# *CHESTER LabNet*

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Standard Operating Procedure
XR-004.01

KEVEX XRF SPECTROMETER CALIBRATION
CHESTER LABNET PROPRIETARY METHOD

Approvals:

| | |
|---|---|
| Author | 11-13-07<br>Date |
| Lead Analyst | 1/8/08<br>Date |
| QA/QC | 1/8/08<br>Date |

Effective from: 1-8-08
Effective until: present

# REVIEW HISTORY

| Review date: | Changes made: | Changes made by: |
|---|---|---|
| | | |
| 11/12/07 | Updated excitation conditions for low density filter media to reflect current practices, typographical errors corrected. | Sheri Heldstab |
| 9/8/05 | Updated to NELAP formatting. | Sheri Heldstab |
| 9/8/04 | Changes made to correct errors in some calculations. Minor changes to text for clarification. | Sheri Heldstab |
| 7/1/03 | Minor changes to text. | Sheri Heldstab |
| 4/22/02 | No changes. Date of origination. Original text found in SOP XR-002, written by Rick Sarver in 1998. | Sheri Heldstab |

# ANNUAL REVIEW

The undersigned attests that this standard operating procedure has undergone annual review for adherence to current practices and the latest QA/QC protocols:

| signature | title | date |
|---|---|---|
| Rick Sarver | Senior XRF Analyst | 1-2.09 |
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# KEVEX XRF SPECTROMETER CALIBRATION CHESTER LABNET PROPRIETARY METHOD

## 1.0 Introduction

1.1 Test Method Reference ID: Chester LabNet Proprietary Method.

1.2 Applicability: This method is applicable to the calibration of Kevex Model Delta 770 energy dispersive XRF (EDXRF) for all thin film matrices analyzed by Chester LabNet.

1.3 Detection Limit: N/A

1.4 Method Performance: N/A

## 2.0 Summary

2.1 Scope and Application: This method is applicable to the calibration of Kevex Model Delta 770 energy dispersive XRF (EDXRF). Chester LabNet currently operates two of these instruments, informally named "770" and "772". The process by which each instrument is calibrated is identical.

2.2 Summary of Method:

2.2.1 The instruments are configured for the analytes of interest, then a series of standards and matrices are analyzed to determine the peak energies of each analyte. A response factor for each element at each excitation utilized condition is determined, and that factor is entered into the IXRF operational software.

2.2.2 Also determined are interelemental interferences and matrix differences. These factors are entered into the LabNet proprietary software, which corrects for these issues.

2.2.3 Full calibration will take a minimum of 2 working days to complete.

2.2.4 Calibration should be performed only by trained operators with experience in data interpretation and instrument functioning. This SOP is designed only as a guide, and should not be used in lieu of proper training.

2.3 Interferences: As with all emissions spectrometry, secondary, tertiary, quarternary emission lines may overlap primary lines depending on the amount of interferent present.

2.4 Sample collection/preservation/shipment/storage: Thin film standards are stored at room temperature in the weigh room.

## 3.0 Safety

3.1 The Chester LabNet Chemical Hygiene plan should be followed. Samples of unknown origin and/or constitution should always be treated as hazardous.

3.2 This method presents no safety risk beyond typical laboratory safety hazards.

3.3 No carcinogenic reagents are used in this method.

3.4 Although X-rays are produced by the instrument at dangerous levels, the X-rays will not turn on unless a built-in interlock is fully locked, thus ensuring the user is shielded from the X-rays. Users should never remove the front cover of the instrument while the X-rays are operational, nor should they ever attempt to disable the interlock device. All users wear dosimeter badges. To date, Chester LabNet has never had a dosimetry badge showing any radiological exposure of any XRF technician.

## 4.0 Pollution Prevention and Waste Management

4.1 The smallest quantity of chemical feasible is removed from its primary container for use.

4.2 Chemicals are used in amounts needed by the method, and excess reagents are not made.

4.3 Chester LabNet is a conditionally exempt small quantity generator and as such does not require formal chemical waste processing.

4.3.1 Acidic and Basic wastes are neutralized prior to disposing of them in the sanitary sewer system.

4.3.2 Organic liquids are usually primarily used for cleaning purposes. Organic wastes are generated in very small quantities, and evaporate off with no need for more formal disposal.

4.4 Larger quantities of known hazards are returned to the client for disposal.

4.5 Expired Chemicals:

4.5.1 Dry chemicals beyond their real or arbitrary expiration date are lab packed and disposed of by a qualified chemical disposal company.

4.5.2 Acids and Bases beyond their real or arbitrary expiration date are neutralized prior to being disposed of via the sanitary sewer system.

4.5.3 Organic liquids beyond their real or arbitrary expiration date are disposed of by a qualified chemical disposal company if the volume or type of liquid warrants such disposal. Disposal of organic liquids is rare.

## 5.0 Apparati, Equipment and Supplies

5.1 Instrument. Kevex Model Delta 770 energy dispersive XRF (EDXRF) equipped with a water-cooled end-window X-ray tube with a rhodium anode and a peak operating power of 60kV and 3.3 mA. The system was modified by IXRF Inc. to operate using Windows based software to control instrument operation. Chester LabNet currently operates two of these instruments, informally named "770" and "772". 770 is the original instrument. Its computer operates on Windows 95 772 operates on Windows 98. Both instruments use an Ethernet connection to transfer data files to the data processing computer.

5.2 Thin Film Calibration Standards

5.2.1 Micromatter Inc. vapor deposit of single or two non-interfering elements onto thin mylar film.

5.2.2 EPA organo-metallic acetate film, usually with two non-interfering elements.

5.2.3 NIST SRM's 1832 and 1833 multi-element vapor deposits on glass films.

## 6.0 Reagents and Standards

6.1 95% ethanol

6.2 Liquid Nitrogen

## 7.0 Preparation, Calibration and Standardization

7.1 Ensure the proper excitation conditions are configured within the IXRF software. Different excitation conditions are used in order to maximize the sensitivity of the analysis over a wide range of elements fluorescing at various energies. If only one energy is listed below, it is used for both instruments. The analytes appearing in **bold** type are in their most favorable excitation condition.

