The Arizona Border Study

An Extension of the
Arizona National Human Exposure Assessment Survey (NHEXAS)Study
Sponsored by the Environmental Health Workgroup of the Border XXI Program

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 824719

Standard Operating Procedure **SOP-UA-L-12.1**

Title: Vacuum Dust Characterization

Source: The University of Arizona

U.S. Environmental Protection Agency
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Form TP-1

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Vacuum Dust Characterization

1.0 Purpose and Applicability

The purpose of this SOP describes the procedures to be followed in aliquoting and analyzing dust samples. This procedure includes splitting samples for independent laboratory analysis within the EPA NHEXAS project of the University of Arizona/Battelle/Illinois Institute of Technology consortia.

2.0 Definitions

2.1 Blue Ice = Commercially produced packages which act as ice blocks but are self contained thus no liquid is generated as they warm.

2.2 DDW = Deionized Distilled Water

2.3 SOP = Standard Operating Procedure

3.0 References

Not applicable

4.0 Discussion

Vacuum dust samples must be split to facilitate analysis in separate laboratories. In splitting the samples each portion must be an accurate representation, in its distribution of different particle sizes, of the whole. Vacuum Dust samples are initially homogenized during the vacuuming process. Further processing and splitting must ensure maintenance of a homogenized sample. Pertinent samples will be analyzed and after splitting, samples will be shipped to the other laboratory sites.

5.0 Responsibilities

5.1 The Project Director will be responsible for:

5.1.1 Final review and approval of this procedure.

5.2 The Project Lab Supervisor will be responsible for:

5.2.1 Insuring SOP procedures are followed by the Project Lab Staff.

5.2.2 Notifying the appropriate technicians with needed repairs. In cases when the item can not be fixed in-house, Project Field Coordinator will generate the appropriate paperwork, notify the appropriate vendor or company, and ship the disfunctional item.

5.3 The Project Lab Staff will be responsible for:

5.3.1 Knowing and following the procedures described in this SOP.
5.3.2 Recording the information as directed in this SOP.
5.3.3 Notifying the Project Lab Supervisor with down equipment and repair supplies needed (where applicable).
5.3.4 Providing the Project Lab Supervisor with down equipment label and isolating the down equipment into the down equipment area.
5.3.5 Insuring proper labeling techniques of down equipment.
5.3.6 Repairing the item (where applicable) in a timely manner.

6.0 Equipment and Materials

6.1 Equipment

6.1.1 Blue Ice blocks
6.1.2 Brass 10 g calibration weight (#9800011)
6.1.3 Brass 5 g calibration weight (#9800017)
6.1.4 Brass 1 g calibration weight (#9800018)
6.1.5 Freezer Bags
6.1.6 Metal tray
6.1.7 Mettler Balance
6.1.8 Nalgene 125 ml bottles
6.1.9 Paper Towels
6.1.10 Plastic XRF cups
6.1.11 Powder free rubber gloves
6.1.12 Teflon coated spatula
6.1.13 Teflon coated tweezers
6.1.14 Weighing paper (3 in^2)
6.1.15 Wire cutters
6.1.16 X-ray mylar film
6.1.17 Ziploc freezer bags

6.2 Materials

6.2.1 DDW

7.0 Procedure

7.1 Work Area Setup

7.1.1. For each sample to be processed fill out a "Vacuum Dust Characterization" (figure 1) form.
7.1.2 Place a vacuum dust aliquot ID# on a 125 ml Nalgene bottle and record that number on the "Vacuum Dust Characterization" form (figure 1), and the "Vacuum Dust Chain of Custody" form (figure 2). Fill out a "Vacuum Dust Aliquot Chain

of Custody" form (figure 3) for the sample.

7.1.3 Clean the work area with DDW and paper towels.

7.1.4 Turn on the fan in the fume hood.

7.1.5 Locate and set up soil sieve stacks containing a solid pan, sieve #230, sieve #10, and a top.

7.1.6 Set up the equipment needed to clean the sieves after each use (UA-L-5.1).

7.1.7 Turn on and calibrate the Mettler AE 166 balance in the lab (UA-L-1.1).

7.1.8 Set up a cold surface to work on by placing "blue ice" bags between two inverted metal trays. The tray surface provides a cold surface to reduce the amount of heating that each sample will experience during processing.

