.970	0.0855	iron	Fe	0.970	0.0855
cobalt	Co	0.0600	0.00529	cobalt	Co	0.0600	0.00529
nickel	Ni	0.0380	0.00335	nickel	Ni	0.0800	0.00705
copper	Cu	0.0650	0.00573	copper	Cu	0.100	0.00882
zinc	Zn	0.500	0.0441	zinc	Zn	0.500	0.0441
gallium	Ga	0.0610	0.00538	gallium	Ga	0.0610	0.00538
germanium	Ge	0.0800	0.00705	germanium	Ge	0.0800	0.00705
selenium	Se	0.660	0.0582	selenium	Se	0.660	0.0582
bromine	Br	0.110	0.00970	bromine	Br	0.110	0.00970
rubidium	Rb	0.0730	0.00640	rubidium	Rb	0.0730	0.00640
strontium	Sr	0.0620	0.00550	strontium	Sr	0.0620	0.00550
yttrium	Y	0.0570	0.00500	yttrium	Y	0.0570	0.00500
molybdenum	Mo	0.570	0.0503	molybdenum	Mo	0.570	0.0503
rhodium	Rh	1.48	0.130	rhodium	Rh	1.48	0.130
palladium	Pd	0.420	0.0370	palladium	Pd	0.420	0.0370
silver	Ag	0.580	0.0511	silver	Ag	0.580	0.0511
cadmium	Cd	0.440	0.0388	cadmium	Cd	0.440	0.0388
indium	In	0.560	0.0494	indium	In	0.560	0.0494
tin	Sn	0.300	0.0265	tin	Sn	0.300	0.0265
antimony	Sb	0.180	0.0159	antimony	Sb	0.180	0.0159
tellurium	Te	0.880	0.0776	tellurium	Te	0.880	0.0776
iodine	I	1.22	0.108	iodine	I	1.22	0.108
cesium	Cs	1.05	0.0926	cesium	Cs	1.05	0.0926
barium	Ba	1.15	0.101	barium	Ba	1.15	0.101
lanthanum	La	1.24	0.109	lanthanum	La	1.24	0.109
cerium	Ce	1.35	0.119	cerium	Ce	1.35	0.119
thallium	Tl	0.130	0.0115	thallium	Tl	0.130	0.0115
lead	Pb	0.210	0.0185	lead	Pb	0.210	0.0185
bismuth	Bi	0.170	0.0150	bismuth	Bi	0.170	0.0150
platinum	Pt	0.200	0.0176	platinum	Pt	0.200	0.0176
zirconium	Zr	0.120	0.0106	zirconium	Zr	0.120	0.0106

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Table 5 Certified mass loading values of selected elements in NIST SRM 2783 for EDXRF

Element	NIST Certified Conc. (µg/cm ²)	NIST Conc. 95% uncertainty (µg/cm ²)	ARB Primary method 12/20/2016 (µg/cm ²)	ARB PC film method 7/21/2016 (µg/cm ²)
Aluminum*	2.33	0.12	2.31	n/a
Potassium	0.53	0.03	0.531	0.495
Calcium	1.325	0.066	1.31	1.19
Titanium	0.15	0.01	0.164	0.141
Chromium	0.014	0.001	0.0177	0.0105
Iron	2.661	0.133	2.70	2.65
Nickel	0.007	0.0004	0.0088	0.0064
Copper	0.041	0.002	0.043	0.0376
Zinc	0.18	0.01	0.192	0.181
Antimony**	0.0072	0.0004	<RL	<RL
Barium**	0.034	0.002	<RL	<RL
Lead	0.032	0.002	0.0315	0.0277

* Aluminum is only reported with conventional method

** Elements with concentrations below reporting limits