7.1.1 Excitation conditions for 'low-density' filter media (Teflon, Nuclepore, Mylar).

7.1.1.1 sp0: direct Rh L$\alpha$ excitation with one thickness of Whatman no. 41 cellulose used as a prefilter to absorb low-energy X-rays from source. Energy: 7.5 kV. Analytes of interest: **Na, Mg, Al, Si, P, S**. Tube geometry (incident beam angle): $\theta = 21°$.

7.1.1.2 sp1: Energy: 20 kV. Analytes of interest: Al, Si, P, **S, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe,** Co, **Cs, Ba, La, Ce.**. Tube geometry: $\theta = 21°$

7.1.1.3 sp2: Ge secondary target. Energy 35 kV for 770, 30kV for 772. Analytes of interest: K, Ca, Ti, V, Cr, Mn, Fe, **Co, Ni, Cu, Zn, Sm, Eu, Tb**. Tube geometry: $\theta = 45°$

7.1.1.4 sp3: direct Rh K$\alpha$ excitation with .1 mm Rh foil prefilter. Energy: 35 kV. Analytes of interest: K, Ca, Ti, V, Cr, Mn, Fe, **Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Hf, Ta, W, Ir, Au, Hg, Pb**. Tube geometry:

$\theta = 21°$

7.1.1.5 sp4: direct Rh bremstrahlung excitation with .1 mm W foil prefilter. Energy: 55 kV. Analytes of interest: **Rh**, **Pd, Ag, Cd, In, Sn, Sb**, **Te, I,** Cs, Ba, La, Ce. Tube geometry: $\theta = 21°$

7.1.2 Excitation conditions for 'high-density' filter media (quartz, glass fiber, carbon impregnated).

7.1.2.1 sp0: Fe secondary target. Energy: 35 kV for 770, 25kV for 772. Analytes of interest: **Al, Si, P, S, Cl, K, Ca, Ti, V, Cr.** Tube geometry: $\theta = 45°$

7.1.2.2 sp1: Ge secondary target. Energy 35 kV for 770, 30kV for 772. Analytes of interest: K, Ca, Ti, V, Cr, **Mn, Fe, Co, Ni, Cu, Zn**. Tube geometry: $\theta = 45°$

7.1.2.3 sp2: direct Rh K$\alpha$ excitation with .1 mm Rh foil prefilter. Energy: 35 kV. Analytes of interest: K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, **Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Mo, Hg, Pb**. Tube geometry: $\theta = 21°$

7.1.2.4 sp3: direct Rh bremstrahlung excitation with .1 mm W foil prefilter. Energy: 55 kV. Analytes of interest: **Rh**, **Pd, Ag, Cd, In, Sn, Sb**, **Te, I, Cs**, **Ba, La, Ce**. Tube geometry: $\theta = 21°$

7.2 Preparation of standards for analysis: standards are permanently mounted in sample holders and stored in a tray labeled "standards" in the XRF lab.

7.3 Characterizing peak energies:

7.3.1 Peak energies should only have to be determined if the instrument is brand new or if the peaks have shifted due to detector "frift", of if the detector has been replaced. Detector "drift", or broadening of the peaks, is usually a prelude to detector failure. Characterization of peak energies should ONLY be performed by analysts with extensive knowledge of the instrument. The description below is only a brief overview of the technique.

7.3.2 Operate the XRF in manual mode.

7.3.3 Set the system up as follows:

7.3.3.1 Pressure = vacuum
7.3.3.2 Energy = defined by elements of interest
7.3.3.3 mA and kV set to approximate that needed by given element
7.3.3.4 set the filter appropriately for the element of interest
7.3.3.5 set the target appropriately for the element of interest.
7.3.3.6 Start the X-rays and acquire spectrum until the peak is of the desired height.

7.4 Obtaining Calibration Counts:

7.4.1 Analyze each standard following the protocols in SOP XR-002 (ELEMENTAL ANALYSIS OF AIR FILTERS BY X-RAY FLUORESCENCE USING A KEVEX-770 SPECTROMETER AND IXRF OPERATING SYSTEM)

7.4.2 Use the QS analytical protocol to analyze the standards at reduced power (mA).

7.5 After transferring the raw data into the Excel workbook, determine the net counts.

## 8.0 Procedure

8.1 Determine energies for each analyte/excitation condition. Energy calibration is performed using Kevex Standard No. 2036 and the Kevex software calibration option in which a low energy line is measured at the Al K$\alpha$ line (1.487 KeV) and a high energy line is measured at the Cu K$\alpha$ line (8.047 KeV). The program will then assign channel number 149 as the Al peak centroid, and channel 805 as the Cu peak centroid, thereby calibrating each channel increment at 10 eV.

8.2 Identify KeV location of peaks: Peak calibration is performed by analyzing thin film standards containing the elements of interest (see section 5.2). Peaks should be gausian-shaped with at least 2000 counts in the central channel (centroid).

8.2.1 Determine channel numbers associated with each analyte:

8.2.1.1 Calculate the Full-Width-at Half-Maximum (FWHM) window for each element by dividing the number of counts in the most intense channel (usually the centroid) by two (HM).

8.2.1.2 Next, find the channel on the low energy side of the peak closest in counts to HM = LEHM (low-energy-at half-max).

8.2.1.3 Repeat this procedure for the HEHM (high-energy-at half-max).

8.2.1.4 The area between and including LEHM and HEHM represents the FWHM window for that element where LEHM and HEHM are defined as channel numbers. The sum of the counts in these channels should represent approximately 70% of the total area of the peak.

8.2.2 Determine peak centroids associated with analytes of interest:

8.2.2.1 Determine the peak centroid for each analyte containted in the standard by the following equation: (LEHM + HEHM) / 2.

8.2.2.2 Plot a graph of atomic number along the x-axis, versus peak centroid on the y-axis.

8.2.2.3 This graph should yield a smoothly varying curve sloping upward.

8.2.2.4 Visual interpretation of the graph, or algebraic regression can be used to approximate peak positions for elements not represented by standards.

8.2.3 Determine peak widths associated with analytes of interest:

8.2.3.1 Determine the peak width for each analyte containted in the standard by the following equation: (HEHM - LEHM) + 1.

8.2.3.2 Plot a graph of atomic number along the x-axis, versus peak width on the y-axis.

8.2.3.3 This graph should yield a smoothly varying curve sloping upward.

8.2.3.4 Visual interpretation of the graph, or algebraic regression can be used to approximate peak widths for elements not represented by standards.

