

# National Human Exposure Assessment Survey (NHEXAS)

## *Arizona Study*

## Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona  
Tucson, Arizona 85721

Cooperative Agreement CR 821560

**Standard Operating Procedure**

**SOP-UA-L-1.2**

**Title:** Calibration and Operation of NHEXAS Balances

**Source:** The University of Arizona

U.S. Environmental Protection Agency  
Office of Research and Development  
Human Exposure & Atmospheric Sciences Division  
Human Exposure Research Branch

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## **Calibration and Operation of NHEXAS Balances**

### **1.0 Purpose and Applicability**

The purpose of this SOP is to describe the procedures used when calibrating and operating Balances during the EPA NHEXAS and Border projects of the University of Arizona/Battelle/Illinois Institute of Technology consortia, as well as future "Health in the Environment" investigations.

### **2.0 Definitions**

- 2.1 Control weight = An identified metallic object of a selected weight used to monitor balance precision over time.
- 2.2 Down Equipment Area = Area in which defective equipment is placed to await either repair in-house or shipment to a repair facility.
- 2.3 Down Equipment Label = Label which is attached to a defective piece of equipment to identify it as being defective.
- 2.4 SOP = Standard Operating Procedure
- 2.5 Standard weight = A reference standard weight that has a known valid relationship with a recognized national standard.

### **3.0 References**

- 3.1 Model AE163 Operating Instructions, Mettler Instrument Corporation, Hightown, NJ, 1982.
- 3.2 Assuring Balance Accuracy, J. Weil, Cahn Instruments, Inc., Cerritos, CA, 1984.
- 3.3 Acculab, Model #V-1200 Operating Instructions, Acculab, Newton PA. No Date.
- 3.4 Mettler Toledo BalanceLink (V2.0) Software. Operating Instructions, Mettler Instrument Corporation, Hightstown NJ, 1994.

### **4.0 Discussion**

This procedure is directly applicable to the calibration and operation of balances used to weigh a variety of materials during the NHEXAS and Border projects, as well as future "Health in the Environment" investigations. The balances include; a Mettler AE 163 (UA ID# A195535) with a readability set at 0 to 162 grams at  $1 \pm 0.5$  mg and 0 to 60 grams at  $0.1 \pm 0.1$  mg. This balance is connected to a computer and readings are digitally transferred to the computer screen during operation; a Mettler AE 166 (UA ID# A201335), with a readability set at 0 to 162 grams at  $0.1 \pm 0.02$  mg and set at 0 to 31 grams at  $0.01 \pm 0.1$  mg; and an Acculab #V-1200 (ID# 50090339) with a readability of  $0.1 \pm 0.1$  g, set to 0 to 600 grams, and  $0.1 \pm 0.2$ , set to read 601 to 1200 g.

## **5.0 Responsibilities**

- 5.1 The Project Director will be responsible for:
  - 5.1.1 Final review and approval of each procedure.
- 5.2 The Project Laboratory Supervisor is 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 dysfunctional item.
  - 5.2.3 Notifying and scheduling appropriate factory calibration for the item of equipment.
- 5.3 The Project Lab Technicians 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 Materials and Equipment**

- 6.1 Materials
  - 6.1.1 Acculab #V-1200 balance (ID# 50090339)
  - 6.1.2 Bachrach humidity/temperature recorder
  - 6.1.3 Balance Log Book for the Mettler AE 163
  - 6.1.4 Balance Log Book for the Mettler AE 166
  - 6.1.5 Balance Log Book for the #V-1200 balance
  - 6.1.6 Class "S" standard calibration weight 100 mg (#9800007)
  - 6.1.7 Class "S" standard calibration weight 20 mg (#9800008)
  - 6.1.8 Mettler model 163 balance (UA ID# A195535)
  - 6.1.9 Mettler model 166 balance (UA ID# A201335)
  - 6.1.10 Mettler Toledo BalanceLink Software (#ME410023)
  - 6.1.11 Standard Brass calibration weight 1000 g (#9800009)
  - 6.1.12 Standard Brass calibration weight 100 g (#9800010)
  - 6.1.13 Standard Brass calibration weight 10 g (#9800011)
  - 6.1.14 Standard Brass calibration weight 5g (#9800017)

