WEIGHTS & MEASURES
CONNECTION
NIST
National Institute of Standards and Technology
U.S. Department of Commerce
Volume 3 Issue 6
November 19, 2012

Inside this Issue

This Month in History

November 1, 1848 - Samuel Gregory founded the first medical school for women, Boston Female Medical School. The first class was 12 students. In 1874, it merged with the Boston University School of Medicine and became the first co-ed medical school.

November 2, 1734 - Daniel Boone (1734-1820) was born in Berks County, Pennsylvania.

November 6, 1860 - President Abraham Lincoln, the first Republican president, was voted into office.

November 10, 1775 - The U.S. Marine Corps was created. It was orginially part of the Navy but became its own service branch on July 11, 1789.

November 26, 1832 - Marks the date of the first horse-drawn streetcar in New York City.

November 29, 1929 - American explorer Richard Byrd completed the first airplane flight to the South Pole.

November 29, 1832 - Author Lousia May Alcott (1832-1888) was born in Philadelphia, Pennsylvania.

Grain Moisture Air-Oven Reference Methods in the United States

Byline: G. Diane Lee

Grain moisture air-oven reference methods are used by many stakeholders in the United States and these reference methods play an important role in measurement accuracy and uniformity of commercial grain moisture meter measurements in the country. This article will provide a basic description of the air-oven reference methods and their use; discuss the importance of grain moisture measurements in legal-for-trade applications; identify the key users of the air-oven reference methods for commercial use; review the origins of these air-oven reference methods; and outline the specific air-oven reference methods used for the major U.S. grains.

What are the grain moisture air-oven reference methods in the United States and how are they used?

The grain moisture air-oven reference methods are procedures used to determine the percent moisture content in grain. Basically, a small sample of grain (ground or unground) that represents a larger sample set of that grain is placed in a small metal dish and the weight of the sample is recorded. The sample is then placed in an oven at a specific temperature and heated for a specific amount of time. The sample is removed from the oven and cooled in a container that prevents loss or gain of moisture during the cooling process. Then the sample is reweighed. The loss in the sample mass is calculated as the percent moisture content of the grain.

Commercial grain moisture meters are devices that provide a rapid prediction of moisture in grain and are used to test the moisture of grains that are bought and sold in the U.S. Calibrations for commercial grain moisture meters are developed by comparing the grain moisture meter reading to grain moisture air-oven reference values. Also, air-oven reference methods determine the reference moisture values of grain samples that verify the accuracy and operation of commercial grain moisture meters.

Why are grain moisture measurements important in commercial (legal-for-trade) applications?

Moisture is a critical commercial measurement in grain because “discounts” to the price per bushel of grain are made at the time of sale, based on the

moisture content of the grain. "Discounts" are reductions to the price per bushel of grain. Two examples of discounts are drying and shrinkage discounts. A grain buyer must dry grain that is too high in moisture to prevent molding during storage. The cost associated with drying the grain or a "drying discount" is determined based on the grain moisture measurement at the time of sale. This discount reduces the amount of money the seller receives for the grain. For example, a buyer may be purchasing corn from a seller at $4.00 per bushel of corn at a target or desired 15 % moisture content. If the seller's corn is higher in moisture content at the time of sale than is targeted or desired, the buyer will reduce the unit price per bushel he will give the seller for the grain. Grains that are too high in moisture may be placed into storage with aeration causing shrinkage. The cost associated with shrinkage or a "shrinkage discount" is also based on the moisture of the grain at the time of sale. Errors in the moisture measurements may result in large losses to either the seller or buyer of grain. See article number C-006, March 2007 "The Economic Impact of Errors in Moisture Measurements, Part 2, Grain Moisture Meter Series," located at:
http://www.nist.gov/pml/wmd/pubs/archives-grain-moisture.cfm, for additional information on discounts.

Who uses air-oven reference methods?

Stakeholders in the United States that use air-oven reference methods as the basis for commercial measurements of moisture in grain include:

- The U.S. Department of Agriculture, Grain Inspection, Packers, and Stockyards Administration (USDA, GIPSA);
- The National Type Evaluation Program (NTEP) Laboratory for Grain Moisture Meters;
- Grain Moisture Meter (GMM) Manufacturers; and
- State Weights and Measures and other state officials.

These organizations work together to ensure equity in the commercial grain market as illustrated in Figure 1, the system of commercial grain moisture measurements in the United States and Figure 2, the movement of grain in the United States and inspection points.