8.2.3.5 Note that separate plots should be made for Kα, Lα, and Lβ peaks.

8.2.3.6 Some peak widths and centroids for LabNet's 770 Kevex instrument are:

| Analyte line | centroid | width |
|---|---|---|
| | (channel #) | (no. of channels) |
| Si Kα | 174.5 | 12 |
| Fe Kα | 640.5 | 16 |
| Pb Lβ | 1263.0 | 19 |
| Cd Kα | 2312.5 | 32 |
| La Kα | 3334.0 | 53 |

8.3 Determine calibration response factors for each analyte of interest:

8.3.1 Determine the calibration factor for each analyte normalized to 1.0 mA. See Section 9.4 for calculations.

8.3.2 Plot the calibration curves associated with analytes of interest:

8.3.2.1 Determine the sensitivity factor at 1mA for each analyte containted in the standard by the following equation: $S_n$ = 1 / (Calibration Factor x Calibration Current (mA)).

8.3.2.2 Plot a graph of atomic number along the x-axis, versus the log of the sensitivity factor on the y-axis.

8.3.2.3 This graph should yield a smoothly varying curve sloping upward, except for Tungsten filtered 55 kV, which slopes downward.

8.3.2.4 Visual interpretation of the graph, or algebraic regression can be used to approximate calibration factors for elements not represented by standards as long as the atomic number of the analyte is within the range represented on the graph. Sensitivity values lying outside the curve should be checked by analyzing additional standards

8.3.2.5 Note that separate plots should be made for $K\alpha$, $L\alpha$, and $L\beta$ peaks.

8.4 Calculate interference factors from the thin film standards used for calibration using the equations in section 10.6.

8.5 Determine the correction for $PM_{2.5}$ particle size for Aluminum.

8.5.1 Analyze the 'fine' fractions of particulate matter deposited onto 37mm Teflon filters in the form of $Al_2O_3$ and $Al_2Si_2O_7$.

8.5.2 Create the deposits by suspending the particulate matter and 'sampling' with a Sierra-Andersen dichotomous sampler. See SOP XR-001 for suspension of particulate matter onto filters.

8.5.3 The particle size attenuation effect for Al is then calculated for each standard following the equations in section 10.7.

8.6 Update the Chester LabNet XRF Data Processing Program with the newly acquired factors as follows:

8.6.1 The peak location data are entered into the file labeled 'peaks' within the LabNet XRF Data Processing Program.

8.6.1.1 The first column contains the element of interest.

8.6.1.2 The next four columns define:

8.6.1.2.1 the low-energy-background (LEB), where L = the low-energy channel,

8.6.1.2.2 H = the high energy channel,

8.6.1.2.3 C = the LEB centroid; and

8.6.1.2.4 # denotes the number of channels in the LEB window.

8.6.1.3 The next four columns represent the analyte peak and the following four columns labeled 'HEB' define the high-energy-background channels.

8.6.2 The calibration factors data are entered into the file labeled 'peaks' within the LabNet XRF Data Processing Program.

8.6.2.1 The column at the far right contains the calibration factors for the elements of interest normalized to 1.0 mA current.

8.6.2.2 The next column to the left contains the calibration factors normalized to the current (mA) used in the analysis of that excitation condition.

8.6.3 Enter the interference factors into the file labeled 'IEC' within the LabNet XRF Data Processing Program.

8.7 Enter the size correction factor for Al into the size/absorption correction algorithm in the LabNet XRF Data Processing Program.

## 9.0 QA/QC

9.1 Curve shapes for all graphs: All graphs of analyte atomic number vs other parameter should yield a smooth quadratic line. If the graph is not quadratic, all operating parameters must be verified and the calibration performed from the beginning

## 10.0 Calculations

10.1 Full-Width-at Half-Maximum (FWHM) window. The area between and including LEHM and HEHM represents the FWHM window for that element. Note that the counts as described here refer to the net counts (background subtracted).

HM = [counts in the most intense channel (usually the centroid)] ÷ 2.
LEHM = [channel on the low energy side of the peak closest in counts to HM]
HEHM = [channel on the high energy side of the peak closest in counts to HM]

where: HM = counts at half maximum
LEHM = Low-energy-half-maximum channel number
HEHM = High-energy-half-maximum channel number

Note that the sum of the counts in these channels should represent approximately 70% of the total area of the peak.

10.2 Peak centroid channel number:

$$\text{Centroid} = (\text{LEHM} + \text{HEHM}) / 2$$

where: LEHM = Low-energy-half-maximum channel number
HEHM = High-energy-half-maximum channel number

10.3 The peak width in channels:

$$\text{Width} = (\text{HEHM} - \text{LEHM}) + 1$$

where: LEHM = Low-energy-half-maximum channel number
HEHM = High-energy-half-maximum channel number

10.4 Calibration factors are calculated as follows:

$$I_n = I_g - I_b$$

where: $I_n$ = net counts
$I_g$ = the gross counts (sum of all counts in FWHM window)
$I_b$ = the background counts (average bkg cts x peak width)

$$F = V / (I_n / t)$$

where: F = the calibration factor [($\mu$g/cm$^2$)/(counts/second)]

t = the counting livetime (seconds)
V = the surface equivalent value (SEV) of the element in the standard. SEV is the absorption corrected value. Absorption effects will be discussed later in the text.

The calibration factor is then normalized to 1.0 mA as follows:

$$F_n = F \times C_c$$

where: $F_n$ = the calibration factor at 1.0 mA
$C_c$ = the current (mA) used during calibration

10.5 Sensitivity factors are calculated as follows:

$S_n = 1 / F_n$ where $S_n$ represents the sensitivity factor at 1.0 mA

10.6 Interference factors are calculated for spectral overlap using the data from the thin film standards used for calibration:

$$L_i = N_i / N_j$$

where: $L_i$ = the interference correction factor (cts/s in the window of element i due to element j)
$N_i$ = net cts/s in window of element i
$N_j$ = net cts/s in window of element j (interfering element)

Most spectral overlap is the result of the Kβ line of element j overlapping the Kα line of element i. Another common interference occurs when an L-line of element j from one excitation condition overlaps the Kα line of element i in a separate excitation condition. In the latter case it is necessary to normalize the correction factor (L) to 1.0 mA per second as follows:

$$L_i = (N_i / C_i T_i) / (N_j / C_j T_j)$$

where: $C_i$ = the current (mA) used in the excitation condition in which element i is measured.

