Table of Contents

WI NO.: AO_1 (Filename: 130_1HR_.r01)		
Title: Single-stage Air Oven Reference Method for Moisture in Grains, Oilseeds, and Commodities		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden Jr.
Chemist
Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall
ARTS Branch Chief
Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

"CONTROLLED DOCUMENT"

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in the grains and oilseeds listed in Table 1, and the processed commodity samples listed in Table 2.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Oil Chemists Society (AOCS) Method Ba 2a-38.
- American Association of Cereal Chemists (AACC) Method 44-15A.
- Association of Official Analytical Chemists International (AOAC) Method 925.10.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, Laboratory Information Management System (LIMS), and Laboratory Management Information Network (LABMIN).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 96C.

h) NIST traceable partial-immersion thermometer, ASTM No. 96C.

i) Laboratory Mill, Wiley Intermediate Model equipped with a 20-mesh (0.850 mm opening) wire sieve (Arthur H. Thomas Company, Philadelphia, PA).

j) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 130 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is

interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.1.f Grinding Mill

1. Monthly, adjust the clearance between the stationary knives and the knives mounted on the rotor so that a sheet of paper of average thickness (0.002 to 0.003 inches) will pass between them without binding. While turning the rotor by hand, the knives should touch the paper, but not tear it.

2. Physically examine the grinder's knives every six months for wear and replace as needed. Replace the rotor at the same time.

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, "Processing the Air Oven Laboratory Wheat Check Sample").

8. Sample Analysis

8.1 Grinding

Samples that arrive in the laboratory pre-ground are analyzed for moisture in order to report other constituents, such as protein and oil, on a moisture-corrected basis. Information regarding the grinding processes used in testing these other constituents in grains or oilseeds may be found in the Grain Inspection Handbook, Books V (wheat) and VI (soybeans), and in the AOCS Methods Ac 3-44 (soybeans) and Ai 3-75 (sunflower and canola).

1. Grind a representative 20 to 30 gram portion of the original sample so that all ground material will pass through a 20-mesh (0.850mm opening) wire sieve.

2. The minimum possible pressure should be used in feeding the sample through the mill.

3. The ground portion should pass directly from the mill into a suitable container not much larger than necessary to hold it.

4. If the sample "packs" in the grinding chamber, the packed material must be discarded, but that which is already ground and in the catch jar may still be used. Any unground portion left in the hopper may also be reused after the grinding chamber has been cleaned out.

5. Clean the grinder before grinding a different sample by vacuuming or dusting the accessible surfaces. (The grinder does not have to be cleaned between grinding several cuts of the same sample.)

6. Anytime any maintenance is performed on the grinder, such as replacing parts, it should be recorded in the Grinder LogBook. The number of samples that were ground on each grinder each day and by whom should also be recorded in the Grinder LogBook.

8.2 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Using a spatula, mix the ground sample well.

4. Weigh 2.5 ± 0.1 grams of the sample into each of two moisture dishes. **Note: For commodity samples, weigh 3 to 5 grams into only one dish.**

5. Immediately cover the dishes.

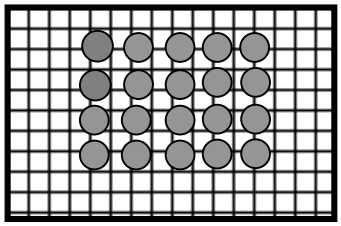
6. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

7. Weigh each dish on the analytical balance.

8. The sample weight should be 2.5 ± 0.1 grams (except for commodity samples which should weigh between 3 to 5 grams). If necessary, adjust the sample weight by adding sample to or removing sample from the dish until the sample weight is within this range.

9. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

10. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 22 small dishes may be placed on a rack***.



Front of Rack

    a. All moisture dishes should be placed on a single shelf in the oven.

    b. An ASTM No. 96C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

11. Insert the rack and dishes into the oven.

12. Dry the samples for one hour (two hours for soybean meal). Timing begins when the oven reaches 129 °C after insertion of the dishes.

13. Remove the shelf containing the dishes and cover the dishes immediately.

14. Transfer the dishes to a desiccator and allow them to cool to room temperature.

15. Weigh the dishes back on the analytical balance.

16. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.3 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

Data for commodity samples are also entered manually into LABMIN.

10.2 Acceptance Criteria

10.2.a Grain and Oilseed Samples

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for all

samples.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an assignable cause.

    b. If an assignable cause is found, correct the problem.

    c. Repeat the analysis.

    d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

    e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

10.2.b Commodity Samples

Repeat the analysis on any sample for which a Quality Control (QC) request is issued through LABMIN or LIMS.

Table 1: GRAINS AND OILSEEDS ANALYZED BY THIS METHOD

Description	Moisture Restriction
Barley	≤ 16%
Lentils	≤ 16%
Oats	≤ 16%
Peas	≤ 16%
Rice, Brown	≤ 16%
Rice, Milled	≤ 16%
Rice, Rough	≤ 13%
Rye	≤ 16%
Sorghum	≤ 16%
Soybean	≤ 10%
Sunflower ground 50/50 with celite	none (for moisture correction of crude oil results)
Triticale	≤ 16%
Wheat	≤ 16%

Table 2: PROCESSED COMMODITIES ANALYZED BY THIS METHOD

Barley - submitted	Macaroni
Bulgur	Macaroni and Cheese
Corn Soy Blend	Rotini
Corn Soya Flour	Rolled Wheat
Corn Soy Milk	Sorghum Grits
Corn - submitted	Soybean Meal
Cornmeal	Spaghetti
Dessert Powder	Wheat Flour
Egg Noodles	Wheat Soy Blend
Hominy Grits	Wheat Soy Milk
Icing Mix Powder	Wheat - submitted
Lasagna	

WI NO.: AO_2 (Filename: 130_2_ST.r01)		
Title: Two-stage Air Oven Reference Method for Moisture in Grains and Oilseeds		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

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Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in grains and oilseeds requiring the two-stage analysis at 130 °C for one hour as listed in Table 1.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Association of Cereal Chemists (AACC) Method 44-15A.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division (TSD) Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 96C.

h) NIST traceable partial-immersion thermometer, ASTM No. 96C.

i) Laboratory Mill, Wiley Intermediate Model equipped with a 20-mesh (0.850 mm opening) wire sieve (Arthur H. Thomas Company, Philadelphia, PA).

j) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 130 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is

interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.1.f Grinding Mill

1. Monthly, adjust the clearance between the stationary knives and the knives mounted on the rotor so that a sheet of paper of average thickness (0.002 to 0.003 inches) will pass between them without binding. While turning the rotor by hand, the knives should touch the paper, but not tear it.

2. Physically examine the grinder's knives every six months for wear, and replace as needed. Replace the rotor at the same time.

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, "Processing the Air Oven Laboratory Wheat Check Sample").

8. Procedure

8.1 Sample Analysis

1. Using the top-loading balance, weigh approximately 15 grams (but no less than 13 grams) of unground sample into two tared moisture dishes, taking care that the difference between the two net sample weights is ≤ 0.4 grams. Make sure that enough room is left in the dishes for the covers to fit snugly.

2. Weigh the covered dish and sample to the nearest 0.1 mg on the analytical balance.

3. Input the weight and the barcode number directly into the computer, or manually enter the data if the sample has no barcode.

4. Uncover the dishes and place them, with their covers underneath, in a warm, well-ventilated place (preferably on top of a heated oven).

   a) The portions should dry reasonably quickly and reach an air-dry condition. This usually takes from 14 to 16 hours when the top of the heated oven is used for this preliminary drying.

   b) In all cases, except for soybeans and rough rice, the moisture content must be reduced to 16 percent or less (10 percent in the case of soybeans and 13 percent in the case of rough rice) in this first stage of drying.

5. Cover the dishes containing the air-dried portions and weigh each of them on the analytical balance as soon as they reach room temperature.

6. Input the weight directly into the computer via the interface.

7. Grind the air-dried samples as soon as possible after weighing and analyze each of the ground portions, in duplicate, as described in the single stage procedure, WI No. AO_1.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

9. Determination

The percentage of moisture in the original sample is calculated by the computer as follows:

$$\%\,moisture = \frac{\frac{E \times B}{D} + C}{A} \times 100$$

Where A = Weight of the original portion used for the test,

B = Weight of the portion after air-drying,

C = Moisture loss due to air-drying = A - B,

D = Weight of the ground subportion of the air-dried portion used for the 130 °C air-oven drying,

E = Loss of moisture due to oven drying.

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for all samples except rough rice containing more than 18% moisture, for which the replicates can differ by up to 0.30%.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an

assignable cause.

b. If an assignable cause is found, correct the problem.

c. Repeat the analysis.

d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

Table 1: GRAINS AND OILSEEDS ANALYZED BY THIS METHOD

Description	Suspected or Known Moisture Level
Barley	> 16%
Lentils	> 16%
Oats	> 16%
Peas	> 16%
Rice, brown	> 16%
Rice, milled	> 16%
Rice, rough	> 13%
Rye	> 16%
Sorghum	> 16%
Soybean	> 10%
Triticale	> 16%
Wheat	> 16%

WI NO.: AO_3 (Filename: Corn&Bea.r01)		
Title: Air Oven Reference Method for Moisture in Corn and Beans		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

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WI NO.: AO_3 (Filename: Corn&Bea.r01)		Page 1 of 9
Title: Air Oven Reference Method for Moisture in Corn and Beans		
Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 1	Replaces: 0	Effective: 04/01/98

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in corn and bean samples.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Association of Cereal Chemists (AACC) Method 44-15A.
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- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter (for all samples except high-moisture corn) of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. The large dishes for high-moisture corn should have a diameter of about 92 mm and a height of about 53 mm. (Note: intermediate sized dishes, 70 mm diameter and 30 mm height, may be used on large kernel samples such as lima beans, cranberry beans, large kernel corn, and confectionery sunflower seeds.) Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

h) NIST traceable partial-immersion thermometer, ASTM No. 94C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 103 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is

interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.2 Check Sample

A corn check sample should be run every time corn or bean samples are analyzed (see WI No. AO_12, "Processing the Air Oven Laboratory Corn Check Sample").