The USDA, GIPSA uses air-oven reference methods to develop the calibrations for the official grain moisture meter(s). These official grain moisture meters are used to measure the moisture of grains at export locations and upon request at interior grain facilities in the United States.

The National Type Evaluation Program (NTEP) Laboratory at the USDA, GIPSA uses these air-oven reference methods when analyzing a national grain sample set and providing manufacturers with data to develop the calibrations for all grain moisture meters used in commercial service in the United States. These reference methods are also used to evaluate commercial (i.e., legal for trade) grain moisture meters in the NTEP laboratory to determine if prototypes of the device meet the requirements of the National Institute of Standards and Technology (NIST) Handbook (HB) 44 "Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices," prior to being used commercially in the United States. See Figure 1, the system of commercial grain moisture measurements in the

Calendar 2012

Registration for training in the NIST Office of Weights and Measures is handled by Yvonne Branden at yvonne.branden@nist.gov.

Course descriptions can be viewed on the Office of Weights and Measures website by clicking on the name of the course.
http://www.nist.gov/pml/wmd/calendar.cfm

December 3 - 7 (5 days)
Administrator Workshop
Class No. 5230
NIST/Gaithersburg, MD

December 3 - 7 (5 days)
Intermediate Metrology
Class No. 5164
NIST/Gaithersburg, MD

December 10 - 14 (5 days - FULL)
Handbook 133 - "Checking the Net Contents of Packaged Goods"
Class No. 5227
Raleigh, NC

2013

January 27 - 30 (4 days)
NCWM Interim Meeting
Charleston, SC
Contact: info@ncwm.net

January 28 - February 1 (5 days)
Volume Metrology Seminar
Class No. 5193
NIST/Gaithersburg, MD

February 25 - March 1 (TENTATIVE)
Lab Administration
NIST/Gaithersburg, MD

March 4 - 15 (2 weeks)
Mass Metrology Seminar
NIST/Gaithersburg, MD

April 4
Webinar - PT Follow-up and RCA

April 8 - 11 (4 days)
SEMAP*
Class No. 5209
Tifton, GA

April 9 - 11 (3 days)
Packaging and Labeling Seminar
Class No. 5224
NIST/Gaithersburg, MD

(continued on page 3)

United States.

State Weights and Measures Officials and other State Officials use these reference methods to determine the moisture in grain samples that are used to test commercial grain moisture meters. Typically, the grain moisture meters tested by State Weights and Measures or other state officials are at local elevators that purchase grain from farmers in their jurisdictions. See Figure 1, the system of commercial grain moisture measurements in the United States and Figure 2, the movement of grain in the United States and inspection points.

Manufacturers of grain moisture meters use these reference air-oven moisture methods when developing the calibrations for grain moisture meters that they sell for use in legal-for-trade applications. (See Figure 1, The system of commercial grain moisture measurements in the United States.)

April 15 - 19 (5 days)
Fundamentals of Metrology
Class No. 5208
NIST/Gaithersburg, MD

April 22
Webinar - Management Review

April 29 - May 2
Webinar - Internal Auditing Best Practices (4 days)

