

MOISTURE ANALYZER & DRY WEIGHT REPORTING

Method: NA

Approvals (Signature/Date):

Carol McNulty 12/2/08
Carol McNulty
Custody Supervisor

Sal Ramos 12/2/08
Salvador Ramos
Health & Safety Manager / Coordinator

Lori Mangrum 12/3/08
Lori Mangrum
Quality Assurance Manager

Keith Blanchard 12/2/08
Keith Blanchard
Laboratory Director

John Shanholtzer 12-2-08
John Shanholtzer
Technical Director

This SOP was previously identified as SOP No. TPA-CU-003 Rev. 0

Copyright Information:

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2008 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. _____

Distributed To: _____

1.0 Scope and Application

- 1.1 The purpose of this SOP is to define procedures for the calibration of the Mettler Toledo HB43 Moisture Analyzer, the measurement of percent moisture and the upload of dry weight results into the LIMS.
- 1.2 In many cases, sample results are calculated on a dry-weight basis. When such data is desired, a portion of the sample will be utilized in Sample Custody for moisture determination.
- 1.3 Procedures defined in this SOP will affect the use of each of the HB43 infrared drying/weighing units located in Sample Custody.
- 1.4 The current Reporting Limit (RL), Method Detection Limit (MDL) and precision and accuracy limits associated with this procedure are given in the Method Limit Group (MLG) in LIMS.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 20 of the Tampa Quality Assurance Manual.

2.0 Summary of Method

The percent moisture determination is accomplished by placing a homogenized soil or sediment sample (with foreign objects such as sticks, leaves and rocks removed) in an infrared drying/weighing unit and measuring its initial weight. The sample is then heated to complete dryness and the final weight is measured. The percent moisture is calculated by the instrument using the difference between the initial and final weights and dividing the result by the initial weight.

3.0 Definitions

Refer to SOP TP-AN-005: Definitions, Terms, and Acronyms for a complete listing of applicable definitions and to the current revision of the Tampa Quality Assurance Manual (TP-QAM).

4.0 Interferences

The primary cause of interference for this method comes from containers that have not been properly cleaned or prepared prior to analysis. The basis of this procedure is the difference in the weight of the aluminum pan containing the sample before and after drying. Therefore, care must be taken to insure that the aluminum pan holder is clean and completely dry for each sample so as to not add or lose weight.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- 5.1.1 The HB43 Moisture Analyzer works with heat! Use caution when reaching into the balance because the sample and sample container, the sample chamber and the draft shield may still be very hot.
- 5.1.2 Ensure sufficient free space around the instrument to avoid heat accumulation and overheating (approx. 1 m free space above the instrument).
- 5.1.3 The HB43 analyzer should be vented because significant contamination of the air may result from drying a heavily contaminated hazardous waste sample. The vent over the sample must never be covered, plugged, taped over or tampered with in any other way.
- 5.1.4 Do not place any combustible materials on, under or next to the instrument when it is connected to the power supply, since the area around the heating module becomes hot.
- 5.1.5 Be very careful if you open the heating module during operation, because the circular heating module or its protective glass can be as hot as 400 °C! For this reason, only ever touch the handles provided on the heating module.
- 5.1.6 No modifications must be made within the heating module. It is particularly dangerous to bend any components or remove them, or to make any other changes.

5.2 Primary Materials Used

There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 **Equipment and Supplies**

6.1 Instrumentation

- 6.1.1 Mettler Toledo HB43 Moisture Analyzer (Infrared Dryer with balance)
- 6.1.2 Mettler Toledo Calibration Kit (Part# 214528)

6.2 Supplies

- 6.2.1 Calibration Weights

6.2.2 Disposable aluminum pans for holding the sample

6.2.3 Disposable spatulas or spoons for stirring and transferring the sample

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Reagent water - de-ionized water Type II

7.1.2 Ottawa Sand - commercially purchased

7.2 Standards

7.2.1 Wetted Ottawa Sand (10% Moisture, 90% Dry Weight) - Add 25mL (g) of reagent water to 225g of clean and dry Ottawa Sand in a soil jar. Seal tightly and refrigerate.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

| Matrix | Sample Container | Min. Sample Size | Preservation | Holding Time | Reference |
|--------|------------------|------------------|--------------------------------|--------------|-----------|
| Soils | Glass | 5 grams | Cool $4 \pm 2^{\circ}\text{C}$ | 7 Days | N/A |

9.0 Quality Control

9.1 Continuing Calibration Verification (CCV) - A sample with known percent moisture must be analyzed **daily** on the HB43 analyzer in order to confirm proper functioning of the infrared dryer. The acceptance range for recovery is 90 - 110%.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Samples for Semivolatile Organics, Inorganics and Metals

Mix the sample thoroughly, especially composite samples, prior to beginning the percent moisture analysis. Discard any foreign objects such as sticks, leaves and rocks.