7.1.9 Locate teflon coated tweezers and spatulas cleaned to specifications outlined in UA-L-5.1.

7.2 Sample Processing Set-up

7.2.1 Prepare the paperwork for each of the samples to be processed. Fill out the date, sample ID#, HHID#, technician code and bubble the scale to be used on a "Vacuum Dust Characterization Form" (Figure 1) for each of the samples to be processed. Fill out similar information on chain of custody records for the pesticide samples and metals samples (figure 3). Place sample ID# lables on the "metals" sample bag and the Nalgene 125 ml pesticide sample bottle. Fill in the metals sample ID# and HHID on an XRF form for each of hte samples to be processed.

7.2.2 Place a large kimwipe onto the cold work surface.

7.2.3 Place four sheets of weighing paper (3 in^2) onto the cold working surface. Weigh each sheet and enter the weight under "weighing paper wt." as value #10, #11, #15 and #18, respectively. Place the weighing papaers on the cold surface. For example, the sheet for the >10 sample is placed in the upper left corner, 10-230 in the upper right corner and the two sheets weighed for pesticide and metals slightly overlapping along the bottom of the tray.

7.2.4 Record the set of screens to be used under "Screen Set" on the "Vacuum characterization Form" (Figure 1).

7.3 Initial Sample Weighing

7.3.1 Receive the " Vacuum Dust" samples from the Materials Technician, maintaining the required temperature by keeping them under refrigeration ($\leq 4\,^{\circ}C$).

7.3.2 Place a 3 in^2 piece of weighing paper on the balance to protect the cleanliness of the balance's weighing surface and tare to 0.0000.

7.3.3 To begin sample processing, take the vacuum dust sample from the freezer, remove from the ziplock bag the filter package (consisting of a filter wrapped

around the collected dust and secured at the top with a plastic cable tie) and place on the balance pan on top of the weighing paper.

7.3.4 Once the scale has stabilized, record the weight and enter it under weight #4 on the "Vacuum dust Characterization" form (figure 1), "Chain of Custody" record (figure 2), and "Aliquot Chain of Custody" record (figure 3). The "total weight (#4)" minus the "Filter + Tie weight (#3)" equals the "Collected weight (#5)". Follow filter weighing protocols (UA-L-9.1) to determine validity.

7.3.5 Place the sample back into its ziplock bag and return to the freezer ($\leq 4°C$).

7.4 Sample Processing

7.4.1 Remove the sample from its plastic bag and place the filter package onto the cold work surface.

7.4.2 Take the wire cutters and snip the plastic tie thus opening the filter package exposing the dust collected in the filter.

7.4.3 weigh the dirty filter and plastic cable tie on the Mettler AE 166 balance. Record the "Dirty filter + Tie Weight (#6)" on the "Vacuum Dust Characterization" form (figure 1), sample "Chain of Custody" record (figure 2), and the "Vacuum Dust Aliquot chain of Custody" record (figure 3). The "Total Weight (#4)"minus the "Dirty Filter + Tie Weight (#6)" equals the "Sample Weight (#7)". Follow filter weighing protocols (UA-L-9.1) to determine validity. Weigh the standard weight closest to the heaviest sample weight and record the value under "total sample" along with the sample ID#.

7.4.4. the vacuum dust sample contains fine particulate material and a mass of fiberous material. A number of steps are involved in the processing of the vacuum dust sample (figure 4). Dump the contents of the filter onto a set of clean (UA-L-5.1) screens (Standard size 10, 230 and a solid bottom pan). Cover, transfer to the fume hood and shake gently at least 1 min. A couple of firm blows to the side of the sieves at the end concentrates each fraction and dislodges loose particle matter caught on each screen. Return to the cold surface to maintain low temperatures in the finest fraction (the bottom pan).

