

Title: Multi-element Analysis of Ambient Aerosols Using Energy Dispersive X-ray Fluorescence Spectrometry		Copy No: ##
Method No.: 6.09/3.5/M	Effective Date: November 19, 2013	Location: ###

QSM Approval: _____

Multi-element Analysis of Ambient Aerosols Using Energy Dispersive X-ray Fluorescence Spectrometry

1. INTRODUCTION and SCOPE

- 1.1. The following describes the method for the multi-element analysis of ambient aerosols and other environmental samples deposited on Teflon[®] membrane filters (47-mm or 37-mm, 2 µm pore diameter, O-ring mounted).
- 1.2. The elements analyzed both quantitatively and semi-quantitatively are found in Appendix A. Detection limits (DLs) for the analysed elements range from 1 to 260 ng/cm². Expanded measurement uncertainties at the Quantitation limits (QLs = 3 x DLs) are in the range of 1 to 15 %.
- 1.3. This methodology is recommended for use only by or under the supervision of analysts experienced in the use of Energy Dispersive X-ray Fluorescence spectrometers (EDXRF) and in the interpretation of the resulting x-ray spectra.

2. SUMMARY of METHOD

- 2.1. EDXRF spectrometry is based on the measurement of fluorescent x-rays produced when an inner shell electron is ejected allowing a higher energy electron to drop into the lower energy orbital to fill the vacancy. The energy of the fluorescent x-ray is characteristic of the emitting element and the area of the fluorescent peak is proportional to the number of atoms in the sample. Quantitative determination is made through comparison with standards.
- 2.2. The two EDXRF spectrometers, used are the Panalytical Epsilon 5 DY714, and Panalytical Epsilon 5 DY827 (hereafter referred to as Panalytical), calibrated with thin metal foils and salts for the quantitative and semi-quantitative analysis of elements.
- 2.3. The spectrometer excitation conditions for elements of interest are optimized following the procedures outlined in:
 - “Panalytical Epsilon 5 System User’s Guide 4th ed. 2005.

Excitation conditions for each spectrometer are found in Appendix B.

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- 2.4. Spectra are acquired using manufacturer's software, and are stored for subsequent processing using the Epsilon 5 software.
- 2.5. Element concentrations and associated detection limits are reported in $\mu\text{g}/\text{cm}^2$ and $\mu\text{g}/\text{m}^3$.

3. SAMPLE REQUIREMENT and PREPARATION

- 3.1. Polytetrafluoroethylene (PTFE/Teflon[®]) membrane filters having a diameter of either 37 mm or 47 mm, with a 2 μm pore size and mounted on PMP o-rings are used.
- 3.2. The particulates are deposited on the opposite side of the o-ring.
- 3.3. EDXRF analysis provides its best performance when thin membrane filters with homogeneous deposits on their surfaces are used.
- 3.4. Particulate loading should be between 15 $\mu\text{g}/\text{cm}^2$ and 150 $\mu\text{g}/\text{cm}^2$.
- 3.5. Samples are stored in petri dishes in the X-ray laboratory until analysis. **Note:** Holding time is not applicable to these types of samples.

4. EQUIPMENT

- 4.1. Two Panalytical Epsilon 5 energy dispersive x-ray spectrometer are used (DY714 and DY827). The Panalytical systems are equipped with a 600 watt Gadolinium target tube.
- 4.2. The Panalytical instruments use secondary and polarizing targets for excitation.
- 4.3. In order to eliminate the attenuation of fluorescent radiation from light elements in the path between the sample and the detector's window, the Panalytical instrument measures samples under vacuum
- 4.4. The detectors, cooled by liquid nitrogen, are:
Panalytical DY714 and DY827 – PAN-32 Germanium with a 8 μm Be window having a resolution of <140eV (2000cps Mn Ka).
- 4.5. The Panalytical instruments are equipped with a 48 position horizontal tray

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5. STANDARDS

5.1. Calibration Standards

- 5.1.1. Energy Calibration is performed using the Panalytical “Beam Stop”-for the Panalytical instruments
- 5.1.2. Intensity Calibration is performed using multiple Micromatter[®] vacuum deposited thin film standards (see Appendix C).

5.2. Control Standards

- 5.2.1. The Breitlander multi element standards are used to verify that the spectrometers are operating within control limits and also for quality control. (see Appendix D)

6. CALIBRATION

- 6.1. There are 2 calibrations to be performed for the EDXRF spectrometers. The first is the energy calibration, which calibrates the electronics of the detecting circuitry, and the second is the intensity calibration, which calibrates the detected counts to correspond to the analytical concentration.