10.1.2 Samples for Volatile Organics

The sample vial for volatile analysis must remain unopened until the time of analysis to avoid the loss of volatile compounds. Therefore, a separate

container (approximately 60g capacity) filled with the same sample should be received for the percent moisture analysis. The contents of the secondary sample container should be mixed well prior to beginning the percent moisture analysis. If the secondary container is not provided, notify the project manager responsible for the sample immediately.

10.1.3 Treatment of "Non-homogeneous" Samples

If the client does not split a "non-homogeneous" sample that will be logged in for more than one analysis (in more than one department), the sample will be homogenized by one of the departments performing the analysis as follows:

- 10.1.3.1 According to SOP TP-AN-070: *Homogenization, Composition and Segregation of Samples*, mix the sample thoroughly.
- 10.1.3.2 Quickly pour the sample into the appropriate sample containers (before it separates again).
- 10.1.3.3 Working with Sample Custody, label the homogenized samples and make sure that they are delivered to the appropriate departments.

10.2 Calibration

10.2.1 Balance Calibration

10.2.1.1 Daily Balance Calibration

- 10.2.1.1.1 Prior to calibration, make sure that the HB43 analyzer is level by looking at the level glass in the back right portion of the instrument. If it is not level, adjust the legs of the unit until the bubble in the glass indicates that it is.
- 10.2.1.1.2 The Oven Balance needs to be calibrated **daily** using the 1.000g and 5.000g weights. Use the tool provided with the weights to handle them (**DO NOT** use your bare hands).
- 10.2.1.1.3 Fill out the Daily Calibration Log (see Attachment 2 for an example). The 1.000g weight has a tolerance of ± 0.004 g and the 5.000g weight has a tolerance of ± 0.005 g. Any time that a weight does not fall in the tolerance range, the HB43 unit can not be used to run samples. Report the failure immediately to the QA Officer for resolution of the problem.
- 10.2.1.1.4 If the analyzer unit is moved after calibration, then the balance must be recalibrated.

10.2.1.1.5 When taking the weight readings, keep the balance free from drafts (as much as possible).

10.2.1.2 Weekly Balance Calibration

10.2.1.2.1 Prior to calibration, make sure that the HB43 analyzer is level by looking at the level glass in the back right portion of the instrument. If it is not level, adjust the legs of the unit until the bubble in the glass indicates that it is.

10.2.1.2.2 The Oven Balance needs to be calibrated **weekly** using the 1.000g, 5.000g, 10.000g, 20.000g, and 30.000g weights. Use the tool provided with the weights to handle them (**DO NOT** use your bare hands).

10.2.1.2.3 Fill out the Weekly Calibration Log (see Attachment 3 for an example). The weight tolerance ranges for each weight are found in Section 11.4. Any time that a weight does not fall in the tolerance range, the HB43 unit can not be used to run samples. Report the failure immediately to the QA Officer for resolution of the problem.

10.2.1.2.4 If the analyzer unit is moved after calibration, then the balance must be recalibrated.

10.2.1.2.5 When taking the weight readings, keep the balance free from drafts (as much as possible).

10.2.2 Infrared Dryer Calibration

Every 3 months, the accuracy of the Infrared Dryer temperature calibration must be checked with the Mettler Toledo Calibration Kit. The instructions for using the calibration kit are included with the kit.

10.3 Sample Analysis

10.3.1 Switch the instrument on by pressing the “**On/Off**” key.

NOTE: The instrument performs a self-test and the status display will show the initial status of the system when the heating module is closed. If the **Auto** mode (utilizing the SmartStart function) is used, it will not be necessary to press the “**Tare-O/T**” key to operate the instrument.

10.3.2 Double -click on the CollectXL 5.1 icon on the desktop.

10.3.3 Select **Open** under the **File** option on the menu toolbar at the top of the screen.

10.3.4 Use the scanner to enter or type the LIMS log number into the appropriate column under the Oven number of the analyzer being used. Make sure to

click on the corresponding empty cell prior to scanning or typing the number.

10.3.5 Open the heating module.

10.3.6 The status display will flash and prompt for loading the empty sample pan.

10.3.7 Position the empty sample pan in the sample pan handler. Place the sample pan handler in the draft shield. Ensure that the tongue of the sample pan handler lies exactly in the slot of the draft shield. The pan must lie flat in the pan holder.

NOTE: Use of the sample pan handler is advised at all times. The pan handler is ergonomic, safe and provides protection against burns due to the hot sample pan.