$C_j$ = the current (mA) used in the excitation condition in which element j is measured.

$T_i$ = livetime (sec) for the excitation condition in which the element is measured.

$T_j$ = livetime (sec) for the excitation condition in which element j is measured.

10.7 The mass absorption effect for Al is then calculated for each particle size attenuation standard as follows:

10.7.1 Mass absorption coefficient

$$\mu = \Sigma (\mu_i w_i)$$

where: $\mu$ = the total mass absorption coefficient of all elements present on the incoming X-rays ($cm^2/g$)

$\mu_i$ = the mass absorption coefficient of each element i on the incoming X-rays

$w_i$ = the weight percent of element i in the deposit ($0 < w_i < 1$)

10.7.2 Total Mass absorption coefficient

$$\mu' = \Sigma (\mu_{i,j} w_i)$$

where: $\mu'$ = the total mass absorption coefficient of all elements present on the exiting (fluorescing) X-rays from element j

$\mu_{i,j}$ = the mass absorption coefficient of each element i on the exiting X-rays from element j.

10.7.3 Total Mass absorption coefficient by area

$$\underline{\mu} = (\mu \ \csc \theta) + (\mu' \ \csc \theta')$$

where: $\underline{\mu}$ = the total mass absorption coefficient ($cm^2/g$)

$\theta$ = the angle at which the incoming X-rays strike the sample

$\theta$ is described in sections 6.1 and 6.2 for each excitation condition

$\theta'$ = the angle at which the exiting X-rays travel to the detector and is fixed at 45°

10.7.4 Mass absorption:

$$A = (1 - e^{-\mu m}) / \mu m$$

where: m = the areal density of the deposit (g/cm$^2$)
A = mass absorption
e = natural log exponent (mathematical e)

10.7.5 The net counts are then corrected for the calculated absorption for each standard as follows:

$$N_c = N / A$$

where: $N_c$ = the absorption corrected intensity (net counts/second)
N = the uncorrected net count counts/second

10.7.6 Determine final size correction:

10.7.6.1 Plot the empirical concentration of Al for each standard along the x-axis versus the absorption corrected intensity along the y-axis.

10.7.6.2 Perform a linear regression, forcing the line through zero, such that

$$y = mx.$$

where: m = the sensitivity factor [(cts/s)/(μg/cm$^2$)].

10.7.6.3 Calculate the size correction factor as follows:

$$A_{i,p} = S_t / S_p$$

where: $A_{i,p}$ = the size attenuation factor for element i at particle size p

$S_t$ = the thin film calibration sensitivity factor for element i

$S_p$ = the particle standard sensitivity factor which is the slope of the line derived from the linear regression above.

Simply stated, the attenuation for mass absorption has been taken into account, so that if there were no additional attenuation due to the size of the particles, then $S_p = S_t$ and $A_{i,p} = 1$. Any additional attenuation would then be due to individual particle size, and $A_{i,p} < 1$. $A_p \cong 0.667$ for Al $PM_{2.5}$, and $A_p \cong 1$ for Si $PM_{2.5}$.

## 11.0 References

11.1 Kevex Operator's Manual

11.2 Practical X-Ray Spectrometry, R. Jenkins and J.L. De Vries, second edition, Philips Technical Library, Springer-Verlag New York Inc.

11.3 X-Ray Fluorescence Analysis of Environmental Samples, Jaklevic, et al, Ed. by T.G. Dzubay, Ann Arbor Sci.

11.4 Self Absorption Corrections for X-Ray Fluorescence Analysis of Aerosols, T.G. Dzubay and R.O. Nelson, in Advances in X-Ray Analysis, Vol 18, 619-631.

11.5 Quantitative Analysis of Aluminum and Silicon in Air Particulate Deposits on Teflon Membrane Filters by X-Ray Fluorescence Analysis, J.A. Cooper, L.M. Valdovinos, J.R. Sherman, W.L. Pollard, R.H. Sarver, and J.K. Weider, report by NEA, Inc., Beaverton, OR, July 15, 1987.

11.6 Round Robin Evaluation: Elemental Analysis of Bulk Samples and $PM_{10}$ Loaded Teflon Filters, presented at the 82nd Annual Meeting of AWMA, Anaheim, CA, June 24-30, 1989.

## 12.0 Definitions

12.1 Analyst: the designated individual who performs the "hands-on" method and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

12.2 Analysts' Notes: Non-essential aspects of a method, which may help the analyst during some phase of the method. Notes may include, but not be limited to, historical aspects of the method, "tricks" of the method, unexpected issues to be aware of, or other facts or opinions related to the method, but not directly part of the procedure.

12.3Blank: a clean aliquot of the same matrix as the digested samples. A blank is subjected to the usual analytical and measurement processes.

12.3.1 Calibration Blank: An unspiked clean matrix of similar constitution as the sample extracts or digests (e.g. DI Water, 5% HNO3 etc) used to establish the zero intercept of the calibration curve.

12.4Calculations (Data Reduction): the mathematical process of transforming raw data into a more useable form.

12.5 Calibrate: to determine, by measurement or comparison with a standard, the correct value of each reading of the instrument.

12.6 Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements where possible.

12.7 Calibration Standard: a substance or reference material used to calibrate and instrument.

12.8Correlation Coefficient: the statistical representation of how closely a set of x,y coordinates comes to a true line. A correlation coefficient of 1.000 is considered a perfectly straight line. Correlation coefficients above 0.995 are usually attainable by most instruments.

12.9Detection Limit: the lowest concentration of an analyte of interest that can be identified, measured and reported with confidence that the analyte concentration is not a false positive value.

12.9.1 Instrument Detection Limit (IDL): The detection limit determined at the instrument using a clean matrix and no preparation.

12.10 Matrix/Matrices: the component or substrate that contains the analyte of interest. In this case, the matrix should consist of a bicarbonate impregnated cellulose filter.

12.11 QA/QC: Quality Assurance/Quality Control. A series of samples or metrics designed to show precision, accuracy and bias of the procedure are within acceptable limits.

12.12 Reagent: a single chemical or combination of chemicals or a chemical solution used in the preparation or analysis of samples.