- 6.1.1. The system energy calibration is necessary for accurate element identification. Energy calibration aligns the amplifier so that for a peak generated from a known element, the centre of distribution of the peak falls on the correct energy for the excitation line of the known element. This is performed following the manufacturer’s guidelines:

“Panalytical Epsilon 5 System User’s Guide 4th ed. 2005, Section 6.6 Detector Calibration”

- 6.1.2. The intensity calibration of both Panalytical spectrometers is achieved by the analysis of a set of Micromatter[®] standards. These are a set of thin film standards on a Mylar substrate with a thickness of 6.3 microns. The standards cover a range of elements from sodium to uranium. Sensitivity/Intensity factors, (the number of x-ray counts/ $\mu\text{g}/\text{cm}^2$ of the element), are determined for each excitation condition. The software on both instruments calculates directly a calibration curve of Intensity vs. concentration for individual elements within the excitation condition, which is used for calculating unknowns.

- 6.1.3. It is useful for obtaining correct calibration coefficients to have as complete a set as possible.

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6.2. Calibration must be performed after any major changes to the instrument (e.g. replacement of the detector and/or the X-ray tube) or when data steadily falls beyond data acceptance limits (Section 9). The instrument's performance is monitored as outlined in SOP 19.03/*.*S, "Control Charts".

7. ANALYSIS and DATA PROCESSING

7.1. Prior to analysis, Teflon[®] membrane filters are weighed and conditioned according to "Determination of the Weight of Particulate Matter Collected on Teflon membrane Filters", Method 6.08/*.*M.

7.2. The potential hazards associated with x-ray fluorescence are outlined:

- "Panalytical Epsilon 5 System User's Guide 4th ed. 2005 Chapter 3 Safety".

7.3. The procedures for obtaining spectra are described in the manufacturer's operating manual and should be reviewed before operating the instrument:

- "Panalytical Epsilon 5 System User's Guide 4th ed. 2005.

7.4. Detailed steps for acquiring spectra, processing and report preparation can be found in:

- SOP 6.19/*.*S, "PANalytical Epsilon 5 EDXRF Spectral Acquisition, Processing and Reporting".

7.5. After analysis, Teflon membrane filters are sent for further analysis according to SOP 6.1/*.*S, "Sample Management".

8. DETECTION LIMITS and METHOD VALIDATION

8.1. Detection limits are calculated following the theory outlined by E.P. Bertin in "Principles and Practice of X-Ray Spectrometric Analysis".

8.2. Detection limits (DLs) are established from at least 30 replicate analyses of blank Teflon membrane filters and analysing the standard counting error of the background intensities.

The Instrument Detection Limit is determined using equation 1.

$$IDL = \frac{3\sqrt{Cts_b}}{Cts_s} \times L_s \quad (\text{Eq. 1})$$

Where,

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Cts_b = the background counts in analyte peak fitting region measured from the blank
 Cts_s = the peak counts in analyte peak fitting region measured from the calibration standard
 L_s = the loading of the analyte from the calibration standard

Detection limits for each element are in units of µg/cm² and µg/m³

- 8.3. The reported detection limits are reviewed at least once a year using historical data, and/or after any major modification of the EDXRF systems by determining detection limits from at least 30 replicate measurements of Blank filters (see the Panalytical Detection Limits files located in \XRF Lab\QC\Detection Limits\ and lab QC binder for printout).
- 8.4. Data validation is accomplished by EDXRF/EDXRF, EDXRF/IC and/or EDXRF/ICP-MS analysis comparison of the same samples and/or standards and by treating known Micromatter standards, of known concentrations, as unknowns.

This method is fit for the intended use.