6.1.15 Standard Brass calibration weight 1g (#9800018)

6.1.16 Teflon coated forceps

## 7.0 Procedure

### 7.1 Preparation

#### 7.1.1 Siting Criteria

Not applicable

#### 7.1.2 Reagents

Not applicable

#### 7.1.3 Samples/Actions:

##### A. Balance Area Preparation

1. Wipe down working area of table adjacent to balance with damp towel.

### 7.2 Analyses/Procedures

#### 7.2.1 Standards/Blanks

- A. Control weights are the same as the "Standard Weights" in 7.2.2 (B)7 through 7.2.2 (B)12 and 7.2.2 (C)3 through 7.2.2 (C)7

#### 7.2.2 Balance Preparation for Operation

##### A. Mettler Balances AE 163 and AE 166

1. Check level of balance via the integral spirit level.
2. Close all balance doors.
3. Turn on balance, 20 minutes prior to use.
4. Depress single control bar to turn display on; wait for 0.00000 to appear in display on the AE 163 and 0.0000 on the AE 166.
5. Set the balance to the lowest range. With the AE 163 the 30 g setting is achieved by holding the control bar down until "rng" is displayed. Depress the control bar to toggle between 30 g and 160 g. The AE 166 automatically switches to the higher mode if material heavier than 60 g is placed on the weighing pan.
6. Depress the control bar to set the following ranges for the greatest weighing accuracy;
  - a. Cal
  - b. Int -3-
  - c. ASd -1-"CAL" is the calibration procedure discussed in 7.2.2.B.
7. Release control bar when 0.00000 reappears on the AE 163 display and 0.0000 reappears on the AE 166 display.
8. Record the start-up data in the balance logbook for the balance to be used, and on the appropriate boxes on the weighing forms (figures 1-6) to be used during each weighing session.

9. To setup the computer link with the AE 163 follow section 7.2.3.
- B. Calibration procedure for the Mettler Balances (AE 163 and AE 166):
  1. Depress and hold down control bar until "CAL" appears; then release the bar.
  2. When "100.000" blinks on the display, slide CAL knob on lower right side of balance to rear stop.
  3. When "Cal 0" blinks, return CAL knob to forward stop.
  4. The following displays should be observed:
    - a. ---- (blinks)
    - b. 0.0000
  5. The balance is now zeroed; observe 0.0000
  6. Select one of the standard weights discussed in step 10 below, and place it in the middle of the balance pan.
  7. Close all balance doors.
  8. Wait 5 seconds after stability indicator dot goes out before recording displayed weight.
  9. Record observed weight and serial number of standard weight in the respective calibration log book.
  10. Repeat steps 7-9 for each of the standard weights used with each scale.
    - a. Class "S" 20 mg, serial #9800008 (AE163)
    - b. Class "S" 100 mg, serial #9800007 (AE 163)
    - c. Standard brass calibration weight 10g, serial #9800011 (AE 166)
    - d. Standard brass calibration weight 5g, serial #9800017 (AE 166)
    - e. Standard brass calibration weight 1g, serial #9800018 (AE 166)
  - 11 For the calibration to be valid the recorded standard and control weights will be  $\pm 5\%$  of their recognized value (Table 1).
- C. Acculab #V-1200
  1. Push the "on/memory" button turning the balance on 20 minutes prior to use.
  2. To Calibrate;
    - a. Press and hold the "CAL" key until 1000.0 appears and flashes on the display.
    - b. Press the "tare" key and the display will read "CAL 0". Pausing for at least two seconds, then place a 1000 g calibration weight gently on the center of the tray top.
    - c. The display momentarily shows "CAL F" and then returns to active weighing. Calibration is complete when the display correctly shows the calibration weight, 1000 g, and the display has stabilized.
    - d. If "CAL E" appears instead of "CAL F", a calibration error is indicated

and the calibration process must be repeated. "CAL E" can occur if the calibration steps are not followed correctly or if an incorrect calibration weight is applied.