May 6 - 9
WRAP*
Class No. 5216
Helena, MT

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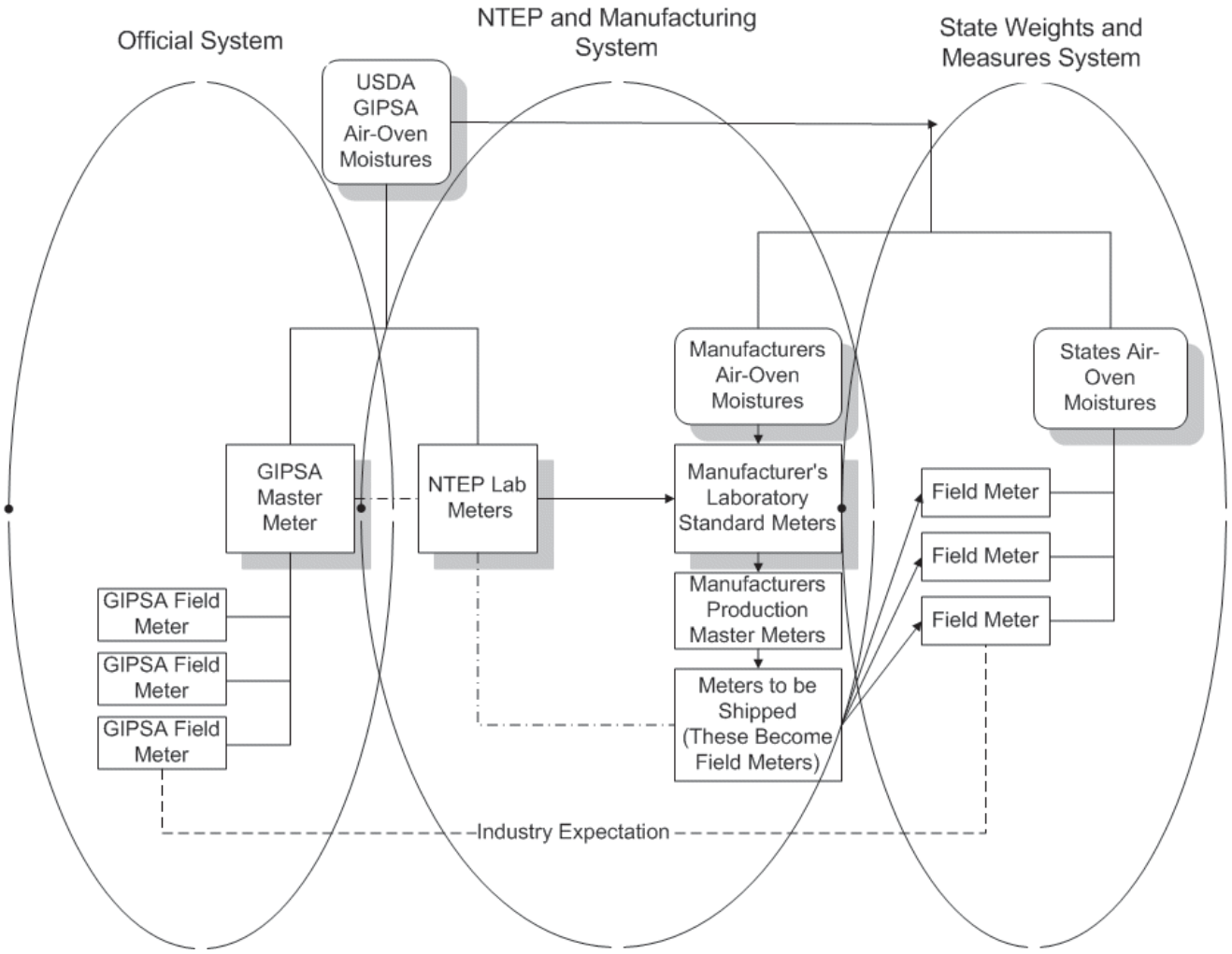

Figure 1
The system of commercial grain moisture measurements in the United States.