9. QUALITY CONTROL

- 9.1. For the Panalytical E5 instruments, quality control consists of monitoring the parameters of a multi-element Breitlander standard and the x-ray tube beam stop. Weekly detector verification is performed after each filling of liquid nitrogen. This verification uses the beam stop and automatically corrects for drifts in peak centre. To confirm that this beam stop detector verification is functioning correctly, the MnO₂ standard is subsequently measured for peak resolution. The Breitlander standard is measured several times during the analysis of a batch of samples. The Mn Ka resolution and peak centre from this standard is plotted on a chart. Detector resolution must be less than 140eV (Mn Ka) as per manufacturer's specification, if not the manufacturer must be contacted for servicing. The Breitlander standard can also be run as a monitor ratio. This will automatically correct for any change in tube intensity below a drift value of 10. If the value is higher than 10, this indicates a hardware or software problem which necessitates contacting the manufacturer for servicing.
- 9.2. For both instruments, any resolution deviation beyond 2 sigma ' warning limit ' for one of the parameters warrants investigation. The raw spectra are compared to the previously measured Breitlander to discover any deformations of the spectra. If none are noted the results are kept. If the spectra are deformed, the standards and samples are re-measured.

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9.3. With any deviation beyond 3 sigma 'control limit' for one of the parameters, the procedures in section 9.3 are followed. If comparison of the spectra does not indicate any deformities, a record of non-conformance is created and the results are kept. If subsequent measurements continue to surpass 3 sigma, or multiple parameters surpass the 'control limit', recalibration may be called for.

9.4. Precision (the degree of mutual agreement among replicate determinations) provides an estimate of random error. Precision is expressed in terms of the percent relative standard deviation (%RSD) between replicate measurements of the Breitlander standards. Analysis precision encompasses the instruments stability, sample geometry and homogeneity. Precision varies with the element and concentration.

9.5. The accuracy (the degree of agreement with the "true" or most reliably known value) for this method is calculated as follows:

$$a = (m - t) / (t) \times 100 \quad \text{where: } a = \text{accuracy}$$

$$m = \text{mean value}$$

$$t = \text{true value}$$

Precision and Accuracy information for the EDXRF spectrometers can be found in the "Panalytical Precision and Accuracy" files located in directory \XRF Lab\Method Validation\QC\Precision and Accuracy\ and lab QC binder for printout.

9.6. Further quality control consists of the repeat measurement of samples. This comprises approximately 20% of all samples throughput, along with Teflon Lab Blank analysis with each batch of samples.

9.7. Two particulate laden Teflon[®] membrane filters are used as quality control standards in the determination of uncertainty (see Section 10).

9.8. Data Acceptance

9.8.1. The Breitlander standards measured with each run are used to monitor both the accuracy and the stability of the system. Accuracy should be within 30% and Precision 10%.

9.8.2. Duplicate samples should have a % Relative Error within 30% (for quantitatively calculated samples at concentrations greater than the QL).

9.8.3. Blanks should be below QL.

9.8.4. If the above data acceptance criteria are not met, further investigation is warranted, leading to repeat measurements of control, verification standards and samples.

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9.8.5. All quality control data is maintained and available for easy reference or inspection.

10. ESTIMATION of MEASUREMENT UNCERTAINTY

10.1. Potential uncertainty sources of this method are:

10.1.1. The EDXRF spectrometers' excitation conditions are set specifically for thin films. For heavily loaded filters, internal absorption of both the incident and emitted x-rays within the sample will cause the x-ray spectrum to be distorted. Therefore, optimum filter loading should be no greater than $150 \mu\text{g}/\text{cm}^2$ (approximately 1 mg/filter for 37-mm filters, and 2 mg/filter for 47-mm filters).

10.1.2. At low concentrations, counting statistics and signal noise will dominate the calculations of elemental concentrations. Filter deposits should be at least $15 \mu\text{g}/\text{cm}^2$.

10.1.3. The x-ray beam is focused on the central area of the filter. Since concentration results are extrapolated to the entire deposit area of the filter during data processing, it is essential that the analyzed area be representative of the entire deposit.

10.1.4. Polychromatic excitation results in a more complex spectrum, resulting in spectral overlap. The major overlap in condition 2 is As ($K\alpha$) and Pb ($L-\beta$). The overlaps in condition 3 are Al ($K-\alpha$) and Br ($L-\alpha$), S ($K-\alpha$) and Mo ($L\alpha$) and Pb ($M-\alpha$), S ($K-\alpha$) and Cl ($K-\alpha$) and P ($K-\alpha$). Other overlaps do exist but are insignificant to this analysis.

10.1.5. Absorption of both incident and emitted x-rays also occurs in the presence of large particles. This will affect the light elements analyzed (Al to S).

10.1.6. Additional uncertainty is introduced due to the potential variation in the manufactured thickness of the Teflon[®] membrane filters. An increased filter thickness will increase x-ray scattering, which increases the spectral background. This will in turn affect accurate background subtraction determination and quantification of light elements during data processing.