12.13 Standard: a solution or matrix of a known amount of analyte(s).

12.13.1 Primary standard: a standard received from a vendor with NIST or equivalent traceability.

## 13.0 Analysts' Notes

13.1 N/A

## Figure 1. Final calibration data sheet

| | LEB | | | | Analyte | | | | HEB | | | | Avg Counts | | | | Calibration | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **SP0** | L | H | C | # | L | H | C | # | L | H | C | # | LEB | HEB | m | b | Factors | |
| Na | 91 | 93 | 92 | 3 | 95 | 110 | 102.5 | 16 | 114 | 116 | 115 | 3 | 69.3 | 61.0 | -0.3623 | 102.6667 | 0.04273 | 0.03205 |
| Mg | 114 | 116 | 115 | 3 | 118 | 133 | 125.5 | 16 | 133 | 134 | 133.5 | 2 | 61.0 | 67.0 | 0.3243 | 23.7027 | 0.01930 | 0.01447 |
| Al | 137 | 139 | 138 | 3 | 142 | 157 | 149.5 | 16 | 158 | 160 | 159 | 3 | 47.7 | 60.0 | 0.5873 | -33.3810 | 0.00932 | 0.00699 |
| Si | 158 | 160 | 159 | 3 | 167 | 182 | 174.5 | 16 | 188 | 191 | 189.5 | 4 | 60.0 | 79.5 | 0.6393 | -41.6557 | 0.00516 | 0.00387 |
| P | 191 | 192 | 191.5 | 2 | 193 | 209 | 201.0 | 17 | 210 | 211 | 210.5 | 2 | 78.0 | 98.5 | 1.0789 | -128.6184 | 0.00336 | 0.00252 |
| S | 199 | 201 | 200 | 3 | 223 | 239 | 231.0 | 17 | 249 | 251 | 250 | 3 | 75.7 | 179.3 | 2.0733 | -339.0000 | 0.00245 | 0.00184 |
| **SP1** | | | | | | | | | | | | | | | | | | |
| Al | 113 | 117 | 115 | 5 | 142 | 157 | 149.5 | 16 | 298 | 302 | 300 | 5 | 88.8 | 63.6 | -0.1362 | 104.4649 | 0.19094 | 0.57283 |
| Si | 113 | 117 | 115 | 5 | 167 | 182 | 174.5 | 16 | 298 | 302 | 300 | 5 | 88.8 | 63.6 | -0.1362 | 104.4649 | 0.10518 | 0.31555 |
| P | 113 | 117 | 115 | 5 | 193 | 209 | 201.0 | 17 | 298 | 302 | 300 | 5 | 88.8 | 63.6 | -0.1362 | 104.4649 | 0.06828 | 0.20484 |
| S | 113 | 117 | 115 | 5 | 223 | 239 | 231.0 | 17 | 298 | 302 | 300 | 5 | 88.8 | 63.6 | -0.1362 | 104.4649 | 0.04921 | 0.14764 |
| Cl | 113 | 117 | 115 | 5 | 254 | 270 | 262.0 | 17 | 298 | 302 | 300 | 5 | 88.8 | 63.6 | -0.1362 | 104.4649 | 0.03526 | 0.10577 |
| K | 298 | 302 | 300 | 5 | 324 | 341 | 332.5 | 18 | 414 | 416 | 415 | 3 | 63.6 | 208.3 | 1.2586 | -313.9652 | 0.01943 | 0.05829 |
| Ca | 298 | 302 | 300 | 5 | 361 | 378 | 369.5 | 18 | 414 | 416 | 415 | 3 | 63.6 | 208.3 | 1.2586 | -313.9652 | 0.01460 | 0.04381 |
| Ti MS | 113 | 117 | 115 | 5 | 443 | 460 | 451.5 | 18 | 524 | 528 | 526 | 5 | 88.8 | 11.4 | -0.1883 | 110.4569 | | |
| **SP2** | | | | | | | | | | | | | | | | | | |
| K | 298 | 302 | 300 | 5 | 324 | 341 | 332.5 | 18 | 421 | 425 | 423 | 5 | 78.2 | 55.4 | -0.1854 | 133.8098 | 0.01706 | 0.05117 |
| Ca | 298 | 302 | 300 | 5 | 361 | 378 | 369.5 | 18 | 421 | 425 | 423 | 5 | 83.2 | 55.4 | -0.2260 | 151.0049 | 0.01300 | 0.03900 |
| Sc | 298 | 302 | 300 | 5 | 401 | 418 | 409.5 | 18 | 421 | 425 | 423 | 5 | 83.2 | 55.4 | -0.2260 | 151.0049 | 0.01026 | 0.03077 |
| Ti | 421 | 425 | 423 | 5 | 443 | 460 | 451.5 | 18 | 517 | 521 | 519 | 5 | 55.4 | 85.4 | 0.3125 | -76.7875 | 0.00809 | 0.02427 |
| V | 421 | 425 | 423 | 5 | 487 | 505 | 496.0 | 19 | 517 | 521 | 519 | 5 | 55.4 | 85.4 | 0.3125 | -76.7875 | 0.00633 | 0.01900 |
| Cr | 512 | 521 | 516.5 | 10 | 533 | 551 | 542.0 | 19 | 564 | 573 | 568.5 | 10 | 79.4 | 123.7 | 0.8519 | -360.6183 | 0.00491 | 0.01474 |
| Fe MS | 316 | 320 | 318 | 5 | 631 | 650 | 640.5 | 20 | 737 | 741 | 739 | 5 | 77.8 | 24.6 | -0.1264 | 117.9843 | | |
| **SP3** | | | | | | | | | | | | | | | | | | |
| Cr | 420 | 424 | 422 | 5 | 533 | 551 | 542.0 | 19 | 564 | 568 | 566 | 5 | 41.4 | 21.4 | -0.1389 | 100.0111 | 0.01279 | 0.03838 |
| Mn | 564 | 568 | 566 | 5 | 581 | 599 | 590.0 | 19 | 726 | 730 | 728 | 5 | 21.4 | 27.6 | 0.0383 | -0.2617 | 0.01033 | 0.03100 |
| Fe | 564 | 568 | 566 | 5 | 631 | 650 | 640.5 | 20 | 726 | 730 | 728 | 5 | 21.4 | 27.6 | 0.0383 | -0.2617 | 0.00826 | 0.02478 |
| Co | 564 | 568 | 566 | 5 | 684 | 703 | 693.5 | 20 | 726 | 730 | 728 | 5 | 21.4 | 27.6 | 0.0383 | -0.2617 | 0.00660 | 0.01981 |