- e. Remove the calibration weight and press the tare key to reset the zero point.
3. Record the success of the calibration in the Acculab #V-1200 log book.
4. Place a standard brass calibration weight on to the scale and wait for the stability indicator to appear.
5. Record the observed weight and serial number in the Acculab #V-1200 calibration log book.
6. Repeat steps 4-5 for each of the following standard weights;
  - a. Standard brass calibration weight, 100g (#9800010).
  - b. Standard brass calibration weight, 10g (#9800011).
  - c. Standard brass calibration weight, 5g (#9800017).
  - d. Standard brass calibration weight, 1g (#9800018).
7. If the standard weights are within 1% of their recognized value (Table 1), the calibration is valid.

#### 7.2.3 Computer Preparation

- A. Turn on the computer and login using your name and password.
- B. Type "**cd\balance**" to go to the "Balance" sub-directory.
- C. Once in the Balance subdirectory, type "**Balink Numeric Stable Right**" to start and setup the balance program. Follow directions in UA-L-9.1 (7.1.2.A-E; 7.5.1 A-J) for computer balance interface use.

#### 7.2.4 Actions

- A. General weighing procedure Mettler Balances (AE 163 and AE 166)
  1. Calibrate each scale prior to each operating session.
  2. Close all doors and zero the balance by momentarily depressing the left side of the control bar. Observe 0.0000
  3. Pass the specimen over a static eliminator bar.
  4. Open the appropriate door and carefully place the sample in the center of the pan.
  5. Close all doors and wait for the stability indicator dot to leave the display.
  6. Record onto the appropriate weighing backup form (figures 1-3, 6) the specimen weight that is displayed 5 seconds after the stability indicator dot goes out. On the AE163, which is connected to the computer, press the hand-switch to transfer the weight to the computer screen. Check that it is recorded with the appropriate sample ID#.
  7. Remove specimen, close all doors, and observe that the scale returns to 0.0000. If not, re-zero the balance between each weighing by momentarily depressing the control bar. Specific quality control issues are discussed

with each sample type (see UA-L-9.1).

8. A control weight of known value is weighed and recorded between each 5 successive specimen weight determinations for accuracy assurance (figures 1-6). If the scale does not record the standard within 5% of its known weight the equipment is labeled as defective and the scale must be recalibrated. All samples that were weighed after the last good accuracy assurance weighing must be re-weighed.
  9. Turn off balance display by momentarily lifting up on control bar when finished.
  10. Return all supplies used during weighing operation to their designated storage areas.
- B. General weighing procedure for the Acculab #V-1200.
1. Perform calibration procedure prior to each operating session.
  2. Place the material to be weighed onto the weighing pan, gently.
  3. Record the specimen weight that is displayed 5 seconds after the stability indicator arrow appears on the display screen.
  4. Remove the specimen and observe that the scale returns to 0.0. Press the "Tare" button to return the scale to 0.0 at any point during a procedure.
  5. A control weight of known value (5g, 10g or 100g) is weighed and recorded between each 5 successive specimen weight determinations for accuracy assurance. If the scale does not record the standard within 5% of its known weight ( $10 \pm 0.5$  g or  $100 \pm 5$  g), the equipment is labeled as defective and must be recalibrated. All samples weighed after the last good accuracy assurance weighing must be re-weighed.
  6. Turn off balance display by pressing the "Off" button.
  7. Return all supplies used during the weighing operation to their designated storage areas.

7.3 Calculations  
Not applicable

7.4 Quality Control

7.4.1 Tolerance limits

A. Mettler Balance AE163

1. At a weighing range of 0 to 31g, the reproducibility (standard deviation) is 0.02 mg.
2. At a weighing range of 0 to 162g, the reproducibility (standard deviation) is 0.1 mg.
3. A failure to reproduce standard weights within 5% of their recorded weight

constitutes an equipment failure and results in recalibration. Three successive equipment failures and recalibration results in labeling the item as "down equipment". A manufacturers representative is then consulted or the equipment is returned for factory calibration.

B. Mettler Balance AE166

1. At a weighing range of 0 to 31g the reproducibility (standard deviation) is 0.02 mg.
2. At a weighing range of 0 to 162g the reproducibility (standard deviation) is 0.1 mg.
3. A failure to reproduce standard weights within 5% of their recorded weight constitutes an equipment failure and results in recalibration. Three successive equipment failures and recalibration results in labeling the item as "down equipment".