10.2. EDXRF uncertainty is calculated based on the Type A approach recommended by the Canadian Association for Laboratory Accreditation (CALA). Refer to SOP 2.10/*.*S for details. Uncertainty is estimated through the use of experimental data, specifically the replicate measurements of two particulate laden Teflon[®]

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membrane filters, the Breitlander standards and a blank Teflon[®] membrane filter.

11. MAINTENANCE

11.1. Liquid nitrogen is added to the detector Dewar weekly in accordance with:

- Panalytical Epsilon 5 System User's Guide 4th ed. 2005, Chapter 7 User Maintenance".

11.2. The vacuum pump oil is changed twice a year, in the Panalytical EDXRF system.

11.3. X-ray tube coolant is added when the liquid level in the reservoir drops to more than 1 inch below the return outlet, in the Panalytical EDXRF system.

12. APPLICABLE METHODS and SOPs

6.08/*./M	"Determination of the Weight of Particulate Matter Collected on Teflon Membrane Filters"
6.01/*./S	"Sample Management"
19.03/*./S	"Control Charts"
2.10/*./S	"Estimation of Uncertainty in Chemical Analysis"
6.19/*./S	"PANalytical Epsilon 5 EDXRF Spectral Acquisition, Processing and Reporting"

13. REVISIONS

- 1995:** Author: David Mathieu
Original Document: ING05 ver1.0, "Multi-element Analysis of Ambient Aerosols Using Pulse Height X-ray Fluorescence Spectrometry"
- Oct. 1997:** New method number assigned (ING05 ver1.0 changed to 3.1/2.0/M)
- Jan. 2002:** New version 3.1/3.1/M
- Oct. 2002:** Added Section 8 "Revisions".
Added reference to SOP 6.1/x.x/S "Sample Management" to Sections 4.2 and 7
New header added
- Oct. 2003:** New method number assigned (3.1/*./M changed to 6.9/1.0/M)
In the title "...Pulse Height..." changed to "...Energy Dispersive..."
Major revision of the format of the Method
Changed in Section 1 "Introduction and Scope"
Added Section 2 "Summary of Method"
Added Section 3 "Sample Requirements and Preparation"
Added Section 5 "Standards"

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- Changed in Sections 6, 7 and 8
- Added Section 9 "Detection Limits", Section 10 "Quality Control", Section 11 "Estimation of Uncertainty" and Section 13 "Applicable Methods and SOPs"
- Sep. 2006:** Major revision to method to include new Jordan Valley EDXRF instrument
 Removal of information from Section 7, to become a separate SOP
 Removal of Section 8 to become a separate SOP
 Subsequent sections renumbered accordingly
 The Kevex EDX-770 (referred to as System 1 - Model EDX-770) has been out of service as of January 27, 2005. This instrument is now used for spare parts, for the Kevex EDX-771.
- Sep. 2006 (2):** Section 3.5: delete "Samples should be analyzed within 3 months of reception to the X-ray Laboratory" and "Note: Holding time is not applicable to these types of samples" is added.
 Section 6.2: "Its performance is monitored as outlined in SOP 2.03/*.*S, "Control Charts" is added.
 Sections 9.3 and 9.4: "Refer to SOP 2.03/*.*S for details" is added
 Section 12: reference to SOP – 2.03/*.*S "Control Charts" is added
- Dec. 2007:** Section 8.1: Second sentence "Detection and Quantitation limits are reported for each element in units of $\mu\text{g}/\text{cm}^2$ " moved to section 8.3
 Section 8.3: Delete "Appendix A outlines typical method detection limits for the EDXRF spectrometers" and insert "Detection limits reported to the client are individual sample detection limits for each element in units of $\mu\text{g}/\text{cm}^2$ "
 Section 8.4: Renumbered to "8.5" and the new 8.4 inserted referring to review of detection limits.
 Appendix A. (Typical detection limits) removed and subsequent appendices corrected accordingly.
- Oct. 2008:** Major revision to method to include the new Panalytical Epsilon 5 spectrometer which replaces the Kevex EDX-771 which is now out of service.
 Section 1.2: Change reference from "Inorganic Section" to "Particulate Characterization". Re-insert reference to Appendix A (DL's) now that DL's are calculated using blanks not on individual samples. Correct all appendices labelling accordingly.
 Section 5.2.3, 9.3 and 9.5: Change reference from NBS Standards to new NIST Standard
 Section 8: All references to calculating individual detection limits for each sample deleted. New method for determining DL's based on blank values added, along with formula.
 Section 9.6: Reference to Accuracy and Precision appendix deleted, added sentence indicating where Accuracy and Precision data can be found on the computer and hardcopy.
 Section 9.1 and 9.2: Reference to Quality Control appendix deleted.
 Section 9.7: Add information on Lab blanks measured with each batch for QC.
 Section 9.9: New section on "Data Acceptance" added.
- Nov. 2008:** Add new reference section 14.6, referring to EPA document.
- Sep. 2009:** Section 3.1: Remove reference to name brand of PTFE filters.