8. Procedure

8.1 Sample Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Place 15 ± 0.2 grams of a representative portion of the unground sample into each of two or more moisture dishes. For high-moisture corn (over 25 %), use 100 ± 0.2 gram portions and the large dishes for the determination. In any case, the sample size should not be so great that it prevents the dish cover from seating snugly on the dish.

4. Immediately cover the dishes.

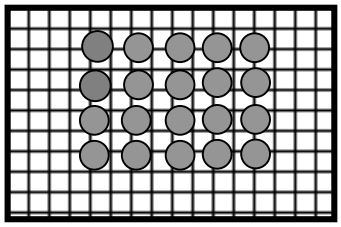
5. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

6. Weigh each dish on the analytical balance.

7. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the

total weight and records the difference as the weight of the portion.

8. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 24 small dishes may be placed on a rack.***



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. An ASTM No. 94C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

9. Insert the rack and dishes into the oven.

10. Dry the samples for 72 hours. Timing begins when the oven reaches 102 °C after insertion of the dishes.

11. Remove the shelf containing the dishes and cover the dishes immediately.

12. Transfer the dishes to a desiccator and allow them to cool to room temperature.

13. Weigh the dishes back on the analytical balance.

14. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be as follows:

≤ 0.25% for corn containing ≤ 15% moisture,
≤ 0.30% for corn containing > 15% moisture, and
≤ 0.20% for all beans.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in

FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an assignable cause.

    b. If an assignable cause is found, correct the problem.

    c. Repeat the analysis.

    d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

    e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

WI NO.: AO_4 (Filename: MUST&RAP.r01)		
Title: Air Oven Reference Method for Moisture in Mustard, Canola, and Rapeseed Samples		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

WI NO.: AO_4 (Filename: MUST&RAP.r01)		Page 1 of 9
Title: Air Oven Reference Method for Moisture in Mustard, Canola, and Rapeseed Samples		
Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 1	Replaces: 0	Effective: 04/01/98

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in mustard, canola, and rapeseed samples.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- International Organization for Standardization (ISO) 665.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

h) NIST traceable partial-immersion thermometer, ASTM No. 94C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, “Calibrating the

Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 103 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-

certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, "Processing the Air Oven Laboratory Wheat Check Sample").

8. Sample Analysis

8.1 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Weigh 10 ± 0.1 grams of a representative portion of the unground sample into each of two moisture dishes.

4. Immediately cover the dishes.

5. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

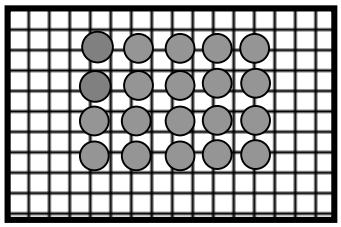
6. Weigh each dish on the analytical balance.

7. The sample weight should be 10 ± 0.1 grams. If necessary, adjust the sample weight by adding sample to or removing sample from the dish until the sample weight is within this range.

8. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

9. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 24 small dishes may be placed on a***

rack.



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. An ASTM No. 94C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 3 inches) and directly over the dishes.

10. Insert the rack and dishes into the oven.

11. Dry the samples for one hour (two hours for soybean meal). Timing begins when the oven reaches 102 °C after insertion of the dishes.

12. Remove the shelf containing the dishes and cover the dishes immediately.

13. Transfer the dishes to a desiccator and allow them to cool to room temperature.

14. Weigh the dishes back on the analytical balance.

15. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

16. Repeat steps 9 and 10.

17. After drying for another hour, remove the shelf containing the dishes and cover the dishes immediately.

18. Repeat steps 13, 14, and 15.

19. If the difference between the second dried weight and the first dried weight is greater than 0.01 grams, the sample must be dried for an additional hour.

20. Repeat steps 9, 10, and 12-15.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for all samples.

1. If the difference between a sample's replicates falls within the tolerance, the data

is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an assignable cause.

    b. If an assignable cause is found, correct the problem.

    c. Repeat the analysis.

    d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

    e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

WI NO.: AO_5 (Filename: FLAXSEED.r01)		
Title: Air Oven Reference Method for Moisture in Flaxseed		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

"CONTROLLED DOCUMENT"

WI NO.: AO_5 (Filename: FLAXSEED.r01)		Page 1 of 9
Title: Air Oven Reference Method for Moisture in Flaxseed		
Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 1	Replaces: 0	Effective: 04/01/98

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in flaxseed.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Association of Cereal Chemists (AACC) Method 44-15A.
- American Society of Agricultural Engineers (ASAE) Method S352.2.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

h) NIST traceable partial-immersion thermometer, ASTM No. 94C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 103 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is

interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, "Processing the Air Oven Laboratory Wheat Check Sample").

8. Sample Analysis

8.1 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Weigh 5 to 7 grams of a representative portion of the unground sample into each of two moisture dishes.

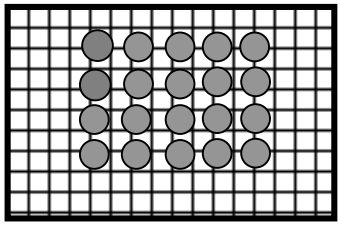
4. Immediately cover the dishes.

5. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

6. Weigh each dish on the analytical balance.

7. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

8. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 24 small dishes may be placed on a rack***.



Front of Rack

   a. All moisture dishes should be placed on a single shelf in the oven.

   b. An ASTM No. 94C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

9. Insert the rack and dishes into the oven.

10. Dry the samples for four hours. Timing begins when the oven reaches 102 °C after insertion of the dishes.

11. Remove the shelf containing the dishes and cover the dishes immediately.

12. Transfer the dishes to a desiccator and allow them to cool to room temperature.

13. Weigh the dishes back on the analytical balance.

14. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for all samples.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an assignable cause.

    b. If an assignable cause is found, correct the problem.

    c. Repeat the analysis.

d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

WI NO.: AO_6 (Filename: SAFFLOWR.r01)		
Title: Air Oven Reference Method for Moisture in Safflower Seeds		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C William Burden Jr.

Approved By: C. William Burden, Jr.
Chemist

3/31/98
Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall

Approved By: Donald C. Kendall
ARTS Branch Chief

4-2-98
Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in safflower seeds.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Society of Agricultural Engineers (ASAE) Method S352.2.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 96C.

h) NIST traceable partial-immersion thermometer, ASTM No. 96C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, “Calibrating the Thermometers Used in the Air Oven Laboratory”).

2. If the temperature is not 130 ± 1 °C, adjust the oven’s controls until this

temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

   a. Record the weights, your initials, and the date in the balance logbook.

   b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

   c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, “Processing the Air Oven Laboratory Wheat Check Sample”).

8. Sample Analysis

8.1 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Weigh 10 ± 0.2 grams of a representative portion of the unground sample into each of two moisture dishes.

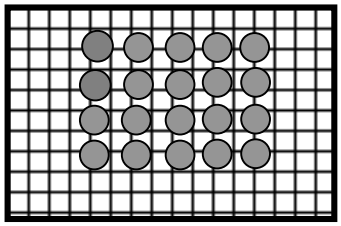
4. Immediately cover the dishes.

5. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

6. Weigh each dish on the analytical balance.

7. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

8. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 22 small dishes may be placed on a rack***.



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. An ASTM No. 96C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

9. Insert the rack and dishes into the oven.

10. Dry the samples for one hour. Timing begins when the oven reaches 129 °C after insertion of the dishes.

11. Remove the shelf containing the dishes and cover the dishes immediately.

12. Transfer the dishes to a desiccator and allow them to cool to room temperature.

13. Weigh the dishes back on the analytical balance.

14. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for all samples.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an assignable cause.

    b. If an assignable cause is found, correct the problem.

    c. Repeat the analysis.

    d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

    e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

WI NO.: AO_7 (Filename: SUNFLOWR.r01)		
Title: Air Oven Reference Method for Moisture in Sunflower Seeds		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr.
Chemist

Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4.2.98

Approved By: Donald C. Kendall
ARTS Branch Chief

Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in sunflower seeds and the Smalley collaborative series soybean samples.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Oil Chemists Society (AOCS) Method Ac 2-41.
- AOCS Method Ai 2-75.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. (Note: intermediate sized dishes, 70 mm diameter and 30 mm height, may be used on large kernel samples such as confectionery sunflower seeds). Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 96C.

h) NIST traceable partial-immersion thermometer, ASTM No. 96C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 130 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is

interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11, "Processing the Air Oven Laboratory Wheat Check Sample").

8. Sample Analysis

8.1 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Weigh 10 ± 0.2 grams of a representative portion of the unground sample into each of two moisture dishes.

4. Immediately cover the dishes.

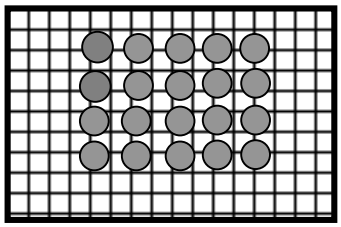
5. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

6. Weigh each dish on the analytical balance.

7. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the

total weight and records the difference as the weight of the portion.

8. Uncover the dishes and center them, with covers beneath, on a room-temperature oven rack (see example below). ***From 1 to 22 small dishes may be placed on a rack***.



Front of Rack

   a. All moisture dishes should be placed on a single shelf in the oven.

   b. An ASTM No. 96C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 3 inches) and directly over the dishes.

9. Insert the rack and dishes into the oven.

10. Dry the samples for three hours. Timing begins when the oven reaches 129 °C after insertion of the dishes.

11. Remove the shelf containing the dishes and cover the dishes immediately.

12. Transfer the dishes to a desiccator and allow them to cool to room temperature.

13. Weigh the dishes back on the analytical balance.

14. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

10.2 Acceptance Criteria

Precision can be verified by comparing the differences between replicate analyses. Differences between replicates should be ≤ 0.20% moisture for soybean samples, and ≤ 0.30% moisture for sunflower samples.

1. If the difference between a sample's replicates falls within the tolerance, the data is made available to the customer by uploading it to the QARDRSLT.dbf file in FoxPro®, or by validating the data in the LIMS.

