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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 United States. 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: 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 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.)

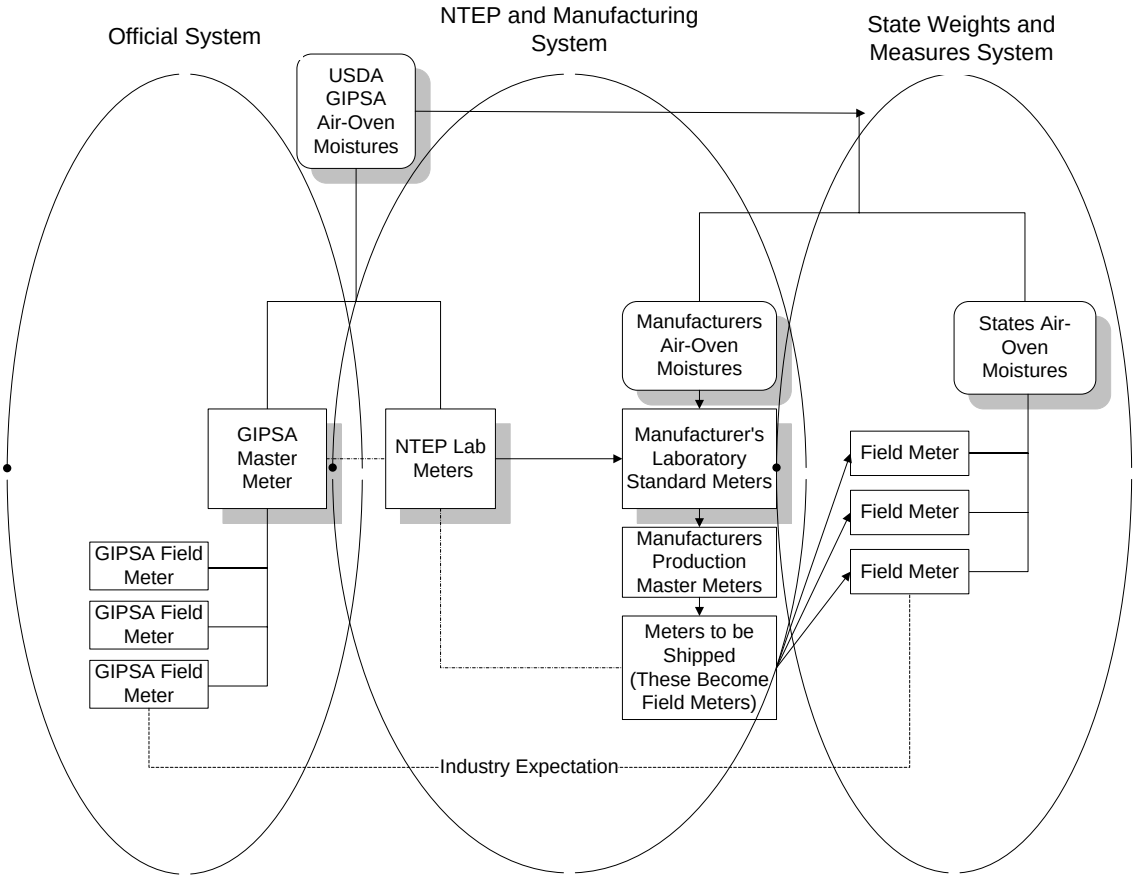

Figure 1.
The system of commercial grain moisture measurements in the United States.