| Ni | 667 | 671 | 669 | 5 | 738 | 758 | 748.0 | 21 | 767 | 771 | 769 | 5 | 20.8 | 27.0 | 0.0620 | -20.6780 | 0.00563 | 0.01690 |
| Cu | 767 | 771 | 769 | 5 | 795 | 815 | 805.0 | 21 | 833 | 837 | 835 | 5 | 27.0 | 44.0 | 0.2576 | -171.0758 | 0.00483 | 0.01450 |
| Zn | 833 | 837 | 835 | 5 | 854 | 875 | 864.5 | 22 | 883 | 887 | 885 | 5 | 44.0 | 102.8 | 1.1760 | -937.9600 | 0.00404 | 0.01211 |
| Sm | 516 | 520 | 518 | 5 | 554 | 572 | 563.0 | 19 | 726 | 730 | 728 | 5 | 34.2 | 27.6 | -0.0314 | 50.4800 | 0.02698 | 0.08095 |
| Eu | 516 | 520 | 518 | 5 | 575 | 593 | 584.0 | 19 | 726 | 730 | 728 | 5 | 34.2 | 27.6 | -0.0314 | 50.4800 | 0.03800 | 0.11401 |
| Tb | 516 | 520 | 518 | 5 | 688 | 708 | 698.0 | 21 | 726 | 730 | 728 | 5 | 34.2 | 27.6 | -0.0314 | 50.4800 | 0.02563 | 0.07690 |
| Ge MS | 725 | 734 | 518 | 10 | 978 | 999 | 988.5 | 22 | 1136 | 1140 | 1138 | 5 | 25.7 | 10.6 | -0.0244 | 38.3158 | | |
| **SP4** | | | | | | | | | | | | | | | | | | |
| Zn | 767 | 771 | 769 | 5 | 854 | 875 | 864.5 | 22 | 891 | 895 | 893 | 5 | 24.2 | 25.4 | 0.0097 | 16.7581 | 0.01733 | 0.05200 |
| Ga | 891 | 895 | 893 | 5 | 915 | 936 | 925.5 | 22 | 1017 | 1021 | 1019 | 5 | 25.4 | 25.6 | 0.0016 | 23.9825 | 0.01483 | 0.04450 |
| Ge | 891 | 895 | 893 | 5 | 978 | 999 | 988.5 | 22 | 1017 | 1021 | 1019 | 5 | 25.4 | 25.6 | 0.0016 | 23.9825 | 0.01298 | 0.03895 |
| As | 1017 | 1021 | 1019 | 5 | 1044 | 1065 | 1054.5 | 22 | 1153 | 1157 | 1155 | 5 | 25.6 | 21.8 | -0.0279 | 54.0721 | 0.01166 | 0.03497 |
| Se | 1017 | 1021 | 1019 | 5 | 1111 | 1133 | 1122.0 | 23 | 1153 | 1157 | 1155 | 5 | 25.6 | 21.8 | -0.0279 | 54.0721 | 0.01086 | 0.03257 |
| Br | 1153 | 1157 | 1155 | 5 | 1181 | 1204 | 1192.5 | 24 | 1227 | 1231 | 1229 | 5 | 21.8 | 19.2 | -0.0351 | 62.3811 | 0.00991 | 0.02973 |
| Rb | 1299 | 1303 | 1301 | 5 | 1327 | 1352 | 1339.5 | 26 | 1376 | 1379 | 1377.5 | 4 | 26.0 | 37.8 | 0.1536 | -173.8268 | 0.00850 | 0.02550 |
| Sr | 1376 | 1379 | 1377.5 | 4 | 1404 | 1430 | 1417.0 | 27 | 1454 | 1458 | 1456 | 5 | 37.8 | 67.8 | 0.3828 | -489.5605 | 0.00784 | 0.02351 |
| Y | 1454 | 1458 | 1456 | 5 | 1481 | 1508 | 1494.5 | 28 | 1533 | 1537 | 1535 | 5 | 67.8 | 105.0 | 0.4709 | -617.8101 | 0.00733 | 0.02200 |
| Zr | 1533 | 1537 | 1535 | 5 | 1562 | 1590 | 1576.0 | 29 | 1615 | 1619 | 1617 | 5 | 105.0 | 158.2 | 0.6488 | -890.8780 | 0.00678 | 0.02034 |
| Hg | 891 | 895 | 893 | 5 | 989 | 1010 | 999.5 | 22 | 1017 | 1021 | 1019 | 5 | 25.4 | 25.6 | 0.0016 | 23.9825 | 0.02201 | 0.06604 |
| Pb | 1222 | 1231 | 1226.5 | 10 | 1252 | 1273 | 1262.5 | 22 | 1296 | 1305 | 1300.5 | 10 | 20.9 | 29.4 | 0.1149 | -119.9818 | 0.02563 | 0.07688 |
| **SP5** | | | | | | | | | | | | | | | | | | |
| Pd | 2061 | 2080 | 2070.5 | 20 | 2095 | 2130 | 2112.5 | 36 | 2145 | 2164 | 2154.5 | 20 | 13.4 | 14.5 | 0.0137 | -14.9961 | 0.01458 | 0.04373 |
| Ag | 2158 | 2177 | 2167.5 | 20 | 2192 | 2229 | 2210.5 | 38 | 2244 | 2263 | 2253.5 | 20 | 13.6 | 13.7 | 0.0012 | 11.0797 | 0.01402 | 0.04205 |
| Cd | 2258 | 2277 | 2267.5 | 20 | 2292 | 2331 | 2311.5 | 40 | 2346 | 2365 | 2355.5 | 20 | 13.8 | 13.7 | -0.0011 | 16.3767 | 0.01375 | 0.04125 |
| In | 2360 | 2379 | 2369.5 | 20 | 2394 | 2435 | 2414.5 | 42 | 2450 | 2469 | 2459.5 | 20 | 14.0 | 15.5 | 0.0161 | -24.1753 | 0.01375 | 0.04125 |
| Sn | 2463 | 2482 | 2472.5 | 20 | 2497 | 2540 | 2518.5 | 44 | 2555 | 2574 | 2564.5 | 20 | 15.9 | 18.5 | 0.0288 | -55.3688 | 0.01415 | 0.04246 |
| Sb | 2569 | 2588 | 2578.5 | 20 | 2603 | 2649 | 2626.0 | 47 | 2664 | 2683 | 2673.5 | 20 | 17.5 | 23.1 | 0.0589 | -134.5458 | 0.01503 | 0.04508 |
| Cs | 3021 | 3040 | 3030.5 | 20 | 3055 | 3112 | 3083.5 | 58 | 3127 | 3146 | 3136.5 | 20 | 39.2 | 49.1 | 0.0934 | -243.8373 | 0.02475 | 0.07424 |
| Ba | 3143 | 3162 | 3152.5 | 20 | 3177 | 3236 | 3206.5 | 60 | 3251 | 3270 | 3260.5 | 20 | 47.5 | 55.0 | 0.0699 | -172.9331 | 0.03037 | 0.09110 |
| La | 3265 | 3284 | 3274.5 | 20 | 3299 | 3361 | 3330.0 | 63 | 3376 | 3395 | 3385.5 | 20 | 58.3 | 58.7 | 0.0041 | 44.9750 | 0.03836 | 0.11508 |