C. Acculab #V-1200

1. At a weighing range of 0 to 600g the reproducibility (standard deviation) is 0.1g.
2. At a weighing range of 601 to 1200g the reproducibility (standard deviation) is 0.2 g.
3. A failure to reproduce standard weights within 5% of their recorded weight constitutes an equipment failure and results in recalibration. Three successive equipment failures and recalibration results in labeling the item as "down equipment". A manufacturers representative is then consulted or the equipment is returned for factory calibration.

7.4.2 Detection Limits

A. Mettler AE163 and AE 166

1. When the balance is set to a range of 0 to 30g it can be read to 0.01 mg.  
When set at a range up to 160g it can be read to 0.1 mg

7.4.3 Corrective Actions

A. Routine Maintenance (Mettler AE 163 and 166)

1. A corporate representative will clean, adjust, and calibrate both balances once a year.
2. After the 3rd failure to reproduce the standard weights within the stated variation, a corporate representative will be called to calibrate and adjust the equipment.

B. Acculab #V-1200

1. Failure to reproduce the standard weights within the stated variation 3 times will constitute a failure and the balance will be sent back to the factory for calibration.

8.0 Records



8.1 Data collected by this procedure.

8.1.1 Balance calibration data

- A. The date, calibration weight serial #, measured calibration weight and operator's initials are recorded in each balance's respective log book at the beginning of each weighing session.
- B. Standard weights measured during the weighing procedure will be recorded in the data files containing the filter weights (UA-L-9.1; 7.5).
- C. Cleaning and Maintenance are recorded in the balance log book.
- D. Balance audits by an outside vendor are recorded in the balance log book, with certificates of accuracy kept on file by the lab supervisor.

8.2 Location/Placement of Log Books and Forms

8.2.1 The "Balance Log Books" will be kept near each respective balance in the weighing room and the wet lab.

- A. Removal of a "Balance Log Book" will be recorded on a sign-out sheet kept with each balance.

8.2.2 Blank sample weighing forms (figures 1-6) are kept in a stacked file located in the lab (Room 130A). Completed forms are returned to the lab supervisor for QA/QC checks and transfer to the data coordinator.

Table 1. Standard weight values recorded during calibration of each of the balances. All values reported in grams.

	Mean	Standard Deviation ( $1\sigma$ )	n
<b>Scale #V-1200</b>			
#9800010	99.97	0.0455	229
#9800011	9.96	0.0486	229
#9800017	4.95	0.0603	175
<b>Scale AE166</b>			
#9800011	10.008	0.00013	190
#9800017	5.000	0.00009	190
#9800018	1.004	0.00008	190
#9800007	0.100	0.00005	66
#9800008	0.020	0.00004	66
<b>Scale AE163</b>			
#9800007	0.09996	0.00051	81
#9800008	0.01994	0.00055	81

## 25mm and 37mm TEFLO FILTER PRE-WEIGHING BACKUP FORM

File Name: \_\_\_\_\_

**All Weights Are Recorded In Grams (g).**

[illegible]

L-9.0-1.1

### 25mm and 37mm TEFLO FILTER POST-WEIGHING FORM

File Name:

**All Weights Are Recorded In Grams (g).**

Comments:

Figure 3. Sentinel Filter Pre-Weighing Form

### SENTINEL FILTER PRE-WEIGHING BACK-UP FORM

Equilibration Start Date \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Equilibration Start Time [ ][ ]:[ ][ ][ ]  
Technician [ ][ ] \_\_\_\_\_  
Code Init. \_\_\_\_\_

Balance  
0 AE 163  
0 AE 166  
0 #V-1200

Weighing Date \_\_\_\_\_/\_\_\_\_\_/\_\_\_\_\_  
Technician [ ][ ] \_\_\_\_\_  
Code Init. \_\_\_\_\_

Temperature [ ][ ][ ] °F  
Relative Humidity [ ][ ][ ] %

File Name: \_\_\_\_\_

All Weights Are Recorded In Grams (g).