2. If the difference between a sample's replicates exceeds the tolerance, the data is marked for deletion in FoxPro®, or stored in the LIMS with the designation of "Fail." Under either scenario, the sample must be repeated.

    a. Investigate the process, equipment, materials, etc. for an

assignable cause.

b. If an assignable cause is found, correct the problem.

c. Repeat the analysis.

d. If the data from the repeated analysis is within tolerance, make it available to the customer as under 1 above.

e. If the data from the repeated analysis exceeds the tolerance, report the average of the original and the repeated results as under 1 above.

WI NO.: AO_8 (Filename: CGF.r01)		
Title: Air Oven Reference Method for Moisture in Corn Gluten Feed		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

"CONTROLLED DOCUMENT"

WI NO.: AO_8 (Filename: CGF.r01)		Page 1 of 8
Title: Air Oven Reference Method for Moisture in Corn Gluten Feed		
Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 1	Replaces: 0	Effective: 04/01/98

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in corn gluten feed samples.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- Method specified through the Corn Refiners Association.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, Laboratory Information Management System (LIMS), and Laboratory Management Information Network (LABMIN).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

h) NIST traceable partial-immersion thermometer, ASTM No. 94C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

6. Materials

Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

7. Quality Control ProceduresError! Bookmark not defined.

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, “Calibrating the Thermometers Used in the Air Oven Laboratory”).

2. If the temperature is not 103 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

   a. Record the weights, your initials, and the date in the balance logbook.

   b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

   c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

7.2 Check Sample

A durum wheat check sample should be run every morning and afternoon when samples are being analyzed for one hour by the Air Oven method (see WI No. AO_11,

"Processing the Air Oven Laboratory Wheat Check Sample").

8. Sample Analysis

8.1 Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Using a spatula, mix the ground sample well.

4. Weigh three to five grams of the sample into the moisture dish.

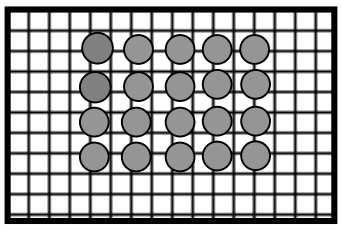
5. Immediately cover the dish.

6. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

7. Weigh the dish on the analytical balance.

8. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

9. Uncover the dish and center it, with cover beneath, on a room-temperature oven rack (see example below). ***From 1 to 24 small dishes may be placed on a rack***.



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. An ASTM No. 94C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 3 inches) and directly over the dishes.

10. Insert the rack and dishes into the oven.

11. Dry the samples for four hours. Timing begins when the oven reaches 102 °C after insertion of the dishes.

12. Remove the shelf containing the dishes and cover the dishes immediately.

13. Transfer the dishes to a desiccator and allow them to cool to room temperature.

14. Weigh the dishes back on the analytical balance.

15. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture using the calculation above.

The data are also entered manually into LABMIN.

10.2 Acceptance Criteria

Repeat the analysis on any sample for which a Quality Control (QC) request is issued through LABMIN or LIMS.

WI NO.: AO_9 (Filename: VAC_70.r02)		
Title: 70 °C Vacuum Oven Reference Method for Moisture in Commodity Samples		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 2	Replaces: 1	Effective: 08/20/01

C William Burden Jr.

Approved By: C. William Burden, Jr.
Chemist

8/20/01
Date

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0437

"CONTROLLED DOCUMENT"

Aaron Johnson for Timothy Norden

Approved By: Timothy D. Norden
ARTS Branch Chief

8/21/2001
Date

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0470

Table of Contents

Changes From Previous Revision

1. In section 3. References:
   Added "Operating Instructions for Mettler Toledo AG Balances (1999)"

2. In section 5. Equipment:
   (b) removed "Microsoft FoxPro" and "LABMIN," and added "Microsoft Excel"
   (e) added "150 mm or…"
   (f) changed "mm" of mercury to "inches," and changed "removable perforated or wire shelf" to "metal shelf"
   (g) changed "mm" of mercury to "inches"
   (i) added NIST-traceable digital thermometer
   (j) added NIST-traceable vacuum test gauge

3. In section 6. Materials:
   (a) removed the sentence, "Anhydrous calcium chloride may be used as an indicator along with the desiccant."
   (b) added Humidity Indicating Cards and indicating silica gel

4. In section 7. Quality Control Procedures:
   (7.1.2) added "…or just the display is off, in which case no warm-up is needed – see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14.)"
   (7.1.4) moved to 7.1.7
   (7.1.5) is now 7.1.4; changed the frequency and reasons for calibrating the balance
   (7.1.6) is now 7.1.5
   (7.1.7) is now 7.1.6; added the sentence, "Checking other weights from the same set in addition to these three is optional."
   (7.1.7.b) is now 7.1.6.b; removed the sentences, "Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the log book."
   (7.1.7.c) is now 7.1.6.c; removed the sentences, "Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the log book."
   (7.1.6.d) new; addresses action(s) to take if balance performance is suspect
   (7.1.7) moved from 7.1.4
   (7.4) new; addresses frequency and method of checking the accuracy of the vacuum oven temperature and vacuum

5. In section 8. Sample Analysis:
   - (8.1.6) changed "barcode wand" to "barcode reader" and added "won't scan, or won't scan correctly" as reasons for manually entering sample ID
   - (8.1.9) added "…(however, hardware or software problems could require the weight to be entered manually)."
   - (8.2.1) added tolerance to oven temperature
   - (8.3.5) added tolerance to oven vacuum
   - (8.4.5) new; addresses method for checking humidity level in desiccator; former numbers 8.4.5 through 8.4.11 have been incremented by one
   - (8.4.6) formerly 8.4.6; added "…(or manually, if necessary)."

6. In section 10. Data Validation:
   - (10.1) removed references to "FoxPro database" and "LABMIN," added "Excel spreadsheet" and changed the sentences to reflect the fact that Excel makes the calculations and the data are then uploaded to LIMS
   - (10.2) removed reference to LABMIN

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in the processed commodity samples listed in Table 1.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- Association of Official Analytical Chemists International (AOAC) Method 925.45.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.
- Operating Instructions for Mettler Toledo AG Balances (1999).

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft Excel, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccators with an inside diameter of approximately 150 mm, or 250 mm, equipped with a porcelain desiccator plate.

f) Vacuum oven capable of maintaining a vacuum of at least 27 inches of mercury, equipped with a metal shelf.

g) Vacuum pump capable of achieving a vacuum of at least 27 inches of mercury.

h) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

i) NIST-traceable digital thermometer.

j) NIST-traceable vacuum test gauge.

6. Materials

a) Activated alumina, “molecular sieves” Type 4A desiccant.

b) Humidity indicating cards. Silica gel with cobalt chloride added may be used as an additional indicator.

7. Quality Control Procedures

7.1 Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one-half hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, or just the display is turned off, in which case no warm-up is needed -- see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. Calibrate the balance if it is disconnected from the power supply or if there is a power outage. When power is restored, "OFF" will appear in the digital display. Also calibrate the balance anytime "CAL" flashes on the digital display as this may indicate excessive temporary drafts or vibrations.

5. Make sure all balance doors are closed before reading weights.

6. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction. Checking other weights from the same set in addition to these three is optional.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) may indicate a malfunction.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, may be indicative of a drift error.

    d. If the measurement results on these known weights or moisture dishes are not stable, reproducible or accurate, then the balance must be removed from service pending rectification of the cause(s) as determined from investigation.

        i. Initial investigation should be conducted by the analyst(s) after consulting the balance operating manual. The Project Leader

should also be consulted.

ii. If rectification can be achieved by appropriate measures suggested in the operating manual, then the balance can be returned to service. It should be noted that if the cause is environmental, rectification might be achieved without removing the balance from service.

iii. If it is determined that the balance should be tested and/or serviced by a qualified service engineer, the Project Leader will be responsible for seeing that this is done.

iv. A certified balance may be used as a replacement if it meets all relevant criteria in this Working Instruction.

v. Record all actions taken in the logbook.

7. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

7.2 Reference Weight Set

Re-certify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.3 Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.4 Vacuum Oven

1. Prior to analyzing samples, verify the accuracy of the oven's thermocouple and temperature controller by comparing the temperature reading to a NIST-traceable thermometer.

2. Adjust the oven's temperature controller if its temperature reading differs from the

NIST-traceable thermometer by more than 2 °C.

3. If the oven cannot be adjusted to agree with the NIST-traceable thermometer, remove it from service until repairs can be made to get it to agree.

4. Also verify the accuracy of the oven's vacuum gauge by comparing its reading to a NIST-traceable test gauge.

5. Adjust the oven's vacuum gauge if its reading differs from the NIST-traceable gauge by more than 2 inches of mercury.

6. If the oven's gauge cannot be adjusted to agree with the NIST-traceable gauge, remove the oven from service until repairs can be made to get it to agree.

7. Record all temperature and pressure readings, differences, and actions taken in the logbook.

8. Sample Analysis

8.1 Sample Weighing

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Using a spatula, mix the ground sample well.

4. Weigh 3 to 5 grams of the sample into the moisture dish.

5. Immediately cover the dish.

6. Enter the sample ID into the computer using a barcode reader, or manually enter the ID if a barcode is not present, won't scan, or won't scan correctly.

7. Weigh the dish on the analytical balance.

8. The sample weight should be 3 to 5 grams. If necessary, adjust the sample weight by adding sample or removing sample from the dish until the sample weight is within this range.

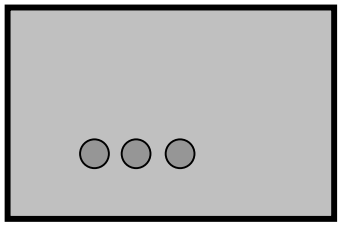
9. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface (however, hardware or software problems could require the weight to be entered manually). The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

8.2 Loading Vacuum Oven

1. Check the temperature setting on oven to make sure the correct oven is selected (70 ± 3 °C).

2. Open the oven door and place the dishes, with the covers underneath, inside on the top oven shelf.

3. The dishes should be loaded starting at the edge of the shelf closest to the door (see below):



Door of Oven

4. Close the door.

5. Make sure the air valve on the oven is closed.

8.3 Vacuum Pump Operation

CAUTION: The following steps must be performed in the prescribed order to ensure that there is no oil suctioned back into the oven. This situation could ruin samples inside the oven and mean a massive clean up of the oven interior.