## Figure 2. Overlap correction data.

| Analyte to be corrected | sp | Interferent | sp | Factor | Amount Subtracted | Corrected Net Counts | | Corrected Uncertainty |
|---|---|---|---|---|---|---|---|---|
| K | 1 | Cd | 5 | 0 | 0.00 | 0 | 0.00 | 60 |
| Sn | 5 | Ag | 5 | 0 | 0.00 | 110 | 0.00 | 40 |
| K | 1 | Sn | 4 | 0 | 0.00 | 0 | 0.00 | 60 |
| Ca | 1 | Sn | 4 | 0 | 0.00 | 0 | 0.00 | 71 |
| Sb | 5 | Cd | 5 | 0.13207 | 1.98 | 12 | 4.41 | 44 |
| K | 1 | Sb | 5 | 0 | 0.00 | 0 | 0.00 | 60 |
| Ca | 1 | SB | 5 | 0 | 0.00 | 0 | 0.00 | 71 |
| Ca | 1 | K | 1 | 0.07171 | 0.00 | 0 | 4.32 | 71 |
| Ca | 2 | K | 2 | 0.07171 | 5.37 | 45 | 3.05 | 50 |
| Sc | 2 | Ca | 2 | 0.092 | 4.12 | 0 | 4.59 | 45 |
| Ba | 5 | I | 5 | 0 | 0.00 | 10 | 0.00 | 78 |
| Ti | 2 | BA | 5 | 0.3332 | 0.00 | 345 | 0.00 | 52 |
| Ti | 2 | Fe MS | 2 | 0.0012 | 381.35 | 0 | 0.68 | 64 |
| V | 2 | BA | 5 | 0.1321 | 0.00 | 0 | 0.00 | 49 |
| V | 2 | TI | 2 | 0.13371 | 0.00 | 0 | 8.58 | 49 |
| Cr | 2 | V | 2 | 0.13568 | 0.00 | 0 | 6.69 | 62 |
| Mn | 3 | CR | 3 | 0.1257 | 18.74 | 65 | 4.15 | 31 |
| Fe | 3 | MN | 3 | 0.08087 | 5.27 | 415 | 2.50 | 37 |
| Cu | 3 | Ge MS | 3 | 0.0017 | 209.02 | 32 | 0.60 | 47 |
| Pb | 4 | SE | 4 | 0.07656 | 0.00 | 0 | 2.32 | 33 |
| S | 1 | PB | 4 | 0.11478 | 0.00 | 0 | 8.20 | 49 |
| Cl | 1 | PB | 4 | 0.02922 | 0.00 | 689 | 2.09 | 55 |
| Cl | 1 | S | 1 | 0.0175 | 0.00 | 689 | 0.86 | 55 |
| Cl | 1 | Ti MS | 1 | 0.0021 | 645.80 | 43 | 1.17 | 85 |
| S | 1 | CL | 1 | 0 | 0.00 | 0 | 0.00 | 49 |
| Co | 3 | FE | 3 | 0.06992 | 29.00 | 0 | 2.61 | 30 |
| Sm | 3 | Cr | 3 | 0.0762 | 11.36 | 0 | 2.51 | 32 |
| Sm | 3 | Mn | 3 | 0.0172 | 1.12 | 0 | 0.53 | 32 |
| Eu | 3 | Cr | 3 | 0.0611 | 9.11 | 0 | 2.02 | 32 |
| Eu | 3 | Mn | 3 | 0.8262 | 53.88 | 0 | 25.50 | 41 |
| Tb | 3 | Fe | 3 | 0.1 | 41.47 | 0 | 3.74 | 31 |
| Tb | 3 | Co | 3 | 0.9369 | 0.00 | 0 | 28.34 | 42 |
| Co | 4 | FE | 4 | 0.06007 | 10.75 | 0 | 2.18 | 32 |
| Ga | 4 | PB | 4 | 0.06488 | 0.00 | 28 | 2.16 | 34 |
| As | 4 | PB | 4 | 1.200648 | 0.00 | 0 | 0.00 | 31 |
| Ge | 4 | ZN | 3 | 0 | 0.00 | 0 | 0.00 | 32 |
| Ge | 4 | Hg | 4 | 0 | 0.00 | 0 | 0.00 | 32 |
| Br | 4 | AS | 4 | 0.04556 | 0.00 | 43 | 1.40 | 32 |
| Rb | 4 | BR | 4 | 0.13207 | 5.73 | 24 | 4.24 | 41 |
| Y | 4 | PB | 4 | 0.06959 | 0.00 | 0 | 1.78 | 67 |
| Y | 4 | RB | 4 | 0 | 0.00 | 0 | 0.00 | 67 |
| Hf | 4 | Pb | 4 | 0.0174 | 0.00 | 0 | 0.58 | 33 |
| Hf | 4 | Cu | 4 | 0.0883 | 13.99 | 0 | 3.05 | 33 |
| Ga | 4 | Ta | 4 | 0.65672 | 41.21 | 0 | 22.59 | 41 |