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Section 5.1.1: Change BRPC3 standard to "beam Stop".
 Section 5.2.1, 9.1: Change BRPC3 to Breitlander.
 Section 5.2.2: Delete section, and place quality control information in section 5.2.1. Where Quality control checks with samples is now being performed with the Breitlander standards.
 Section 8.2: Add equation for calculating detection limit.
 Section 9.2: Add Breitlander standards as QC check for JV instrument.
 Section 9.3, 9.9.1: Change NIST to Breitlander.
Aug. 2011: Reviewer: John Colless, David Mathieu
 Section 1.2: Remove reference to Appendix A containing detection limit values.
 Section 2.4: Edited software name for both Instruments
 Section 5.2.1: Deleted references to NIST standards
 Section 5.2.2: Removed section as this set of Micromatter standards is now part of the Intensity Calibration.
 Section 9.1: Edited information to better reflect the process of "Quality Control"
 Section 9.5: Deleted reference to "NIST SRM" standard. Added "pure Copper" standard reference.
 Section 9.6: Edited pathway name to reflect where the appropriate files are located.
 Section 9.9.1: Added "pure Copper" to standards being used. Deleted "for the Breitlander standards"
 Appendix A: Change from specific detection limits to list of elements analysed.
 Appendix D: Removed "NIST and" from title and deleted chart "NIST SRM 2783"
 Appendix E: Combine information with Appendix C
 Appendix F: Delete the appendix showing specific Uncertainties and add range of uncertainties to section 1.2. Remove reference to this appendix from 10.2.

July 2013: Reviewer: John Colless, David Mathieu
 Minor revision caused by the retirement of the Jordan Valley spectrometer and addition of the new Panalytical Epsilon 5 DY827 spectrometer.
 Section 1.1 and subsequent: the word "membrane" to better describe the Teflon Filters
 Section 2.0: Removed reference to Jordan Valley spectrometer and addition of the Panalytical Epsilon 5 DY827 spectrometer.
 Section 4.0: Remove reference to Jordan Valley spectrometer and added Panalytical Epsilon 5 DY827 spectrometer.
 Section 5.1.1: Removed reference to Jordan Valley spectrometer.
 Section 6.1.1: Removed reference to Jordan Valley manual.
 Section 6.1.2: Removed reference to Jordan Valley spectrometer.
 Section 7.2: Removed reference to Jordan Valley manual.
 Section 7.3: Removed reference to Jordan Valley manual.
 Section 7.4: Removed reference to Jordan Valley SOP.
 Section 8.2: Changed-lower limit of detection (LLD) to Instrument Detection Limit (IDL).
 Section 8.3: Removed reference to Jordan Valley LLD file location.
 Section 9.1: Change calibration to verification

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Section 9.2: Removed reference to Jordan Valley quality control process, renumbered subsequent section.
 Section 9.4: Removed reference to Jordan Valley's pure copper standard.
 Section 9.5: Removed reference to Jordan Valley Precision and Accuracy file location
 Section 9.6: Removed reference to "over weekend" repeat measurements
 Section 9.7: Edited reference to Section 10 from Section 11.
 Section 9.8.1: Removed reference to Jordan Valley's pure copper standard.
 Section 10.1.3: Edited information to better reflect x-ray beam focus area.
 Section 10.1.5: Edited information to better reflect light elements analyzed.
 Section 10.2: Edited information to better reflect how uncertainty is calculated.
 Section 11.1: Removed reference to Jordan Valley manual.
 Section 11.4: Removed reference to Jordan Valley maintenance.
 Section 12.0: Removed reference to Jordan Valley SOP.
 Section 14.3: Removed reference to Jordan Valley manual.
 Section 14.4: Removed reference to Jordan Valley manual.
 Appendix A: Edited to reflect list of elements analysed.
 Appendix B: Edited to reflect excitation conditions that are in elements analysed.