Note: the oil in the vacuum pump should be checked once a quarter and changed once a year or as needed. The used oil should be discarded in accordance with the TSD Chemical Hygiene Plan.

1. Turn on the vacuum pump.
2. Connect the hose from the vacuum pump to the hose from the oven.
3. Open the vacuum outlet valve on the oven.
4. Make sure the oven door seals completely by listening for the hissing of air leaking into the oven around the door seal.
5. Continue pulling the vacuum on the oven until the oven gauge reading is 27-30 inches of mercury.
6. Turn off the vacuum outlet valve on the oven.
7. Disconnect the vacuum pump hose from the oven hose.
8. Turn off the vacuum pump.

8.4 Sample Drying

1. Dry the samples for two hours.
2. Reopen the oven by SLOWLY opening the air inlet valve on the oven to release the vacuum.
3. When the vacuum is fully released, open the door, remove the dishes, and cover them immediately.
4. Transfer the dishes to a desiccator and allow them to cool to room temperature.

5. Use a humidity-indicating card to check the humidity level in the desiccator. If the humidity is higher than 10%, replace the desiccant with fresh molecular sieves.

6. Weigh the dishes back on the analytical balance.

7. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface (or manually, if necessary).

8. Following the same procedure, return the dishes to the oven and dry for another hour.

9. Repeat steps 2 through 7.

10. If the second dried weight is within 0.01 gram of the first dried weight, the analysis is done.

11. If the second dried weight is not within 0.01 gram of the first dried weight, return the dishes to the oven and dry for another hour.

12. Repeat steps 2 through 7.

8.5 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to an Excel spreadsheet via the interface (unless hardware or software problems require the weights to be entered manually). The computer then calculates the moisture using the calculation above. All data are uploaded directly to the LIMS via the interface.

10.2 Acceptance Criteria

Repeat the analysis on any sample for which a Quality Control (QC) request is issued through LIMS.

Table 1: PROCESSED COMMODITIES ANALYZED BY THIS METHOD

Cereal Bars
Cornflake – (CBAR/I)
Rice – Cornflake – (CBAR/II)
Fruit-Cornflake – (CBAR/III)
Granola Bar – (CBAR/IV)

Bakery Mixes
Chocolate Brownie
Pancake Mix
Roll Mix
Cornbread Mix
Sweet Dough Mix
Sugar Cookie
Chocolate Cookie
Oatmeal Cookie
Gingerbread
White Cake
Yellow Cake
Devil's Food Cake
Biscuit Mix
Donut Mix
ASCS Biscuit Mix

WI NO.: AO_10 (Filename: VAC_100.r02)		
Title: 100 °C Vacuum Oven Reference Method for Moisture in Commodity Samples		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 2	Replaces: 1	Effective: 08/20/01

C. William Burden Jr. 8/20/01

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0437

"CONTROLLED DOCUMENT"

Aaron Johnson for Timothy Norden 8/21/2001

Approved By: Timothy D. Norden Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0470

Table of Contents

Changes From Previous Revision

1. In section 3. References:
   Added "Operating Instructions for Mettler Toledo AG Balances (1999)"

2. In section 5. Equipment:
   (b) removed "Microsoft FoxPro" and "LABMIN," and added "Microsoft Excel"
   (e) added "150 mm or…"
   (f) changed "mm" of mercury to "inches," and changed "removable perforated or wire shelf" to "metal shelf"
   (g) changed "mm" of mercury to "inches"
   (i) added NIST-traceable digital thermometer
   (j) added NIST-traceable vacuum test gauge

3. In section 6. Materials:
   (a) removed the sentence, "Anhydrous calcium chloride may be used as an indicator along with the desiccant."
   (b) added Humidity Indicating Cards and indicating silica gel

4. In section 7. Quality Control Procedures:
   (7.1.2) added "…or just the display is off, in which case no warm-up is needed – see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14.)"
   (7.1.4) moved to 7.1.7
   (7.1.5) is now 7.1.4; changed the frequency and reasons for calibrating the balance
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- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.
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e) Glass vacuum desiccator with an inside diameter of approximately 150 mm, or 250 mm, equipped with a porcelain desiccator plate.

f) Vacuum oven capable of maintaining a vacuum of at least 27 inches of mercury, equipped with a metal shelf.

g) Vacuum pump capable of achieving a vacuum of at least 27 inches of mercury.

h) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

i) NIST-traceable digital thermometer.

j) NIST-traceable vacuum test gauge.

6. Materials

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b. Humidity indicating cards. Silica gel with cobalt chloride added may be used as an additional indicator.

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7.1 Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the

balance must be allowed one-half hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, or just the display is turned off, in which case no warm-up is needed -- see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. Calibrate the balance if it is disconnected from the power supply or if there is a power outage. When power is restored, "OFF" will appear in the digital display. Also calibrate the balance anytime "CAL" flashes on the digital display as this may indicate excessive temporary drafts or vibrations.

5. Make sure all balance doors are closed before reading weights.

6. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction. Checking other weights from the same set in addition to these three is optional.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) may indicate a malfunction.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, may be indicative of a drift error.

    d. If the measurement results on these known weights or moisture dishes are not stable, reproducible or accurate, then the balance must be removed from service pending rectification of the cause(s) as determined from investigation.

        i. Initial investigation should be conducted by the analyst(s) after consulting the balance operating manual. The Project Leader should also be consulted.

        ii. If rectification can be achieved by appropriate measures suggested in the operating manual, then the balance can be returned to service. It should be noted that if the cause is environmental, rectification might be

achieved without removing the balance from service.

iii. If it is determined that the balance should be tested and/or serviced by a qualified service engineer, the Project Leader will be responsible for seeing that this is done.

iv. A certified balance may be used as a replacement if it meets all relevant criteria in this Working Instruction.

v. Record all actions taken in the logbook.

7. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

7.2 Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.3 Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

7.4 Vacuum Oven

1. Prior to analyzing samples, verify the accuracy of the oven's thermocouple and temperature controller by comparing the temperature reading to a NIST-traceable thermometer.

2. Adjust the oven's temperature controller if its temperature reading differs from the NIST-traceable thermometer by more than 2 °C.

3. If the oven cannot be adjusted to agree with the NIST-traceable thermometer, remove it from service until repairs can be made to get it to agree.

4. Also verify the accuracy of the oven's vacuum gauge by comparing its reading to a NIST-traceable test gauge.

5. Adjust the oven's vacuum gauge if its reading differs from the NIST-traceable gauge by more than 2 inches of mercury.

6. If the oven's gauge cannot be adjusted to agree with the NIST-traceable gauge, remove the oven from service until repairs can be made to get it to agree.

7. Record all temperature and pressure readings, differences, and actions taken in the logbook.

8. Sample Analysis

8.1 Sample Weighing

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Using a spatula, mix the ground sample well.

4. Weigh 3 to 5 grams of the sample into the moisture dish.

5. Immediately cover the dish.

6. Enter the sample ID into the computer using a barcode reader, or manually enter the ID if a barcode is not present, won't scan, or won't scan correctly.

7. Weigh the dish on the analytical balance.

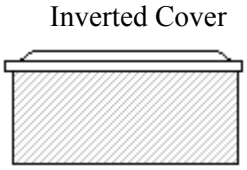
8. The sample weight should be 3 to 5 grams. If necessary, adjust the sample weight by adding sample or removing sample from the dish until the sample weight is within this range.

9. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface (however, hardware or software problems could require the weight to be entered manually). The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

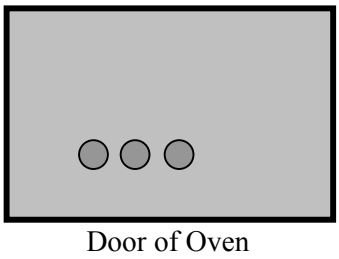
8.2 Loading Vacuum Oven

1. Check the temperature setting on oven to make sure the correct oven is selected (100 ± 3°C).

2. Open the oven door and place the dishes inside on the top oven shelf with the covers placed upside down on top of the dishes as shown below:



3. The dishes should be loaded starting at the edge of the shelf closest to the door (see below):



4. Close the door.

5. Make sure the air valve on the oven is closed.

8.3 Vacuum Pump Operation

CAUTION: The following steps must be performed in the prescribed order to ensure that there is no oil suctioned back into the oven. This situation could ruin samples inside the oven and mean a massive clean-up of the oven interior.

Note: the oil in the vacuum pump should be checked once a quarter and changed once a year or as needed. The used oil should be discarded in accordance with the TSD Chemical Hygiene Plan.

1. Turn on the vacuum pump.
2. Connect the hose from the vacuum pump to the hose from the oven.
3. Open the vacuum outlet valve on the oven.
4. Make sure the oven door seals completely by listening for the hissing of air leaking into the oven around the door seal.
5. Continue pulling the vacuum on the oven until the oven gauge reading is 27-30 inches of mercury.
6. Turn off the vacuum outlet valve on the oven.
7. Disconnect the vacuum pump hose from the oven hose.
8. Turn off the vacuum pump.

8.4 Sample Drying

1. Dry the samples for five hours.
2. Reopen the oven by SLOWLY opening the air inlet valve on the oven to release the vacuum.
3. When the vacuum is fully released, open the door, remove the dishes, and cover them immediately.
4. Transfer the dishes to a desiccator and allow them to cool to room temperature.
5. Use a humidity indicating card to check the humidity level in the desiccator. If

the humidity is higher than 10%, replace the desiccant with fresh molecular sieves.

6. Weigh the dishes back on the analytical balance.

7. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface (or manually, if necessary).

8.5 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

10. Data Validation

10.1 Data Entry

All weights are sent directly to an Excel spreadsheet via the interface (unless hardware or software problems require the weights to be entered manually). The computer then calculates the moisture using the calculation above. All data are uploaded directly to the LIMS via the interface.

10.2 Acceptance Criteria

Repeat the analysis on any sample for which a Quality Control (QC) request is issued through LIMS.