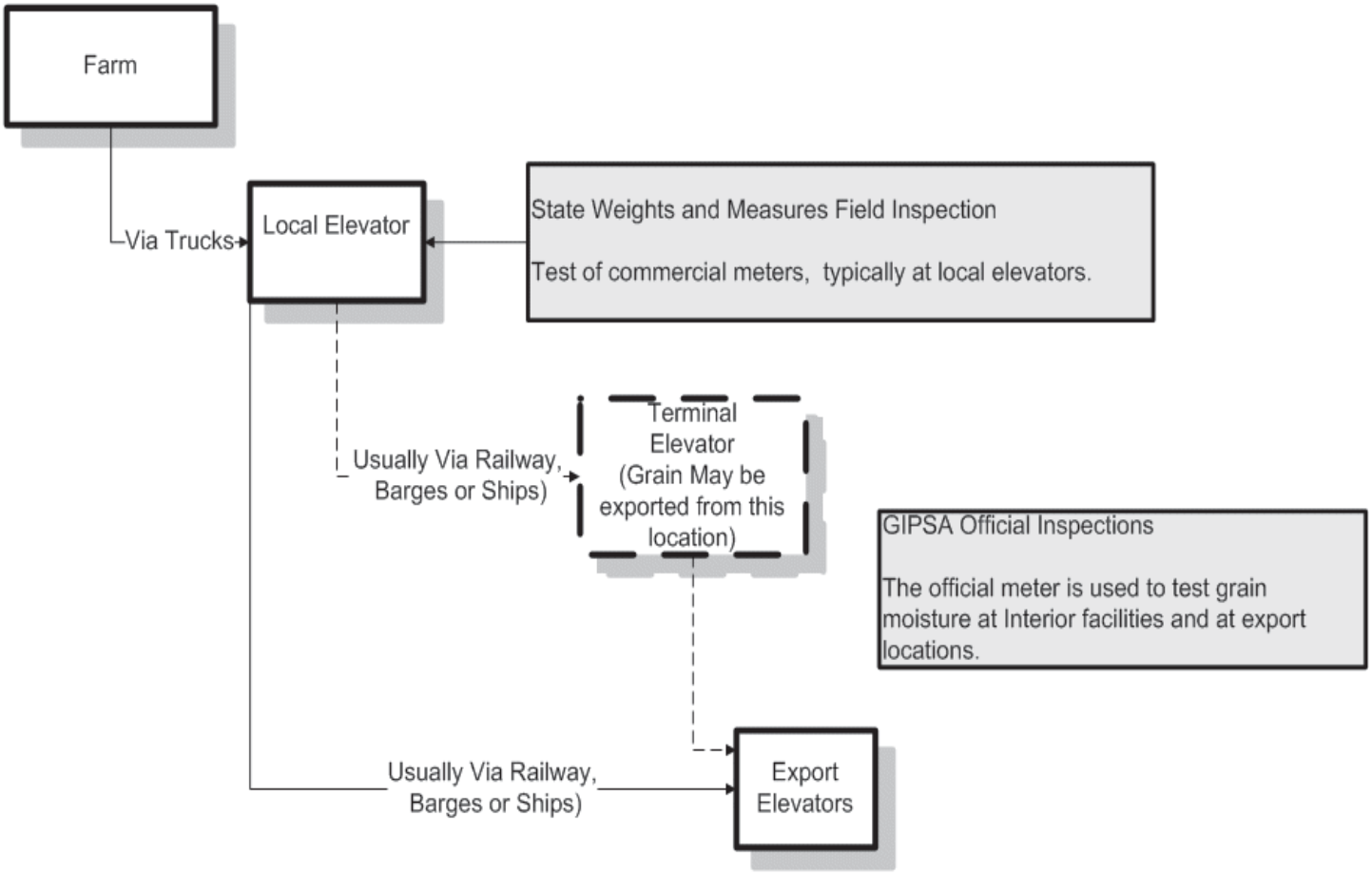

Figure 2

The movement of grain in the United States and Inspection points.

History of Air-oven Reference Methods in the United States

The most widely recognized reference methods for determining moisture content in grain are based on drying known weights of grain in various types of ovens and calculating moisture content from the weight lost in the drying operation.[1] The USDA, GIPSA, identifies the "air-oven methods" as its official reference methods for determining moisture in grain and oil seeds. According to the history of changes to USDA, GIPSA grain standards, these air-oven reference methods have been in use since July 1, 1935. The USDA, GIPSA air-oven reference methods were accepted by the American Association of Cereal Chemists (AACC, currently known as AACC International), Method 44-15A. Although oven methods are subject to error due to the loss of substances other than water during the drying process, Hart and Neustadt adapted the use of Karl Fisher (KF) Titration in 1957 to test the accuracy of the USDA air-oven moisture methods.[2] The USDA air-oven methods were designed to give results which agree with those obtained by KF Titration. KF Titration provides moisture values by extracting water from grain with the use of a solvent, such as methanol, and is more specific for grain moisture. Historical records have noted that KF Titration required more time for testing than was practical and, due to the test process at that time, there were repeatability issues from operator to operator due to portions of the test that were not automated. Frank E. Jones and Carroll S. Brickenkamp, National Bureau of Standards (NBS, now known as the National Institute of Standards and Technology), National Engineering Laboratory, noted that techniques were developed to automate KF Titration to address some of the earlier disadvantages of this method. Jones and Brickenkamp also noted that, although the KF Titration is more specific for moisture, additional tests are needed to determine if other substances react with the solvent used to extract water.[3] The air-oven reference methods remains the reference methods for moisture in the United States; this is likely because these methods have been compared to KF Titration and/or other more definitive tests and have proven to provide very repeatable measurements.

Air-oven Reference Methods for Major U.S. Grains

There are currently eight air-oven moisture reference methods used to determine moisture in the major U.S. grains and these methods are recognized internationally. These reference methods vary per grain type, moisture, and commodity and are maintained at the USDA, GIPSA in the Technology and Science Division's Laboratory Work Instructions. These work instructions include specific procedures, guidance on test sample size, equipment and material, and acceptance criteria needed for each test. Because these air-oven reference methods vary per grain type, the following tables are provided to summarize the different air-oven reference methods used in the United States.