7.4.5 Set aside the top two screens onto a plastic tray. Return the finest fraction in the bottom pan to the cold surface for processing of the pesticide and metals sample aliquots.

7.5 Pesticide Aliquot

7.5.1. Approximately 1 gram of fine material (< #230 sieve) is needed to determine the pesticide content of the "Vacuum Dust Sample".

7.5.2 Remove the bottom pan containing the fine fraction and dump the contents onto the topmost piece of weighing paper.

7.5.3 Using a teflon coated spatula, split the sample in half scraping the material onto the underlying piece of weighing paper. If there appears to be less than 2 grams of material the pesticide sample is not split-out, with all of the material used for the metals sample.

7.5.4 Weigh the split on the overlying piece of weighing paper, recorded as "Weighing Paper Wt.(#10)", on the Mettler AE 166 balance. Record the weight of the sample under "Split Wt.(#8)" on the "Vacuum Dust Characterization" form (figure 1), the vacuum dust "Chain of Custody" record (figure 2) and the vacuum dust aliquot "Chain of Custody" record (figure 3). Follow the filter weighing protocols (UA-L-9.1) to determine validity.

7.5.5. Place the vacuum dust pesticide aliquot material into a pre-labled 125 ml nalgene bottle, tighten the cap and return to the freezer. Transfer the sample to the Material Tech to await shipment for pesticide analysis (UA-F-7.1).

7.6 Metals Aliquot

7.6.1 The remaining fine fraction is used for the metals aliquot

7.6.2 Weigh the remaining split of the underlying piece of weighing paper, recorded as "Weighing Paper Wt.(#11)", on the Mettler AE 166 balance. Record the weight of the sample under "Split Wt.(#9)" on the "Vacuum Dust Characterization" form (figure 1), the vacuum dust "Chain of Custody" record (figure 2) and the vacuum dust aliquot "Chain of Custody" record (figure 3). Follow the filter weighing protocols (UA-L-9.1) to determine validity. The "Split Wt.(#9)" minus the " Weighing Paper wt.(#11)" is the "Metals Aliquot Weight (#13)". Follow the filter weighing protocols (UA-L-9.1) to determine validity.

7.6.3 Weigh the standard weight which is closest to the weight of the samples and record the value and ID# under “Aliquots”.

7.6.4 The fine fraction is analyzed using the XRF (UA-L-10.1) then transferred to other laboratories for further analysis. From 1 to 2 grams of material is needed. The metals aliquot should easily fit into a plastic XRF cup.

7.6.5 Transfer the fine fraction XRF sample to a numbered XRF cup. Place X-ray mylar film over the top and secure with a plastic side ring. Fit the top ring onto the cup ensuring that the mylar film is stretched tight and flat. Record the cup number on the "Vacuum Dust Characterization" form (figure 1) and fill out the header information on an "XRF Analysis" form (figure 5) for that particular sample.

7.6.6 Place the XRF cup into a sample tray and place onto one of the stacked shelves next to the XRF (see SOP UA-L-10.1). Place the "XRF Analysis" form onto the top of the sample tray.

7.6.7 After XRF analysis, pour the sample into a small plastic bag. Label with the sample ID# and size fraction. Archive (room 130B) with the other size fractions

or ship to another laboratory for further analysis.

7.6.8 Repeat procedures 7.6.1 through 7.6.7 for each sample.

7.7 Larger Sieve Fractions

7.7.1 Weigh each remaining fraction, material from the #230 and #10 sieve, individually using the Mettler AE 166 balance. Enter the " >10 Screen Wt.(#14)" and "10-230 Screen Wt.(#17)" on the "Vacuum Dust Characterization" form (figure 1). The ">10 Screen Wt.(#14)" minus the "Weighing Paper Wt.(#15)" equals the ">10 Sample Wt.(#16)" The "10-230 Screen Wt.(#17)" minus "Weighing Paper Wt.(#18)" equals "10-230 Sample Wt.(#19)" Follow the filter weighing protocols (UA-L-9.1) to determine validity.