Table 1: PROCESSED COMMODITIES ANALYZED BY THIS METHOD

Description
Cornflakes
Corn Grits
Crackers
Farina
Feed
Flavored Cold Ration
Flavored Tray Pack
Hominy Feed
Popcorn
Rolled Oats Quick Cook
Rolled Oats Instant
Rolled Wheat Quick Cook
Rolled Wheat – Domestic

WI NO.: AO_11 (Filename: DURUMCHK.r02)		
Title: Processing the Air Oven Laboratory Wheat Lab Control Sample		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 2	Replaces: 1	Effective: 09/16/02

C. William Burden Jr.

Approved By: C. William Burden, Jr.
Chemist

9/12/01
Date

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0437

"CONTROLLED DOCUMENT"

Approved By: Timothy D. Norden
ARTS Branch Chief

9/15/02
Date

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0470

Changes From Previous Revision

1. In section 3. References:
   Added *SPC for the Rest of Us* and "Operating Instructions for Mettler Toledo AG Balances (1999)."

2. In section 5. Equipment:
   (b) Replaced reference to Microsoft FoxPro 2.0 with Microsoft Excel and Access.
   (l) Added 8.75" x 17.75" x 6 mil polyethylene sample bags.

3. In section 6. Materials
   (1) Removed reference to anhydrous calcium chloride's use as an indicator.
   (2) Added humidity indicating cards and silica gel with cobalt chloride's use as an additional indicator.

4. In section 7.1.c Analytical Balance:
   (2) Replaced "…less than ten minutes, in which case the balance will reach equilibrium more quickly…" with "…less than ten minutes, or just the display is off, in which case no warm-up is needed..."
   (4) Moved to 7.
   (5) Moved to 4 and rewritten to better describe when calibration is required.
   (6) Moved to 5.
   (7) Moved to 6 and added "Checking other weights from the same set in addition to these three is optional."
   (7.b) Changed "…indicates a malfunction" to "…may indicate a malfunction", and removed the last two sentences.
   (7.c) Changed "…is indicative of a drift error" to "…may be indicative of a drift error", and removed the last two sentences.
   (7.d) Added to describe the actions to take if the balance weighings are not stable, reproducible, or accurate.

5. In section 7.1.f Grinding Mill:
   (3) Replaced "…for sharpness and wear…" with "…for wear…"

6. In section 8.1 Bulk Sample:
   (4) Moved to 5. New 4 added to describe double bagging the samples.

7. In section 8.2 Parameters for Use:
   Added definition of "batch."
   (1) Rewritten to better describe under what circumstances the LCS must be analyzed.
   (2) Added "…(at least one hour)…"
   (3) Replaced "…two portions are required per day…" with "…one portion may be required per day…", added "…for no longer than two weeks…", and changed "…days' morning sample" to " …days' samples."
   (4) Added to emphasize the importance of getting a moisture result on the LCS as early as practical.

8. In section 9.1 Sample Analysis:
   (6) Replaced "…it is the wheat daily check sample" with "…it is the wheat LCS."
   (9) Added "…(however, hardware or software problems could require the weight to be entered manually)."
   (15) Moved to 16. New 15 added to describe the use of humidity indicating cards.
   (16) Moved to 17 and added "…(or manually, if necessary)."

9. In section 11.1 Data Entry:
   Replaced reference to FoxPro with Excel and clarified that the moisture results are calculated and uploaded to LIMS through Excel.

10. In section 11.2 Acceptance Criteria:
   (a) Moved to b. New "a" section added to describe precision verification.
   (b) Completely reworked to describe the development of a control chart and the rules for using it.

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the determination of moisture in the wheat Lab Control Sample (LCS).

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the difference between replicate analyses of a sample meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any sample that exceeds this tolerance.

3. References

- American Association of Cereal Chemists (AACC) Method 44-15A.
- Association of Official Analytical Chemists International (AOAC) Method 925.10.
- ISO Guide 25.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.
- Pitt, H., 1993: *SPC for the Rest of Us*. Addison-Wesley, 419 pp.
- Operating Instructions for Mettler Toledo AG Balances (1999).

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, and data capturing software such as Microsoft® Excel®, Microsoft® Access®, or Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 96C.

h) NIST traceable partial-immersion thermometer, ASTM No. 96C.

i) Laboratory Mill, Wiley Intermediate Model equipped with a 20-mesh (0.850 mm opening) wire sieve (Arthur H. Thomas Company, Philadelphia, PA).

j) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

k) Boerner grain divider.

l) Polyethylene sample bags, 6” x 13” x 6 mil, and 8.75” x 17.75” x 6 mil.

m) Poly bag impulse sealer.

6. Materials

a. Activated alumina, "molecular sieves" Type 4A desiccant.

b. Humidity indicating cards. Silica gel with cobalt chloride added may be used as an additional indicator.

c. Bulk sample of Durum wheat, minimum size: 1500 grams.

7. Quality Control Procedures

7.1 Instrument Parameters

7.1.a Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 130 ± 1 °C, adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month (see WI No. AO_13, "Air Oven Uniformity Check").

7.1.b Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.1.c Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, or just the display is turned off, in which case no warm-up is needed -- see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. Calibrate the balance if it is disconnected from the power supply or if there is a power outage. When power is restored, "OFF" will appear in the digital display. Also calibrate the balance anytime "CAL" flashes on the digital display as this may indicate excessive temporary drafts or vibrations.

5. Make sure all balance doors are closed before reading weights.

6. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction. Checking other weights from the same set in addition to these three is optional.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) may indicate a malfunction.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, may be indicative of a drift error.

    d. If the measurement results on these known weights or moisture

dishes are not stable, reproducible or accurate, then the balance must be removed from service pending rectification of the cause(s) as determined from investigation.

i. Initial investigation should be conducted by the analyst(s) after consulting the balance operating manual. The Project Leader should also be consulted.

ii. If rectification can be achieved by appropriate measures suggested in the operating manual, then the balance can be returned to service. It should be noted that if the cause is environmental, rectification might be achieved without removing the balance from service.

iii. If it is determined that the balance should be tested and/or serviced by a qualified service engineer, the Project Leader will be responsible for seeing that this is done.

iv. A certified replacement balance may be used as a replacement if it meets all relevant criteria in this Working Instruction.

v. Record all actions taken in the logbook.

7. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

7.1.d Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

7.1.f Grinding Mill

1. Monthly, adjust the clearance between the stationary knives and the knives mounted on the rotor so that a sheet of paper of average thickness (0.002 to 0.003 inches) will pass between them without binding. While turning the rotor by hand, the knives should touch the paper, but not tear it.

2. Physically examine the grinder's knives every six months for wear and replace as needed. Replace the rotor at the same time.

7.2 Check Sample

A durum wheat LCS should be analyzed as described in this WI.

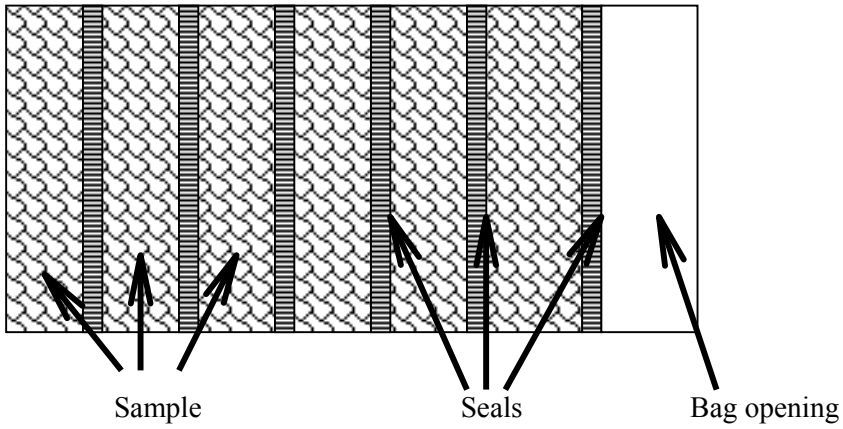
8. Sample Preparation

8.1 Bulk Sample

1. Mix the bulk durum wheat sample thoroughly.

2. Cut the bulk sample into portions of approximately 15 grams each using the Boerner divider.

3. Put the individual portions into a plastic sample bag and seal. One bag can accommodate six portions as long as the bag is sealed after the addition of each successive portion as shown below:



4. Place the individually sealed portions inside a second plastic bag and seal. Reseal the outer bag each time a portion is removed for testing.

5. Keep the samples in refrigerated storage until needed.

8.2 Parameters for Use

Note: a batch of samples is defined as all samples analyzed on the same workday.

1. One portion must be ground and analyzed with every batch of samples that include any of the grain types listed in Table 1 of WI No. AO_1, and which are to be analyzed using the Single Stage method (or the second stage of the Two-Stage method).

2. The portions must be retrieved from the refrigerated storage and be allowed to come to room temperature (at least one hour) before grinding.

3. Although only one portion may be required per day, up to six may be kept outside the refrigerated storage, for no longer than two weeks, in order to prevent delays in processing the next few days' samples.

4. Because the disposition of the rest of the day's samples relies on the status of the LCS, it should be ground and analyzed as early as practical in the day.

8.3 Grinding

Note: Anytime any maintenance is performed on the grinder, such as replacing parts, it should be recorded in the Grinder logbook. The number of samples that were ground on each grinder each day and by whom should also be recorded in the Grinder logbook.

1. Grind an entire 15 gram portion of the original bulk sample so that all ground material will pass through a 20-mesh (0.850mm opening) wire sieve.

2. The minimum possible pressure should be used in feeding the sample through the mill.

3. The ground portion should pass directly from the mill into a suitable container not much larger than necessary to hold it.

4. If the sample "packs" in the grinding chamber, the packed material must be discarded, but that which is already ground and in the catch jar may still be used. Any unground portion left in the hopper may also be reused after the grinding chamber has been cleaned out.

5. Clean the grinder before grinding a different sample by vacuuming or dusting the accessible surfaces. (The grinder does not have to be cleaned between grinding several cuts of the same sample.)