AO 1

Single-Stage Air-Oven Reference Method for Moisture in Grains, Oilseeds, and Commodities is the difference in weight after drying an ground sample of grain for one hour at 130 °C ± 1 °C (drying time is two hours for soybean meal). The following grains and commodities are tested using this

Grains and Oilseeds		Commodities
Description	**Moisture Restriction**	**Description**
Barley	≤ 16 %	Barley, submitted
Lentils	≤ 16 %	Bulgur
Oats	≤ 16 %	Corn Soy Blend
Peas	≤ 16 %	Corn Soya Flour
Rice, brown	≤ 16 %	Corn Soy Milk
Rice, milled	≤ 16 %	Corn, submitted
Rice, rough	≤ 16 %	Corn Meal
Rye	≤ 16 %	Dessert Powder
Sorghum	≤ 16 %	Egg Noodles
Soybeans	≤ 10 %	Hominy Grits
Sunflower ground 50/50 with celite	None (for moisture correction of crude oil results	Icing Mix Powder
Triticale	≤ 16 %	Lasagna
Wheat	≤ 16 %	Macaroni
		Macaroni and Cheese
		Rotini
		Rolled Wheat
		Sorghum Grits
		Soybean Meal
		Spaghetti
		Wheat Flour
		Wheat Soy Blend
		Wheat Soy Milk
		Wheat - submitted

Cited References: AOCS Method Ba 2a-38; AACC Method 44-15A; AOAC Method 925.10; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 2

Two-Stage Air-Oven Reference Method for Moisture in Grains and Oilseeds is the difference in weight after air-drying a unground sample of grain until its moisture content is 16 % (10 % for soybeans and 13 % for rough rice), then drying the ground, air-dried sample for one hour at 130 °C ± 1 °C (two hours for soybean meal). The following grains are tested using this method:

Grains and Oilseeds	
Description	**Moisture Restriction**
Barley	≤ 16 %
Lentils	≤ 16 %
Oats	≤ 16 %
Peas	≤ 16 %
Rice, brown	≤ 16 %
Rice, milled	≤ 16 %
Rice, rough	≤ 16 %
Rye	≤ 16 %
Sorghum	≤ 16 %
Soybeans	≤ 16 %
Triticale	≤ 16 %
Wheat	≤ 16 %

Cited References: AACC Method 44-15A;, ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 3

Air-Oven Reference Method for Moisture in Corn and Beans is the difference in weight after drying an unground sample of grain for 72 hours at 103 °C ± 1 °C . The following grains are tested using this method:

Grains	
Description	**Moisture**
Corn	Any
Beans	Any

Cited References: AACC Method 44-15A; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

Happy Thanksgiving

May 6 - 9
Northeastern Weights and Measures Association (NEWMA)
Saratoga Springs, NY
Contact: James Cassidy at jcassidy@cambridgema.gov

May 13 - 24 (2 weeks)
Mass Metrology Seminar
NIST/Gaithersburg, MD

May 20 - 23
Central Weights and Measures Association (CWMA)
Overland Park, KS
Contact: Maureen Henzler at maureen.henzler@kda.ks.gov

June 17
Webinar Week - SOP 8, Part I

June 17
Webinar Week - Uncertainty Budgets for SOP 4

June 18
Webinar Week - Management Reviews

June 18
Webinar Week - SOP 8, Part II

June 19
Webinar Week - SOP 18, Part I

June 19
Webinar Week - Uncertainty Budgets for SOP 5

June 20
Webinar Week - SOP 18, Part II

June 20
Webinar Week - Undertainty Budgets for SOP 19

June 21
Webinar Week - Calibration Report Evaluation

June 21
Webinar Week - Uncertainty Budgets for SOP 14

July 14 - 18
NCSL International
Nashville, TN - Contact: info@ncsli.org

July 14 - 18
NCWM Annual Conference
Louisville, KY
Contact: info@ncwm.net

July 29 - August 1
Webinar - Calibration Report Evaluation

(continued on page 7)

AO 4

Air-Oven Reference Method for Moisture in Mustard, Canola and Rapeseed is the difference in weight after drying an unground sample of grain for one hour at 103 °C ± 1 °C (two hours for soybean meal), repeat the drying and if the difference between the 1st and 2nd weight after each drying is greater than 0.01 g the sample is dried for an additional hour. The following grains are tested using this method:

Grains	
Description	**Moisture**
Mustard	Any
Canola	Any
Rapeseed	Any

Cited References: ISO 665; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 5

Air-Oven Reference Method for Moisture in Flaxseed is the difference in weight after drying an unground sample of seed for four hours at 103 °C ± 1 °C. The following grains are tested using this method:

Grain	
Description	**Moisture**
Flaxseed	Any

Cited References: AACC Method 44-15A; ASAE Method S352.2; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 6

Air-Oven Reference Method for Moisture in Safflower seeds is the difference in weight after drying an unground sample of seed for one hour at 130 °C ± 1 °C. The following grain is tested using this method:

Grain	
Description	**Moisture**
Safflower Seeds	Any

Cited References: ASAE Method S352.2; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

National Conference on Weight and Measures Interim Meeting

January 27 - 30, 2013
Charleston, South Carolina

Addtional Information at:
http://www.ncwm.net/content/2013-ncwm-interim-meeting

August 5 - 8
Webinar - Contract Review

August 12 - 15
Webinar - Document Control and Recordkeeping

August 19 - 23 (5 days)
Fundamentals of Metrology
NIST/Gaithersburg, MD

September 9 - 12
NEMAP*
Class No. 5218
Harrisburg, PA

September 16 - 19
MidMAP*
Class No. 5219
Springfield, IL

September 22 - 25
Western Weights and Measures Association (WWMA)
Kalispell, MT
Contact: Doug Deiman at
doug.deiman@alaska.gov

September 23 - 26
SWAP*
Class No. 5220
Phoenix, AZ

October 7 - 9
Southern Weights and Measures Association (SWMA)
Charleston, WV
Contact: Steve Benjamin at
steve.benjamin@ncagr.gov

October 7 - 10
Webinar - Annual Submission Process*

October 21 - November 1 (2 weeks)
Mass Metrology Seminar
NIST/Gaithersburg, MD

October 28 - 31 (4 days)
Webinar - Internal Auditing Best Practices

November 18 - 22 (5 days)
Volume Metrology Seminar
NIST/Gaithersburg, MD

*Invitation Only
**Limited to State Laboratory Program Participants

AO 7
Air-Oven Reference Method for Moisture in Sunflower seeds is the difference in weight after drying an unground sample of seed for three hours at 130 °C ± 1 °C. The following grain is tested using this method:

Grain	
Description	**Moisture**
Sunflower Seeds	Any

Cited References: AOAC Method Ac 2-41; AOAC Method Ai 2-75; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 8
Air-Oven Reference Method for Moisture in Corn Gluten Feed is the difference in weight after drying a ground sample for four hours at 103 °C ± 1 °C. The following sample is tested using this method:

Grain	
Description	**Moisture**
Corn Gluten Feed	Any

Cited References: Method of the Corn Refiners Association; ASTM; NIST; Kenyon, A.S., Black, J.C., & Layloff, T.P. (1995); J. Assoc. Off. Anal. Chem. 78, 1109-1111.

AO 10 Through AO 15 are reference methods for other commodities and processes for laboratory check samples and equipment maintenance:
AO 10 100 °C Vacuum Oven Reference Method for moisture in Commodity Samples
AO 11 Processing the Air-Oven Laboratory Wheat Check Sample
AO 12 Processing the Air-Oven Laboratory Corn Check Sample
AO 13 Air-Oven Uniformity Check
AO 14 Calibrating the Thermometers Used in the Air-Oven Laboratory
AO 15 Moisture Dish Maintenance and Tare Weight Check Schedule

Many studies of air-oven reference methods have been conducted over the years since the first use of these reference methods. These studies resulted in changes to the air-oven reference methods that improved the accuracy of grain moisture meter measurements. The air-oven reference methods are well established in the U.S. Stakeholders use these reference methods to develop the calibrations for all commercial grain moisture meters and to test these devices to provide uniformity in the market place.

NIST, OWM greatly appreciates the review and comments of Dr. Richard Pierce, USDA, GIPSA during the development of this article.

For additional information concerning this article, please contact Diane Lee by e-mail at diane.lee@nist.gov.

ENDNOTES

[1, 2] Storage of Cereal Grains and Their Products, Chapter 1: Moisture - Its Significance, Behavior, and Measurement, Haward Hunt and S.W. Pixton.

[3] Journal of AOAC, Volume 64, Number 6, 1981, Frank E. Jones and Carroll S. Brickenkamp.

New Publications Released

- Handbook 44, *Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices* (2013)
- Handbook 130, *Uniform Laws and Regulations in the Areas of Legal Metrology and Engine Fuel Quality* (2013)
- Handbook 133, *Checking the Net Contents of Packaged Goods* (2013)

Links to these publications can be found at:

http://www.nist.gov/pml/wmd/pubs/index.cfm