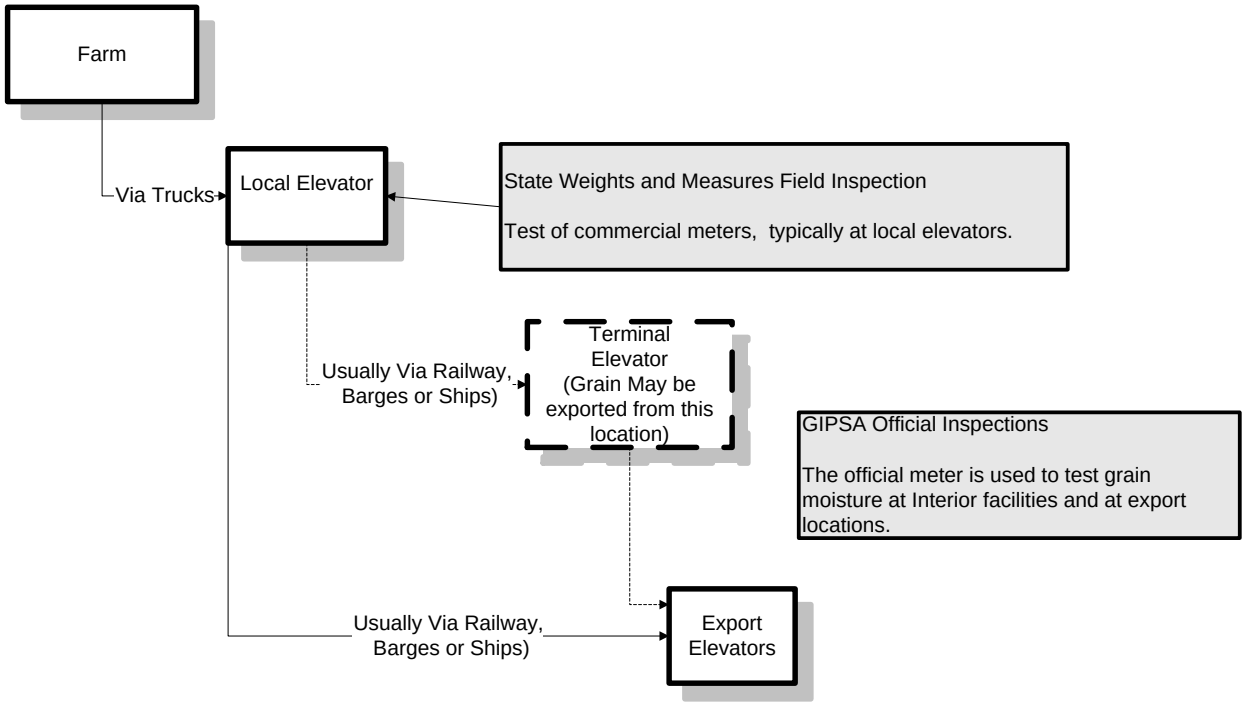

Figure 2.
The movement of grain in the United States and inspection points.

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.[i] 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.[ii] 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.[iii] 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 a ground sample of grain for one hour at 130 ºC ± 1 ºC (drying time is two hours for soybean meal). The following grains and commodities are tested using this method:

Grains and Oilseeds		**Commodities**
Description	**Moisture Restriction**	**Description**
Barley	≤ 16 %	Barley - submitted
Lentils	≤ 16 %	Bulgur
Oats	≤ 16 %	Corn Soy Blend
Pea	≤ 16 %	Corn Soya Flour
Rice, brown	≤ 16 %	Corn Soy Milk
Rice, milled	≤ 16 %	Corn - submitted
Rice, rough	≤ 13 %	Corn Meal
Rye	≤ 16 %	Dessert Powder
Sorghum	≤ 16 %	Egg Noodles
Soybeans	≤ 10 %	Hominy Grits
Sunflower ground 50/50 with celite	Non (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

AO_1
Single-Stage Air-Oven Reference Method for Moisture in Grains, Oilseeds, and Commodities: is the difference in weight after drying a ground sample of grain for one hour at 130 ºC ± 1 ºC (drying time is two hours for soybean meal). The following grains and commodities are tested using this method:

Grains and Oilseeds		Commodities
Description	**Moisture Restriction**	**Description**
		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 ºC ± 1 ºC (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	> 13 %
Rye	> 16 %
Sorghum	> 16 %
Soybeans	> 10 %
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 ºC ±1 ºC. 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.

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 ºC ±1 ºC (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 ºC ±1 ºC. 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 ºC ±1 ºC. 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.

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 ºC ±1 ºC. 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 the ground sample for four hours at 103 ºC ±1 ºC. 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 º 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 United States. 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.

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For additional information concerning this article, please contact Diane Lee by e-mail at diane.lee@nist.gov or the Office of Weights and Measures at (301) 975-4004, e-mail at owm@nist.gov.

[i] [i] Storage of Cereal Grains and Their Products, Chapter 1: Moisture–Its Significance, Beahavior, and Measurement, W. Harward Hung and S.W. Pixton.

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

Editor's Note: July 18, 2016, content updates were made to the moisture allowance in several tables.