9. Procedure

9.1 Sample Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Using a spatula, mix the ground sample well.

4. Weigh 2.5 ± 0.1 grams of the sample into two moisture dishes.

5. Immediately cover the dishes.

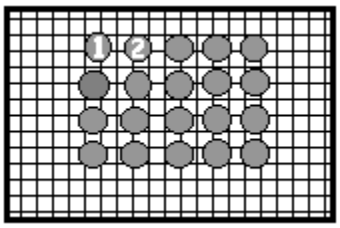
6. Enter the sample ID into the computer by recording that it is the wheat LCS.

7. Weigh each dish on the analytical balance.

8. The sample weight should be 2.5 ± 0.1 grams. If necessary, adjust the sample weight by adding sample to or removing sample from the dish until the sample weight is within this range.

9. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface (however, hardware or software problems could require the weight to be entered manually). The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

10. Uncover the dishes and place them, with covers beneath, on a room-temperature oven rack in positions one and two along with the regular samples that are to be tested:



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. An ASTM No. 96C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

11. Insert the rack and dishes into one of the ovens operating at 130 °C.

12. Dry the samples for one hour. Timing begins when the oven reaches 129 °C after insertion of the dishes.

13. Remove the shelf containing the dishes and cover the dishes immediately.

14. Transfer the dishes to a desiccator and allow them to cool to room temperature.

15. Use a humidity-indicating card to check the humidity level in the desiccator. If the humidity is higher than 10%, replace the desiccant with fresh molecular sieves.

16. Weigh the dishes back on the analytical balance.

17. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface (or manually, if necessary).

9.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

10. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

11. Data Validation

11.1 Data Entry

All weights are sent directly to an Excel® spreadsheet via the interface (unless hardware or software problems require the weights to be entered manually). The computer then calculates the moisture using the calculation above. All data are uploaded to the LIMS via the interface.

11.2 Acceptance Criteria

11.2.a Precision can be verified by comparing the difference between replicate analyses. The difference between replicates should be ≤ 0.20% moisture.

1. If the difference between the LCS's replicates falls within the tolerance, the average of the two replicates is checked for accuracy as under 11.2.b below.

2. If the difference between the LCS's replicates exceeds the tolerance, the data is stored in the LIMS with the designation of "Fail" and the sample must be repeated (unless an assignable cause is known to have caused one of the two replicates to be in error, in which case the unaffected replicate may be considered "Valid" and used in the accuracy check below).

11.2.b Accuracy can be verified by comparing the replicate analyses to the target value.

1. Determining the Target Value

   a. In order to obtain a good estimate of the method's variability and have a control chart in place for a new LCS by the time the old LCS is expended, an action date should be estimated, and twenty portions of the new LCS should be analyzed in duplicate (one portion per day over a period of two-to-four weeks). If for any reason this cannot be accomplished before the new LCS is needed, a less robust estimate can be developed by analyzing ten portions of the new LCS in duplicate on the same day.

   b. Calculate the average value and standard deviation of the results.

   c. The average is the target value for all future analyses of the LCS.

   d. The standard deviation value is used to set the acceptable tolerance of all future analyses of the LCS.

2. Comparing the LCS to the Target Value

   a. The LCS result is acceptable and data for all samples within the batch will be validated and reported to the customer (pending agreement of the other samples' replicates with the tolerance specified in the Working Instructions -- see applicable WI) if none of the following is true:

      i. The result is outside the 3 standard deviation lines
      ii. The result is the 8^{th} point in a row on one side of the average line
      iii. The result is the 6^{th} point in a row all going up or down
      iv. The result is outside the 2 standard deviation lines, along with more than 5% of the others

   b. If any of the above scenarios are true, the data for all samples within the batch are stored in the LIMS with the designation of "Fail" and the process, equipment, materials, etc. are investigated for an assignable cause.

      i. If an assignable cause is found, document and correct the problem.

      ii. Whether or not an assignable cause is found, grind and analyze

another portion of the LCS, along with all of the samples from the batch.

1. If the LCS does not violate any of the four rules above the entire batch will be validated and reported to the customer.

2. If the LCS again violates one of the four rules above, the Project Leader (or, in his/her absence, the technician) must use discretion in deciding how to proceed.

WI NO.: AO_12 (Filename: CORNCHEK.r02)		
Title: Processing the Air Oven Laboratory Corn Lab Control Sample		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 2	Replaces: 1	Effective: 09/16/02

C. William Burden Jr. 9/12/02

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0437

"CONTROLLED DOCUMENT"

Timothy D. Norden 9/16/02

Approved By: Timothy D. Norden Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0470

WI NO.: AO_12 (Filename: CORNCHEK.r02)		Page 1 of 15
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Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 2	Replaces: 1	Effective: 09/16/02

Changes From Previous Revision

1. In section 3. References:
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2. In section 5. Equipment:
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   (k) Added 8.75" x 17.75" x 6 mil polyethylene sample bags.

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   (2) Added humidity indicating cards and silica gel with cobalt chloride's use as an additional indicator.

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   (4) Moved to 7.
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b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, and data capturing software such as Microsoft® Excel®, Microsoft® Access®, or Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter (for all samples except high-moisture corn) of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. The large dishes for high-moisture corn should have a diameter of about 92 mm and a height of about 53 mm. (Note: intermediate sized dishes, 70 mm diameter and 30 mm height, may be used on large kernel samples such as lima beans, cranberry beans, large kernel corn, and confectionery sunflower seeds.) Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

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i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

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l) Poly bag impulse sealer.

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a. Activated alumina, "molecular sieves" Type 4A desiccant.

b. Humidity indicating cards. Silica gel with cobalt chloride added may be used as an additional indicator.

c. Bulk sample of corn, nominal size: 1500 grams.

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3. Record the corrected temperature readings in the Oven Temperature Logbook.

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2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, or just the display is turned off, in which case no warm-up is needed -- see Kenyon et al., and the Operating Instructions for Mettler Toledo AG Balances, page 14).

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5. Make sure all balance doors are closed before reading weights.

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    b. Gross deviation from the known weights (i.e., a milligram or more) may indicate a malfunction.

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7.1.e Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture Dish Maintenance and Tare Weight Check Schedule”).

7.2 Check Sample

A corn check sample should be analyzed as described in this WI.

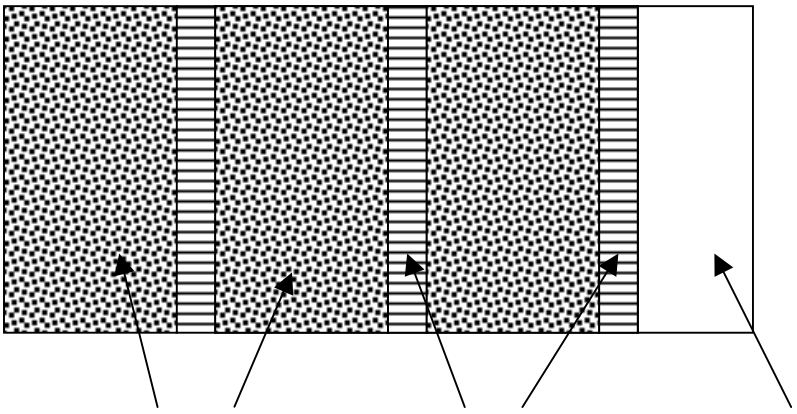
8. Sample Preparation

8.1 Bulk Sample

1. Mix the bulk corn sample thoroughly.

2. Cut the bulk sample into portions of approximately (but no less than) 30 grams each using the Boerner divider.

3. Put the individual portions into plastic sample bags and seal. One bag can accommodate three portions as long as the bag is sealed after the addition of each successive portion as shown below:



4. Place the individually sealed portions inside a second plastic bag and seal. Reseal the outer bag each time a portion is removed for testing.

5. Keep the samples in refrigerated storage until needed.

8.2 Parameters for Use

Note: a batch of samples is defined as all samples whose analysis began on the same workday.

1. One portion of the bulk corn sample must be analyzed with every batch of corn or bean samples that are analyzed using WI No. AO_3.

2. The portions must be retrieved from the refrigerated storage and be allowed to come to room temperature (at least two hours) before weighing.

3. Although only one portion may be required per batch, up to three may be kept outside the refrigerated storage (for no longer than two weeks) in order to prevent delays in processing the next few days' samples (if needed).

9. Procedure

9.1 Sample Analysis

1. Place a dry, clean, empty moisture dish onto the top-loading balance.

2. Tare the balance by pressing down the tare button. The tare weight for each moisture dish and its cover is already stored in the computer under that dish's number.

3. Place 15 ± 0.2 grams of a representative portion of the bulk sample into each of two moisture dishes.

4. Immediately cover the dishes.

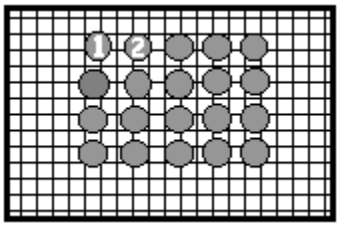
5. Enter the sample ID into the computer by recording that it is the corn LCS.

6. Weigh each dish on the analytical balance.

7. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface (however, hardware or software problems could require the weight to be entered manually). The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

8. Uncover the dishes and place them, with covers beneath, on a room-temperature oven rack in positions one and two along with the regular samples that are to be tested:

Front of Rack



   a. All moisture dishes should be placed on a single shelf in the oven.

   b. An ASTM No. 94C thermometer should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

9. Insert the rack and dishes into one of the ovens operating at 103 °C.

10. Dry the samples for 72 hours. Timing begins when the oven reaches 102 °C after insertion of the dishes.

11. Remove the shelf containing the dishes and cover the dishes immediately.

12. Transfer the dishes to a desiccator and allow them to cool to room temperature.

13. Use a humidity-indicating card to check the humidity level in the desiccator. If the humidity is higher than 10%, replace the desiccant with fresh molecular sieves.

14. Weigh the dishes back on the analytical balance.

15. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface (or manually, if necessary).

9.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

10. Determination

The calculation for the percentage of moisture in the sample is as follows:

$$\%\ moisture = \frac{(Wt_1 - Wt_2)}{sample\ weight} \times 100$$

Where Wt_1 = weight of dish + sample (before oven),
Wt_2 = weight of dish + sample (after oven).