Nov. 2013: Reviewer: John Coltess,
 Minor editorial changes are made throughout the document.
 Section 6.2: Changed to include details of conditions that may alter the sensitivity of the instrument; the reference to SOP 2.03/*.* /S changed to SOP 19.03/*.* /S;
 Sections 9.2 and 9.3: Reference to SOP 19.03/*.* / is removed

14. REFERENCES

- 14.1. "Panalytical Epsilon 5 System User's Guide 4th ed. 2005"
- 14.2. "Panalytical EDXRF Epsilon 5 Training Manual. 2005"
- 14.3. "Principles and Practice of X-Ray Spectrometric Analysis" by E.P. Bertin, 2nd edition.
- 14.4. "EPA Quality Assurance Document, Compendium Method IO-3.3, Determination of Metals in Ambient Particulate Matter using X-Ray Fluorescence (EDXRF) Spectroscopy, EPA/625/R-96/010a, June 1999"

Lead Reviewer: David Mathieu, John Coltess
 Title: X-ray Analysis Laboratory, Particulate Characterization

Approved by: Ewa Dabek
 Title: Head, Particulate Characterization

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Appendix A
EDXRF Elements Reported

Al *	Se *
Si	Br
S	Rb
K	Sr
Ca	Cd
Ti	Sn
V	Sb
Cr	Cs
Mn	Ba
Fe	Pb
Ni	
Zn *	

*Elements concentrations are semi-quantitative due to spectral interferences

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Appendix B
EDXRF Spectrometer Excitation Conditions

Panalytical
Epsilon 5

Condition	Excitation	Target	Atmosphere	Acquisition (sec)	Potential (kV)	Current (mA)	Transmission Filters	Range KeV	Resolution (eV/Channel)
1	Secondary	CaF ₂	Vacuum	600	40	15	none	20	5
2	Secondary	Fe	Vacuum	400	75	8	none	20	5
3	Secondary	Ge	Vacuum	400	75	8	none	20	5
4	Secondary	Zr	Vacuum	400	100	6	none	20	5
5	Secondary	Mo	Vacuum	200	100	6	none	20	5
6	Secondary	CsI	Vacuum	400	100	6	none	80	5
7	Secondary	BaF ₂	Vacuum	200	100	6	none	80	5
8	Secondary	Al ₂ O ₃	Vacuum	400	100	6	none	80	5

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Appendix C Micromatter Standards Composition