10.3.8 Close the heating module. The built-in balance will then automatically set itself to zero.

NOTE: If the "**Tare-O/T**" key is used, the status display will prompt for closure of the heating module. Tare the pan and the status display prompts for the addition of the sample to the sample pan.

10.3.9 Open the heating module.

10.3.10 Add the sample (3 -5g) to the sample pan. Make sure that the sample is distributed evenly to obtain thorough drying and accurate analysis results.

10.3.11 The status display now prompts for the start of the drying process. Close the heating module and the instrument will automatically start the drying and measuring process. The progress of the drying process (current temperature, elapsed drying time and the current drying value) will be continuously updated in the status display until it is finished.

10.3.12 As soon as the preselected switch-off criterion is met (or the selected drying time has elapsed), an audio signal sounds.

10.3.13 The percent moisture measurement can now be read off of the status display (The result and time display remain at their final values, whereas the temperature continues to be updated). The initial and final weights of each sample are automatically uploaded from the instrument to the Excel spreadsheet.

10.3.14 It is a good idea to save the data from time to time in case of power outages, distractions, etc. Save the file by clicking the Floppy disk-shaped icon in the toolbar (The computer will prompt that this is a "**Read-only file**", Click **OK**) or by selecting **Save As** under the **File** option on the menu toolbar at the top of the screen.

10.3.15 Browse to the **Desktop** heading and save the data by changing the filename from **Method Template** to a filename containing the date and a letter (i.e. 09.15.08A).

10.3.16 Open the heating module. The measurement result is now deleted from the status display.

NOTE: If the analyzer is being manually operated, the measurement result can be cleared by pressing either the "Tare-O/T" or "Reset" keys.

10.3.17 Carefully remove the sample pan handler from the sample chamber. To remove the sample pan from the handler, lift the pan slightly from below and pull it sideways out of the handler (if you no longer need the sample and the pan, you can simply tilt the handler until the pan slides out).

Warning: Pan and sample may still be hot! Allow them to cool before removing the pan from the handler!

10.3.18 The reset instrument is now ready for the next measurement. Repeat Sections 10.3.2 to 10.3.12 for each sample.

10.3.19 When the analysis is complete and all the data has been saved under a unique filename, follow the instructions in Attachment 1 to upload the data into LIMS.

11.0 Calculations / Data Reduction

11.1 Percent Dry Weight Calculation

The formula for the EPA (SW-846) procedures is equivalent to the calculation performed by the Infrared Dryer.

EPA SW-846 Formula for Percent Dry Weight

$$\% \text{ Dry Weight} = \frac{\text{Weight (g) of dry sample}}{\text{Total weight (g) of sample}} \times 100$$

HB43 Infrared Dryer Calculation

$$\% \text{ Dry Weight} = \frac{\text{Dried weight (g) of sample}}{\text{Wet weight (g) of sample}} \times 100$$

11.2 Dry weight results will be uploaded to LIMS daily. See attachment 1 for detailed instructions for the LIMS upload process.

- 11.3 The dry weight results will be used to adjust sample quantitation results and reporting limits on all solid samples.
- 11.4 The acceptance ranges for the weights used for daily and weekly calibrations are listed in the following table.

| ACCEPTANCE RANGES | |
|-------------------------|---------------|
| ASTM Class 1 Weights | Tolerance |
| 1.0 g | ± 0.004 g |
| 5.0 g | ± 0.004 g |
| 10 g | ± 0.005 g |
| 20 g | ± 0.005 g |
| 30 g | ± 0.005 g |

12.0 Method Performance

This section is not applicable to this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Tampa's current revision of SOP TP-HAZ-001 *Waste Management*).

15.0 References / Cross-References

- 15.1 *Installation and Operating Instructions, Mettler HB43 Moisture Analyzer, Mettler © Mettler-Toledo GmbH 2005 1 1780531A Printed in Switzerland 0512/2.12 Mettler-Toledo GmbH, Laboratory & Weighing Technologies, CH-8606 Greifensee, Switzerland Phone +41-1-94422 11, Fax +41-1-9443060, Internet: www.mt.com*
- 15.2 *Manual for the Certification of Laboratories Analyzing Drinking Water, EPA 815-B-97-001, March 1997.*
- 15.3 *Laboratory Operations and Quality Custody Manual, EPA Region IV, Sep 1990.*
- 15.4 *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW846), EPA SW-846, most current revision.*
- 15.5 *TestAmerica's Environmental Health & Safety Manual, CW-E-M-001, most current revision.*
- 15.6 *Tampa's Quality Assurance Manual (TP-QAM), most current revision.*
- 15.7 *Balance Calibration and Use, TestAmerica Tampa SOP TP-AN-012, most current revision.*
- 15.8 *Homogenization, Composition and Segregation of Samples, TestAmerica Tampa SOP TP-AN-070, most current revision.*

16.0 Method Modifications

This section is not applicable to this SOP.