11. Data Validation

11.1 Data Entry

All weights are sent directly to an Excel® spreadsheet via the interface (unless hardware or software problems require the weights to be entered manually). The computer then calculates the moisture using the calculation above. All data are uploaded to the LIMS via the interface.

11.2 Acceptance Criteria

11.2.a Precision can be verified by comparing the difference between replicate analyses. The difference between the corn LCS replicates should be ≤ 0.25% moisture (or, ≤ 0.30% if the LCS moisture level is > 15%).

1. If the difference between the LCS's replicates falls within the tolerance, the average of the two replicates is checked for accuracy as under 11.2.b below.

2. If the difference between the LCS's replicates exceeds the tolerance, the data is

stored in the LIMS with the designation of "Fail" and the sample must be repeated (unless an assignable cause is known to have caused one of the two replicates to be in error, in which case the unaffected replicate may be considered "Valid" and used in the accuracy check below).

11.2.b Accuracy can be verified by comparing the replicate analyses to the target value.

1. Determining the Target Value

   a. In order to obtain a good estimate of the method's variability and have a control chart in place for a new LCS by the time the old LCS is expended, an action date should be estimated, and twenty portions of the new LCS should be analyzed in duplicate (one portion every couple of days over a period of eight-to-ten weeks). If for any reason this cannot be accomplished before the new LCS is needed, a less robust estimate can be developed by analyzing ten portions of the new LCS in duplicate on the same day.

   b. Calculate the average value and standard deviation of the results.

   c. The average is the target value for all future analyses of the LCS.

   d. The standard deviation value is used to set the acceptable tolerance of all future analyses of the LCS.

2. Comparing the LCS to the Target Value

   a. The LCS result is acceptable and data for all samples within the batch will be validated and reported to the customer (pending agreement of the other samples' replicates with the tolerance specified in WI No. AO_3) if none of the following is true:

      i. The result is outside the 3 standard deviation lines
      ii. The result is the 8^{th} point in a row on one side of the average line
      iii. The result is the 6^{th} point in a row all going up or down
      iv. The result is outside the 2 standard deviation lines, along with more than 5% of the others

   b. If any of the above scenarios are true, the data for all samples within the batch are stored in the LIMS with the designation of "Fail" and the

process, equipment, materials, etc. are investigated for an assignable cause.

i. If an assignable cause is found, document and correct the problem.

ii. Whether or not an assignable cause is found, analyze another portion of the LCS, along with all of the samples from the batch.

1. If none of the four scenarios above are true for the repeated LCS result, the entire batch will be validated and reported to the customer.

2. If any of the four scenarios above are true for the repeated LCS, the Project Leader (or, in his/her absence, the technician) must use discretion in deciding how to proceed.

WI NO.: AO_13 (Filename: UNIFORM.r01)		
Title: Air Oven Uniformity Check		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden 3/31/98

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C. Kendall 4-2-98

Approved By: Donald C. Kendall Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

"CONTROLLED DOCUMENT"

Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the monthly uniformity check of the Air Ovens used in the reference moisture laboratory.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

c) The analyst is responsible for checking that the uniformity of the ovens meets the tolerance specified in this WI. The analyst is also responsible for repeating the analysis on any oven that exceeds this tolerance.

3. References

- ISO Guide 25.
- Hunt, W.H., & Neustadt, M.H. (1966) *J. Assoc. Off. Anal. Chem.* **49**, 757-763.
- Hunt, W.H., & Pixton, S.W. "Moisture -- Its Significance, Behavior, and Measurement," *Storage of Cereal Grains and Their Products*, ed. Clyde M. Christensen (St. Paul: American Association of Cereal Chemists, 1974), pp. 1-55.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).
- Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995) *J. Assoc. Off. Anal. Chem.* **78**, 1109-1111.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, and Laboratory Information Management System (LIMS).

c) Top-loading balance accurate to 0.01 gram.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and be maintained as a set.

e) Glass vacuum desiccator with an inside diameter of approximately 250 mm, equipped with a porcelain desiccator plate.

f) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

g) Partial-immersion thermometer, ASTM No. 94C.

h) Partial-immersion thermometer, ASTM No. 96C.

i) Reference weight set, NIST Class S or ASTM Class 1, including 1g, 10g, and 100g weights.

j) Polyethylene sample bags, 6” x 13” x 6 mil.

k) Poly bag impulse sealer.

6. Materials

1. Activated alumina, “molecular sieves” Type 4A desiccant. Anhydrous calcium chloride may be used as an indicator along with the molecular sieves.

2. Five pound bag of commercially available wheat flour, cut down in approximately 300 gram portions, repackaged and sealed (via the impulse sealer) individually in six-mil plastic bags. The amount of flour cut out from the five pound bulk can be adjusted if fewer or more than nine ovens are to be checked (approximately thirty grams of flour is needed per oven).

7. Instrument Parameters

7.1 Air Ovens

1. The temperature of each oven in use should be read daily from the thermometer inserted in the top of the oven, being careful to apply any necessary correction (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

2. If the temperature is not 103 ± 1 °C or 130 ± 1 °C (whichever is the normal working temperature of the oven), adjust the oven's controls until this temperature is achieved (see oven operating manual). If the oven heating is erratic and this temperature range cannot be held, remove the oven from service until corrective action is taken. Record the action taken in the Oven Temperature Logbook.

3. Record the corrected temperature readings in the Oven Temperature Logbook.

4. The uniformity of oven conditions at shelf level in each oven must be checked within the first five working days of every month by following this WI.

7.2 Thermometers

The accuracy of the ASTM thermometer used in each oven must be checked within the first five working days of every month (see WI No. AO_14, "Calibrating the Thermometers Used in the Air Oven Laboratory").

7.3 Analytical Balance

1. Refer to the balance operating manual for information regarding the operation and calibration of the balance.

2. Leave the power to the balance on at all times to ensure that the balance is at temperature equilibrium with the surroundings. If power is interrupted, the balance must be allowed one hour to return to equilibrium before a weighing is made (unless the interruption is brief, e.g., less than ten minutes, in which case the balance will reach equilibrium more quickly -- see Kenyon et al.).

3. Locate the balance in an area that is as free as possible of drafts and temperature fluctuations, preferably on a stone balance table with vibration-damping pads separating the table top from the legs.

4. The balance must be cleaned, serviced, and calibrated with NIST-certified weights twice a year by a qualified service engineer.

5. Calibrate the balance each day before using (see balance operating manual for instructions on calibrating the balance).

6. Make sure all balance doors are closed before reading weights.

7. On the first work day of the week, weigh the 1, 10, and 100 gram weights from a set of NIST class S or ASTM Class 1 weights to check the balance for drift or other malfunction.

    a. Record the weights, your initials, and the date in the balance logbook.

    b. Gross deviation from the known weights (i.e., a milligram or more) indicates a malfunction. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

    c. A small deviation from the known weights that is continuously in the same direction on all three weights, and that grows larger over time, is indicative of a drift error. Remove the balance from service and have it repaired by a qualified service engineer. Record the action taken in the logbook.

7.4 Reference Weight Set

Recertify the calibration of the reference weights once a year by sending them to a facility equipped to provide the service using NIST-traceable standards.

7.5 Moisture Dishes

The moisture dishes must be cleaned and re-tared periodically (see WI No. AO_15, “Moisture

Dish Maintenance and Tare Weight Check Schedule").

8. Sample Analysis

8.1 Analysis

1. Open one sealed bag of wheat flour.

2. Pour the flour into a screw-top plastic or glass bottle that is large enough to hold the entire amount and still leave adequate headspace to allow for vigorous stirring.

3. Using a spatula, stir the flour vigorously.

4. Weigh 2.5 ± 0.1 grams of the sample into nine moisture dishes.

5. Immediately cover the dishes.

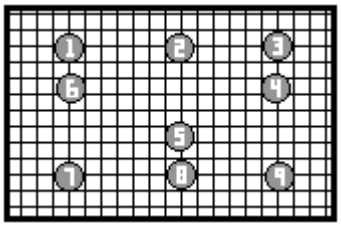
6. Enter the sample ID into the computer by passing a barcode wand across the barcode on the sample bottle, or manually enter the ID if the sample has no barcode.

7. Weigh each dish on the analytical balance.

8. The sample weight should be 2.5 ± 0.1 grams. If necessary, adjust the sample weight by adding or removing flour from the dish until the sample weight is within this range.

9. The final weight, to the nearest 0.1 mg, is input directly into the computer via the interface. The computer subtracts the weight of the dish and its cover from the total weight and records the difference as the weight of the portion.

10. Uncover the dishes and place them, with covers beneath, on a room-temperature oven rack in the order shown below.



Front of Rack

a. All moisture dishes should be placed on a single shelf in the oven.

b. A thermometer (ASTM No. 94C or 96C, depending on the normal working temperature of the oven) should be positioned in the oven such that its immersion mark is aligned with the oven's ceiling and its bulb is close to (approximately 1-3 inches) and directly over the dishes.

11. Insert the rack and dishes into the oven.

12. Dry the samples for one hour. Timing begins when the oven reaches 102 °C or 129 °C (depending on the normal working temperature of the oven) after insertion of the dishes.

13. Remove the shelf containing the dishes and cover the dishes immediately.

14. Transfer the dishes to a desiccator and allow them to cool to room temperature.

15. Weigh the dishes back on the analytical balance.

16. Input the weight, to the nearest 0.1 mg, directly into the computer via the interface.

8.2 Cleanup

1. Brush out the dishes with a soft-haired brush.

2. If necessary, due to excessive residue, wash and re-tare the set of dishes (see WI No. AO_15, "Moisture Dish Maintenance and Tare Weight Check Schedule").

9. Determination

1. Calculate the average moisture and range of the nine portions for each oven as described below.

$$\bar{x} = \frac{x_1 + x_2 + \ldots + x_n}{n}$$

$$r = \max_P - \min_P$$

Where
- $\bar{x}$ is the average moisture of the nine portions in an oven,
- x_n is the moisture of the nth (last) portion,
- x_i is the moisture of the ith portion (of n total),
- n is the number of portions in each oven,
- r is the range of moisture results within each oven,
- $\max_P$ is the largest moisture result in each oven, and
- $\min_P$ is the smallest moisture result in each oven.