Micromatter Standard	Element 1	Conc. $\mu\text{g}/\text{cm}^2$	Element 2	Conc. $\mu\text{g}/\text{cm}^2$	Micromatter Standard	Element 1	Conc. $\mu\text{g}/\text{cm}^2$	Element 2	Conc. $\mu\text{g}/\text{cm}^2$		
16378	MgF2	Mg	5.8	F	9.1	16417	PrF3	Pr	9.3	F	3.7
16379	Al	Al	12.7			16418	NdF3	Nd	10.0	F	4.0
16380	SiO	Si	10.1	O	5.7	16419	SmF3	Sm	12.1	F	4.6
16381	GaP*	Ga	10.0	P	4.4	16420	EuF3	Eu	9.9	F	3.7
16382	CuS	Cu	10.3	S	3.5	16421	GdF3	Gd	12.4	F	4.5
16383	NaCl	Na	5.4	Cl	8.4	16422	TbF3	Tb	12.4	F	4.4
16384	KCl	K	7.3	Cl	6.6	16423	DyF3	Dy	10.7	F	3.8
16385	CaF2	Ca	7.3	F	6.9	16424	HoF3	Ho	10.5	F	3.6
16386	ScF3	Sc	6.6	F	8.3	16425	ErF3	Er	10.3	F	3.5
16387	Ti	Ti	13.4			16426	TmF3	Tm	13.5	F	3.4
16388	V	V	11.7			16427	YbF3	Yb	12.5	F	4.1
16389	Cr	Cr	9.4			16428	LuF3	Lu	10.9	F	3.6
16390	Mn	Mn	14.5			16429	WO3	W	13.1	O	3.4
16391	Fe	Fe	13.9			16430	Pt	Pt	11.4		
16392	Co	Co	12.9			16431	Au	Au	14.7		
16393	Ni	Ni	10.5			16432	TlCl	Tl	11.8	Cl	2.0
16394	Cu	Cu	11.7			16433	Pb	Pb	10.2		
16395	ZnTe*	Zn	5.2	Te	10.1	16434	Bi	Bi	15.9		
16396	Ge	Ge	12.8			16435	ThF4	Th	9.6	F	3.1
16397	GaAs*	Ga	7.5	As	8.1	16436	UF4	U	12.4	F	4.0
16398	Se	Se	12.7			16437	MgF2	Mg	20.6	F	32.1
16399	CsBr	Cs	8.0	Br	4.8	16438	Al	Al	39.0		
16400	RbI	Rb	5.4	I	8.0	16439	SiO	Si	24.9	O	14.2
16401	SrF2	Sr	9.5	F	4.1	16440	GaP*	Ga	26.7	P	11.8
16402	YF3	Y	9.0	F	5.7	16441	CuS	Cu	44.3	S	12.4
16403	Nb2O3	Nb	9.8	O	2.5	16442	NaCl	Na	18.4	Cl	28.3
16404	MoO3	Mo	10.1	O	5.1	16443	KCl	K	23.9	Cl	21.6
16405	Rh	Rh	12.7			16444	CaF2	Ca	24.1	F	22.9
16406	Pd	Pd	15.4			16445	ScF3	Sc	21.2	F	26.9
16407	Ag	Ag	15.2			16446	Ti	Ti	45.1		
16408	CdSe*	Cd	9.3	Se	6.6	16447	V	V	48.1		
16409	In	In	15.0			16448	Cr	Cr	48.2		
16410	Sn	Sn	16.0			16449	Mn	Mn	45.2		
16411	Sb	Sb	12.7			16450	Fe	Fe	45.3		
16412	Te	Te	13.2			16451	Co	Co	45.2		
16413	KI	K	3.3	I	10.7	16452	Ni	Ni	44.2		
16414	BaF2	Ba	11.6	F	3.2	16453	Cu	Cu	46.7		
16415	LaF3	La	10.9	F	4.5	16454	ZnTe*	Zn	18.1	Te	35.2
16416	CeF3	Ce	10.7	F	4.3	16455	Ge	Ge	46.7		

The following information is valid for the Micromatter® standards:
 Deposit diameter 31mm
 O-ring thickness 1.5mm
 Outside diameter 37mm
 Inside diameter 30mm

* Not Stochiometric

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Appendix C Continued
Micromatter Standards Composition

Micromatter Standard	Element 1	Conc. µg/cm ²	Element 2	Conc. µg/cm ²
2436	Mg	48		
2437	Al	57		
2438	SiO	29.94		
2439	GaP	12.3	Ga	27.7
2441	CuS	50	S	13
2442	NaCl	20.1	Cl	30.9
2443	KCl	25.2	Cl	22.8
2444	CaF ₂	27.7	F	26.3
2445	ScF ₃	16.8	F	21.2
2446	Ti	37		
2447	V	33		
2448	Cr	62		
2449	Mn	51		
2450	Fe	37		
2451	Co	54		
2452	Ni	66		
2453	Cu	52		
2454	ZnTe	13.9	Te	27.1
2455	Ge	49		
2456	GaAs	7.6	As	16.4
2458	Se	46		
2459	CsBr	29.4	Br	17.6
2460	RbNO	24.9		
2461	SrF ₂	35.6	F	15.4
2462	YF ₃	29.9	F	19.1
2463	Nb ₂ O	33.4	O	8.6
2464	MoO ₃	36	O	18
2465	Rh	49		
2466	Pd	54		
2467	Ag	36		
2468	CdSe	24.7	Se	17.3
2469	In	49		
2470	Sn	47		
2471	Sb	57		
2472	Te	50		
2473	KI	11.8	I	38.2