17.0 Attachments

Attachment 1: Instructions for LIMS Data Upload

Attachment 2: Daily Balance Calibration Logbook Page

Attachment 3: Weekly Balance Calibration Logbook Page

18.0 Revision History

Revision 0, dated 1 December, 2006

Original Release

Revision 1, dated 30 November, 2008

Complete rewrite, updated to TestAmerica format

Attachment 1

Instructions for LIMS Data Upload

1. Click on the **TA** logo at the top of the Excel spreadsheet (this will automatically activate a macro that will import the data to LIMS).
2. A prompt will request the name of the file to be used for the batch data in LIMS. Enter a file name using the same format as that used for the Excel spreadsheet containing the data to be uploaded. Click **OK** when finished.
3. When prompted, enter the number of samples for Oven #1 and click **OK**, then enter the number of samples for Oven #2 and click **OK**.
4. When the "Upload Complete" message appears, click **OK**.
5. Exit Microsoft Excel by clicking on the **X** in the upper right corner of the screen or by selecting **Exit** under the **File** option on the menu toolbar at the top of the screen.
6. The computer will ask "Do you want to close the workbook or Microsoft Excel? This will terminate data collection.". Click **Yes**.
7. The computer will then ask "Do you want to save the changes to **MethodTemplate.xls**". Click **No**.
8. Click on the **TA LIMS** icon on the desktop and expand the **Equipment** subfolder by clicking on the plus sign.
9. Select the **Moisture** subfolder and expand it by clicking on the plus sign. The newly created data batch should be listed at the top with the date of upload next to a "briefcase-looking" icon.
10. Double-click on the batch to open it.
11. Select the **Edit** option for the batch. A popup box will appear stating "There are some samples in the batch that require further processing. Would you like them processed now?". Click **Yes**.
12. Right-click anywhere in the samples field and select **Batch Information** from the popup box that appears.
13. Click on the box icon next to the analyst's name. A popup box will appear with a list of analyst names. Select the correct name and click **Select** to continue.
14. In the **Batch Comment** field, type "Results based on tared tin".
15. Click on the **Worksheet** tab at the bottom of the screen and check to make sure that there are no "err" values or other errors in the data (A small g entered for the **DishWeight** is correct).

16. Click on the **Batch Results** tab at the bottom of the screen and check to make sure that there are no "err" values or other errors in the data.
17. Click on the **Save** button, and, when prompted, enter the LIMS password to complete the save process.
18. Click on the **Samples** tab, and then click on the **Edit** option.
19. Highlight all the samples and right-click on the list. A popup box that will appear. Select **Sample Review** and another popup list will then appear. Select **1st Level Reviewed**.

NOTE: A yellow icon may appear next to some samples to indicate that they are not log-in approved. It will be necessary to return to the batch later to perform the 1st and 2nd Level Reviewing.
20. Highlight all the samples again and right-click on the list. A popup box that will appear. Select **Sample Review** and another popup list will then appear. Select **2nd Level Reviewed**.
21. Click on the **Save** button, and, when prompted, enter the LIMS password to complete the save process.
22. Data upload and review are now complete. Exit **TA LIMS** until the next set of data is ready to be uploaded.

Attachment 2

Daily Balance Calibration Logbook Page

[illegible]

Attachment 3

Weekly Balance Calibration Logbook Page

| | | | | | | | |
|---------------------------|--|--|--|--|--|--|--|
| Month/Year: _____ | | | | TOLERANCE: 30g, 20g, 10g \pm 0.005 g 5.0g, 1.0 g \pm 0.004 g | | | |
| BALANCE ID #: HB43 | | | | | | | |
| WEIGHTS 06- | | | | | | | |
| SERIAL #: J67933 | | | | | | | |

| Oven#1 | | | | Oven#2 | | | |
|----------|------|---------------------|----------------------|----------|------|------------------|----------------------|
| INITIALS | DATE | TRUE MASS (g) | MEASURED MASS (g) | INITIALS | DATE | TRUE MASS (g) | MEASURED MASS (g) |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |
| | | 30 | | | | | |
| | | 20 | | | | | |
| | | 10 | | | | | |
| | | 5.00 | | | | | |
| | | 1.00 | | | | | |
| | | | | | | | |

***If the weights exceed tolerance, notify QA Manager immediately.**