7.7.2 Place each of the samples into a small ziplock bag and return them to the original sample bag.

7.7.3 Weigh the standard weight which is closest to the weight of the samples and record the value and ID# under "Aliquots".

7.8 Calculations

7.8.1 A number of weights are taken throughout the processing of samples. These weights are used to calculate the sample size for later concentration calculations.

(#4)Total Wt. - (#3)Filter + Tie Wt. = (#5)Collected Wt.

(#4)Total Wt. - (#6)Dirty Filter + Tie Wt. = (#7)Sample Wt.

(#8)Split Wt. - (#10)Weighing Paper Wt. = (#12)Pesticide Aliquot Wt.

(#9)Split Wt. - (#11)Weighing Paper Wt. = (#13)Metals Aliquot Wt.

(#14)>10 Screen Wt. - (#15)Weighing Paper Wt. = (#16)>10 Sample Wt.

(#17)10-230 Screen Wt. - (#18)Weighing Paper Wt. = (#19)10-230 Sample Wt.

(#12) Pesticide Aliquot Wt. + (#13) Metals Aliquot Wt. + (#16) >10 Sample Wt. + (#19) 10-230 Sample Wt. = (#20)Total Screened Wt.

[(#7) Sample Weight - (#20) Total Screened Weight] *100 = % Dust Loss

7.9 Quality Control

7.9.1 Tolerance Limits

A. The "% Dust Loss" during processing is calculated to allow evaluation during data analysis.

7.9.2 Detection Limits

A. Each sieve screen is certified at the factory. The certificate of authenticity is on file in the Lab Supervisors office. An example of the authentication certificate appears in figure 6.

7.9.3 Corrective Actions

A. Invalid samples are discarded with no further processing.

8.0 RECORDS

8.1 Data Collected by this Procedure

8.1.1 The weights recorded on each "Chain of Custody" record (figure 2 and 3) will be transferred with the sample.

8.1.2 "Vacuum Dust Characterization" forms (figure 1) will be checked for completeness and then transferred to the data coordinator for further data processing.

8.2 Location/Placement of Forms

8.2.1 Blank forms will be kept with the Laboratory (room 130A) until needed.

8.2.2 Completed forms will be forwarded to the Lab Supervisor and onto the Data Coordinator for further data processing.

Figure 1. Vacuum Dust Characterization Form

VACUUM DUST CHARACTERIZATION FORM

Technician: _____ [ ][ ] Sample ID [ ][ ][ ][ ][ ][ ][ ] Status [ ]
Init Code
Start Date: ____/____/____ HHID [ ][ ][ ][ ][ ][ ][ ] FS [ ]

Total Sample √ √√

(#4)_____ g. - (#3) _____ g. = (#5) _____ g. 0 0
Total Wt. Filter + Tie Wt. Collected Wt.

(#4)_____ g. - (#6) _____ g. = (#7) _____ g. 0 0
Total Wt. Dirty Filter + Tie Wt. Sample Wt.

Screen Set [ ][ ] Archive Code [ ][ ]-[ ]-[ ][ ][ ][ ][ ]

Aliquots (<230 Fine Fraction)

Pesticide Sample ID# [ ][ ][ ][ ][ ][ ][ ]

(#8)_____ g. - (#10)_____ g. = (#12)_____ g. 0 0
Split Wt. Weighing Paper Wt. Pesticide Aliquot Wt.

Metals Sample ID# [ ][ ][ ][ ][ ][ ][ ]

(#9)_____ g. - (#11)_____ g. = (#13)_____ g. 0 0
Split Wt. Weighing Paper Wt. Metals Aliquot Wt.

XRF Cup # [ ][ ][ ] XRF Form Header Completed Y [ ] N [ ]

Other Fractions

(#14)_____ g. - (#15)_____ g. = (#16)_____ g. 0 0
>10 Screen Wt. Weighing Paper Wt. >10 Sample Wt.

(#17)_____ g. - (#18)_____ g. = (#19)_____ g. 0 0
10-230 Screen Wt. Weighing Paper Wt. 10-230 Sample Wt.