**Figure 3. Interelement energy absorption table (see section 10.7.2)**

KeVex Absorption Table

| | Incom | Incom | Exit | Exit | Exit | Exit | Exit | Exit | Exit | Exit | Exit | Exit |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Rh La | Ti Ka | Na Ka | Mg Ka | Al Ka | Si Ka | P Ka | S Ka | Cl Ka | K Ka | Ca Ka | Ti Ka |
| csc | 1.071 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 | 1.4142 |
| C | 116 | 26 | 1780 | 1050 | 640 | 400 | 263 | 175 | 125 | 61 | 45 | 26 |
| O | 275 | 63 | 4000 | 2440 | 1520 | 965 | 633 | 435 | 300 | 162 | 115 | 63 |
| Na | 650 | 163 | 8160 | 4925 | 3395 | 2100 | 1480 | 1030 | 715 | 366 | 276 | 163 |
| Mg | 850 | 212 | 580 | 350 | 4050 | 2660 | 1794 | 1330 | 930 | 490 | 367 | 212 |
| Al | 1035 | 263 | 850 | 500 | 330 | 3170 | 2223 | 1610 | 1130 | 595 | 450 | 263 |
| Si | 1265 | 328 | 1230 | 740 | 480 | 315 | 2645 | 1960 | 1375 | 735 | 556 | 328 |
| P | 1435 | 389 | 1640 | 1015 | 650 | 435 | 300 | 2180 | 1570 | 850 | 648 | 389 |
| S | 1770 | 453 | 2100 | 1320 | 795 | 525 | 355 | 250 | 1920 | 1030 | 671 | 453 |
| Cl | 190 | 512 | 2500 | 1570 | 960 | 635 | 435 | 300 | 210 | 1140 | 870 | 512 |
| K | 280 | 689 | 3425 | 2120 | 1300 | 855 | 593 | 425 | 305 | 162 | 606 | 689 |
| Ca | 320 | 780 | 3850 | 2380 | 1500 | 980 | 710 | 480 | 345 | 185 | 142 | 780 |
| Ti | 425 | 114 | 4680 | 2975 | 2000 | 1300 | 905 | 645 | 455 | 250 | 190 | 114 |
| V | 480 | 129 | 5050 | 3260 | 2200 | 1460 | 1020 | 730 | 520 | 280 | 215 | 129 |
| Cr | 550 | 153 | 5480 | 3510 | 2470 | 1670 | 1165 | 835 | 590 | 320 | 247 | 153 |
| Mn | 675 | 171 | 5895 | 3790 | 2700 | 1920 | 1310 | 935 | 665 | 360 | 279 | 171 |
| Fe | 700 | 193 | 6275 | 4100 | 2910 | 2040 | 1465 | 1070 | 760 | 410 | 317 | 193 |
| Co | 735 | 198 | 6640 | 4380 | 3070 | 2195 | 1565 | 1160 | 790 | 435 | 341 | 198 |
| Ni | 830 | 244 | 6800 | 4540 | 3140 | 2225 | 1675 | 1260 | 900 | 490 | 379 | 244 |
| Cu | 885 | 262 | 7550 | 5035 | 3450 | 2415 | 1780 | 1350 | 960 | 530 | 419 | 262 |
| Zn | 1050 | 292 | 0 | 5235 | 3645 | 2510 | 1915 | 1460 | 1130 | 595 | 480 | 292 |
| Ga | 1145 | 300 | 0 | 0 | 3810 | 2645 | 2025 | 1575 | 1225 | 700 | 508 | 300 |
| Ge | 1210 | 325 | 0 | 0 | 3995 | 2750 | 2145 | 1670 | 320 | 760 | 543 | 325 |
| As | 1310 | 340 | 1580 | 1020 | 0 | 2880 | 2490 | 1795 | 1420 | 820 | 595 | 340 |
| Se | 1380 | 380 | 1740 | 1110 | 0 | 3010 | 2620 | 1930 | 1530 | 925 | 660 | 380 |
| Br | 1410 | 480 | 1875 | 1190 | 840 | 0 | 2528 | 2060 | 1615 | 1025 | 703 | 480 |
| Rb | 1630 | 455 | 2200 | 1380 | 950 | 710 | 0 | 2330 | 1810 | 1190 | 805 | 455 |
| Sr | 1740 | 180 | 2400 | 1500 | 1020 | 760 | 0 | 2465 | 1910 | 1260 | 875 | 180 |
| Y | 1875 | 515 | 2555 | 1820 | 1080 | 810 | 648 | 0 | 2020 | 1310 | 938 | 515 |
| Zr | 1940 | 580 | 2755 | 1740 | 1155 | 855 | 688 | 0 | 2125 | 1390 | 995 | 580 |
| Mo | 0 | 720 | 3180 | 2005 | 1315 | 990 | 788 | 630 | 0 | 1540 | 1190 | 720 |
| Pd | 550 | 890 | 4100 | 2590 | 1675 | 1260 | 1000 | 800 | 590 | 0 | 683 | 890 |
| Ag | 585 | 925 | 4230 | 2700 | 1800 | 1350 | 1058 | 860 | 625 | 355 | 1365 | 925 |
| Cd | 620 | 1025 | 4660 | 2930 | 1880 | 1415 | 1120 | 890 | 665 | 385 | 0 | 1025 |
| In | 650 | 1065 | 4880 | 3080 | 1975 | 1480 | 1175 | 930 | 700 | 400 | 170 | 1065 |
| Sn | 720 | 1020 | 5300 | 3360 | 2280 | 1475 | 1235 | 990 | 760 | 435 | 348 | 1020 |
| Sb | 730 | 0 | 5510 | 3500 | 2355 | 1660 | 1310 | 1050 | 780 | 455 | 368 | 0 |
| Te | 770 | 0 | 5825 | 3650 | 2500 | 1740 | 1375 | 1100 | 825 | 480 | 385 | 0 |
| I | 805 | 265 | 0 | 3870 | 2645 | 1840 | 1453 | 1160 | 870 | 505 | 405 | 265 |
| Cs | 0 | 0 | 0 | 0 | 0 | 2010 | 0 | 0 | 0 | 0 | 0 | 0 |
| Ba | 940 | 310 | 0 | 0 | 0 | 2130 | 1680 | 1345 | 1010 | 585 | 473 | 310 |
| La | 980 | 325 | 0 | 0 | 0 | 2210 | 1760 | 1400 | 1055 | 610 | 495 | 325 |
| Hg | 0 | 880 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1435 | 880 |
| Pb | 0 | 935 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1500 | 935 |
| Σ | 35506 | 17549 | 131275 | 92680 | 72480 | 64755 | 51324 | 44600 | 37550 | 24576 | 23043 | 17549 |