2. Calculate the range of the oven averages for all the ovens of similar temperature as described below.

$$R = \max_O - \min_O$$

Where
- R is the range of moisture averages among the ovens,
- $\max_O$ is the largest average moisture among the ovens, and
- $\min_O$ is the smallest average moisture among the ovens.

10. Data Validation

10.1 Data Entry

All weights are sent directly to a FoxPro® database or to the LIMS via the interface. The computer or LIMS then calculates the moisture average and standard deviations using the calculations above.

10.2 Acceptance Criteria

10.2.a Within Oven Tolerance

1. The moisture range of the nine portions within an oven should be less than or equal to 0.1300.

2. If any one of the nine portions does not meet this tolerance, the uniformity check for that oven must be repeated (unless a portion's result is an obvious gross error – differing from the average by more than 0.50% -- in which case that result is discarded and the remaining eight are used).

    a. Check to make sure the temperature setting is correct, the air vents have not been moved, and that the door makes a good seal when closed.

    b. Repeat the uniformity check on the oven.

    c. If the portion is still out of tolerance, note the position that the portion occupied on the oven rack. Do not use this position for testing until corrective action is taken.

    d. Record the action taken in the Oven Maintenance Logbook.

10.2.b Between Oven Tolerance

1. The range of moisture averages of the ovens should be less than or equal to 0.1500.

2. If the results are not in tolerance, the check must be repeated on any oven that is out of tolerance.

a. Check to make sure the temperature setting is correct, the air vents have not been moved, and that the door makes a good seal when closed.

b. Repeat the uniformity check on the oven(s) that exceeded the tolerance.

c. If the repeated analysis is done the same day as the original uniformity check, calculate the new range using the repeated data and the averages of the ovens that were originally within tolerance.

d. If the repeated analysis is done at a later date, one of the ovens that originally met the tolerance must be repeated along with the oven(s) that exceeded it. Calculate the new range using only the data from these repeated ovens.

e. If the oven(s) is still out of tolerance, remove it from service until corrective action is taken.

f. Record the action taken in the Oven Temperature Logbook.

WI NO.: AO_14 (Filename: THERMOS.r02)		
Title: Calibrating the Thermometers Used in the Air Oven Laboratory		
Lab: USDA,GIPSA,FGIS,TSD,ARTS		Program: Moisture Reference Program
Revision: 2	Replaces: 1	Effective: 08/20/01

C William Burden Jr. 8/20/01

Approved By: C. William Burden, Jr. Date
Chemist

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0437

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Aaron C. Johnson for Timothy Norden 8/21/2001

Approved By: Timothy D. Norden Date
ARTS Branch Chief

USDA/GIPSA
10383 N. Ambassador Drive
Kansas City, MO 64153
(816) 891-0470

Table of Contents

Changes From Previous Revision

1. In section 6. Procedure:
   (5) moved to 6 and added procedure for checking data obtained while thermometer may have been out of tolerance

moved to 5

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in calibrating the thermometers used in the Air Oven reference moisture laboratory.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

3. References

- ISO Guide 25.
- American Society of Testing Materials (ASTM).
- National Institute of Standards and Technology (NIST).

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Mechanical convection (forced-draft) oven capable of maintaining a reasonably uniform temperature (within one degree of the normal working temperature of the oven) throughout the chamber, and equipped with adjustable vents and removable perforated or wire shelves.

b) Partial-immersion thermometer, ASTM No. 94C.

c) National Institute of Standards and Technology (NIST) traceable partial-immersion thermometer, ASTM No. 94C.

d) Partial-immersion thermometer, ASTM No. 96C.

e) NIST traceable partial-immersion thermometer, ASTM No. 96C.

6. Procedure

This monthly thermometer calibration check must be done within the first five working days of every month.

1. Read the oven temperature of an empty, temperature-stable oven from the ASTM thermometer (the "working" thermometer) in use for that oven and record the reading.

2. Replace the thermometer with the NIST-traceable thermometer of the same ASTM number, wait for a constant reading, and record the reading.

3. Record the difference between the NIST-traceable thermometer and the working thermometer. Apply a correction factor to the working thermometer as follows:

    a. If the NIST-traceable thermometer reads higher than the working thermometer, add the magnitude of the difference to the working thermometer's reading.

    b. If the NIST-traceable thermometer reads lower than the working thermometer, subtract the magnitude of the difference from the working thermometer's reading.

4. This correction factor is recorded and used every time the working thermometer is read, daily, until the next monthly check is performed and a new factor is determined.

5. Repeat this procedure for each working thermometer.

6. If the working thermometer differs from the NIST-traceable thermometer by more than ± 1°C, remove it from service and follow this procedure to determine the validity of sample results obtained while the thermometer may have been out of tolerance:

    a. Identify three samples which were tested during the previous three days when

b. analyses were done prior to checking the thermometer.

c. Analyze the three samples again using a thermometer that has been verified as being in tolerance with the NIST-traceable thermometer.

d. If the results of the repeated samples agree with the original results by the tolerance listed under section 10.2, Acceptance Criteria, of the Working Instructions corresponding to the sample type, record this information in the Nonconformity log – no further action is required.

e. If the repeated results do not agree with the originals within this tolerance, record that information in the Nonconformity log, determine which customer(s) submitted samples for analysis during the time period in question, alert them of the discrepancy, and request their input for resolution.

7. The NIST-traceable thermometers must be re-certified every eighteen months by NIST or another organization which uses calibration and testing instrumentation and standards that are traceable to NIST, **OR** new NIST-traceable thermometers may be purchased each every eighteen months as replacements.

7. Determination

The calculation to determine the temperature correction factor is as follows:

Working thermometer reading - NIST thermometer reading = Correction Factor.

WI NO.: AO_15 (Filename: TINS.r01)		
Title: Mositure Dish Maintenance and Tare Weight Check Schedule		
Lab: USDA,GIPSA,FGIS,TSD,ARTS	Program: Moisture Reference Program	
Revision: 1	Replaces: 0	Effective: 04/01/98

C. William Burden Jr. 3/31/98

Approved By: C. William Burden, Jr.
Chemist

Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0437

Donald C Kendall 4-2-98

Approved By: Donald C. Kendall
ARTS Branch Chief

Date

USDA/GIPSA
10383 N. Executive Hills Boulevard
Kansas City, MO 64153
(816) 891-0463

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Table of Contents

1. Purpose and Scope of Application

The purpose of this Working Instruction (WI) is to establish the operational parameters, methodology, and requirements for the quality assurance and acceptability of data in the maintenance and tare weight verification of the moisture dishes used in the determination of moisture in all samples.

2. Analyst Qualifications and Responsibilities

a) The analyst will receive on-the-job training in the conduct of this WI from a technician who has prior experience and training in using the WI, and/or from the Supervisory Chemist (or Project Leader).

b) The analyst will follow the WI as written. The Supervisory Chemist (or Project Leader) is responsible for ensuring this WI is followed, and modified as necessary or appropriate. All revisions to this WI must be approved by the Branch Chief of the Analytical, Reference and Testing Services Branch, prior to implementation.

3. References

- Hunt, W.H., & Neustadt, M.H. (1966) *J. Assoc. Off. Anal. Chem.* **49**, 757-763.
- ISO Guide 25.

4. Safety and Hazardous Waste

Refer to the Technical Services Division Chemical Hygiene Plan.

5. Equipment

a) Analytical balance accurate to 0.1 mg and equipped with RS-232 serial data interface.

b) Computer and software, IBM-compatible, equipped with a barcode wand, RS-232 serial interface, Microsoft® FoxPro®, Laboratory Information Management System (LIMS), and Laboratory Management Information Network (LABMIN).

c) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 55 mm and a height of about 15 mm, and supplied with tightly fitting slip-in covers. Both the dish and its cover should be identified by the same number and maintained as a set.

d) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 70 mm and a height of about 30 mm, and supplied with tightly fitting slip-on covers. Both the dish and its cover should be identified by the same number and maintained as a set.

e.) Moisture dishes (tins) constructed of heavy-gauge aluminum, with a diameter of about 92 mm and a height of about 53 mm, and supplied with tightly fitting slip-on covers. Both the dish and its cover should be identified by the same number and maintained as a set.

6. Parameters

1. The small moisture dishes are washed and weighed every twelve months. The tare weights are stored in FoxPro®, LIMS, and/or LABMIN along with the dish ID number and the date stored.

2. The dishes are reweighed every six months in sets of ten.

3. The large and intermediate sized moisture dishes are handled in the same way except that they are weighed only once a year. Because these dishes are used very infrequently, there is no mandatory time frame within which they must be rewashed.

7. Procedure

1. At the beginning of each work week, check the database files to identify the dishes that are within four weeks of coming due to be checked or washed. Special attention should be directed to any dishes that are less than one week from their maintenance due date.

2. Weigh the dishes no later than one week beyond their due date (grace period). Dishes that have not been checked within this time frame cannot be used until the maintenance is performed on them.

3. Compare the actual weights to the values stored in the tare weight file.

4. Generate a report that shows the dish ID number, the stored tare weight and date stored, the

actual weight and date weighed, and the difference between the two weights.

5. If the difference between the actual weight and stored tare weight of each of the ten dishes within a set is within tolerance, the actual weights are stored as the new tare weights for the dishes, along with the date when they were weighed.

6. If the difference between the actual and stored weights of any one of the dishes exceeds the tolerance, all ten of them are washed and reweighed. This is done to keep the date on which the new tare weights are updated the same for all of the dishes within the same group.

7. The actual weights of the freshly washed dishes are stored as the new tare weights along with the date they are entered and the date they were washed.

8. ALL small dishes must be washed every twelve months whether or not their actual weights are in tolerance to the stored tare weights.

8. Determination

$$\Delta = A-B$$

Where Δ is the change in tare weight,
A is the old tare weight,
B is the new tare weight

9. Data Validation

9.1 Data Entry

All weights are sent directly to a FoxPro® database or to a LIMS via the interface. The computer or LIMS then calculates the weight difference using the calculation above.

The weights are also entered manually into LABMIN.

9.2 Acceptance Criteria

To be within tolerance, the difference between the actual and stored tare weight of each moisture dish must be less than 0.0040 grams.