Sample ID#	Weight	Status	QA/QC
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0

[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0
[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ][ ][ ][ ][ ][ ][ ]	[ ][ ][ ]	0 0

Standard Weight \_\_\_\_\_ g. Number \_\_\_\_\_ 0 0  
Standard Weight \_\_\_\_\_ g. Number \_\_\_\_\_ 0 0

Comments:

## SENTINEL FILTER POST-WEIGHING BACK-UP FORM

0 #V-1200

$$[\quad][\quad]\%$$

**All Weights Are Recorded In Grams (g).**

L-9.0-4.1

Figure 5. Vacuum Dust Filter "Chain of Custody Form"

<b>Sample Weights and Chain of Custody Sheet</b> <b>NHEXAS Arizona</b> <b>Vacuum Dust Sample</b>				
<b>Sample ID# :</b> _____				<b>QA/QC</b> 0 0
Filter Weight : _____ g. Tie Weight : _____ g. Filter + Tie Weight: _____ g				0 0
Total Wt.(#4) : _____ g. -- Filter + Tie Wt.(#3) _____ g. = Collected Wt.(#5) _____ g.				0 0
<b>Pesticide Aliquot ID#:</b> _____				0 0
Split Wt.(#8) _____ g. -- Weigh Paper(#10) _____ g. = Aliquot Wt.(#12) _____ g.				0 0
<b>Metals Aliquot ID#:</b> _____				0 0
Split Wt.(#9) _____ g. -- Weigh Paper(#11) _____ g. = Aliquot Wt.(#13) _____ g.				0 0
Standard Weight #: _____ Weight: _____ g.				0 0
<b>Custody Record</b>				
Relinquished or Received	Signature	Date mo./day/yr.	Time	Action
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	
[Rel] [Rec]		____/____/____	____:____	

Figure 6. Vacuum Dust Characterization Form

<b>VACUUM DUST CHARACTERIZATION FORM</b>										Balance	
Technician _____ [ ][ ]				Start Date ____/____/____				0 AE 163			
Init. Code								0 AE 166			
Samp. ID [ ][ ][ ][ ][ ][ ][ ]				HHID [ ][ ][ ][ ][ ][ ]				F.S. [ ]		Status [ ]	
								0 #V-1200			
<b>1. Total Sample</b>											
Total Wt.(#4) _____ g. - Filter + Tie Wt.(#3) _____ g. = Collected Wt.(#5) _____ g.										0 0	
Total Wt.(#4) _____ g. - Dirty Filter + Tie Wt.(#6) _____ g. = Sample Wt.(#7) _____ g.										0 0	
Screen Set [ ][ ] Archive Code [ ][ ]-[ ][ ]-[ ][ ][ ][ ][ ]											
<b>2. Aliquots</b>											
Pesticide Sample ID# [ ][ ][ ][ ][ ][ ][ ]											
Split Wt.(#8) _____ g. - Weigh Paper Wt. (#10) _____ g. = Pest Samp. Wt.(#12) _____ g.										0 0	
Metals Sample ID# [ ][ ][ ][ ][ ][ ][ ]											
Split Wt.(#9) _____ g. - Weigh Paper Wt. (#11) _____ g. = Pest Samp. Wt.(#13) _____ g.										0 0	
XRF Cup # [ ][ ] XRF Form Header Completed Y [ ] N [ ]											
<b>3. Other Fractions:</b>											
>10 Wt. (#14) _____ g. - Weigh Paper Wt. (#15) _____ g. = >10 Samp. Wt.(#16) _____ g.										0 0	
10-230 Wt.(#17) _____ g. - Weigh Paper Wt.(#18) _____ g. = 10-230 Samp. Wt.(#19) _____ g.										0 0	
<b>4. Standard Weights: ID#</b>											
Total Sample: [ ][ ][ ][ ][ ][ ][ ]								Weight _____ g		0	
Aliquots: [ ][ ][ ][ ][ ][ ][ ]								Weight _____ g		0	
Other Fractions: [ ][ ][ ][ ][ ][ ][ ]								Weight _____ g		0	
<b>5. QA/QC Check</b>											
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 _____										0 0	
Weight _____ g. _____ g. _____ g.										0 0	
QA/QC Signature; _____										Date; ____/____/____	