QA/QC Check

Total Screened Weight (#20)_____ g. 0 0

[(#7)_____ g. - (#20)_____ g.] * 100 = _____ 0 0
Sample Wt. Total Screened Wt. % Dust Loss

	Total Sample	Aliquots	Other Fractions	
Standard Wt. Number	_____	_____	_____	
Weight	_____ g	_____ g	_____ g	0 0

QC Initial _____ Date; ____/____/____

Figure 2. Vacuum dust sample chain of custody form.

Sample Weights and Chain of Custody Sheet
NHEXAS Arizona
Vacuum Dust Sample

√ √√

Sample ID#: _____ **HHID**: _____ **FS**: ____ **Status**: ____ 0 0

(#1)Filter:_____ g. (#2)Tie:_____ g. (#3)Filter + Tie:_____ g.
wt. wt. wt.

(#4)Total Wt.:_____ g. - (#3)_____ g. = (#5)_____ g. 0 0
Filter + Tie wt. Collected wt.

(#4)Total Wt.:_____ g. - (#6)_____ g. = (#7)_____ g. 0 0
Dirty Filter + Tie wt. Sample wt.

Standard Wt. #: _____ Weight: _____ g. 0 0

Pesticide Aliquot ID#: [ ][ ][ ][ ][ ][ ][ ] 0 0

(#8)Split Wt. _____ g. - (#10)_____ g. = (#12)_____ g. 0 0
Weighing Paper wt. Aliquot wt.

Metals Aliquot ID#: [ ][ ][ ][ ][ ][ ][ ] 0 0

(#9)Split Wt. _____ g. - (#11)_____ g. = (#13)_____ g. 0 0
Weighing Paper wt. Aliquot wt.

Standard Wt. #: _____ Weight: _____ g. 0 0

Custody Record

Relinquished or Received	Signature	Date mo./day/yr.	Time	Action
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	

Figure 3. Vacuum dust aliquot chain of custody form.

Sample Weights and Chain of Custody Sheet
NHEXAS Arizona
Vacuum Dust Aliquot Sample

√ √√

Sample ID#: _____ **HHID:** _____ **FS:** ____ **Status:** ____ 0 0

(#1)Filter:_____ g. (#2)Tie:_____ g. (#3)Filter + Tie:_____ g.
wt. wt. wt.

(#4)Total Wt.:_____ g. - (#3)_____ g. = (#5)_____ g. 0 0
Filter + Tie wt. Collected wt.

(#4)Total Wt.:_____ g. - (#6)_____ g. = (#7)_____ g. 0 0
Dirty Filter + Tie wt. Sample wt.

Standard Wt. #: _____ Weight: _____ g. 0 0

Pesticide Aliquot ID#: [ ][ ][ ][ ][ ][ ][ ] 0 0

(#8)Split Wt. _____ g. - (#10)_____ g. = (#12)_____ g. 0 0
Weighing Paper wt. Aliquot wt.

Metals Aliquot ID#: [ ][ ][ ][ ][ ][ ][ ] 0 0

(#9)Split Wt. _____ g. - (#11)_____ g. = (#13)_____ g. 0 0
Weighing Paper wt. Aliquot wt.

Standard Wt. #: _____ Weight: _____ g. 0 0

Custody Record

Relinquished or Received	Signature	Date mo./day/yr.	Time	Action
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	
[Rel] [Rec]		___/___/___	___:___	

Figure 4. Flow of the sample and splits through sample processing.