Micromatter Standard	Element 1	Conc. µg/cm ²	Element 2	Conc. µg/cm ²
2474	BaF ₂	32.1	F	8.9
2475	LaF ₃	41.1	F	16.9
2476	CeF ₃	24.9	F	10.1
2477	PrF ₃	37.7	F	15.3
2478	YbF ₃	38.4	F	12.6
2479	LuF ₃	42.2	F	13.8
2480	WO ₃	43.6	O	11.4
2481	Pt	43		
2482	Au	46		
2483	AgHg	15.4	Hg	39
2484	TlCl	40.9	Cl	7.1
2485	Pb	26		
2487	Bi	41		
2488	ThF ₄	39.9	F	13.1
2489	UF ₄	34.1	F	10.9
6496	ZnTe	17.14	Te	33.46
6497	CsBr	31.66	Br	19.04
6499	KI	10.76	I	34.94
6501	CuS	17	S	5.2
6502	ZnTe	6.71	Te	13.09
6506	Pb	18.8		
6507	CuS	17.1	S	5.5
6508	ZnTe	6.44	Te	12.56
6509	CsBr	13.24	Br	7.96
6511	KI	5.42	I	17.58
6512	Pb	19.1		
6513	NdF ₃	33.19	F	13.11
6514	SmF ₃	31.9	F	12.1
6515	EuF ₃	35.64	F	13.36
6516	GdF ₃	33.18	F	12.02
6517	TbF ₃	34.08	F	12.22
6518	DyF ₃	31.31	F	10.99
6519	HoF ₃	33.96	F	11.74
6520	ErF ₃	33.26	F	11.34
6521	TmF ₃	33.5	F	11.3

The following information is valid for the Micromatter® standards:

- Deposit diameter 31mm
- O-ring thickness 1.5mm
- Outside diameter 37mm
- Inside diameter 30mm



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**Appendix D
 Breitlander Standards Composition**

Breitlander Silicate Glass Disks

Constituent	A3	B2	C3	D2	E2	F2	Constituent	A3	B2	C3	D2	E2	F2
	Mass %	Mass %	Mass %	Mass %	Mass %	Mass %		Mass %	Mass %	Mass %	Mass %	Mass %	Mass %
B2O3	4.2	-	19.1	22.2	4	2	Y2O3	-	-	-	-	0.18	0.45
F	0.17	1.4	-	-	1.3	5	ZrO2	0.15	-	-	0.34	-	0.74
Na2O	0.13	0.09	7.9	9.6	14.6	1.2	Nb2O5	-	-	0.6	-	0.05	0.38
MgO	3.2	0.23	-	7.4	-	0.82	MoO3	-	-	2	0.87	-	-
Al2O3	15.8	6.75	27.1	20	13.2	3.85	Ag2O	-	-	-	-	0.13	-
SiO2	36.5	42.9	9.9	5.3	46.4	57.6	CdO	0.39	-	0.16	-	-	0.96
P2O5	0.58	2.1	15.6	5.8	-	-	In2O3	0.04	-	-	-	0.09	0.26
K2O	2.16	0.04	6.9	0.09	0.95	18.4	SnO2	-	0.92	-	-	0.6	0.2
CaO	0.83	21.4	0.03	14.4	0.6	2.84	Sb2O3	-	-	-	1.85	0.43	0.86
TiO2	3.9	1.2	0.1	0.03	0.02	0.04	TeO2	0.04	0.08	-	-	0.03	-
V2O5	0.01	-	0.26	0.86	-	1.7	Cs2O	0.04	-	-	-	-	0.13
Cr2O3	0.15	-	-	-	0.56	0.27	BaO	2	0.04	1	-	4.6	0.34
MnO	20.3	0.89	0.47	-	0.2	-	La2O3	-	-	-	0.88	0.4	-
Fe2O3	1.16	12.3	5.4	0.58	0.03	0.07	Ce2O3	-	-	-	0.84	-	0.39
CoO	-	1.62	-	-	0.74	0.25	Pr2O3	-	-	0.2	-	-	-
NiO	-	0.79	0.29	-	1.85	-	Nd2O3	-	-	0.46	-	-	-
CuO	-	0.25	-	-	0.82	1.8	Sm2O3	-	-	-	-	-	0.18
ZnO	7.4	0.45	-	3.7	0.92	-	Ta2O5	-	0.85	-	-	0.05	0.36
Ga2O3	-	-	-	0.46	-	0.09	WO3	-	1.85	0.9	0.32	-	-
GeO2	0.08	-	0.27	0.41	-	-	PbO	-	4.4	-	1.7	0.45	0.05
As2O3	-	-	0.78	1.86	0.44	-	Bi2O3	-	-	0.5	0.18	0.08	-
Rb2O	0.04	-	-	-	-	0.16	ThO2	0.04	-	-	0.18	-	0.33
SrO	0.71	0.008	-	0.13	0.31	-	UO2	0.01	-	-	-	0.44	0.27

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