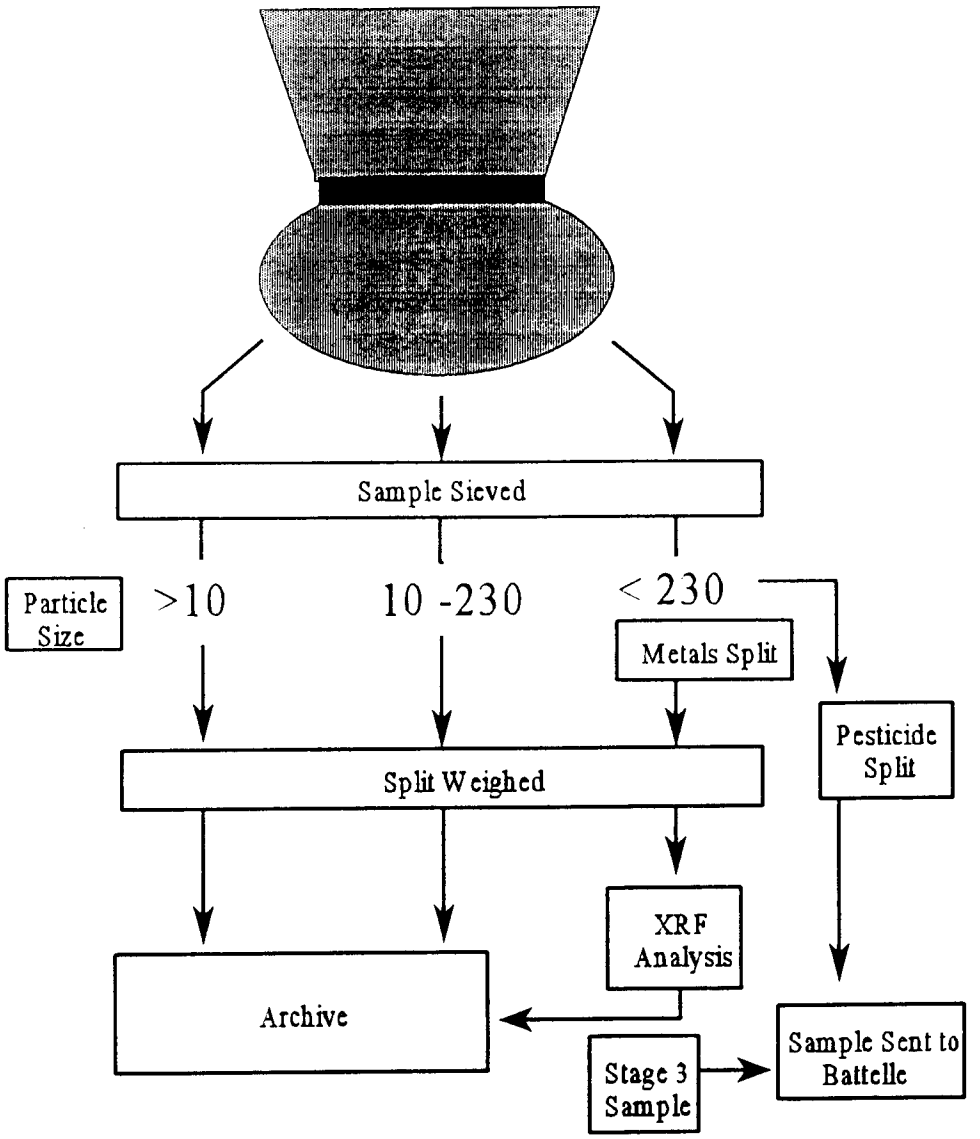

Figure 5. XRF Analysis Form.

XRF ANALYSIS

Technician Init._____ Code [ ][ ] Date _____/_____/_____ Time [ ][ ]:[ ][ ]

Sample ID# [ ][ ][ ][ ][ ][ ][ ] Source Time: Fe-55 [ ][ ][ ]sec. Cd-109 [ ][ ][ ]sec. Am-241 [ ][ ][ ]sec. Application: Thin Film [ ] Soils [ ]

Element	Reading (μg/cm²; ppm)	Standard Deviation
Pb	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
As	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Cd	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ni	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Cr	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ba	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Mn	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Se	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
V	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Cu	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Zn	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
K	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ca	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Co	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Fe	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Mo	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Tl	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ag	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Sr	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
U	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Th	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Sn	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
W	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ti	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Rb	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Ir	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]
Hg	[ ][ ][ ][ ].[ ][ ][ ][ ]	[ ][ ][ ][ ].[ ][ ][ ][ ]

QA/QC Signature: _____ [ ] [ ]